Vol. 6 No. 1 MARCH 2012

FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

POWDER INJECTION MOULDING INTERNATIONAL

in this issue

Understanding defects in PIM

Company visit: Formatec

Developments in Titanium PIM

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POWDER INJECTION MOULDING INTERNATIONAL

For the metal, ceramic and carbide injection moulding industries

New opportunities for PIM in 2012

Welcome to the March issue of *PIM International*. 2012 is set to be a busy year, with a host of major events presenting an opportunity to promote the capabilities of PIM technology to a global audience. Following the MIM2012 Conference in San Diego, important industry events include China's PM-Expo (Shanghai, April 24-25), Ceramitec (Munich, May 22-25), PowderMet (Nashville, June 10-13), Euro PM2012 (Basel, September 17-19) and last but not least, the PM2012 World Congress (Yokohama, October 14-18). We'll be exhibiting at all these events and we look forward to seeing you during the year.

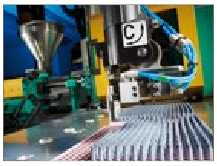
Over the past three decades our industry has experienced tremendous growth on the back of PIM technology's ability to deliver complex, high-volume, high-performance net-shape metal and ceramic components at lower costs than other processes. As with all technologies, however, problems can and do arise. In this issue Prof. Randall German reviews the subject of defects in PIM parts and discusses the appropriate remedial actions (page 33). As PIM processing continues to increase in sophistication, such defects are thankfully rare.

A rapidly growing market for Ceramic Injection Moulded (CIM) products can be found in luxury consumer goods. Thanks to the capabilities of CIM technology, ceramics are now regarded as a must-have material for high-end watches, telephones, jewellery and automotive interiors, to name just a few applications. Dutch CIM producer Formatec has enjoyed considerable success in this area and we report on a recent visit (page 47).

Also in this issue, we present a report on innovations in Ti-PIM from December's successful "Powder Processing, Consolidation and Metallurgy of Titanium" conference held in Brisbane, Australia (page 53).

Our technical papers in this issue reflect positively on the ongoing evolution of PIM. The state-of-the-art in the simulation of injection moulding in PIM is reviewed by Sigmasoft (page 65), and research on the 2C MIM of titanium (page 69), and the PIM processing of zirconium silicate (page 75), is also presented.

Nick Williams
Managing Director and Editor



Cover image
Green ceramic components being stacked following injection moulding (Courtesy Formatec, The Netherlands)

March 2012

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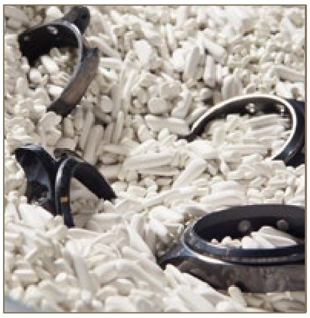

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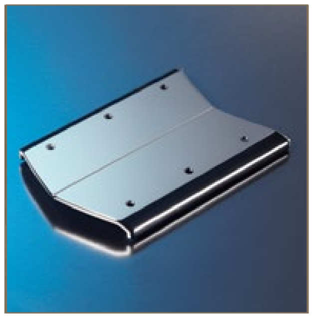

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Over the past 30 years PIM has experienced tremendous growth on the back of the technology's ability to deliver complex high-volume, high-performance net-shape metal and ceramic components at lower costs than alternative processes. As with all technologies, however, problems can and do arise. Professor Randall German reviews the causes of a number of defects experienced during more than two decades of troubleshooting in PIM, along with the necessary corrective actions.

47 Innovation helps producer of CIM products for luxury applications expand into new markets
Some of the world's most elegant CIM products are manufactured by Formatec Technical Ceramics B.V., based in the Netherlands. The company is a specialist manufacturer of luxury CIM products made from zirconia, which have a high gloss, aesthetically appealing surface finish. In addition, Formatec specialises in complex technical components manufactured from zirconia, alumina and silicon nitride. Dr Georg Schlieper reports on a recent visit to Formatec for *Powder Injection Moulding International*.

53 Titanium Powder Injection Moulding (Ti-PIM): Australian conference reviews developments
The international conference on "Powder Processing, Consolidation and Metallurgy of Titanium", took place at The University of Queensland, Brisbane, Australia on 5-7 December, 2011. Dr David Whittaker reports for *PIM International* on a number of international papers that addressed the latest developments in the metal injection moulding of titanium and its alloys.

61 Hot Isostatic Pressing (HIP) of PIM parts: Material properties and increased productivity
Hot Isostatic Pressing (HIP) of PIM parts is an effective route to achieving full density and improving mechanical properties for a range of PIM materials. The latest innovations in HIP technology, such as Uniform Rapid Cooling, URC™ , now make the case for the technology more appealing thanks to significantly faster processing times. Dr Anders Eklund, from Avure Technologies AB, Sweden, reviews the process and its advantages for PIM producers.

Technical papers

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Industry News

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

Proposed new ASTM WK35394 specification for MIM commercially pure Ti components for implant applications

A proposed new specification for ASTM WK35394, relating to metal injection moulded (MIM) commercially pure titanium components for surgical implant applications, is under development.

The specification covers the chemical, mechanical, and metallurgical requirements for four different grades of metal injection moulded commercial titanium components from unalloyed titanium and Titanium-6Aluminum-4Vanadium powders, for use in the manufacture of surgical implants. There are two types of materials within these four grades, as-sintered or with an additional densifying process such as hot isostatic pressing (HIP).

The Standard will use the nomenclature of the ASTM F 2885 Standard Specification for MIM Ti-6A-4V components for surgical implant applications and the chemical values of the ASTM F67 Standard Specification for unalloyed titanium for surgical implant applications demanded by the medical device industry in order to use CP Ti MIM parts for implantable applications.

The title and scope are in draft form and are under development within the ASTM Committee F04.12.

www.astm.org

BorgWarner to boost Hungarian turbo output

BorgWarner Turbo Systems Kft has announced plans to increase capacity by 50% at its turbocharger manufacturing plant in Oroszlany, Hungary. The expansion will add a further 140 workers and is expected to be operational by 2015. BorgWarner Turbo Systems Kft currently employs 700 workers and manufactures turbochargers for passenger car engines.

MIM is widely used to produce complex turbocharger components from materials that are difficult to process via other routes, whilst also offering improved properties.

www.borgwarner.com

PolyMIM GmbH adds catalytic feedstock system to its existing water soluble range

PolyMIM GmbH, the feedstock supplier of water based feedstock systems based in Bad Sobernheim, Germany, has announced the development of its own catalytic feedstock system. PolyMIM GmbH states that it is currently the only feedstock manufacturer on the market offering two different binder systems for Metal Injection Moulding (MIM).

The catalytic debinding system, branded polyPOM, offers high green part strength and fast debinding times. The company's alternative water soluble binder system, polyMIM®, offers superior carbon control and environmental friendliness. The PolyMIM® feedstock system, states the company, can also be used for special alloy manufacturing such as Cu-alloys and tungsten carbides.

"Only by offering both feedstock systems will it be possible to address all the needs of customers when selecting a solution for their respective component," stated PolyMIM's Sabine Pätow-Molz.

Low alloy and stainless steel grades are now available in the polyPOM range, in addition to the wider polyMIM® range.

www.polymim.com

CIM Expert Group to focus on improving technology awareness and process enhancement

The German Expert Group for Ceramic Injection Moulding (Expertenkreis Keramikspritzguss), is to organise a series of CIM workshops during 2012 in collaboration with a number of German universities. The aim is to increase awareness of the technology amongst the next generation of engineers.

Dr. Tassilo Moritz, IKTS Dresden, told *PIM International*, "The marketing committee inside the CIM-Expertenkreis is committed to pursuing the goal of increasing both awareness of the potential of CIM technology and the general level of understanding of the process. The question of how to spread the knowledge about such a powerful production method is a difficult one to answer. Our approach is to plant the seed of knowledge in the next rising generation of engineers."

The group made a successful start on this mission during 2011 with the first ceramic injection moulding workshop and seminar held at the University of Applied Sciences Koblenz, Höhr-Grenzhausen, Germany, in April 2011. In close cooperation with the university the event, for both students and engineers from regional industry, attracted 120 people. Lectures were given by expert group members regarding the injection moulding process, the equipment used, the parts produced and the potential of the technology. Subsequently the ceramic injection moulding process was presented as a live demonstration on an operating injection moulding machine.

In addition to pursuing technology promotion, the CIM expert group has also been addressing the need to further develop an understanding of specific aspects of the CIM process.

A dedicated committee has been responsible for the establishment of a public funded project, Pro-CIM, which aims to increase the knowledge of non destructive control systems for CIM-parts. A multi-purpose trial mould was developed for testing ceramic feedstocks and their properties, in order to achieve a deeper understanding of the injection process. Models from 3D-simulation software are then verified against this and data collected for evaluation and improvement.

The group has also indicated that the existing supplier market is not currently meeting the specific requirements for CIM technology. CIM part producers, it is suggested, cannot easily maximise the potential of the process because of the lack of suitable suppliers. Specifically, the limited availability of special customer tailored ceramic high-end powders in appropriate amounts at affordable prices, plus appropriately shaped sintering furniture, is highlighted. The development teams inside the CIM Expert Group aim to address these challenges, enabling members to take full advantage of this innovative ceramic shaping process.

The CIM Expert Group was founded in 2008 and its members include both CIM producers and suppliers, as well as ceramic and materials research institutes.

www.keramikspritzguss.eu ■

CIM EXPERT GROUP AT CERAMITEC 2012

Almost all CIM Expert Group members will be exhibiting at CERAMITEC 2012, Munich, Germany, 22-25 May 2012.

www.ceramitec.de ■

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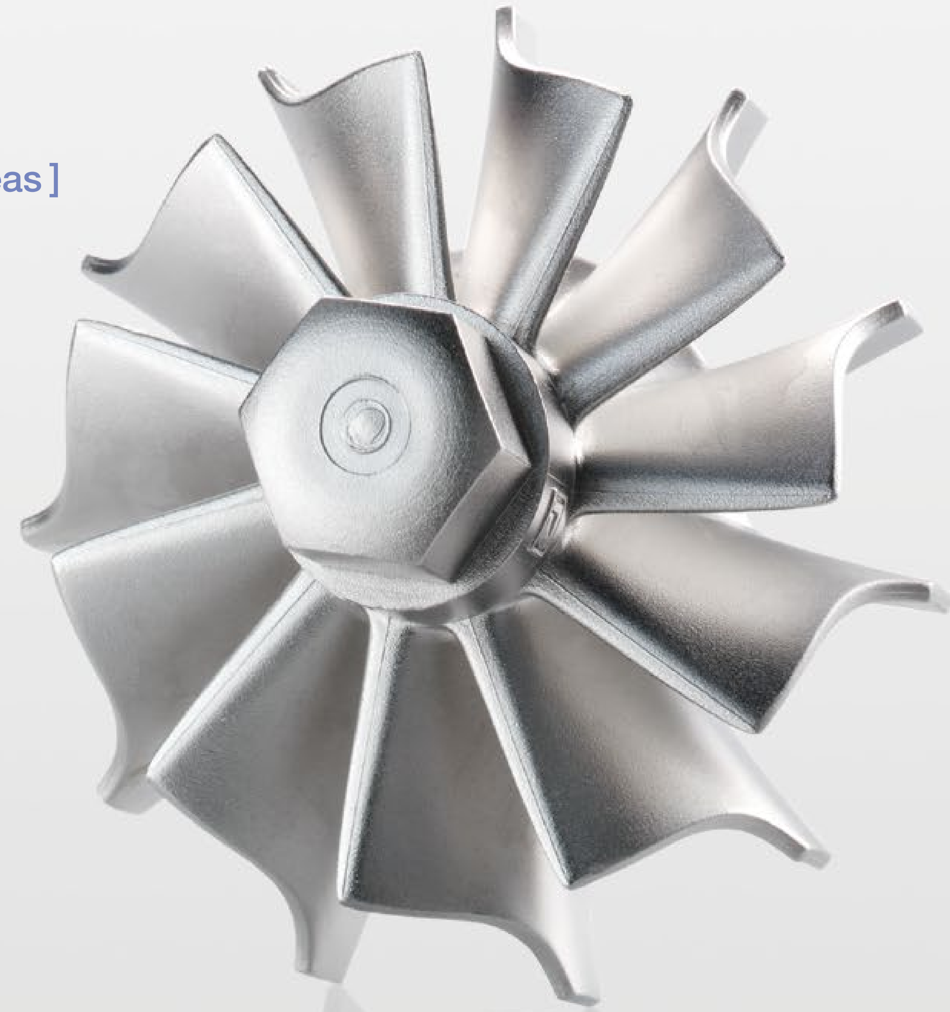

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Epson Atmix to triple its fine powder production capacity with new plant

Seiko Epson Corporation has announced that its subsiduary, Epson Atmix Corporation, a world leader in the manufacture of superfine alloy powder, is to invest 3.2 billion yen in a new plant at the Hachinohe Kita-Inter Industrial Park in Aomori Prefecture, Japan. The new plant will approximately triple Atmix's current production capacity in water-atomised superfine alloy powder, enabling it to meet expanding demand from growing markets for goods such as smart-phones and other high-performance mobile devices, automobiles, and medical equipment.

"We are very excited to announce the establishment of our new plant," stated Satoshi Oguchi, President, Epson Atmix. "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."

To accommodate these expanding markets, Epson Atmix will invest in the new plant, which will increase Atmix's magnetic powder and MIM powder production capacity to approximately 10,000 tons per year, or about triple its current capacity. The company plans to break ground on the new facility in the first half of fiscal 2012 and begin operations in the second half of 2013.

The new plant will have a factory floor area of approximately 3,300 m^2 on a 20,000 m^2 lot.

Epson Atmix's superfine alloy powders are divided into two main types according to the materials from which they are made and their uses, magnetic powder and metal injection moulding (MIM) powder. 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. The atomised metal is then rapidly cooled, producing a powder with regularly-sized, micron-order particles, and uniform composition and characteristics.

Magnetic powder is used in electronic components such as inductors, choke coils, and reactors that are needed to control voltages in smartphones, notebook PCs, and other high-performance mobile equipment. Epson Atmix states that its magnetic powder exhibits particularly good energy conductance and thus contributes significantly to reducing the power consumption and size of voltage control components, as well as supporting high frequencies and large currents.

The expanding global mobile equipment market is not the only market driving demand for magnetic powder. This powder is also attracting considerable attention from the likes of the automotive industry and other industries demanding efficient power consumption, which see the potential for an expanding number of new applications.

Molten metal being transferred into a ladle at Epson Atmix prior to atomisation (PIM International, Vol.2 No. 1 March 2008)

The company's MIM powder is used widely in applications that require parts with complex shapes yet high accuracy and strength. Applications range from special medical equipment to automobile engines. Epson Atmix has a broad lineup of MIM powders that includes, for example, stainless steel and low-alloy steel.

In addition, the size of powder particles can be adjusted to suit a given application, helping to increase the strength of metal injection moulded parts. There is expected to be steady future demand for MIM powder as the markets grow in the medical, automotive and other industry sectors.

www.atmix.co.jp

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THE LINDE GROUP

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Proven performance in MIM production

SINTERFLEX™ is a tried and tested technology that brings proven benefits for MIM producers.

Philip J. McCalla, Operations Manager at leading MIM producer Megamet Solid Metals Inc., commented; "Based on our experience, SINTERFLEX™ has greatly improved our ability to control the carbon content in our carbon steel materials."

"Megamet has been very satisfied with Linde's service and technical knowledge. We are confident, with Linde's expertise, SINTERFLEX™ will meet our expectations."

Megamet Solid Metals, Inc.

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Polymer Technologies Inc. acquires injection mould toolshop, Polmold

Polymer Technologies, Inc. (PTI) has announced the acquisition of Polmold, a Wallington, NJ, USA, based manufacturer and repair shop for injection moulds, tools, dies and fixtures. The acquisition was expected to close by the end of February 2012.

PTI is a custom plastic and metal injection moulding company that provides contract manufacturing to the aerospace, defence, medical/surgical, automotive, commercial, consumer electronics and dental industries. The company offers design, engineering and manufacturing services supported by Mold Flow™ analysis. Its Clifton, NJ, USA, based facility is home to 24 moulding machines ranging from 38 to 650 ton clamping force and employs a staff of 71 across its 24 hours a day, five days a week operations.

Polmold manufactures precision injection moulding tooling for both plastic and metal injection moulding programmes. Additionally, the company has extensive experience in construction and repair of moulds for compression and investment casting applications, as well as providing complementary services for associated tools, dies and fixtures. The purchase will strengthen PTI's position as a 'one-stop shop' contract manufacturing solutions provider. The company has five CAD/CAM work stations, four late-model computer numerical control (CNC) machining centres, seven CNC milling machines, two lathe machines, two wire and three "sinker" electric discharge machines (EDM), six grinders and QC inspection equipment to guarantee the quality of the value-added services they supply.

"The acquisition of Polmold complements our existing product offering and continues our strategic plan to offer streamlined contract manufacturing services to our customers in the USA and internationally," stated PTI President Neal Goldenberg. "Our vertical integration of this critical technology into our operation will truly make us a complete solutions provider for any engineered injection moulding application."

Melvyn Goldenberg, PTI founder and Neal's father added, "We have been pleased to see a strong resurgence of tooling work being done here in the United States. Much of this is due to the realisation that the cost-savings from work done overseas in places such as China didn't pan out the way people had hoped it would when variables such as lost time and additional costs of rework were not a part of the original equation. Further, given the size of some of the tooling, transportation and import fees, outsourcing has become very cost prohibitive."

Polmold was founded by Henry Marzec in the 1980's and today employs six highly-skilled workers whose combined 80+ years of experience in injection moulding tool design and construction and use of their state-of-the art equipment will ensure the tools built will meet or exceed the stringent requirements of PTI's customer base. Marzec will assume the role of Polmold Tool Inc. General Manager.

"We are very pleased to join our forces with PTI and the Goldenberg's. We still intend to support our existing clients with the same level of superior quality, workmanship and service for which they have come to rely on us, however, now we can also offer a new value-proposition," stated Marzec. "Through utilisation of PTI's injection moulding expertise, we can prototype and troubleshoot these tools prior to being shipped to the client. This will significantly reduce the debugging time associated with new tool manufacturing and increase our client's speed-to-market."

www.polytechinc.com

Successful MIM seminar held in India

MATE, a technology consulting company specialising in PM and MIM, organised its first MIM seminar in India at the Victor Menezes Convention Centre, at the Indian Institute of Technology (IIT), Bombay, on December 10th 2011.

In technical presentations which covered all process steps of metal injection moulding, MATE President Michael Godin and his colleague Roelof van Dijk provided a theoretical introduction to the MIM process. The seminar also included a MIM market overview and an introduction to several options for a successful MIM production start-up.

Prof. Parag Barghava, of the Indian Institute of Technology, gave a presentation on the ongoing MIM research at IIT in Mumbai, and Prashant Joshi of BASF presented the Catamold® feedstock system. From Germany, Elino presented its latest solutions for catalytic debinding and continuous sintering furnaces. LÖMI also presented its solvent debinding equipment with integrated vacuum drying and solvent recovery systems. Chinese company Lingqi presented its batch type furnaces for small volume MIM production, whilst another Chinese suppler, Only, presented its injection moulding machines ranging from 10 to 150 ton.

Delegates at the MIM seminar in Bombay

Prakash Khole with MATE's Michael Godin

The seminar was attended by representatives of various Indian companies from the ceramics, defence, medical and hard metal industries, which, state the organisers, are planning to start their own MIM production in the near future.

The seminar ended with a Q&A and discussion session in which the 30 invited attendees addressed their challenges in starting their own MIM production.

The organisers state that this first MIM seminar in India was made possible with the support of Prakash Khole of KSS and the Indian Institute of Technology in Mumbai.

There are currently only three MIM companies in India, however it is expected that due to the significant interest in MIM from various industries, the market in India will grow significantly over the next few years.

www.matepm.com

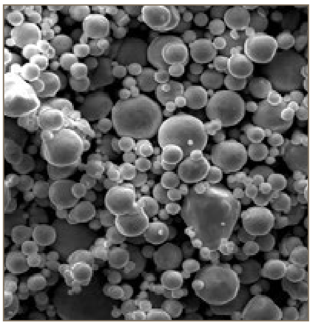
Erasteel's Metallied facility targets high quality speciality MIM powders

Metallied's General Manager Javier Martinez

With its new state-of-the art vacuum induction melt (VIM) gas atomiser, Metallied, based in Irun, Spain, and owned by Erasteel, is offering fine powders suitable for Metal Injection Moulding and other applications such as Additive Manufacturing and Laser Cladding.

The factory specialises in both standard and tailor-made alloys, available in small batches starting from 5 kg and suitable for the production of small series of metal parts or R&D activities. Javier Martinez, General Manager of the unit, commented, "We can supply a wide range of alloys with a fine powder size such as nickel superalloys, cobalt, copper, iron-base alloys and silver alloys. Thanks to the vacuum melting facility and argon atomisation, we can process superalloys with additions of reactive metals such as titanium or aluminium." Metallied was created in 2007 as a spin-off of the CEIT R&D Centre and was acquired in October 2011 by Erasteel, a subsidiary of the ERAMET group.

www.erasteel.com / powder@eramet-erasteel.com



An atomisation chamber at Metallied and MIM fine powder SEM image

Leroxid wins award for wear-resistant tool inserts made of spark erodable ceramic

Leroxid, based in Hochdorf, Germany, won a gold award at EuroMold late last year for its new "Dimacer" tool inserts made from erodible ceramics. The innovative ceramic material, used for in mould components, extrusion dies, gears and threaded components has a level of electrical conductivity that allows the ceramic mould parts to be produced by the spark erosion process. A further advantage is that the material's coefficient of thermal expansion and thermal conductivity values are close to those for steel.

Leroxid also states that the material outperforms steel with its superior abrasion resistance, protecting critical areas such as mould gates as well as fragile features in micro moulds. The company also suggests that the material is suited to the ceramic and metal injection moulding of filigree and sharp-edged parts.

www.leroxid.de

Ceramitec 2012: Organisers offer a forum for PIM and PM in Munich

As well as being the largest exhibition dedicated to the diverse world of ceramics technology, Ceramitec 2012, to be held in Munich, Germany, 22-25 May, will once again be an essential event for the technical ceramics, Powder Injection Moulding (PIM) and Powder Metallurgy (PM) communities.

In addition to featuring a large exhibition area dedicated to PIM, PM and technical ceramics, the Fachverband Pulvermetallurgie (FPM), the trade association of for the German speaking PM community, is presenting a "Powder Metallurgy Forum" on the afternoon of Tuesday 22 May 2012.

The forum, entitled "Powder Metallurgy Today and Tomorrow", will be moderated by Prof. Paul Beiss, RWTH Aachen, and is scheduled to include the following presentations:

- Current status and future prospects of Metal Injection Moulding technology
  Dr.-Ing. Frank Petzoldt, Fraunhofer IFAM, Bremen, Germany
- Hot Isostatic Pressing (HIP) of Powder Metallurgy parts
  Prof. Dr.-Ing. Christoph Broeckmann, RWTH Aachen, Germany
- Inert gas furnaces for PM component production
  Prof. em. Dr.-Ing. Paul Beiss, RWTH Aachen, Germany
- Optimised process for the microwave assisted debinding of carbides using a hybrid technique
  Dipl.-Ing. Ingo Cremer, Cremer Thermoprozessanlagen GmbH, Germany & Daniel Rumo, Extramet AG, Switzerland
- Rapid sintering of large components – Industrial applications
  Dr. Jürgen Hennicke, FCT Systeme GmbH, Germany
- Current status of PVD hard coatings
  Dr.-Ing. Oliver A. Lemmer, CemeCon, Germany
- Current status of CVD hard coatings
  Dr. Helga Holzschuh, SuCoTec AG, Switzerland

Around 15,000 trade visitors from more than 90 countries attended Ceramitec 2009

Visiting the Ceramitec exhibition

The Ceramitec 2012 exhibition will feature numerous suppliers of processing technology and materials for PIM, as well as specialist component suppliers. The exhibition is open from 22–25 May 2012. *PIM International* will be exhibiting on stand 118, Hall B6.

Around 15,000 trade visitors from more than 90 countries attended Ceramitec 2009, with more than 60% of visitors based overseas.

www.ceramitec.de

Wittmann Battenfeld increases production capacity at its Kottingbrunn facility

In response to a continuing positive demand for its products, and in particular strong demand for its new large machine model, the MacroPower, Wittmann Battenfeld GmbH has announced that it will be increasing its production capacity in 2012.

MacroPower, the company's large model was introduced for the first time in 2010 and is now available with clamping forces ranging from 500 to 1100 t and has, states the company, met with an extremely positive response from the market. The company expects that strong demand for its machines will continue into the future. Thanks to the good order book situation, the company's manufacturing facility was already utilised to full capacity in 2011.

The company will be extending the production and warehouse areas at the Kottingbrunn facility, Austria, by 3000 m². The assembly capacity will be increased by roughly 1,600 m² with a new building, the pre-assembly and warehouse capacity has already been expanded by about 1,400 m² through remodelling existing buildings.

The extension of the assembly space will be implemented by adding a new bay to the existing assembly hall. The entire equipment of this new bay will be laid out for producing large machines and thus complement the current assembly area. The planning phase has already been initiated and construction will start in spring. The new hall is expected to be completed by the end of September 2012.

Because of the expansion of the Kottingbrunn facility, the company's open-house "Competence Days" event, originally planned for Spring 2012, is being re-scheduled to a date in Autumn when the new hall will be completed.

www.wittmann-group.com

New "Standard Test Methods for Metal Powders and PM Products" published by MPIF

The Metal Powder Industries Federation (MPIF) has published the 2012 Edition of "Standard Test Methods for Metal Powders and Powder Metallurgy Products". The most current versions of these standards, which are used in the manufacture of both metal powder and PM products, are required by Quality Assurance programmes in order to maintain full compliance.

The MPIF Standard Test Methods publication contains standards covering terminology and recommended methods of testing for metal powders, PM and MIM parts, metallic filter and powder metallurgy equipment.

These standards, intended to present and clarify PM technology as an aid in conducting business, relate to those activities that concern designers, manufacturers, and users of PM parts.

www.mpif.org

2nd European Titanium Conference to take place in UK, March 27-28

Following the success of the 1st European Titanium Conference held in 2009, the Titanium Information Group will be hosting the two day 2nd European Titanium Conference on 27-28 March in Bristol, UK.

The organisers state that the event will showcase the latest in technology, research and applications for titanium and will feature presentations from the leading research institutions, titanium producers and end-users across Europe and worldwide.

The event will provide a unique opportunity to learn about the latest developments in the titanium industry from the perspective of various stakeholders. In addition to a packed two-day schedule of presentations, the conference will offer opportunities for networking with senior commercial, operational and technical decision makers across the titanium sector and will include an exhibition and conference dinner.

Chief Executive of NAMTEC, Alan Partridge, explained, "The last event was a huge success and it was wonderful to see such a breadth of international titanium related businesses in attendance. Delegates immediately recognised opportunities within the market and benefitted from the chance to network with senior figures from the industry who are able to provide worldwide market access, the latest developments in research, company exposure and industry leads. The next conference will feature the latest materials and manufacturing developments, including microstructure and texture, near net shape manufacturing, welding and joining, surface finishing and machining and with such a full and interesting programme we are expecting the 2012 event to be an even greater success."

The two-day conference will feature over 40 presentations from high profile speakers in parallel and plenary sessions, including Boeing, Messier-Dowty and Rolls-Royce plc.

www.namtec.co.uk

EPMA PM Summer School: Programme now available

The programme for the European Powder Metallurgy Association's 2012 PM Summer School has been published. The event will take place at CEIT, in San Sebastian, Spain, from 25–29 June 2012.

The association's annual residential summer school for young materials and design engineers has been designed to offer participants from all parts of the EU an advanced teaching of PM and MIM's advantages by some of the leading academic and industrial personnel in Europe.

The programme consists of a five day course that has been coordinated by Prof José Torralba from the Universidad Carlos III Madrid together with Prof Francisco Castro and his team from CEIT. All lectures and laboratory work will take place at CEIT. The course is open to graduates under the age of 35 who are European citizens. The deadline for applications is 30 March 2012. Full programme details are available on the event website.

www.epma.com/summerschool

Arcast Inc. offers new atomisation system for refractory and reactive metal powders

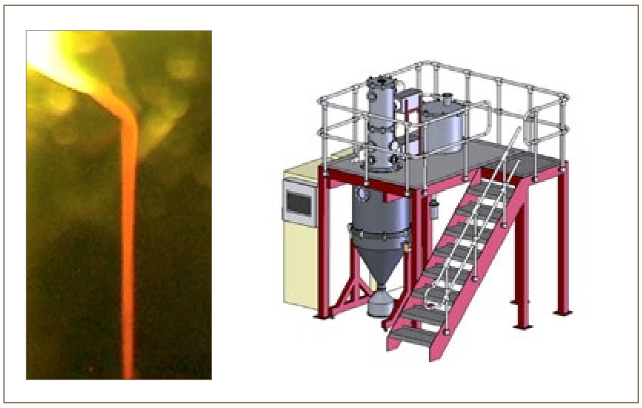
Arcast Inc., based in Oxford, Maine, USA, is introducing a new method for producing refractory and reactive metal powders and castings from elemental feedstock. The company, which produces research and production metallurgical equipment with a focus on cold crucible vacuum furnaces, is using its experience and technology to fully develop its revolutionary hybrid induction and plasma arc alloying furnace. The development is part of a National Science Foundation (NSF)/ Small Business Innovation Research (SBIR) funded research project.

The system can accommodate various forms of melt stock, from elemental powders/granules to scrap components. Arcast states that it can achieve full alloying of elemental feedstock and develop a controlled melt stream for casting or atomising. The hybrid furnace design allows full control of alloy composition. Melting can be conducted under a range of regimes, from high vacuum through to over pressure with various gases. This can help control alloy constituents with high vapour pressures. There is no trapped frozen metal skull that could cause alloy segregation, and the process is continuous so there is virtually no minimum or maximum quantity per run cycle. Moreover, the company states that the cost of capital equipment and services is minimal.

The Arcast system obviates the need to provide relatively expensive bar stock with a limited alloy selection that is required by other (ceramic free) production routes. There are no refractory ceramics that can come into contact with the melt, so there is no risk of contamination from such a source. The process can be stopped and restarted in mid process to allow a run sample to be taken and analysed to allow checks on alloy composition.

The focus of the project is to produce titanium-based shape memory alloys with refractory metal constituents and also refractory metals and intermetallics. Although this is the principal focus of the current project, the furnace is capable of producing a wide range of metal alloys from titanium to tungsten. The target for this technology is to offer a direct production route for novel alloys or those that are not commonly available. The intention is to help bridge the gap between laboratory research and pilot scale production, giving a route to market for all the promising alloys developed in research establishments and university laboratories.

The atomising options are either 'free-fall' type, high velocity gas jet or centrifugal (spinning disk or cup). Arcast states that its initial focus is on gas atomisation because of the greater demand for this product. A realistic target median particle size (D_{50}) of 100 µm for most alloys can be expected. This will produce a good fraction of the finer particle sizes suitable for MIM powders. Oversized product can always be reprocessed if this cannot be used. Because the Arcast furnace achieves full homogeneity in the melt, there is little need to mix powders in a subsequent process in order to achieve desired properties in the pressed or sintered material.



Arcast Inc. titanium alloy melt stream from a cold crucible, with a typical Arcast atomiser arrangement

Over the next year or two, the company states that it will be looking to improve the median particle size of its 'as atomised' powders. In particular, it will be exploring the application of 'close-coupled' gas atomisation. The consumption of electricity while producing titanium-based alloys is around 1.5 kWh/kg of powder. This, combined with the most economical feedstock, offers a very cost-effective finished product.

In the coming year, Arcast Inc. will offer toll-based, in-house production of powders and castings and will also sell licensed equipment for castings and powder production. Options will be available with hot gas (for greater atomisation efficiency) and gas recovery systems, where this may be economically desirable. A range of system sizes will be available to meet customers' performance requirements and budgetary constraints.

www.arcastinc.com

Powder Metallurgy thesis competition launched

The European Powder Metallurgy Association (EPMA) has launched its 2012 Powder Metallurgy (PM) Competition for theses in both Diploma (Masters) and Doctorate (PhD) levels. The aim of the competition is to develop an interest in and to promote powder metallurgy among young scientists at European academic establishments, and to encourage research at under-graduate and post-graduate levels.

In order to be accepted, the subject of the thesis must be capable of being classified under the topic 'Powder Metallurgy'. The thesis must have been officially accepted or approved by the applicant's teaching establishment during the 2009/2010, 2010/2011 or 2011/2012 academic years. Applicants must be graduates of a European university. The entry deadline is May 4 2012. Winners will be awarded their prizes at the opening Plenary Session of the Euro PM2012 Congress & Exhibition in Basel, Switzerland on Monday, September 17 2012.

Each winner will receive a cheque for €750 for the Diploma/Masters category and €1000 for the Doctorate/PhD category. Höganäs AB is once again sponsoring the competition prizes. Winners will have the opportunity of having their work published in the journal "Powder Metallurgy", and both winners will have free registration to the Euro PM2012 Congress and Exhibition.

www.epma.com/thesiscompetition

Dresden to host major international ceramics symposium

The 10th International Symposium on Ceramic Materials and Components for Energy and Environmental Applications (CMCEE) will take place in Dresden, from May 20-23 2012. The conference is hosted by the Fraunhofer Institute for Ceramic Technologies and Systems (IKTS), based in Dresden, in cooperation with the Deutsche Keramische Gesellschaft e.V., Ceramitec 2012 and the American Ceramic Society. The conference features a carefully selected technical programme consisting of invited lectures and contributed papers and posters. Conference sessions will focus on the following areas:

- SOFC materials and technology
- Energy harvesting systems
- High-temperature ceramic filters and membranes
- Advanced structural ceramics for energy and environmental technology
- Ceramic materials and systems for energy conversion and storage

Dresden will host the 10th International Symposium on Ceramic Materials and Components for Energy and Environmental Applications

- Ceramic coatings for structural, environmental and functional applications
- Novel, green and energy efficient processing and manufacturing technologies
- Advanced functional ceramic materials and systems
- Nanoscaled ceramic powders and fibers, their properties and applications
- Precursor derived ceramics
- Ceramic matrix composites (CMC)
- Transparent ceramics

For full details of all presentations visit the event website.

www.cmcee12.de

Sandvik celebrates 150 years

On January 31 2012 staff at Sandvik AB celebrated the 150th anniversary of the formation of the company by Göran Fredrik Göransson in Sandviken, Sweden.

Sandvik was the first company in the world to use the Bessemer process, which would revolutionise steel manufacturing and quickly became one of the world's leaders in its industry. Today it has 50,000 employees across the globe and is a major force in the hard materials industry. A special anniversary flag was hoisted and a spectacular fireworks display took place in Sandviken.

The group's Sandvik Osprey business is today a leading supplier of gas atomised powders for the MIM industry, offering more than 400 different grades, from low-alloy steels to stainless steels and special alloys such as nickel, cobalt and copper.

An anniversary issue of the Group magazine "Meet Sandvik" has been published in 14 languages and a book presenting the company's successful development will be released in March.

www.sandvik.com/150

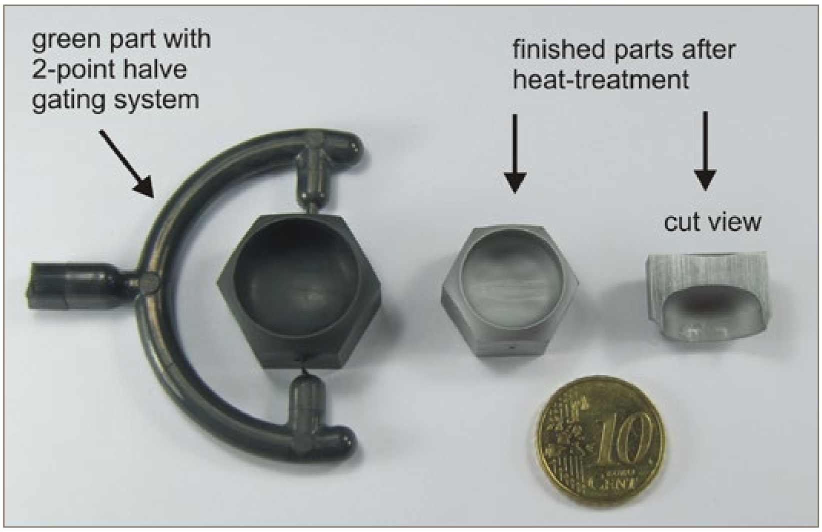
KIT investigates the mass production and joining of divertor parts for nuclear fusion power plants via multicomponent tungsten PIM

At Karlsruhe Institute of Technology (KIT), Germany, divertor design concepts for use in future nuclear fusion power plants, beyond the International Thermo-Nuclear Experimental Reactor (ITER), are being intensively investigated. The KIT divertor design concept for the future Demonstration Reactor for Commercial Power Production (DEMO) power reactor comprised applied research and the development of structural and armour materials, as well as fabrication technologies.

The divertor is one of the most important plasma-facing components of the reactor and has to withstand high surface heat loads (up to 10 MW/m²). The main function of the divertor is to remove impurities and eroded particles from the fusion plasma.

Tungsten and tungsten alloys are presently considered as the most promising materials, primarily thanks to their typical material properties such as high temperature strength and low erosion rate, to withstand extreme conditions. The manufacturing of parts by mechanical machining, such as milling and turning is, however, extremely cost and time intensive because tungsten is very hard and brittle.

Powder Injection Moulding (PIM) has been adapted to tungsten processing at KIT for a number of years. This production method is



Green part with gating system (left) and finished parts after heat-treatment (right) deemed promising in view of the requirement for the large-scale production of near-net-shape high precision tungsten parts. PIM therefore offers a significant cost-saving advantage compared to conventional machining. The properties of the successfully manufactured divertor parts, consisting only of pure tungsten, were a high density >98 % T.D., a hardness of 457 HV0.1, a grain size of approximately 5 µm and a microstructure without cracks or porosity.

Based on these results a new two-component PIM tool has been developed. This new fully automatic multicomponent PIM tool enables, in one step, the replication and joining together of different materials without the need for brazing, as well as the creation of composite materials. This tool will also enable the further development and assessment of new custom-made tungsten materials as well as allow further scientific investigations on prototype materials, for use in general R&D, and for developing industrial products for a wide range of applications.

For more information contact Steffen Antusch, email steffen.antusch@kit.edu

www.kit.edu

Alexander Michaelis receives American Ceramic Society award

Prof. Alexander Michaelis, Director of the Fraunhofer Institute for Ceramic Technologies and Systems (IKTS) was awarded the ACerS "Bridge Building Award" at the 36th International Conference and Exposition on Advanced Ceramics and Composites (ICACC) in Daytona Beach, USA, at the end of January. The event, which attracted more than 1,100 participants from more than 50 countries, is one of the most important in the advanced ceramics calendar.

The award recognises individuals who have made outstanding contributions to engineering ceramics and thus significantly contributed to the visibility of the field. The work of Michaelis and his team covers all aspects of advanced ceramics from preliminary basic research to applications. The focus is on the development and application of modern advanced ceramic materials, the development of industrial powder metallurgical technologies, and the manufacturing of prototypical components. Structural ceramics, functional ceramics and cermets are the main topics with emphasis on innovative complex systems which are applied in many industry sectors. The award, in particular, recognises Michaelis' contribution in the field of energy and environmental technology.

The 48-year-old scientist and university professor, who has already won several awards, has been a member of the World Academy of Ceramics (WAC) since 2010. He is chairman of the 10th International Symposium on Ceramic Materials and Components for Energy and Environmental Applications (CMCEE), which takes place in Dresden from May 20-23, 2012.

www.ikts.fraunhofer.de

Submitting News

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

Morgan Technical Ceramics highlights potential for CIM in energy efficient motors

Typical high volume CIM components produced by Morgan Technical Ceramics

The first requirements for electric motors due to the European Energy Using Products (EuP) directive came into effect in the summer of 2011 and many manufacturers, states Morgan Technical Ceramics, are now reviewing ways to make their products more efficient.

The aim of the directive is to reduce the environmental impact of energy using products and puts new restrictions in place for energy efficiency. It is relevant for all motors in the power range 0.75kW to 375kW and introduces a new mandatory scale for their efficiency. All motors must now meet the IE2 high efficiency standard and any motors not achieving this standard will be prohibited. The motor efficiency ratings are based on the efficiency classes defined in the IEC 60034-30 standard published by the International Electrotechnical Commission (IEC).

The requirements are being introduced in three stages. Tougher regulations for achieving higher energy efficiencies will be implemented in a second phase in 2015 and a final phase in 2017, whereby all 0.75 – 375 kW motors must be able to meet the IE3 standard, or meet the IE2 standard and be equipped with a variable frequency drive.

Approximately 70% of industrial energy demand comes from electric motors and the directive is expected to result in a dramatic reduction in CO_2 emissions. In addition, it is estimated that changes made to energy using products will cut EU annual electricity consumption by 5%, resulting in energy savings worth around €12 billion.

Increasing energy efficiencies through motor design is just one consideration and further efficiencies can be achieved by looking at all aspects of product design, the manufacturing process and involving trusted suppliers at the initial design stages.

For example, choice of materials can have a significant impact on efficiencies and many manufacturers are turning to ceramic components to help. Morgan Technical Ceramics has been working with leading manufacturers, such as Grundfos, who fully realise the benefits of ceramic and pro-actively design the material into their products.

The company's ceramic pump components are being used in circulator pumps. The requirements of circulator pumps, according to the EuP directive, will lead to a reduction in electricity consumption of 23 TWh a year by 2020 in the EU. This savings potential corresponds to the electricity consumption of 14 million people.

Stainless steel and other material components (such as shafts and bearings) continue to remain common in pump design. However, the material combination does not always offer the best abrasive wear resistance to limescale and black iron oxide particles found in heating systems. The gradual wearing of these components increases noise levels, reduces efficiency and can lead to pump failure.

Technical ceramic materials can be engineered to feature hardness, physical stability, extreme heat resistance and chemical inertness; all important characteristics to increase pump life and the efficiency of the whole system. The life cycle cost of the pump is significantly reduced by using ceramic shafts and bearings.

Ceramic is well known for being extremely hard (Rockwell Hardness of 75-86 R45N), second only to diamond.

As such, it is incredibly hard wearing and hence ceramic components used in pumps have a long lifetime despite high speeds of more than 3000 rpm. Ceramic also has exceptional corrosion resistance in aqueous based pumping applications and is not affected by corrosion inhibitors or aggressive environments.

Shaft / bearing clearances can be manufactured within 10µm to ensure minimal pump running noise and provide optimum hydrodynamic lubrication. With the negligible wear of ceramic components over many years (unlike steel) the tolerance fit is maintained, which results in less vibration and less drain on the motor. As a result it delivers optimum efficiency.

Ceramic can also be machined to micron precision tolerances using state of the art diamond processing and grinding wheel technology. For example, on a rod 0.5 mm in diameter and 200 mm in length, tolerances of 0.5 µm roundness and 2 µm straightness can be achieved. In addition, the components can be produced in high volume.

While the mechanical properties of ceramic make it the ideal material choice, Ceramic Injection Moulding (CIM) is enabling the design of more complex ceramic components to further increase efficiencies.

For components requiring high precision and medium to high volumes, CIM can offer a solution when component complexity goes beyond the boundaries of more basic forming technologies such as dry pressing and is an alternative to CNC machining of ceramics when higher volumes are not viable.

The latest manufacturing techniques are enabling the design of more complex shafts to high precision, for example, smaller, fluted shafts with multiple grooves. Morgan Technical Ceramics has facilitated the design of a rotor that can be easily attached to the shaft by injection over-moulding. This enables manufacturers to reduce costs associated with assembly and the carbon footprint from manufacturing, while reducing time to market.

Chris Paine, Sales & Application Engineer at Morgan Technical Ceramics, suggests that key to the design process is engaging with a trusted supplier early in the development stages, which enhances design capabilities. Working together, engineers from both companies can review proposals and enhance the vision and aspirations for the project. This increases innovation and creates more open thinking in the preliminary design stages, eliminates waste from the design review cycle and provides better manufacturability and a more predictable product.

With greater demand to increase efficiencies and the introduction of the EuP motor directive, manufacturers are reviewing every element of product design. By using ceramic components, the latest manufacturing techniques and involving knowledgeable partners early in the design stage, increased product efficiencies and reduced life cycle costs can be achieved.

Morgan Technical Ceramics is a market leader in pump components, extrusions and precision seals, as well as technologies such as CIM. It specialises in medium to high volume production of technical ceramic components, providing engineered ceramic solutions to customers around the world and enabling them to increase product efficiencies.

The company has recently been selected as one of Grundfos' Top 5 "Best Performance Suppliers" for 2010. This accolade demonstrates the exceptional operational and commercial performance Morgan Technical Ceramics consistently delivers to its customers.

www.morgantechnicalceramics.com

MV Multi-Trap® Vacuum Inlet Trap keeps vacuum moulding processes operating

MV Products, of North Billerica, MA, USA, has introduced a multiple stage vacuum inlet trap that protects vacuum pumps on metal injection moulding, vacuum compression moulding and vacuum hydraulic press processes which produce by-products that can damage vacuum pumps and create production slowdowns.

The MV Multi-Trap® Vacuum Inlet Trap is designed to keep vacuum moulding presses operating by protecting their vacuum pumps from waxes, polymers, volatile vapours and moisture from the gas stream. Providing a knock-down stage for collecting heavy solids and condensables, plus an optional cooling coil, users can precisely match this trap to their own process requirements.

Featuring 4.5" and 9" H user-replaceable filter elements, the MV Multi-Trap® Vacuum Inlet Trap can be configured with two stages of 10 elements or one stage of five.

www.massvac.com

"PM: A Global Market Review" to be published by Inovar Communications


Powder Metallurgy: A Global Market Review

The global Powder Metallurgy industry regained its growth momentum in 2010 and 2011 after the turbulence caused by the financial crisis of 2008 and the ensuing economic recession in 2009. Ferrous PM part production was set to break through the one million tonnes barrier in 2011 and the prospects for 2012 remain positive.

The latest edition of *Powder Metallurgy: A Global Market Review*, published by Inovar Communications, presents some of the key statistics in regional and global shipments relating to ferrous and non-ferrous PM products, hardmetals (cemented carbides), diamond tools, PM semi-products and powder-based magnets. Two inset features review PM's special relationship with the automotive industry and the continuing rapid growth of powder injection moulding (PIM).

Powder Metallurgy: A Global Market Review is included in the 15th Edition *International Powder Metallurgy Directory*, to be published in late March 2012, but will also be available to purchase as a separate download for £125 from April 2012.

Fully updated from the previous edition, the 12,500+ word review includes data on both part and powder production and features more than 50 charts and tables.

The review has been compiled by Bernard Williams, former Executive Director of the European Powder Metallurgy Association (EPMA) and a Consulting Editor of *Powder Injection Moulding International* and the leading PM industry news source, *ipmd.net*.

www.ipmd.net

PowderMet 2012 Nashville: Conference Programme now available

The Metal Powder Industries Federation has published the conference programme for the PowderMet 2012 International Conference on Powder Metallurgy & Particulate Materials, to be held in Nashville, Tennessee, June 10-13 2012.

Over 150 worldwide industry experts will present the latest in powder metallurgy and particulate materials, whilst a trade exhibition will showcase leading suppliers of powder metallurgy and particulate materials processing equipment, powders, and products.

Special interest programmes will focus on the following areas:

- Magnesium Powders & Composites: Technologies & Applications
- Delubrication Science, Problems & Development
- Spark Plasma Sintering (SPS) and Associated Processing Technologies
- Dual-Phase Materials: Processing, Microstructure & Properties

In addition to the conference sessions and special interest seminars, a number of management-focused programmes will be offered throughout the conference for MPIF members.

Powder Injection Moulding International will be exhibiting on booth #207, see you there!

www.mpif.org

The Gaylord Opryland Hotel Nashville, venue for PowderMet 2012

UK powder characterisation firm establishes US subsidiary

Building on a decade of successful business in North America, powder characterisation specialist Freeman Technology has formally established a new US subsidiary, Freeman Technology Inc.

This new organisation will take on responsibility for sales, service and applications support for the ever growing number of FT4 Powder Rheometer users in the US, and will work with prospective customers across all industrial markets. Concurrent with this, Mike Delancy has been appointed as National Sales Specialist.

Sales of the Freeman Technology FT4 Powder Rheometer have grown year on year since the instrument's introduction over ten years ago, with North America an increasingly active market within the powder processing sectors.

www.freemantech.co.uk

Carpenter Technology publishes strong Q2 results

Carpenter Technology has published positive second quarter results, with the company stating that second quarter net sales, excluding raw material surcharge, were up 19% from a year earlier due to pricing and mix optimisation on 7% lower overall volume.

Carpenter Technology Corporation reported net income attributable to Carpenter of $23.6 million for the quarter ended December 31, 2011 (net income $9.3 million in Q2 2011). Costs in the quarter relating to the Latrobe Specialty Metals transaction were $2.4 million.

Powder metal product sales were up 15%, and the company states that sales grew for high value materials required in turbo charger, gasket and fuel system applications, used in smaller, higher efficiency turbo charged engines, particularly in Europe, where the company reported a 28% increase in sales.

"Our solid second quarter results reflect continued execution of our strategy to optimise the core business by growing premium product volume and improving our overall profit per pound through pricing and mix management actions," said William A. Wulfsohn, President and Chief Executive Officer.

"Within our overall top-line results for the quarter, revenues increased 12% on 4% higher volume for our premium products, including special alloys, titanium and powder metals, while revenues for our stainless products increased 31% on 12% lower volume. Our success in driving more premium volume through our limited capacity and actions to improve our product mix enabled us to more than double our profit per pound from a year ago."

www.cartech.com

China announces rare earth quotas for 2012

The Chinese Ministry of Commerce (MOFCOM) released the first batch of rare earth export quotas for 2012 on December 27, 2011.

According to the Head of Department of Foreign Trade at MOFCOM, the Chinese government has taken management measures over the exploration, production and export of rare earth minerals, as required for the protection of resources and the environment. The department announced that in order to "guarantee international market demand and keep rare earth supplies stable", export quotas for 2012 would remain at the same level as 2011.

China's rare earth exports totalled 14,750 tons in the first 11 months of 2011, accounting for 49% of total export quotas. Large quantities of most export quotas still lay idle the department stated.

China is the major producer of rare earths, accounting for around 97% of global production. Rare earth minerals are used in many applications, from smart phones to wind turbines, with around 26% (35,000 t) used in the production of magnets. The use of neodymium-iron-boron (NdFeB) permanent magnets, produced via the powder metallurgy (PM) process, has seen spectacular growth since the technology was developed in the early 1980's

Gordon Dowson, luminary of Powder Metallurgy, dies age 99

Gordon Dowson, a popular and much respected figure in the PM industry, died in Bromley, Kent, UK, on December 18 2011. An enthusiastic supporter of *PIM International*, he was a regular guest in Shrewsbury and followed the development of MIM and CIM technology with much interest.

Gordon was a prominent figure on the European PM stage during the 1970's after he took over the helm of Powder Metallurgy Ltd based in Stratford, in the East End of London, in 1969 at the age of 57. As Managing Director of one of the then leading copper and copper-based powder manufacturers in Europe, Gordon made significant contributions to improving the efficiency of the atomisation plant, which had been established by PM pioneer Dr. W. D. Jones in the mid-1940's. He also became involved in the development of blending technology to produce large tonnage lots of press-ready bronze premixes for the PM industry. Whilst at Powder Metallurgy Ltd he was also responsibile for a monthly abstracts journal, *Metal Powder Report*, which was, at that time, published by the company.

His expertise in atomisation technology was again put to good use at PM bearing producer Manganese Bronze Ltd in Ipswich in the early 1980's, where he acted as a consultant in the design of a water atomisation plant to produce copper-based powders and press-ready bronze premixes.

Having graduated from Corpus Christi College, Cambridge in 1934, he joined Baker Platinum in 1937 to work on precious metal materials, but his career here was interrupted by the Second World War when he moved to ICI in Billingham to do research on materials for the construction of a plant for the separation of isotopes of uranium. This was a critical process in the manufacture of uranium fuel for nuclear power, also for the creation of uranium-based nuclear weapons. For a time Gordon was the world authority on the subject.

It was when he rejoined Baker Platinium, just before the end of the War, that he got his first taste of PM through his involvement in the production of thoriated platinum/4% tungsten wire used as an electrode in spark plugs in aircraft engines. Whilst at Baker Platinum Gordon also invented an atomisation process for producing spherical particles of a 'special alloy' used to make fountain pen tips. In 1955 he became Professor of Chemistry and Metallurgy at the Royal Naval College in Greenwich.

Gordon Dowson on a walk above Dolgellau, North Wales in 2006, age 94

Gordon prepared information on PM patents for *MPR* until he was 95 years of age. He had also published a successful book *Powder Metallurgy - the Process and its Products* (Adam Hilger, 1990) and contributed to a number of publications including the *International PM Directory (IPMD)* ■

Understanding defects in Powder Injection Moulding: Causes and corrective actions

Over the past 30 years Powder Injection Moulding (PIM) has experienced tremendous growth on the back of the technology's ability to deliver complex high-volume, high-performance net-shape metal and ceramic components at lower costs than alternative processes. As with all technologies, however, problems can and do arise. Professor Randall German reviews the causes of a number of defects experienced during more than two decades of troubleshooting in PIM, along with the necessary corrective actions.

I would love to report that powder injection moulding (PIM) is successful without defects, but sadly this is not always the case. In this article, based on troubleshooting the causes and cures for many defects, categorisation is provided on where some of the defects arise and more importantly how to cure these defects. As a general categorisation, often the defect cause is several steps prior to the point where the defect is observed.

Introduction

Every year frustrated PIM customers seek legal redress due to missed promises - cost overruns, missed deadlines, defective components, or inadequate quality. Defects are an unfortunate aspect of PIM that are not well documented as to causes and cures. If we can expedite delivery of proper quality components, then it might be possible to avoid redress via legal action. Hence this set of illustrations based on 25 years of consulting with the industry.

It is unfortunate that defects arise during PIM production. By way of introduction, consider the following situations that briefly tell some of the stories.

Case Study 1: Cracked CIM parts

One challenging situation arose when a large multinational firm selected a vendor to ramp up production to a target 100 million ceramic parts per year. This was a large step for the moulder who had little experience outside simple crucibles and similar shapes. Unfortunately, after sintering the complex multiple level components were cracked. Many experiments were conducted changing tooling, moulding, and sintering parameters, but the cracks remained. When the green bodies were examined in light microscopy, there were no cracks. However, after a solvent wash to remove surface binder and a fluorescent penetrant exposure, giving the image shown in Fig. 1, the issue was identified as binder separation along weld lines.

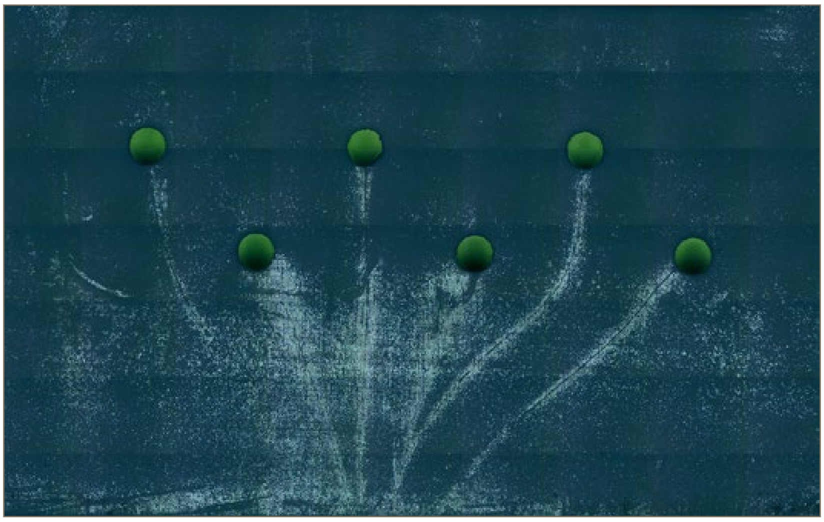


Fig. 1 This is a montage picture of a green ceramic injection moulded part that showed cracks after sintering, but on inspection fluorescent penetrant showed the weld lines were not properly bonded due to oil segregation from the binder

Up to that point, the proprietary binder formulation had been an untouchable parameter during the experiments. It was eventually revealed that the binder included an oil ingredient that never froze. As a consequence, the weld lines accumulated oil and never froze, a defect not noted earlier in simple shapes. However, by this time the frustrated customer walked away from PIM after much expense trying to fix moulding, debinding, and sintering cycles for a poorly conceived binder.

Case Study 2: Dimensional variations

A captive MIM operation forming steel firearm sights had a high scrap rate due to dimensional variations after sintering. For a year the firm performed batch sintering experiments to hone in on the highest yield of acceptable components. Thus, they were able to centre the critical tolerances by the degree of sintering, but still yields and densities were low, sometimes in the 60% range. Measurements at the MIM moulding machine determined the moulded components were widely varying in mass. In statistical studies it was determined that sintered dimensional variation was mostly due to moulded mass variation. The higher mass parts shrank less and were too large, while the low mass parts shrank more and were too small. The inclusion of a die cavity pressure transducer enabled the moulding machine to improve mass uniformity and what followed were significantly tighter dimensional parameters after sintering. In this case, the problem was not in sintering, but in loosely controlled moulding.

'In statistical studies it was determined that sintered dimensional variation was mostly due to moulded mass variation.'

Case Study 3: Rusting parts

In a recent situation, a medical device firm launched a large programme involving a dozen MIM components, but the sintered stainless steel parts were out of size and, more frustratingly, were rusted on delivery. Rusting parts are not acceptable for medical devices. The moulder blamed everything on the tooling. Lawyers got involved in determining who was at fault. The culprit was an improper sintering cycle. The MIM shop apparently had limited equipment capabilities. Had the proper time-temperature-atmosphere been applied during sintering, then rusting and poor sintering could be avoided. Instead, it was easier to blame the tool maker. The customer pulled the project and went elsewhere with a negative opinion of MIM.

Case Study 4: Tooling design

A scientific pump housing exhibited sintering cracks. The MIM shop announced they needed a new and very expensive batch sintering furnace, to be paid for by the customer. Prior to parting with the $600,000 to buy a furnace, the customer asked for an independent analysis. On microscopic

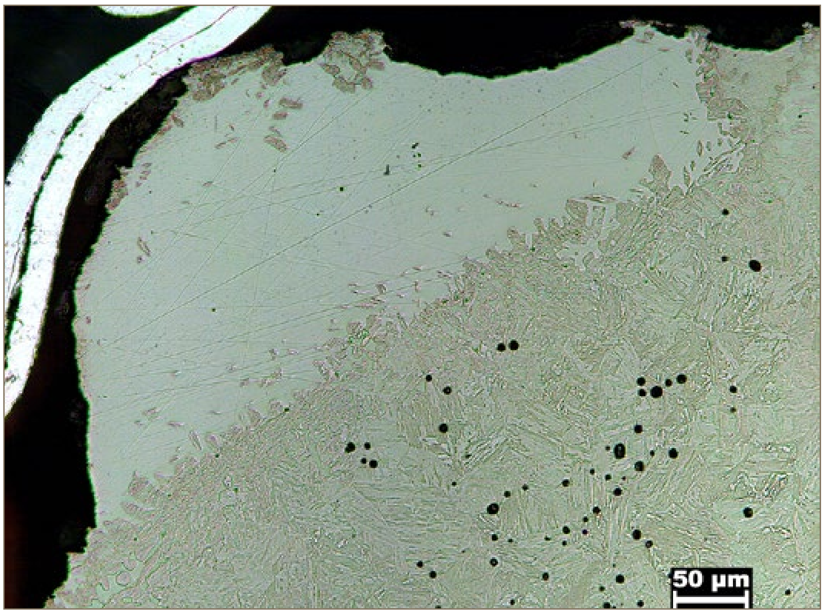


Fig. 2 Surface decarburisation on a gun sight near an outer edge. The very left portion of this image is a retainer ring used for polishing

inspection, cracks were detected in the green body, but the cracks were very small and not easily identified. Sintering opened the cracks, but did not cause the cracks; sintering just made them visible. A new furnace was not the proper cure. Examination of the tooling draft angle determined the parts were sticking on ejection and cracked, but once the ejection stress was relaxed the cracks sealed and were "hidden". An adjustment to the tooling cured the problem.

Case Study 5: Binder design

Another example came during large scale production of disk drive components. Cracks were noted after batch vacuum sintering. Many batch sintering experiments were conducted to statistically isolate if there was a time, temperature, or furnace location that contributed to the 10 to 20% loss on each run. Much effort was directed to the sintering furnace. In the end the cracks were traced to moulding and the failure of the binder to include a mould release surfactant, leading to difficult and inconsistent moulding. When the surfactant was added to the binder, a new issue arose. The parts now moulded too easily. Since the feedstock was underloaded (initially to compensate for the high viscosity associated with the missing plasticiser-surfactant) flashing occurred with the new, lower viscosity feedstock. Of course attention then turned to reducing the packing pressure, but the sintering problem went away.

Case Study 6: New powders

In trials with a new low cost powder replacement for carbonyl iron, an urgent report came in that the moulding machine froze. After assessing the situation, it turned out the powder had microporosity. Those pores very slowly absorbed molten binder and in doing so effectively removed the binder from its lubrication role during mould filling. Effectively the solids loading slowly increased over time in the moulding machine barrel. The problem first occurred when the moulding technician left the moulding machine hot while taking a lunch break. Accordingly, the powder was modified with a tumbling treatment to seal surface connected pores.

Case Study 7: Changing binders

During development of a water-containing binder, standard alumina parts were being evaluated for conversion from wax-polymer to agar-water binders. The moulding technician ran the standard moulding cycle only to see the ejector pins push all the way through the soft feedstock in the tooling. Since part production costs depend on moulding cycle time, the customer was unwilling to change cycle times. Thus, a newly formulated binder was required that included a calcium salt as a gelation accelerant to fit the customer's cycle time demands.

Case Study 8: Decarburisation

In the case of a MIM gun sight, a surface decarburisation problem

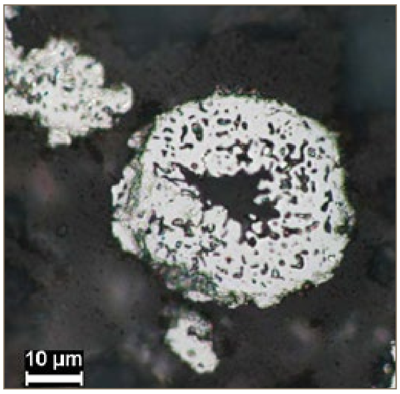


Fig. 3 Polished cross-section through an iron particle illustrating an example of a powder with internal pores

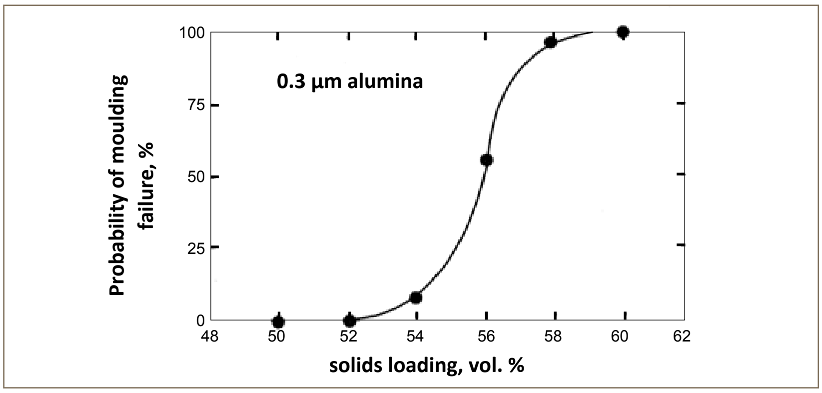


Fig. 4 A plot of moulding defects for an alumina component versus the feedstock solids loading. Typically laboratory rheology experiments would identify the proper solids loading and avoid the need to chart moulding defects

showed up during surface blackening as shown in Fig. 2. Of course the coating process was blamed, but once samples were obtained and cross-sectioned, it was evident the steel was exhibiting surface decarburisation during sintering. Changes were made to the firing cycle in terms of support trays and atmosphere, but by this time the customer went to a new MIM vendor. In a related situation, black gun metal firearm components were growing whitish surface films weeks after production. All of the experimental work was on meeting dimensional specifications, but the sintered density was low with about 6% porosity. This resulted in surface connected pores that retained the hydroxide salt solution used in blackening. Over time the salt retained in the pores oxidised to generate a surface growth that caused the delayed discolouration.

Case Study 9: Controlling properties

An automotive supplier was including a MIM deflector in an air-bag sensor system to detect if a passenger was present. The MIM stainless steel part actuated the on/off condition, but fractured when a heavy passenger sat in the seat. Tests for the material showed density and chemistry were acceptable, but the crystal phase was wrong. Ferrous alloys can be sintered in temperature ranges where they can be body-centred cubic ferrite, face-centred cubic austenite, or at high temperatures body-centred cubic delta-ferrite, and of course depending on cooling rates it is possible to form body-centred tetragonal martensite.

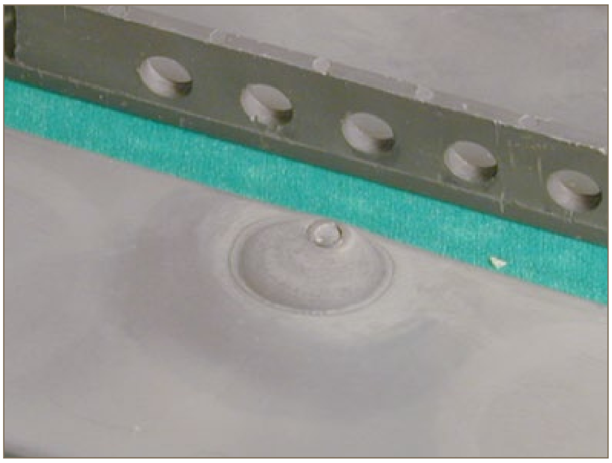


Fig. 5 A blossom of powder-binder separation near the gate

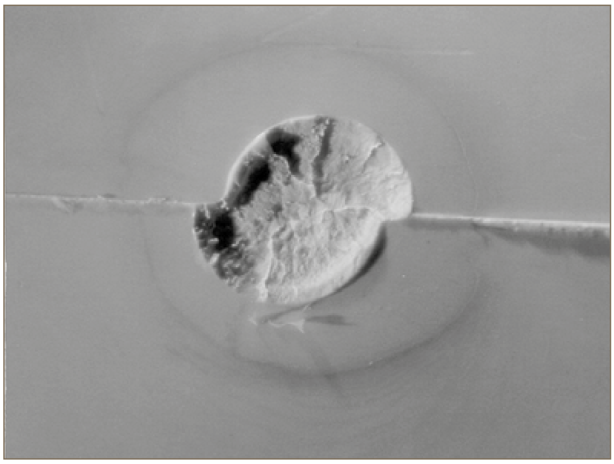


Fig. 6 This gate shows several issues, including some penetration of the parting line, poor alignment of the gate, and powder-binder separation at the gate

The impact toughness, performed using standard ASTM Charpy tests revealed the material was glass-like, with literally no toughness. Thus, when a heavy person dropped into the passenger seat the deflector exhibited brittle failure. The cure was to modify the sintering cycle for a lower peak temperature. But before the MIM firm could explore sintering cycle options the customer jumped to a backup production route.

Corrective actions for key process areas

We recognise PIM and MIM as effective net-shape technologies. This short listing of problems is not meant to degrade the technology successes, but to help lay in place some logic for finding root causes and making appropriate corrections. This article is based on real problems. After doing the obvious trials everyone becomes frustrated. It is common to see many expensive experiments with several furnace loads of scrapped components prior to calling in expertise. In one case, 57 expensive hot isostatic pressing runs were explored with variations in time, pressure, temperature, and so on, trying to reach full density. From that data a statistical regression showed the proper run conditions, but only after significant waste.

This article gives categorisation to some common PIM problems, outlining the symptoms, root causes, and corrective actions. Many other examples are being uncovered daily, but remain anonymous. Unfortunately, the problems are real and represent some very frustrating efforts; the names are not disclosed to protect the guilty and innocent. The defects are organised around a few main causes associated with the feedstock (binder and powder), moulding (operating conditions and tooling), debinding, and sintering.

Feedstock

Early in metal powder injection moulding, a new atomised alloy powder was offered to the industry. Initially it was touted as a new supply variant but it did not sinter well. Careful inspection of the powder in scanning electron microscopy showed no problems. However, in cross-section the powder proved to have pores as seen in Fig. 3. Such pores trap gas that proves difficult to remove by sintering. The cure is to specify the particles as dense, and to verify the absence of internal pores.

During development of an alumina injection moulding capability, relying on a standard powder and binder, variations in solid loading were used to adjust final dimensions. However, defects were noted at the moulding machine. This is a case where tooling was generated too early in the process development. A simple plot of frequency of defects versus solids loading, such as shown in Fig. 4, provides guidance on the proper solids loading. It is best to design the tooling after the solids loading is determined.

A high solids loading leads to moulding defects of short shots, cracks, and sticking. On the other hand, a low solids

Problem	Observation	Cause	Cure
Part sticks in mould	Difficulty with ejection	Packing pressure too high	Reduce packing pressure to allow for lower retained pressure
		Ejection temperature too high	Delay ejection in cooing cycle to contract part more
Short shots	Part freezes prior to filling cavity	Low filling rate	Fill faster to avoid freezing during filling, use larger gate if necessary
		Mould too cold	Provide heat to mould or better control mould temperature
		Low feedstock temperature	Increase nozzle temperature
Air pockets or voids	Green component has large pores	Jetting in moulding	Filling is too fast and feedstock blocks vent
		Inadequate compression in moulding	Screw taper is wrong, generally compression ratio of 2 is best to remove voids
		Gate is too small	Reduce filling rate by enlarging gate diameter
		Granule size or shape causing bridging in hopper	Large pores in feed results in air pockets, use uniform and rounded granules
Sink marks	Sample is not filled to proper size	Packing pressure	Feedstock contracts in cooling and shrinks from mould and needs higher packing pressure
Internal cracks	Often seen near core rods or pins	Check taper on tooling and alignment of tooling	Tool motions are not aligned or tapered ejectors or cores are doing damage during motion

Table 1 Moulding defects, causes and cures

loading also leads to powder-binder separation. This is usually seen near a small gate, as pictured in Fig. 5.

Moulding

Plastic moulding has an array of well recognised defects that include short shots, unhealed weld lines, gate blemishes, parting lines, and blocked vents (trapped air). These are treated in standard textbooks. Usually the tool witness line is small, but in a few cases where the tool halves are not properly aligned the defect is quite large, as illustrated in Fig. 6. This picture shows a gate mark with the two halves offset, indicative of poor alignment and further there is discolouration around the gate from powder-binder separation.

A more subtle difficulty comes from part differences in multiple cavity moulding. Usually a balanced runner system is employed to ensure each cavity fills and packs the same, but curiously similar attention to balance is sometimes missing on the cooling system. It takes special care to ensure each cavity has the same coolant temperature and same flow pattern. Isolation of the dimensional variation from unbalanced cooling channels took considerable effort to isolate.

Delamination cracks are sometimes evident after moulding. One cause is sticking to the mould, possibly from excess packing pressure. A relief angle is recommended, otherwise the low elastic modulus of the moulded feed-stock gives bending on ejection, leading

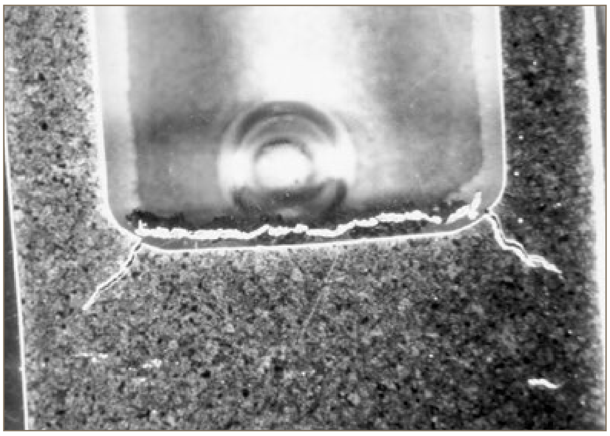


Fig. 7 Internal cracks observed in a sintered component, but traced to tooling alignment issues in moulding

Fig. 8 Cracked tooth brush vibrator weight, made from tungsten, found to result from residual stress from a fast cooling cycle and high molecular weight backbone binder ingredient

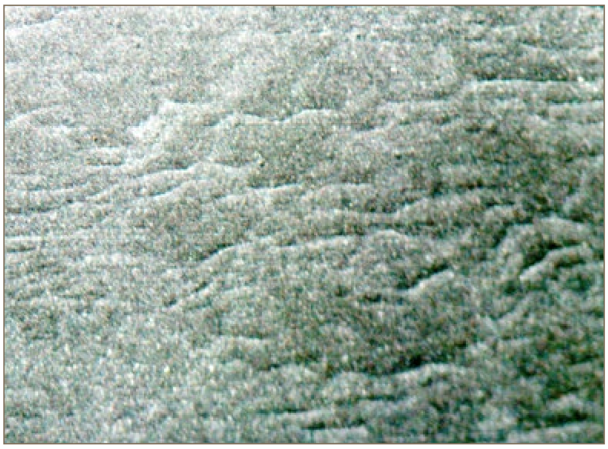


Fig. 9 Picture of the rough surface formed when the feedstock is too cold and slips on the tool surface

Fig. 10 Picture of moulded body with a previously frozen gate caught up in the melt front to form a defect

to internal cracks. Fig. 7 is a micrograph of internal cracks formed by ejection, seen here after sintering. Another cause is from the surfactant (or missing surfactant) in the binder. Ingredients that act as plasticisers also provide mould release. In some operations the solution is to apply mould release periodically, but a more effective solution is to add a surfactant to the binder formulation, stearic acid for example.

Moulding cracks are a common difficulty, usually associated with too fast a fill and too high a pressure. Feedstock that is over-pressurised cracks on ejection or has a high residual stress. One MIM manufacturer let the moulded parts sit for two days prior to debinding to eliminate cracking. This is because the longer molecules that serve as the backbone require time to relax, and as the filler phase in the binder is removed the softened part was responding to the residual stress with delayed cracking. To avoid an "aging" treatment requires reformulation of the feedstock with a shorter molecular weight backbone. In polymers there is a property termed the stress relaxation time constant, based on the viscosity divided by the elastic modulus. If the cooling cycle is fast compared to the stress relaxation time constant, then the component has residual stress that can cause distortion or even cracking, as illustrated in Fig. 8.

Staining is often observed near a small gate. This is due to powder-binder separation and is associated with a small gate and high shear rate in moulding. It is exacerbated by an underloaded feedstock, where the powder and binder take different trajectories due to density differences. In some cases the situation can be corrected using a redesigned fan or tangential gate.

A defect seen after moulding is a rough part surface, as pictured in Fig. 9. This is due to a cold mould and the binder freezing without flowing over the tooling. The frozen feedstock partially slips on the mould walls due to pressure, but is not properly flowing. A related difficulty comes from situations where a cold gate from a prior shot is captured in the flow front and carried into the next part, giving a dramatic defect such as seen in Fig. 10. The size of the defect is similar to the gate size, a first indication of the problem.

A few other defects observed in moulding are listed in the accompanying Table 1.

Debinding

With a wide variety of binders comes an equally diverse range of debinding routes. The major debinding route is still pyrolysis. In thermal debinding there are occasions of blister formation as shown in Fig. 11. Blisters are observed after a rapid heating cycle, especially on thicker sections. There are two major root causes. The first is from simply too rapid heating for the section thickness. For each component there is a map of critical or maximum heating rate versus temperature that identifies when the binder breakdown is too rapid. Indeed, several binders are designed around thermogravimetric analysis (TGA). Mass change is recorded

Problem	Observation	Cause	Cure
Large pores	Microstructure shows large voids	Delayed reaction between impurities	Reactions generate CO, CO_2, or H_2O, so removal of oxygen is required via slower heating
		Packing pressure	Low packing pressure or jetting fails to expel air in moulding; adjust moulding pressure
		Moulding temperature	Feedstock cooling causes sink mark or internal voids, evaluate lower nozzle temperature
		Feedstock mixing	Polymer particles need to be thoroughly melted in mixing, otherwise form large pores
Warpage	Component distorted	Thermal gradient	Components tend to warp or curl toward heat source
		Density gradient	Moulding induces solids gradient in body that causes differential but constrained shrinkage, modify moulding conditions to avoid density gradients
		Thick-thin sections	Stress arises since thin section densifies first, best to slow heating rate
		Setter design	Design conformal setters to provide dimensional support during heating and shrinkage
Distortion	Thin edges bend	Backbone polymer has residual stress	Slow cooling in moulding cycle or move to lower molecular weight backbone
		Powder selection	Low interparticle friction requires addition of irregular powder
Cracks	Surface layer delaminates	Internal vapour generation under skin	Surface pores need to be perforated, possibly using solvent debinding first and less backbone
	Cracks near tool features	Unhealed weld lines, damage from ejection	Crack is introduced by tooling but is difficult to detect, sintering opens crack to be visible
Porosity	Sintered density is low	Insufficient peak tempera-ture	Higher peak temperature is required to induce more densification
Rounded edges	Sharp features are rounded after sintering	Sintering naturally pulls particles together and rounds edges and corners	Lower peak sintering temperature with sacrifice of sintered density
Carbon issues	High or low carbon	Surface carburisation or decarburisation	Carbon chemical potential needs control, carbon gain from graphite furniture or carbon loss from oxidation during thermal cycle, possibly from atmosphere or substrate
		Binder contamination	Remove binder ingredients that convert to graphite instead of burning out, such as cellulose, agar, and polystyrene
Surface finish	Rough surface	Particle size	Sintered surface roughness is proportional to particle size, use smaller particles for smoother surface
Component melted	Part shape is lost, formed puddle	Temperature sensing	Use witness temperature indicators to verify all regions are at selected temperature
		Reaction with substrate	Low melting phase formed, such as ferrous parts on graphite tray inducing liquid during sintering

Table 2 Debinding defects, causes and cures

Fig. 11 Blisters on an alumina PIM component after thermal debinding

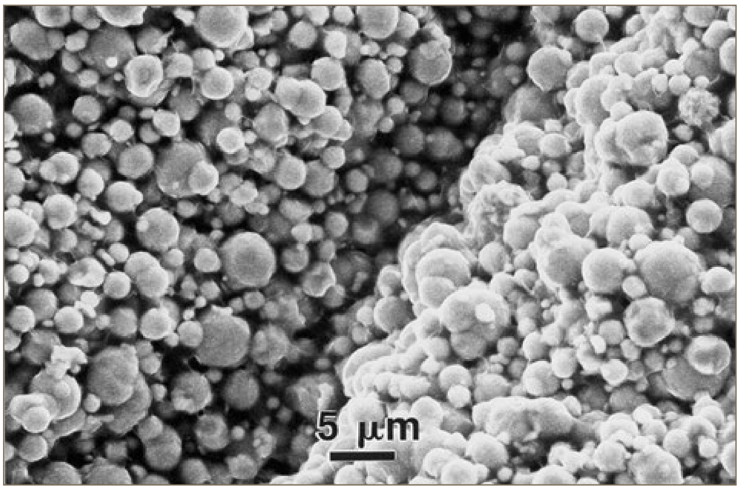


Fig. 12 Environmental scanning electron microscope image of a weld line that has not properly bonded. The sintered component showed cracks in these same locations, indicating that sintering opened this small defect

during heating in the TGA device, and such an analysis enables in situ mapping of temperatures where slow heating is mandated. The slowest heating rate is in the temperature range where the fastest rate of mass loss occurs. As the section thickness increases, heat transport into the component interior lags, so even slower heating rates become necessary with thicker sections.

A debinding defect difficult to detect is binder migration. At temperatures over the binder melting point, but below the binder evaporation point, the binder is a liquid and flows within the porous body. It is possible for the migrating binder to seal previously opened surface connected pores, trapping expanding binder inside the component. This leads to blisters. Usually slower heating cures blisters, but in the case of migrating binder it is appropriate to use first stage solvent debinding or change the binder ingredient ratio to generate more pores early in debinding. Often heating rates of about 1°C/min or slower at 200°C are required. This is a difficult heating rate to control at this low temperature, especially in vacuum, so a process atmosphere is recommended to provide heat conduction to the parts.

Cracking is also seen when binder is trapped inside the component at temperatures in the 250°C range. Poor heat transfer is often an issue. This is a reason for the high heat conduction atmosphere of hydrogen, even if there is no low temperature thermodynamic benefit. Cracking in debinding is also seen if the particles are flat faced. As the binder melts, the flat faces pull together and in making contact generate stresses that nucleates cracking. If the backbone binder is softened during debinding, then the particles can move and initiate cracks, as evident in Fig. 12, for an under-loaded system. A special situation arises in components with thick-thin section combinations. Stress arises at the interface, where the thin region is debound while the thick section has remaining binder. Although the stress is low, still the component is weak and susceptible to the stress gradient. More information on debinding defects, causes and cures is given in Table 2.

'Sintering issues abound in PIM, but still many are due to the fact that sintering amplifies defects introduced earlier in mixing, moulding, or debinding'

Sintering

Sintering issues abound in PIM, but still many are due to the fact that sintering amplifies defects introduced earlier in mixing, moulding, or debinding. Changes to the sintering cycle are ineffective in fixing such root defects. For example, Fig. 13 depicts a large pore in partially sintered steel; most likely this pore resulted from a polymer particle inclusion in an improperly mixed feedstock.

Cracks found after sintering often happen earlier in the process,

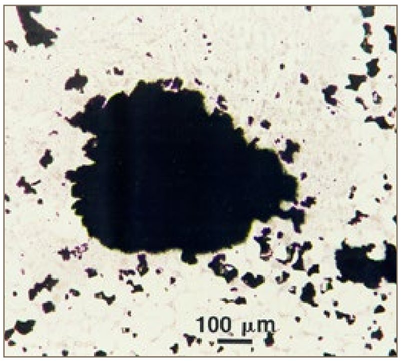


Fig. 13 This large pore in a partially sintered MIM steel is a defect indicative of improper mixing, for example retaining a polymer pellet in the feedstock

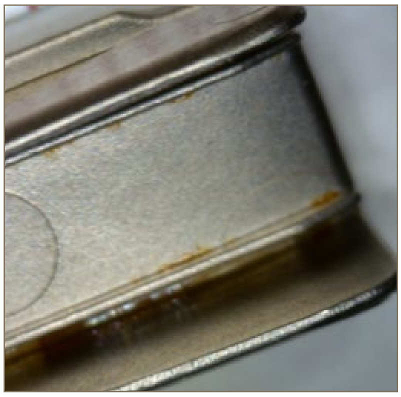


Fig. 14 A picture of the surface rust observed on an as-received MIM stainless steel component, indicating that the vacuum sintering conditions were improper for meeting medical device needs

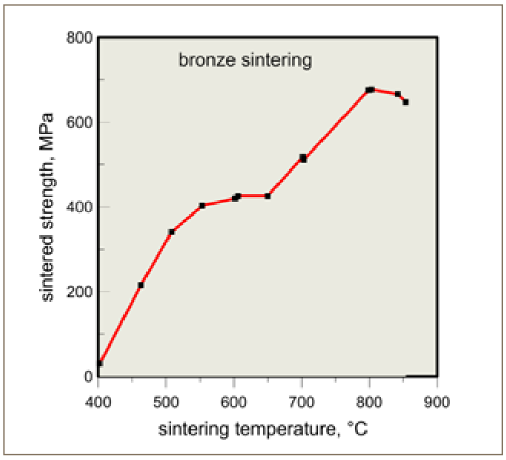


Fig. 15 The sintered strength of MIM bronze powder versus the peak temperature, illustrating how properties increase due to densification in sintering, but subsequently fall due to grain growth

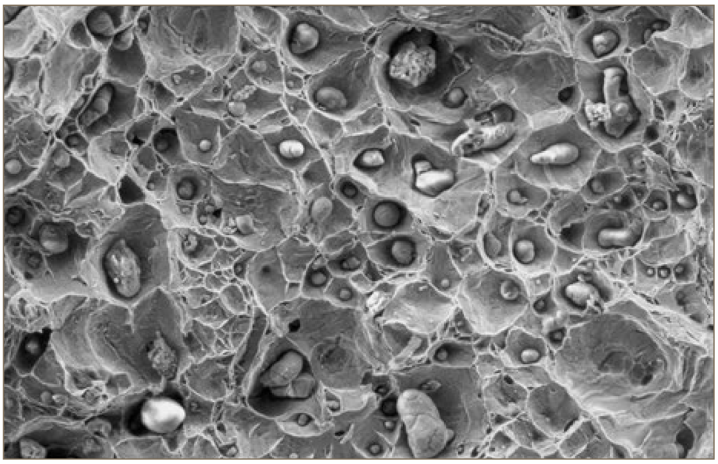


Fig. 16 Limited ductility is attained in this MIM stainless steel because of a reaction between silicon, chromium, and oxygen during sintering to produce inclusions that nucleate failure at low strains

sintering simply opens the crack. More than anything else, the most common cause of sintering cracks is powder-binder separation at weld lines. Careful examination of the green part for separation will show if the difficulty lies in moulding. Other sources of cracks are listed in the tables, and they include combinations ranging from thick-thin sections to improperly designed substrates. Recognise that long cracks happen early in processing while the material toughness is low, prior to sintering.

Not all sintering operations are the same, and that is most evident with respect to the corrosion resistance and impact toughness of MIM stainless steels. The wrong choice of a sintering atmosphere can result in chromium loss or compounding in an oxide, carbide, or nitride, removing the chromium from its role in preventing rust. For stainless steels, the safest atmosphere is hydrogen. Vacuum sintering is common, but vacuum preferentially removes surface chromium from the component since chromium has a higher vapour pressure versus nickel or iron. In some cases MIM parts show surface rust even prior to shipment if sintered in vacuum, as pictured in Fig. 14. Nitrogen in the sintering atmosphere causes chromium nitride precipitation during cooling, and in doing so removes chromium from its passivation role. Carbon contamination from the binder, heating elements, or sintering substrate leads to chromium carbide and degrades the material. Oxygen is not specified in MIM stainless steel, but it has a similar effect on corrosion resistance and must be kept low. Thus, the best solution for high quality sintered stainless steel is to sinter in dry hydrogen.

> *'Statistical experiments usually show differences in sintered dimensions depending on location in the furnace. Special effort is required to ensure uniform heat transfer'*

Warpage during sintering is common with thick-thin sections. The solids loading in the thin section is lower than in thick section, from what is termed the wall effect, so during sintering a stress arises as the thin sections shrinks more and is resisted by the thick section. The shrinkage resistance induces warpage or even cracking.

Most of the binder systems are designed to burnout cleanly during heating to the sintering temperature. But some stronger backbone polymers tend to form graphite and should be avoided. For example, early in MIM there were impressive binders that relied on polystyrene, starch, or cellulose as the backbone, but they failed due to carbon contamination.

Likewise, sintering substrates are a concern. The lower cost ceramic trays contain silica that leads to contamination during sintering. Silica has a high vapour pressure and decomposes, contaminating the material being sintered and the furnace, resulting in surface discolouration. Although more costly, alumina or zirconia are better choices to avoid contamination. Curiously, recent work has traced oxide contamination to small oxide particles trapped in the polymers from polymerisation.

Statistical experiments usually show differences in sintered dimensions depending on location in the furnace. Special effort is required to ensure uniform heat transfer; parts on the outside of the furnace have significantly different pathways versus parts on inside. This is especially evident in large batch vacuum furnaces where heat exchange is poor until the heating elements reach 500 to 600°C and emit significant thermal radiation. Components sitting in shadowed regions will be undersintered. In a continuous furnace the incoming gas flow can create local cool spots with less sintering.

Sooting is seen when heating is rapid. The binder decomposes to form graphite that rains on the parts. It can then discolour the component. Cures for graphite sooting are to reduce the heating rate, increase the gas flow, or to shift to lower molecular weight binder species. Other discolouration might come from contamination of

Sintering time min	Tensile strength MPa	Elongation %
15	920	16.5
30	930	18.2
90	940	22.0
120	845	15.6

Table 3 Strength and ductility for 95W-3.5Ni-1.5Fe sintered at 1500°C

Purchase quantity per year	Piece price, $
1,000	15.69
10,000	8.95
100,000	4.05

Table 4 Pricing versus production quantity

the feedstock. For example copper particles discolour steel. Stainless steel contamination is easily tested by dipping the part in household chlorine bleach.

Sintered microstructures tell a story on the heating trajectory. Grain growth and pore growth accompany sintering densification, so it is common to see large grains and even large, spherical pores in the microstructure. If heating is rapid, then there is a danger of component warpage and rapid grain growth. Once grain boundaries separate from the pores all densification stops; more heating fails to improve density and indeed properties are degraded. The cure is slow heating at the higher temperatures. If full density still is not achieved, then a problem might be trapped gas in the pores. Sintering in inert gases, such as helium and argon, will give incomplete densification since the gas is insoluble and resists final pore annihilation. Reactions between impurities during sintering produce spherical pores in the microstructure, often separated from the grain boundaries. The reaction products form insoluble gases, such as C and O giving CO or CO_2 or H and O giving H_2O.

For alloys formed using mixed powders, incomplete alloying results in a splotchy microstructure. Alloy homogenisation during sintering tends to be slower than densification, so an acceptable sintered density might have a heterogeneous appearance. There are three cures – use smaller additive particles to cut diffusion distances, increase temperature to promote faster diffusion (probably with more grain growth), or increase peak temperature hold time with similar grain growth complications.

In the case of sintered properties, the three factors of concern are sintered density, grain size, and impurities. Assuming good processing without contamination, then low sintered properties are associated with the time-temperature combination. A long sintering time can give a higher density, but it causes enlarged grains. Grain growth is rapid as pores are annihilated during sintering, but both density and grain size are important to properties.

Too short of a sintering time or too low a sintering temperature results in incomplete densification. For example, Table 3 gives data on a 95% tungsten alloy sintered for various times at 1500°C. Note how both strength and ductility pass through a peak at 90 min sintering time.

Another example is plotted in Fig. 15 for sintering MIM bronze at various temperatures; the sintered strength declines at the higher temperatures (heated at 10°C/min), tested for strength on cooling. There is a temperature-time combination that defines a sweet spot for property optimisation. In the case where density and strength are acceptable, but ductility is low, contamination should be considered. Another situation arises from an improper microstructure. Most of the work has been with 17-4 PH stainless steel. In one case low ductility was traced to hard ceramic inclusions analysed as silicon-chromium oxides. This came from the powder atomisation treatment. At high temperature the silicon impurity reacted with chromium to form oxide inclusions that nucleated premature fracture. This is seen on the fracture surface shown in Fig. 16. The cure is to find a powder with low silicon content.

Finally, if sintering densification remains low in terms of sinter density, and properties tend to decline with higher temperatures or longer times, then something is preventing pore annihilation in sintering. Most likely this is due to a trapped gas (such as argon) in the pores. Densification is critically dependent on annihilation of pores using grain boundaries, so anything that resists pore annihilation (gas pressure) or increases the diffusion distance (pore-boundary separation during grain growth) will halt sintering. The cure is to slow heating and to ensure all atmosphere ingredients are soluble in the material.

Nebulous

Even when the overall PIM process seems to be operating properly, there are still some opportunities for those nebulous complaints. Probably the most common is cost: the component is too expensive. This is not really a defect, yet is consistently a problem as final negotiations take place.

'A few factors dominate PIM component cost – production quantity, material, tolerances, and feature combinations - all need to be addressed for optimised pricing'

If the piece price is high, then the parts producer can ask for reduced tolerances, a change in features to increase yield, redesign to decrease mass, or a substitute material. Also the buying quantity is important as illustrated for a stainless steel medical device in Table 4. The piece price decreases by nearly a factor of four in going from 1,000 to 100,000 per year. A few factors dominate PIM component cost – production quantity, material, tolerances, and feature combinations - all need to be addressed for optimised pricing.

Another of the nebulous defects comes in the form of low mechanical properties – the strength is low or the fracture toughness is low. In polymorph materials, such as titanium or iron, the distribution of phases in the sintered product influences the mechanical properties. If the weaker phase is continuous, then it provides a path of easy fracture. It is appropriate to analyse the microstructure, take microhardness traces across the phases, and adjust heating and cooling cycles to favourably influence the properties via the microstructure.

Another common difficulty relates to dimensional capabilities. Using 62 different studies, the coefficient of variation averages 0.2% (coefficient of variation is standard deviation in size divided by mean size). Several factors contribute to dimensional variation, and control improves with better mixing and better moulding. Moulding control is sampled using mass variation at the moulding machine. The anticipated sintered dimensional variation is about one-third the green mass variation (as percent). Cavity pressure and temperature transducers improve moulded component uniformity. Other factors that improve dimensions are feedstock homogeneity (measured using capillary rheometry).

Usually process yield is not an issue once a component is stabilised. Yields are typically 95 to 98%. For lower process yields, the introduction of measurement and control tools pays off; remember "you get what you measure" so the addition of selected test points in production helps stabilise the product simply because employees are alerted to the issues. Some common tests to improve yield are to measure viscosity (melt index or capillary rheometry) after mixing, add instrumentation to moulding in terms of mould temperature and pressure transducers, do dimensional and mass checks after moulding and debinding, and include witness samples and temperature indicators in sintering to ensure all parts of the furnace are performing the same.

'Readers of this article are urged to remember all of the successes in PIM. Although several poor examples are given here, it is important to recognise the many successes'

Summary

Readers of this article are urged to remember all of the successes in PIM. Although several poor examples are given here, it is important to recognise the many successes. If PIM were easy, then everyone would be practicing the technology. Instead, there are complexities and historically it took the advent of microprocessor controls on moulders and furnaces to stabilise PIM sufficiently to see commercial success. As we add more process control metrics, some of the difficulties listed here are hopefully in the past.

Suggested resources

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Innovation helps producer of CIM products for luxury applications expand into new markets

Some of the world's most elegant CIM products are manufactured by Formatec Technical Ceramics B.V., based in the Netherlands. The company is a specialist manufacturer of luxury CIM products made from zirconia, which have a high gloss, aesthetically appealing surface finish. In addition, Formatec specialises in complex technical components manufactured from zirconia, alumina and silicon nitride. Dr Georg Schlieper reports on a recent visit to Formatec for *Powder Injection Moulding International*.

In the far south of the Netherlands, within a stone's throw of the Belgian border, lies the small town Goirle, home to the ceramic injection moulding (CIM) company Formatec Ceramics. The company was started 15 years ago as a spin-off of a plastics injection moulding company which still operates next door. The factory owner, Peter Kuijpers, was looking for new products with more added value and he realised that ceramic injection moulding was a good addition to the existing business. Today the company is owned by a group of local investors and Kuijpers has retired from the daily business, however he still acts as a management consultant offering advice for long term strategies.

Formatec's Managing Director, Michiel de Bruijcker, is a mechanical engineer who acquired much of his professional experience during a two year spell as a production manager at Biosensors International in Singapore, a manufacturer of heart catheters. "This overseas assignment brought me a great deal of experience in all aspects of business operations. Effective cost accounting, implementation of lean management and obtaining the ISO certificates - all these were very valuable lessons for me before I joined Formatec in 2007," stated de Bruijcker. Although his previous position was not related to injection moulding, de Bruijcker feels that it was a useful preparation for his role at Formatec.

CIM production

From the start, Formatec's CIM technology was based around BASF's Catamold® feedstock system. The necessary knowledge about sintering ceramic materials was acquired in close cooperation with the Dutch research institutes TNO and the Energy Research Centre of the Netherlands (ECN). For the majority of its products Formatec still uses the Catamold® feedstock system, but is also well experienced using a binder system provided by Tosoh Corporation, the Japanese supplier of ceramic powders. Current feedstock consumption is approximately 3000 kg per year.

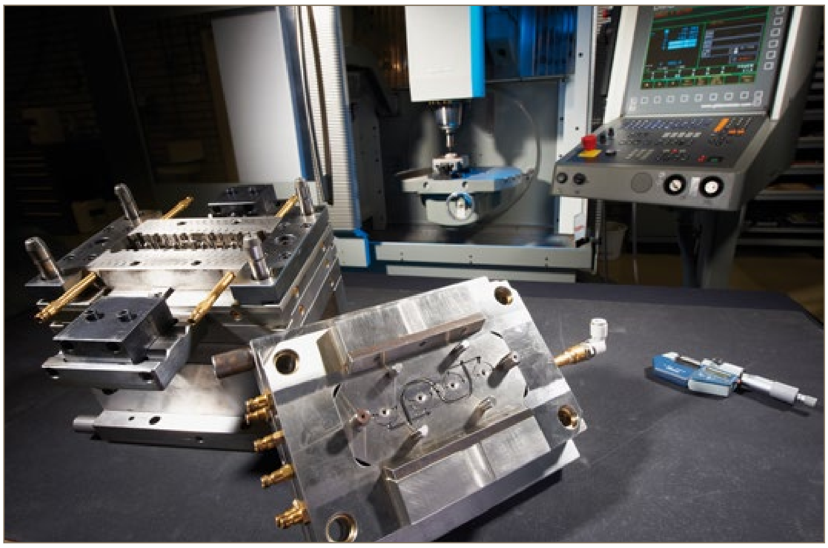

Fig. 1 CIM tooling at Formatec

Fig. 2 View of Formatec's injection moulding shop

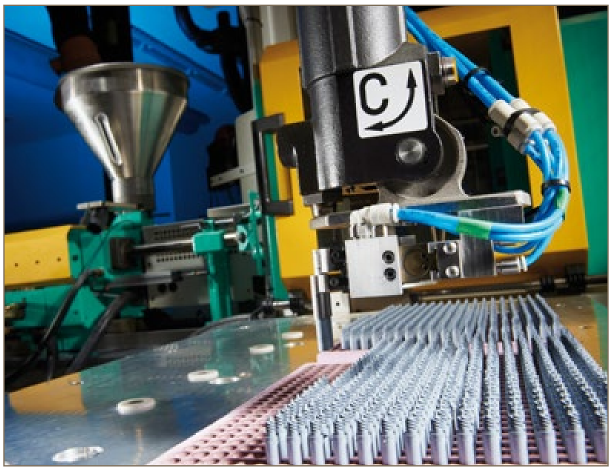

Fig. 3 Green gears being placed on ceramic trays by a pick-and-place system (Courtesy Formatec)

A great deal of injection moulding experience was available during the startup phase thanks to the company's roots in the neighbouring plastic injection moulding business. Multiple cavity tooling, with a maximum number of six cavities, is in use. Hot runner systems are also used, but these are primarily used for technical products. Harrie Sneijers, Sales Manager at Formatec, explained, "The use of hot runner systems for aesthetic applications is difficult as these systems have been designed for plastics. They have corners and recesses where feedstock can be held for some time and when it is released, this causes minor inconsistencies which are harmless for plastics and technical CIM parts, but unacceptable for the high optical requirements of our aesthetic products."

Computer simulation software for CIM is regarded by the company as being still at an early stage, commenting that the simulation software that exists for plastic injection moulding appears not directly applicable to CIM. Formatec follows developments, but to-date a decision has not been made to invest in such software.

An in-house tool shop is available for a flexible and fast response to customer demands by manufacturing prototype moulds and for maintenance (Fig. 1). More complex moulds are supplied from an external tool manufacturer. Tool wear, suggests Formatec, is not a serious issue due to a very efficient coating on the mould.

The company operates six injection moulding machines from 25 to 50 tons clamping force within its 1400 m² plant (Fig. 2). Parts are removed from the moulds and placed on trays for debinding by fully automated pick-and-place systems (Fig. 3).

Formatec sees itself as a specialist in taking full advantage of the ability to machine CIM parts in the green state after the first in-line quality control inspection. The company initially assesses the quality of the green preforms for problems that might arise later on in the process. In the majority of cases this quality control step is performed by visual inspection, if necessary under magnification.

Green machining by turning or milling has progressed to a sophisticated level and is used as a way to make low volume CIM production financially viable, as well as a route to achieve more complex features (Fig. 4). If the production volume of a part is low, it is frequently more economical to machine some geometrical features than to build prohibitively expensive tooling. Sneijers commented, "Of course, the break-even point depends on the complexity of the part and the quantity of parts needed. Therefore it is important that we develop a feeling for the market potential of the final product that is manufactured by our customer. We show them both options and then decide together with the customer."

Debinding is performed using nitric acid in two debinding units. Because

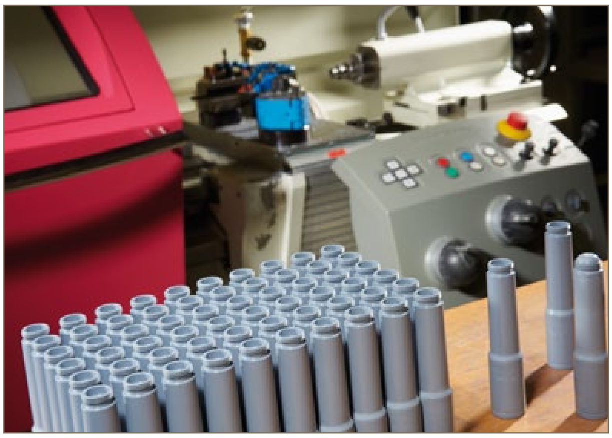

Fig. 4 Green machining sensor housings on a lathe (Courtesy Formatec)

Fig. 5 Sintering furnaces at Formatec

Fig. 6 *Watch cases in a vibratory polishing device (Courtesy Formatec)*

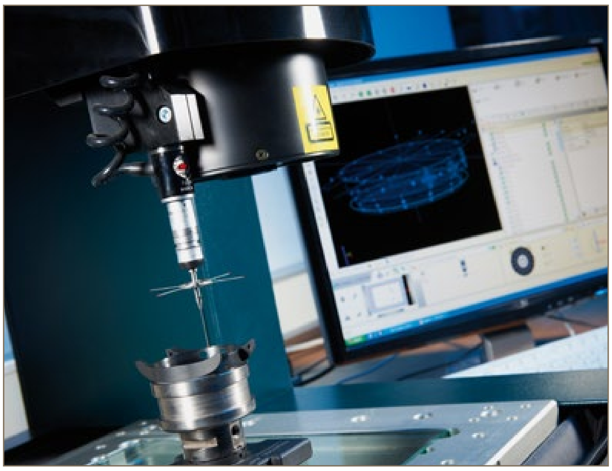

Fig. 7 *3D measurement performed on a watch case (Courtesy Formatec)*

of the fine powders used in CIM, the process takes several hours depending on the wall thickness of the parts. The parts are then sintered in one of the three batch sintering furnaces (Fig. 5). In order to serve the market requirements for white products, Formatec reserves another sintering furnace specifically for these materials.

The sinter support trays used at Formatec are made from silicon carbide. The stability of the binder system, combined with exact process control, enables reproducible shrinkage within 0.3% of the final dimension and remarkable accuracies can be achieved for small sized products.

The company does not set minimum production volumes, believing that these depend on the economic viability of competing manufacturing processes. The highest annual production volume for a component is currently 100,000 parts per year.

Post sintering operations

A wide variety of post-sintering operations is available in-house, most of which focus on surface finishing and tolerances. Besides these two aspects, Formatec also performs assembly services related to the ceramic components and carries out customer specific test procedures where and if required.

Post-sinter machining of ceramic products is done by grinding in order to meet critical dimensional tolerances that cannot be achieved after sintering. "The steps that we have taken on this aspect of our operation over the past 18 months are tremendous," commented Michiel de Bruijcker. "All the efforts made by our development team working on the grinding process have paid off and resulted in a steady and reliable output of high precision ceramic watch cases, all of which are ground in-house."

Equally important is the polishing operation which is a speciality of Formatec, with high gloss polishing being the last processing step (Fig. 6). In order to continue to develop its capability in this area, a new cleanroom has recently been installed which will enter service in the first quarter of 2012.

Final quality control is set up in a dedicated room, featuring special lighting for visual inspection, and operated by well-trained visual inspectors who are familiar with the requirements of the customer. The room also includes a 3D measurement system for critical dimensions (Fig. 7).

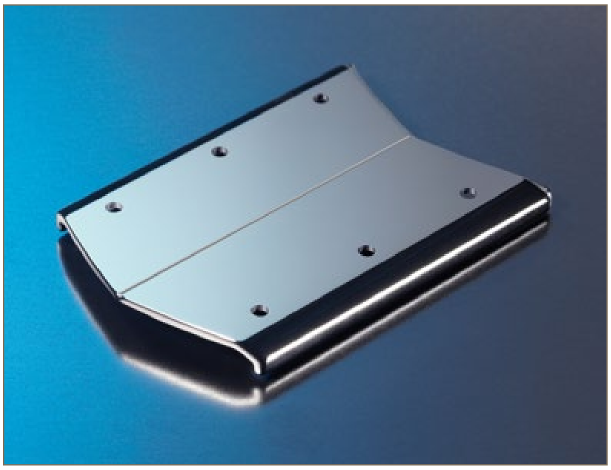

Fig. 8 *Cover plate for cell phones (Courtesy Formatec)*

Fig. 9 *Anchor for fixing dental crowns (Courtesy Formatec)*

The Vertu milestone

A milestone in the company's history was the development of cover pieces for exclusive high-end Vertu mobile phones made from black zirconia (Fig. 8). These components can be up to 10 cm long and 6 cm wide with thin walls. At the same time the components have to meet exacting dimensional tolerances. A specific challenge was the required flatness and uniformity, which imposed particular challenges on the

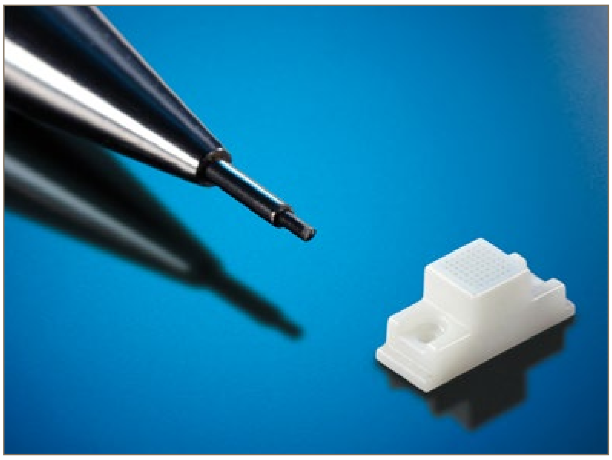

Fig. 10 Telecom connector featuring 64 micro holes (Courtesy Formatec)

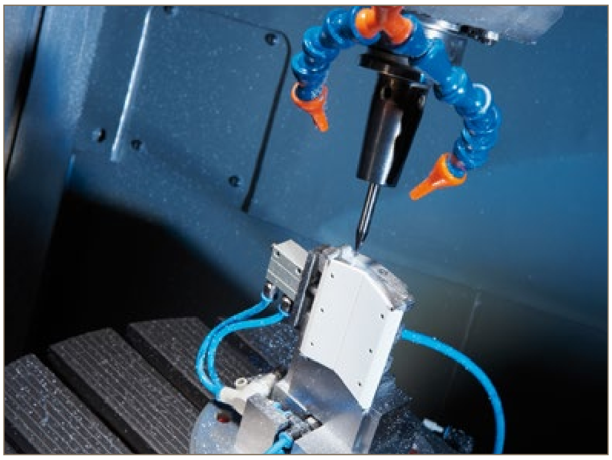

Fig. 11 Machining in the green state (Courtesy Formatec)

ceramic injection moulding process. Formatec perfected its injection moulding processes and was able to optimise the balance between wall thickness and material flow inside the mould cavity. The required flatness was not only produced on the green compacts, but was preserved through the debinding and sintering processes.

In addition, the surface finish had to be completely free from defects, such as irregularities and scratches, and polished to a high gloss finish. Formatec installed polishing equipment to create the required surface finish and developed particular skills in this technology. The result is a surface roughness of around 30 nanometres. This gives the products the glossy appearance which is highly valued by customers. A further improvement of surface quality and a reduction of the reject rate is expected as a result of the new cleanroom, which will accommodate the final polishing processes.

Further aesthetic products

The unique material properties of zirconia, particularly the attractive surface appearance of the products, created further demand so that other aesthetic applications followed. These included luxury writing utensils, watch cases for high-end Swiss watches (Fig. 9) and jewellery.

To manufacture watch cases was a challenge because these products, to some extent, contradict the design guidelines for injection moulding, which recommend a uniform wall thickness wherever possible. In many instances, the watch cases feature significant differences in wall thickness. Further, the expectations of the customers on the quality of these high-end watch cases were so high that it took some time to develop the necessary skills to meet these requirements.

"There is no single way to be successful," de Bruijcker stated. "In the market we see different approaches. For instance, keeping the injection moulding tooling rather simple and achieving the geometrical details by grinding the sintered product. We have another philosophy. We try to come as close as possible to the final geometry by injection moulding and, if necessary, green machining and avoid subsequent machining in the hard state wherever possible. We can achieve this with our extensive knowledge of injection moulding technology and our control of the subsequent processes. It is our belief that this is a very cost efficient approach."

Looking to the future

Formatec recently critically evaluated its business model in relation to market developments, including its product and customer portfolio, and developed an updated strategy for new research initiatives. The majority of Formatec's revenues come from products used for aesthetic applications. In order to further serve the market with ceramic products used for technical applications, market opportunities were explored and balanced with research plans.

Silicon Nitride

Current material development is focused on silicon nitride, a ceramic material with a lower density and higher hardness and heat resistance than zirconia, as well as higher thermal conductivity and temperature shock resistance (Table 1). The characteristics of silicon nitride are closer to alumina than to zirconia, but at the same time it can attain the same attractive surface appearance as zirconia.

It is therefore expected that the technology to manufacture CIM parts from silicon nitride will open up new opportunities for Formatec. Silicon nitride is known to be used in specific high-tech applications such as pump bearings in the space shuttle, turbo charger rotors and certain medical instruments. A demand is also antici-

Material characteristics	Alumina	Silicon nitride	Zirconia
Density g/cm³	3.98	3.26	5.95
Young's modulus GPa	410	320	200
Hardness (Vickers)	2000	1600	1350
Thermal conductivity W/m*K	36	30	2

Table 1 Characteristics of ceramic materials produced at Formatec

pated for applications in the luxury accessories market, where there is a distinct advantage to address the desire for new, exciting and functional materials.

Formatec started this innovation by looking to the starting blocks of the CIM process, the development of the binder and qualification of the feedstock. Much effort went into attaining a high level of shape stability, high dimensional accuracy and excellent surface finish. The strength of the compacts is sufficient to allow machining in the green state. This development is aimed at both aesthetic and complex technical products. Three different colours are available, bronze gold, black, and the material's natural grey colour. The same glossy surface finish that is possible with zirconia can be achieved with silicon nitride.

"Having a qualified binder system for silicon nitride on hand will uniquely position Formatec in the ceramic market. It complements our product portfolio further, and fits our strategy of more product diversity", commented de Bruijcker.

Micro Ceramic Injection Moulding

Micro Ceramic Injection Moulding (MicroCIM), a term used for extremely small CIM products, and larger products with extremely fine details and close dimensional tolerances, is also a speciality at Formatec. The part shown in Fig. 9 is an anchor for fixing dental crowns. The cylindrical section is cemented in a hole that has been drilled into a healthy tooth and the triangular part holds the crown. Two of these parts are necessary to hold a crown from two sides. The part is made from zirconia with critical dimensional tolerances of less than 0.015 mm. It is produced in a six cavity mould and the high precision is attained after sintering without any additional machining operation.

Another example of Formatec's MicroCIM capabilities is the telecoms connector shown in Fig. 10. This features 64 octagonal micro holes, each with a diameter of just 125 microns, which are arranged on a surface area of 2x2 mm. The dimensional tolerance on the diameter of the holes is ±2 microns. Each hole serves to lead a fine wire and the holes are tapered in order to guide the wires through the holes.

Fig. 12 Zirconia can be offered in various colours (Courtesy Formatec)

Prototyping

As has already been discussed, machining in the green state is an efficient extension and supplement to injection moulding, particularly for small to medium production volumes. A high precision five axis CNC milling centre is available, allowing the company to produce complex shapes with a high surface quality. The smoothness achieved by CNC milling is comparable to injection moulding. This is regarded as not only ideal for producing regular parts, but also for prototyping. With this equipment Formatec believes that is has added a new dimension to its options for making ceramic products.

State-of-the-art CAD/CAM software is used for setting the machine parameters and special attention was given to the milling tools which, in combination with the high rotational speed of the spindle, are essential for the high degree of surface finish required.

Although Formatec's core business is CIM components, the development of prototypes is also an essential part of the operation. Today Formatec follows the philosophy to develop prototypes for a new component by pressing and machining in the green state (Fig. 11), but there must be the intention to manufacture the final products by ceramic injection moulding.

Coloured products

The most frequently manufactured material at Formatec is zirconia, thanks to its distinct glossy black surface. Besides black, zirconia is produced in a variety of different colours (Fig. 12). Parts for technical applications are more often made from alumina.

Technical applications

The development of new technical applications for ceramic components in industrial machinery is still very difficult because industrial design engineers initially either consider steel or high-performance plastic parts. It is only where the inherent advantages of engineering ceramics (high strength, hardness and wear resistance even at elevated temperatures, corrosion resistance, electrical and thermal insulation, low weight, excellent surface finish etc) are required that ceramic components have a chance in technical applications.

"Formatec is dedicated to overcome these difficulties and to develop exciting new products and acquire new customers in markets were ceramics are currently not used. This has been successfully accomplished in the past and we are confident that it will continue to be done in the future," concluded de Bruijcker.

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Titanium Powder Injection Moulding (Ti-PIM): Australian conference reviews developments

The international conference on "Powder Processing, Consolidation and Metallurgy of Titanium", took place at The University of Queensland, Brisbane, Australia on 5-7 December, 2011. Dr David Whittaker reports for *PIM International* on a number of international papers that addressed the latest developments in the metal injection moulding of titanium and its alloys.

It would be impossible to dispute the organiser's claim that the international conference on "Powder Processing, Consolidation and Metallurgy of Titanium" was the world's largest ever gathering to date dedicated solely to the topic of processing of titanium by Powder Metallurgy routes.

The event was co-sponsored by Materials Australia (MA), Titanium Industry Development Association, New Zealand (TiDA), The Minerals, Metals & Materials Society (TMS), Japan Society of Powder and Powder Metallurgy (JSPM) and the Chinese Society for Metals (CSM) and was organised largely through the efforts of Dr Ma Qian of The University of Queensland, Australia. It attracted 127 registrants from 15 countries for a packed three-day programme. Key papers, some of which are reviewed here, concerned the latest international developments in the MIM of titanium and its alloys.

Low cost MIM titanium alloy

Concepts in binder selection and in thermal treatment procedures, aimed at delivering a combination of enhanced properties and cost reductions in titanium alloy parts, processed by MIM, were described in a paper by **Xiao Ping An, Hunan University, China**.

The quest for cost reduction centred on two issues, the use of low cost raw materials and the reduction in process times and consequent energy consumptions, while the use of "thermohydrogen processing" (THP) was studied as a potential means of enhancing sintered properties.

As indicated in Fig. 1, a blended feedstock, based on TiH_2 powders with masteralloy additions, was selected and two processing variants were studied, one involving a conventional sintering process and the second involving the use of a THP comprising hydrogenation, solid solution annealing + aging and final dehydrogenation.

The benefits of using a naphthalene containing wax based binder in allowing high powder loadings and an accelerated solvent debinding rate were reported. Table 1 compares debinding rates for feedstocks incorporating binders with and without naphthalene.

Solvent debinding was followed

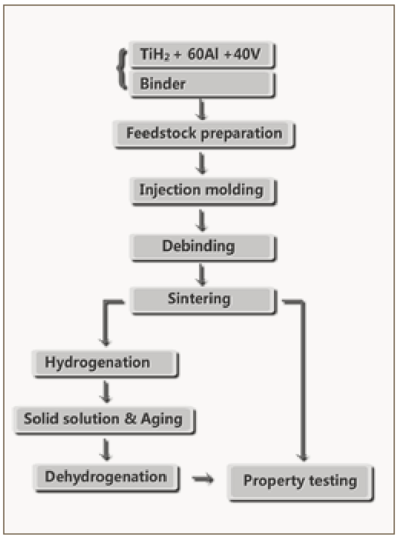

Fig. 1 Process flow diagram

Binder	Time (hour)					
	1	2	3	4	5	6
A (Containing naphthalene)	39.0%	53.5%	66.1%	73.0%	76.4%	77.7%
B (without naphthalene)	15.1%	30.3%	40.4%	49.7%	54.9%	59.1%

Table 1 Binder removal by solvent debinding at 40°C

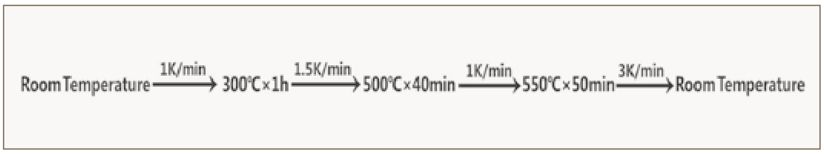

Fig. 2 Thermal debinding schedule

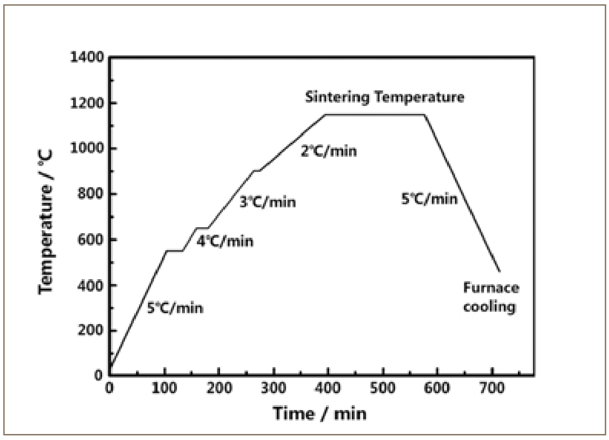

Fig. 3 Schematic of sintering schedule

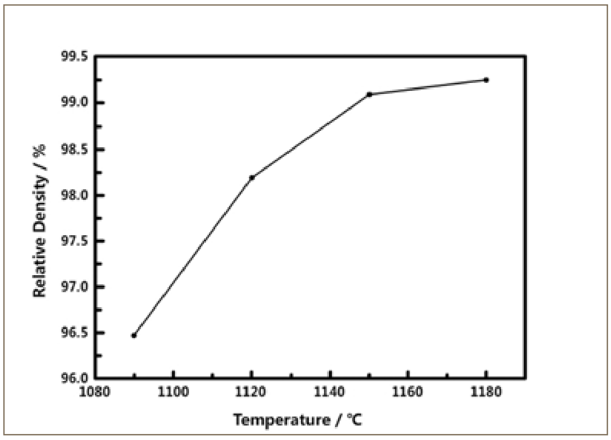

Fig. 4 Influence of sintering temperature on density

Property	Relative Density (%)	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)	Impurity Content (%)			
					C	O	N	H
Data	99.10	587	439	4.9	0.12	0.21	0.0024	0.0049

Table 2 Properties of as-sintered Ti-6Al-4V

Property	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)
After THP	661	496	6.1
As-sintered	587	439	4.9
Increased by	13%	12.6%	19.6%

Table 3 Enhancement of properties by THP

by the thermal debinding schedule defined in Fig. 2 and test-pieces were conventionally sintered according to the schedule described in Fig. 3.

As shown in Fig. 4, Ti-6Al-4V MIM samples were processed to density levels over 99% at sintering temperatures below 1200°C. The microstructures, produced on sintering over a range of temperatures from 1090 to 1180°C, are shown in Fig. 5 and typical as-sintered mechanical property levels and interstitial contents are presented in Table 2. The hydrogenation stage of THP was seen as potentially involving transformations, such as:

$$\alpha_H \rightarrow \alpha_H + \beta_H$$

$$\beta_H \rightarrow \beta_H + \delta\text{-TiH}_2$$

The microstructural modifications, produced by the subsequent solid solution annealing + aging, are presented in Fig. 6.

Finally, the potential transformations in the dehydrogenation of hydrogenated Ti-6Al-4V are:

$$\delta\text{- TiH}_2 \rightarrow \alpha + H_2$$

$$\alpha_H \rightarrow \alpha + H_2$$

$$\beta_H \rightarrow \alpha + \beta + H_2$$

The beneficial influence on mechanical properties of thermohydrogen processing (THP) over conventional sintering is summarised in Table 3.

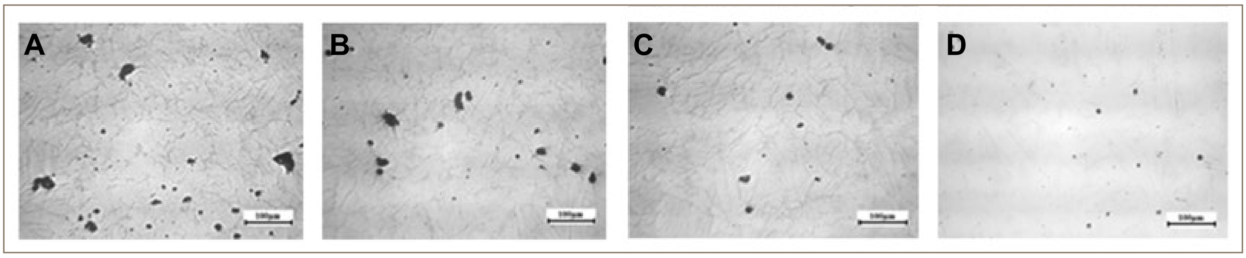

Fig. 5 As-sintered microstructures: Optical micrographs of unetched Ti-6Al-4V alloys sintered at (a) 1090°C (b) 1120°C (c) 1150°C and (d) 1180°C

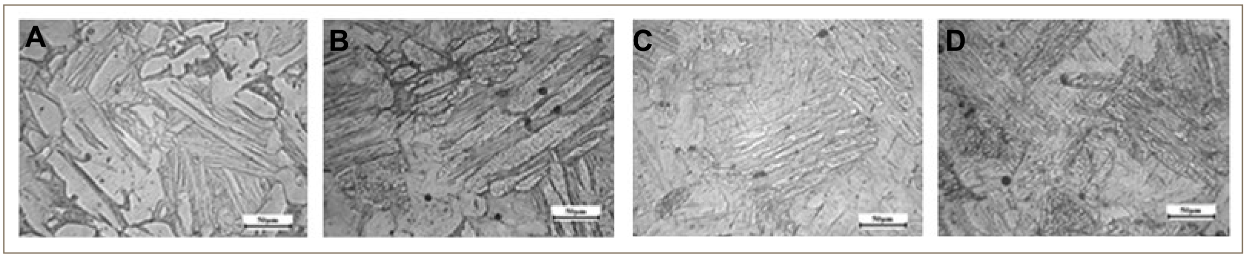

Fig. 6 Microstructure of hydrogenised Ti-6Al-4V after solid solution annealing and aging, with hydrogen content: (a) 0.22%, (b) 0.30%, (c) 0.43% and (d) 0.51%

Strengthening of MIM processed Ti-6Al-4V by fine elemental additions

The use of fine elemental additions to enhance the sintered properties of a Ti-6Al-4V alloy, processed by Metal Injection Moulding (MIM), was described in a paper delivered by **Hideshi Miura, Kyushu University, Japan** on behalf of his co-authors, **Hyungoo Kang and Yoshinori Itoh**.

The elemental additions studied were the β-stabilisers Mo, Fe and Cr, which were judged to have the potential to create solid solution strengthening in the base Ti-6Al-4V alloy. Fe and Cr, particularly, also carried the benefit of cost-effectiveness in comparison to titanium powder.

In this study, the base Ti-6Al-4V composition was created through the blending of elemental Ti powder and an Al-V masteralloy. The further elemental additions were made as fine Mo or Cr powders or fine carbonyl iron powder. The chemical compositions, particle sizes and SEM images of these various powders are shown in Table 4 and Fig. 7 respectively.

The levels of additions of the Mo, Fe or Cr powders were decided on the basis of "Molybdenum Equivalence", using the equation:

$$[Mo]eq = [Mo]+[Ta]/5+[Nb]/3.6+[W]/2.5+[V]/1.5+1.25[Cr]+1.25[Ni]+1.7[Mn]+1.7[Co]+2.5[Fe]$$

Previous studies by this group had indicated that a molybdenum equivalence addition of around 7 allowed sintered elongation levels above 10% to be maintained. The addition levels, chosen for the reported study, were therefore 4 mass% of Mo (Mo_{eq} = 6.7), 2 mass% of Fe (Mo_{eq} =7.7) or 4 mass% of Cr (Mo_{eq} = 7.7).

The various powder additions were mixed and prepared into a MIM-feedstock with a 65 vol% powder loading and the feedstock was then injection moulded into tensile test specimens with a length of 75 mm, a width of 5 mm and a thickness of 2 mm.

Two-stage solvent and thermal debinding was carried out and, finally, samples were sintered in high vacuum (of the order of 10^{-2} Pa) at various temperatures up to 1623°K (1350°C) for 4 hours.

Fig. 8 shows the effect of sintering temperature on the relative density of the Ti-6Al-4V alloy compacts with the various fine elemental additions. In all cases, relative density increased with increasing sintering temperature and reached a level around 99% at 1573°K (1300°C). The relative densities of the compacts with the fine additions were generally around 1% higher than those of the base material in the sintering temperature range 1423-1523°K (1150-1250°C). It was proposed that this was due to an enhancement of densification by the additional fine particles.

Figs. 9 and 10 show the effect of the fine elemental additions on the sintered tensile strength and elonga-

Powder	O (mass%)	C (mass%)	Fe (mass%)	Particle size (µm)
Ti	0.13	0.008	0.044	-45
Al-40V	0.47	0.02	0.16	-20
Mo	0.20	---	0.008	1.59*
Fe	0.38	0.74	val.	4.31*
Cr	0.75	0.025	0.22	2.97*

** mean particle sze*

Table 4 Chemical compositions and particle sizes of the powders used in this study

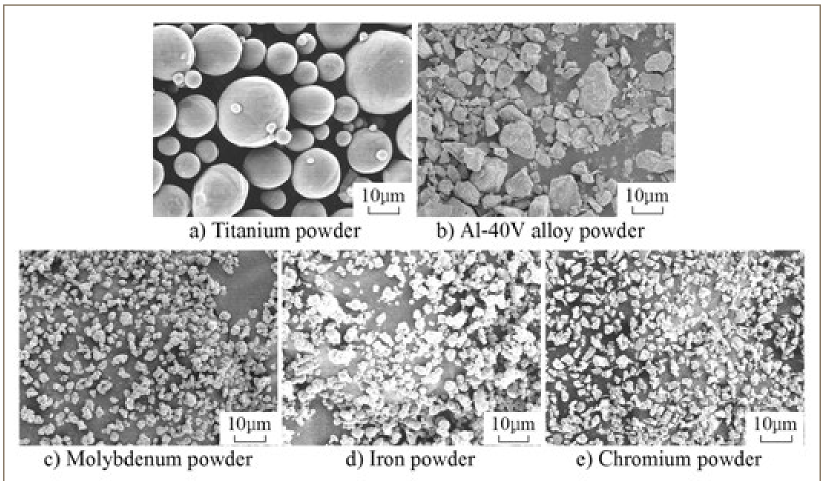

Fig. 7 SEM images of a) Titanium, b) Al-40V alloy, c) Molybdenum, d) Iron and e) Chromium powders used in this study

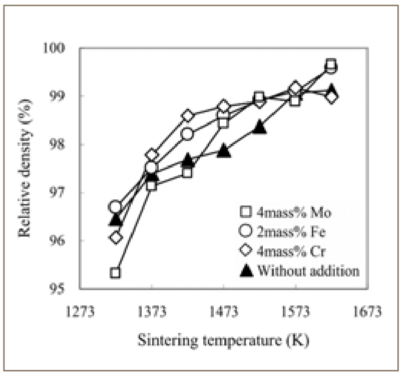

Fig. 8 Effect of sintering temperature on the relative density of Ti-6Al-4V alloy compacts with added Mo, Fe or Cr

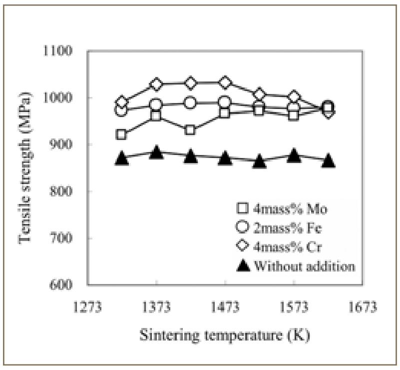

Fig. 9 Effect of sintering temperature on the tensile strength of Ti-6Al-4V alloy compacts with added Mo, Fe or Cr powder

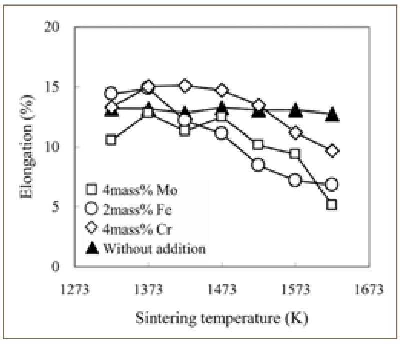

Fig. 10 Effect of sintering temperature on the elongation of Ti-6Al-4V alloy compacts with added Mo, Fe or Cr powder

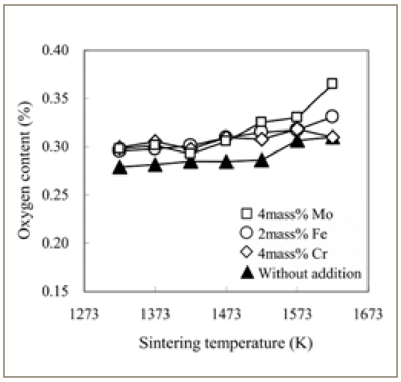

Fig. 11 Effect of sintering temperature on the oxygen content of Ti-6Al-4V alloy compacts with added Mo, Fe or Cr powder

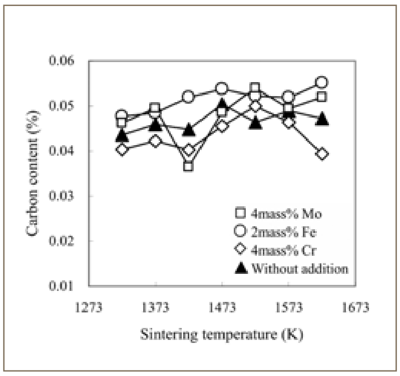

Fig. 12 Effect of sintering temperature on the carbon content of Ti-6Al-4V alloy compacts with added Mo, Fe or Cr powder

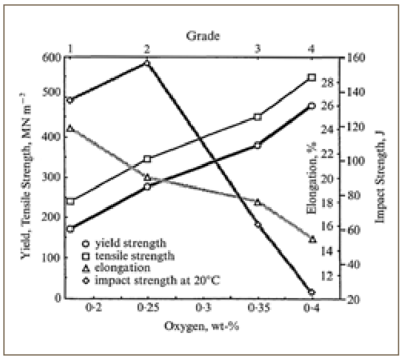

Fig. 13 Mechanical properties of CP-Ti grades 1 to 4

tion of Ti-6Al-4V, respectively. All of the elemental additions increased tensile strength, with the effect being greater for Cr than for Mo or Fe. The increments in tensile strength were around 90 MPa with the 4 mass% Mo addition, around 110 MPa with the 2 mass% Fe addition and around 150 MPa with the 4 mass% Cr addition. The tensile strength of the 4 mass% Cr material reached 1030 MPa.

Although the elongation of the sintered compacts with the fine powder additions decreased at high sintering temperatures, these sintered compacts showed excellent ductility above 13 % elongation at lower sintering temperature. In particular, the elongation levels of the sintered compacts with Fe or Cr powder additions were around 15 % even at low sintering temperature.

Fig. 11 and Fig. 12 show the effect of sintering temperature on the oxygen and carbon contents of Ti-6Al-4V alloy compacts with the fine elemental powder additions. The oxygen content of these sintered compacts had a tendency to increase slightly with increasing sintering temperature, whereas the carbon content of all the compacts was nearly constant with increasing sintering temperature. Although the oxygen content of the sintered compacts slightly increased with the addition of the Mo, Fe or Cr powder, these values were considered to be at a low enough level to have little detrimental effect on the mechanical properties of sintered compacts.

On the basis of these reported results, it was concluded that the addition of fine Mo, Fe or Cr exerted a significant influence on the mechanical properties of sintered Ti-6Al-4V compacts.

> *'to penetrate such markets, the MIM sector is being forced to aim at the same limits as have been established for wrought or cast titanium.'*

Interstitial control in MIM Ti alloys

Increasing interstitials in wrought titanium alloys, especially oxygen, results in an increase in yield strength, tensile strength, hardness and fatigue resistance at a given stress level, but have a detrimental effect on ductility. Nitrogen has the most potent effect, but it is oxygen that causes most concern, because of its high solubility in titanium.

Fig. 13 shows the mechanical properties of CP-Ti grades 1 to 4 with respect to permissible oxygen contents.

In the development of MIM titanium for high performance applications, this leads to two areas of concern:

- The need for careful control of the many potential sources of interstitial contamination in the MIM process – in the starting powder, in the decomposable substances in the binder and the potential pick-up from the atmosphere in the debinding and sintering processes.
- The relative lack of understanding of the relationship between interstitial levels and mechanical properties in MIM titanium parts has led to a situation where, to penetrate such markets, the MIM sector is being forced to aim at the same limits as have been established for wrought or cast titanium.

A paper presented by **Albert Sidambe, University of Sheffield** on behalf of his co-authors, **Fatos Derguti and Iain Todd**, addressed both of these areas of concern. This research group is associated with the University's Mercury Centre and has a well-established reputation for its work in this area of MIM of titanium.

The reported work was based on the use of CP-Ti and Ti-6Al-4V powders, whose morphologies are illustrated in

	Composition (wt%)						
	O	C	N	H	Fe	Al	V
CP-Ti (Sub45μm)	0.12	0.01	0.003	0.004	0.05	-	-
Ti-64 (sub 45μm)	0.14	0.01	0.01	0.003	0.11	6.35	3.9
Ti-64 (sub 25μm)	0.11	0.02	0.01	0.005	0.11	6.0	3.8

Table 5 Titanium Powder Composition. Ti=balance in all cases

Fig. 14 and whose compositions are defined in Table 5.

These powders were mixed with a binder with a major fraction of polyethylene glycol (PEG), a minor fraction of polymethyl-methacrylate (PMMA) and a surfactant addition of stearic acid (SA). Handling of the materials was carried out in an inert argon atmosphere to make feedstocks with powder loadings of 66 and 69 volume% and these feedstocks were injection moulded at 120°C into standard tensile test bars.

The PEG component of the binder was then removed by solvent extraction and the remaining, backbone PMMA component was removed by thermal debinding.

Sintering was then carried out in the same furnace as was used for thermal debinding.

Thermogravimetric analysis (TGA) results, shown in Fig. 15, indicated that oxidation dramatically increases above 400°C, but that, below that temperature, oxidation is not significant, even in air. These results demonstrated that thermal debinding should be carried out at a temperature below 400°C, but that, at higher temperatures, sintering atmosphere control is important.

Sintering temperature selection was an item of compromise. To minimise interstitial contamination, sintering temperature should be as low as possible, but, to achieve the required densification, higher sintering temperatures (e.g. 1300°C) and longer sintering times (2 hours) are needed.

Using optimum processing conditions, it was found that oxygen pick-up in MIM could be limited to around 0.06 wt% for sub-25 µm Ti-6Al-4V ELI, but to as little as 0.03wt% for sub-45µm Ti-6Al-4V and CP-Ti.

The influence of oxygen content on as-sintered mechanical properties is shown in Figs. 16 and 17.

Fig. 16 shows the influence on the elongation levels of CP-Ti and Ti-6Al-4V. There was a wide range of elongation levels in the lower oxygen interstitial range, but the expected reduction of elongation with increasing oxygen content can be observed in the higher oxygen range for CP-Ti. Ti-6Al-4V elongation levels appear not to reduce dramatically with increasing oxygen level and there is some scatter in the data.

Fig. 17 shows that the tensile strengths of both the CP-Ti and Ti-6Al-4V MIM samples increase with increasing interstitial oxygen content, but that the level of tensile strength of CP-Ti is high in comparison to the titanium grades 1 to 4.

CP-Ti MIM parts containing low interstitials but with high tensile strengths have been successfully produced and sufficiently strong Ti-6Al-4V MIM parts, meeting the requirements of ASTM F2885-11, can be manufactured with low interstitial levels.

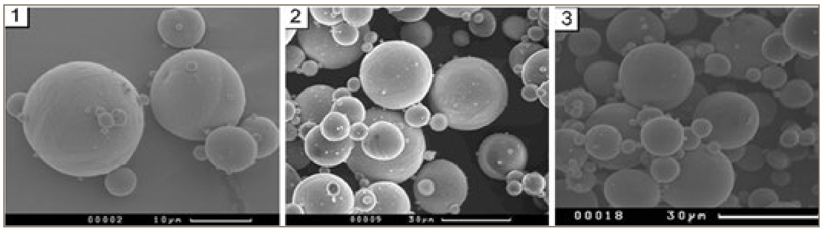

Fig. 14 SEM's of titanium powder; (1) CP-Ti sub 45 micron, (2) Ti-64 sub 45 µm and (3) Ti-64 sub 25 µm ELI

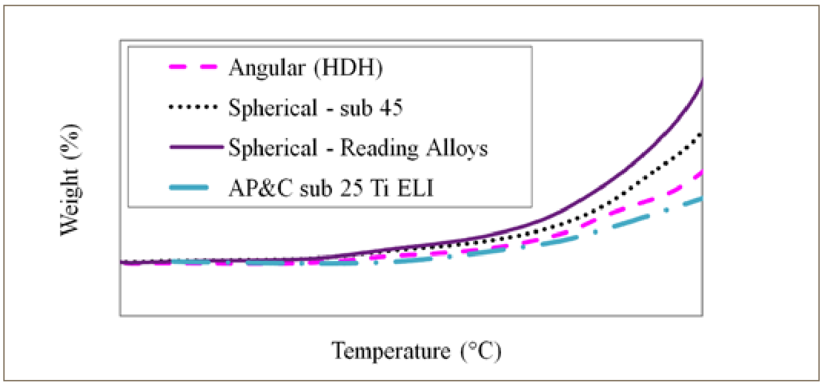

Fig. 15 TGA analysis showing powder oxidation rates. Powder was heated from 50°C at 10°C/min in air at 20.0 ml/min

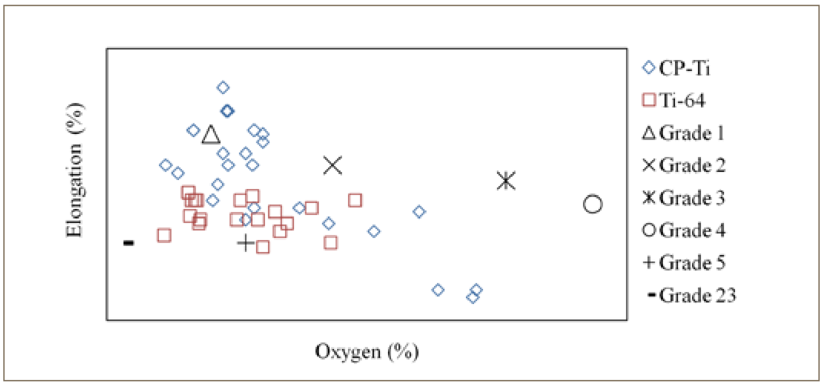

Fig. 16 Effect of oxygen on CP-Ti and Ti-64 elongation

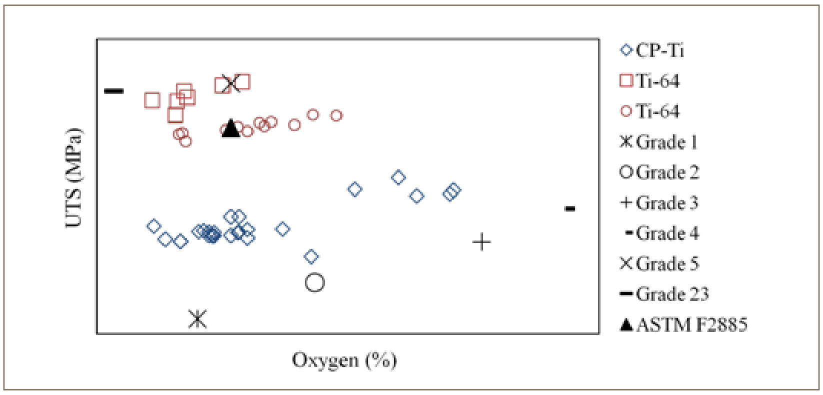

Fig. 17 Effect of oxygen on CP-Ti and Ti-64 UTS

Binder developments for MIM titanium

A group of three conference papers, all from New Zealand-based research groups, reported on studies of binders for metal injection moulding (MIM) of titanium alloys.

In all cases, the binders comprised polyethylene glycol (PEG) as a water soluble component and stearic acid (SA) as a surfactant, but with a range of polymers as the backbone component.

The paper from **G Thavanayagam, D L Zhang, K L Pickering** and **S Raynova**, The **University of Waikato, New Zealand** assessed the use of polyvinylbutryl (PVB) as the backbone component.

Due to its strong intra-molecular interactions, PVB shows high viscosity. However, it facilitates good wetting characteristics with powders and provides homogeneous feedstocks. It holds powder particles and aids shape retention during solvent debinding.

PVB has been used extensively as a binder component in ceramic injection moulding of materials such as alumina or zirconia, but there has been little reported use in metal injection moulding in general or MIM of titanium in particular.

The objectives of this study were to determine optimum formulation procedures for preparing PVB-based feedstocks, containing HDH Ti-6Al-4V powder, and to understand the effects of parameters, such as powder loading and mixing conditions on the rheological properties of the feedstock.

Formulation trials were carried out using three different mixing systems; a compounder, a modified mechanical mixer and a twin screw extruder.

As shown in Fig. 18, the most homogeneous distribution of the binder was achieved by the use of the modified mechanical mixer followed by extrusion.

The superiority of the feedstock, prepared by this method, was also confirmed through thermogravimetric analysis (TGA) of the thermal degradation characteristics of the studied mixing variants.

The density of feedstocks with different powder loadings was measured and plotted as shown in Fig. 19. It can be seen that the measured density of feedstock deviates from its theoretical density at the powder loading of 68%. Hence, it was derived that the critical powder loading was 68 vol.%. This result was further validated by torque analysis, where a sudden peak in mixing torque was observed when the powder loading reached 72 vol.% as shown in Fig. 20. This sudden peak torque at 72 vol.% corresponds to the critical powder loading. From these results, it can be determined that the critical powder loading can be taken as 68 vol.% and the optimum powder loading was deduced to be 63 vol.%.

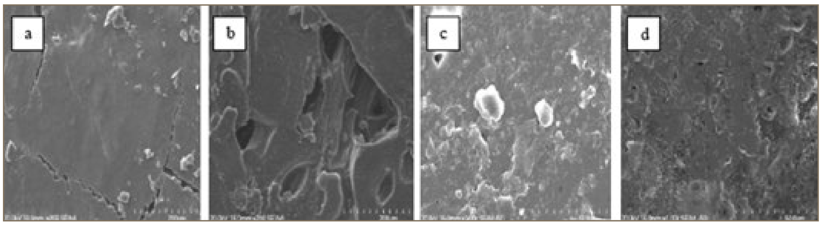

Fig. 18 SEM images showing the microstructures of binder-5 formulated under different conditions: (a) 30 min mixing using a compounder; (b) 90 min mixing using a modified mechanical mixer; (c) mixing by three passes of extrusion; (d) 90 min mixing using a modified mechanical mixer followed by extrusion

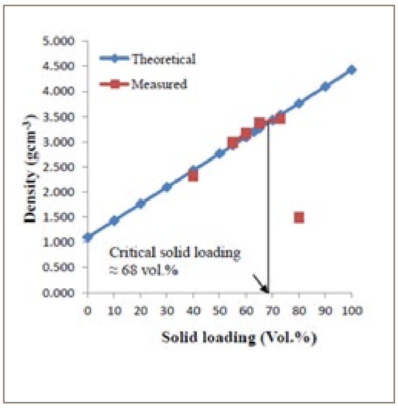

Fig. 19 Density analysis of various feedstocks

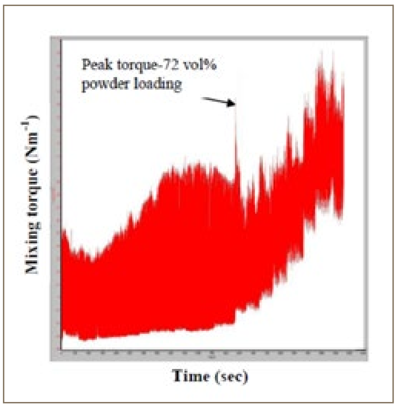

Fig. 20 Mixing torque analysis of feedstocks

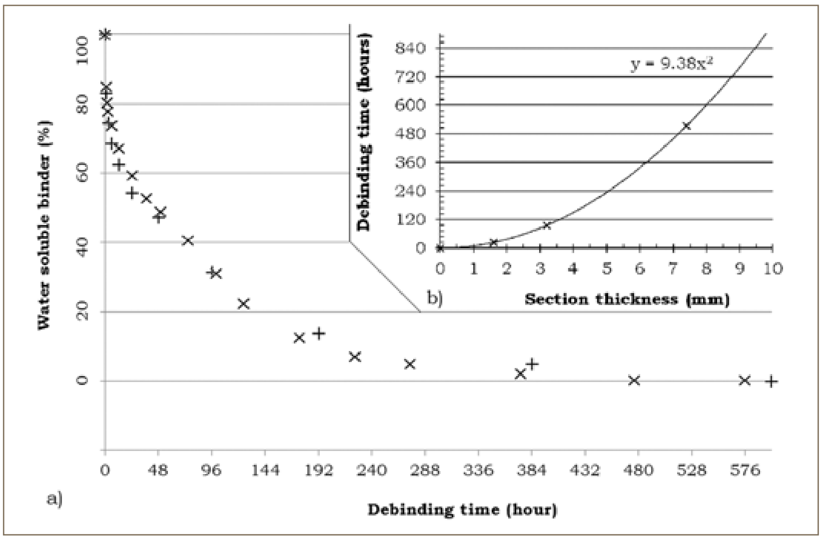

Fig. 21 a) Mass loss of water soluble PEG from Ti-6Al-4V sample parts with powder loading of 0.60 (x) and Ti sample parts with powder loading of 0.65 (+) (each data set is the average value for 11 samples); and b) the solvent debinding time as a function of part section thickness

A second paper from **Paul Ewart** and **Deliang Zhang**, **The University of Waikato, New Zealand** and **Seokyoung Ahn**, **Pusan National University, South Korea** focused on a binder system that also contained PEG, but with an undefined polymer as the backbone component, which was formulated into feedstocks containing HDH CP Ti powder (-325 mesh) or HDH Ti-6Al-4V powder (-200 mesh).

This study assessed the removal rate of the water soluble component during the solvent debinding of compacts produced from these feedstocks.

Fig. 21a shows the mass loss of PEG over time for the sample Ti and Ti-6Al-4V parts. It can be seen that the PEG dissolution time for the Ti-6Al-4V parts is slightly higher than that for the Ti parts but these times are within ± 4% of each other and the rate is the same. From previous work, PEG dissolution for Ti parts with powder loading of 0.60 and section thickness of 1.6 mm completed in 24 hours, and those with the same powder loading and a section thickness of 3.2 mm completed in 100 hours. The PEG dissolution time for the parts in this study, which have the same powder loading and average thickness of 7.4 mm was ~500 hours. These data allowed the authors to correlate the solvent debinding time (y) for removing PEG with the thickness (x) of the moulded parts by the formula $y = 9.38x^2$, as shown in Fig. 21b.

The final paper in this group came from **Gang Chen, Guian Wen, Neil Edmonds** and **Peng Cao**, **The University of Auckland, New Zealand** and **Yimin Li, Central South University, China**. This paper reported the study of debinding kinetics of a water soluble binder system for titanium alloy MIM. The studied binder system again contained PEG and SA, but, in this case, the backbone component was polymethyl methacrylate (PMMA). The powders incorporated in the feedstocks were HDH Ti-6Al-4V and argon atomised NiTi. The feedstocks were ground to powder and were pressed to green compacts with various thicknesses at a range of compaction pressures, in a die pre-heated to 60°C. The reported results were as follows:

<u>Influence of compaction pressure</u>

Fig. 22 (left) shows the binder removal as a function of debinding time for Ti-6Al-4V (<74 μm) specimens with 69.5 vol.% powder loading at different pressures. It can be seen that the binder removal percentage did not noticeably change with the shaping pressure. 300 MPa compaction pressure was used, which is much higher than normal injection moulding pressure (~ 100 MPa) for MIM. At such a high pressure, there will be no significant difference in green densities. For example, the open-cell porosity of the green part after 300 MPa pressing was 0.279 % and the green density was 3.233 g/cm^3, as compared with the theoretical density of 3.445 g/cm^3, indicating the compacted part was fully compacted. In the case of 700 MPa compaction pressure, the green density was 3.252 g/cm^3, which is very close to that attained at 300 MPa. Therefore, the inter-particle spacing and the particle mobility appear to be independent of the compaction pressure. Instead, the inter-particle spacing is mainly dependent on the solid loadings and metal particle size. Nevertheless, the binder removal percentage increased with the soaking time and the extraction is significant in the first 10 h after which it levels off.

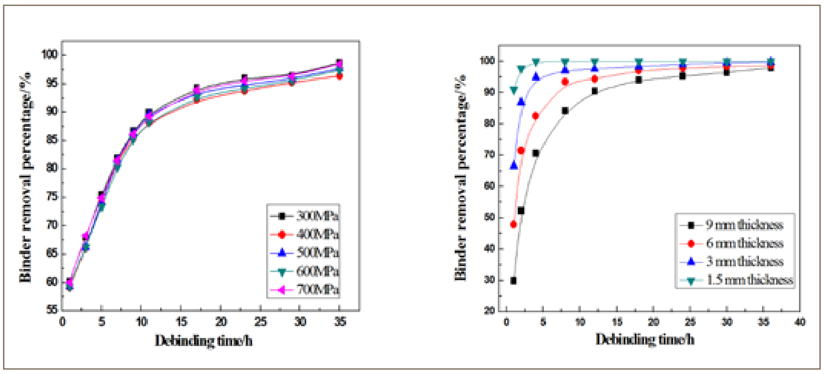

Fig. 22 Influence of (left) pressure and (right) thickness on binder removal behaviour of Ti-6Al-4V feedstock

<u>Influence of green part thickness</u>

The binder removal behaviour also depends on the sample thickness. This is shown in Fig. 22 (right) with 69.5 vol.% powder loading specimens. It is clear that the thicker the sample, the smaller the binder extraction percentage. However, the difference in the extraction percentage for samples with various thicknesses levels off quickly after 15 h of debinding time.

For industrial applications the binder removal is completed as soon as 75.0 % of the weight loss is reached. The required binder removal time for the 1.5, 3, 6 and 9-mm-thick Ti-6Al-4V specimens is approximately 1, 1.5, 3 and 6 hours respectively.

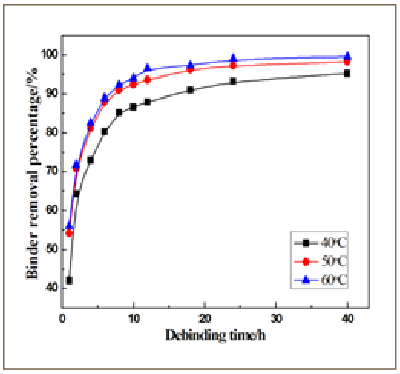

Fig. 23 Influence of water bath temperature on binder removal behaviour for Ti-6Al-4V feedstock

<u>Influence of water bath temperature</u>

Debinding is known to be influenced by temperature. Molecular mobility is generally faster at higher temperatures and hence the binder removal percentage increases with increasing water bath temperature.

'Molecular mobility is generally faster at higher temperatures and hence the binder removal percentage increases with increasing water bath temperature'

Fig. 23 shows the isothermal binder removal behaviour for 6-mm-thick Ti-6Al-4V (<74 μm) green parts with 69.5 vol.% powder loading at different temperatures. The obtained removal percentages clearly show the influence of binder removal temperature on the required binder removal time. The specimens reached 75.0 % of binder loss after 5, 3 and 2.5 h at temperatures of 40, 50 and 60°C, respectively. A 50% debinding time reduction can be achieved by increasing temperature from 40 to 60°C. Increasing temperatures further is not recommended because too

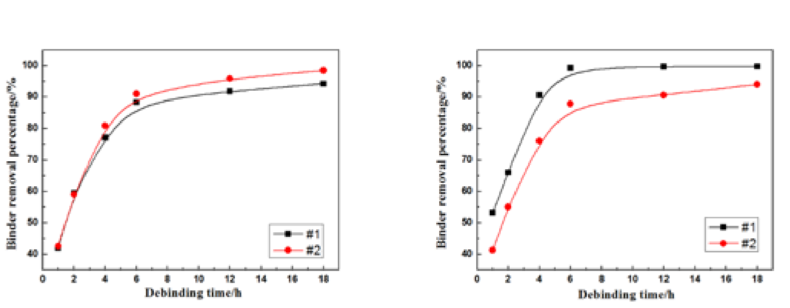

Fig. 24 Influence of (left) particle morphology and (right) particle size on binder removal behaviour: (left) (#1) 65 vol.% Ti-6Al-4V (<47μm) and (#2) 65 vol.% NiTi (20~40 μm), (right) (#1) 60 vol.% NiTi (80~100 μm) and (#2) 60 vol.% NiTi (20~40 μm)

Powder Processing, Consolidation and Metallurgy of Titanium, Brisbane, Australia, 5-7 December 2011

Professor James C Williams, Ohio State University, USA, Dr Hailian Bi, University of Auckland, New Zealand, Dr David Whittaker, UK, and Dr Ma Qian (Conference Chair) The University of Queensland, Australia

Dr M Ashraf Imam, Naval Research Laboratory, Washington, USA

Professor Orest M. Ivasishin, Institute for Metal Physics, Ukraine, and Professor James C Williams, Ohio State University, USA

The clock tower at St. Johns College, The University of Queensland, where many delegates stayed

The Brisbane skyline

quick a debinding rate would result in cracking and loss of mechanical integrity in the green parts.

Influence of powder morphology.
In this study, two different powder morphologies with similar particle size were used: spherical particles for the NiTi alloy and irregular particles for the Ti-6Al-4V alloy powder. Fig. 24 (left) shows the influence of powder morphology on binder removal percentage of NiTi and Ti-6Al-4V specimens with the same 65 vol.% powder loading. It can be seen that the debinding rate decreases with an increase in the content of fines in the powder. For instance, the binder removal percentage of HDH-Ti-6Al-4V green parts was 98.0 % as compared with 94.0 % for atomised NiTi samples with the same powder loading and similar particle size, when the debinding time was 18 h. As irregular particles usually have a lower packing density and hence a larger inter-particle spacing, it is therefore anticipated that the binder in the Ti-6Al-4V feedstock will be extracted faster than that in the NiTi feedstock.

Influence of particle size
Fig. 24 (right) presents the influence of particle size on binder removal percentage of NiTi specimens with the same 60 vol.% powder loading. As shown in Fig. 24 (right), the binder removal percentage decreases with decreasing particle size for the NiTi specimens. The binder removal percentage for 80~100 μm particle size range was 99.0 % as compared with 94.0 % for 20~40 μm particle size range when the debinding time was 18 h. Increasing particle size leads to a reduced packing density and increased inter-particle space, giving rise to a faster debinding rate. Similarly, the debinding process for the larger particle feedstock is faster than that for the smaller particle feedstock if the particles have a similar morphology.

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Hot Isostatic Pressing (HIP) of PIM parts: Material properties and increased productivity

Hot Isostatic Pressing (HIP) of PIM parts is an effective route to achieving full density and improving mechanical properties for a range of PIM materials. The latest innovations in HIP technology, such as Uniform Rapid Cooling, URC™ , now make the case for the technology more appealing thanks to significantly faster processing times. Dr Anders Eklund, from Avure Technologies AB, Sweden, reviews the process and its advantages for PIM producers.

Hot Isostatic Pressing (HIP) technology has been in existence for more than 50 years and can today be considered as a standard production route for many applications. The HIP process applies high pressure and high temperature to the exterior surface of parts via an inert gas. The elevated temperature and pressure cause sub-surface voids to be eliminated through a combination of plastic flow and diffusion. The challenge is to reach the highest possible theoretical density and still have good to excellent productivity.

Uniform Rapid Cooling, URC™, is a route for thin-walled HIP units to increase productivity by up to 70% compared with natural cooling, whilst increasing the density of PIM parts to more than 99% of theoretical value for many alloys. Examples will be shown in this article.

A closer look at the process

HIP is a fabrication process for the densification of castings, powder metal products such as metal injection moulded (MIM) parts, tool steels (TS) and high speed steels (HSS), as well as ceramic injection moulded (CIM) products for dental and medical applications, to name just a few. HIP technology was introduced in the beginning of the 1950's and has since gained acceptance in many application areas, with many more looking to take advantage of the process.

Typically the pressure applied in a HIP cycle is between 100 to 200 MPa using pure argon gas. However, both lower and higher pressures can be used for specific applications. Other gases such as nitrogen and helium are also commonly used, while gases such as hydrogen and carbon dioxide are more seldom put into use in production units. Additionally, combinations of the above mentioned gases can be applied. The application will, however, determine which gas is used for which purpose, especially since helium is quite expensive and hydrogen in the wrong concentrations is very explosive. The parts that are to be HIPed are initially heated either at high pressure, low pressure, or in vacuum. Again the material composition itself, and suggested HIP cycle to be applied, will govern the start procedure. The two main advantages with HIP are as follows:

An increase in density:

- HIP encapsulated loose powder or green compacts to full density
- HIP non-encapsulated, sintered parts from 95% to 100% of theoretical density
- Elimination of internal porosity.

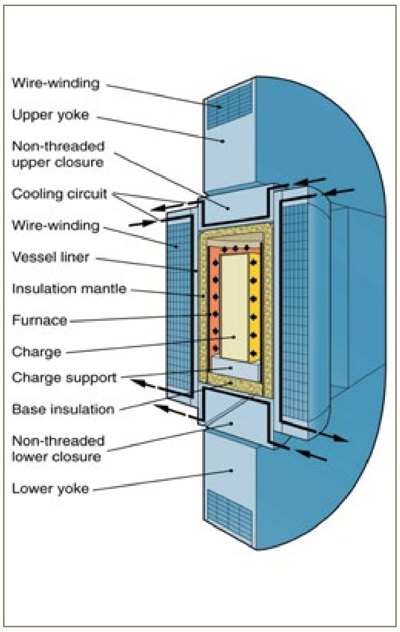

Fig. 1 Wire-wound hot isostatic press

An improvement of mechanical properties:

- Fatigue life increased up to ten times, depending on the alloy system
- Decrease in variation of properties
- Increase in ductility and toughness
- Formation of metallurgical bonds.

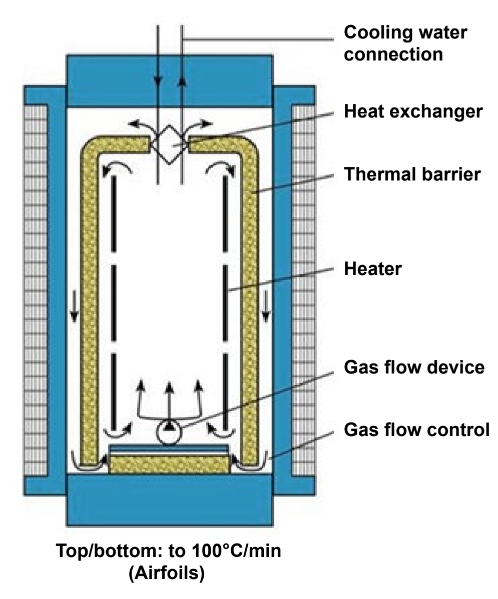

Fig. 2 HIP furnace illustrating the advanced rapid cooling system

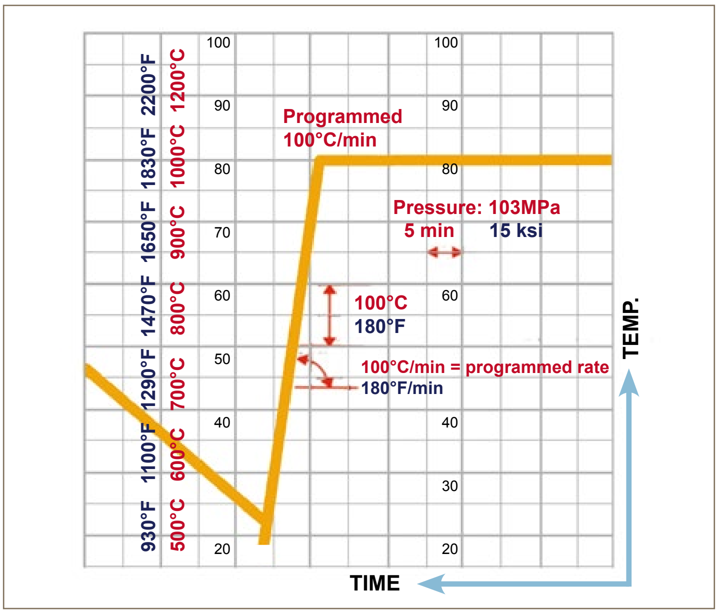

Fig. 3 Uniform Rapid Cooling (URC™) curve. HIP with work zone 1250 x 2500 mm, load weight of 2500 kg

Uniform Rapid Cooling (URC™)

The demand from industry has always been to have shorter cycle times, thereby increasing the productivity of the HIP unit and offering better pay-back on an investment. The HIP pressure vessel itself plays an important role in increasing the cooling rates through efficient removal of the heat generated in the furnace (Fig. 1). The wire-wound technology shown here provides several advantages to enhance heat removal.

Heat removal in this thin-walled vessel is achieved by the wire-winding and an internal water cooled liner, thereby producing an effective heat exchanger between gas-gas and gas-water. Without this thin-wall solution the cooling rates would be significantly lower and true rapid cooling could not be achieved. An extra advantage with the rapid and uniform cooling is that the wear life of the furnace itself is increased dramatically.

The design criteria for the wire-wound vessel are ASME code Section VIII, Div. 3, "leak-rather than-burst". This means that if the vessel was to have a failure, the gas would leave the vessel at high speed, but more importantly it will not crack open and explode.

The flow pattern of the inert gas in a HIP furnace is illustrated in Fig. 2. Parts are heated and cooled more efficiently by flow devices located at the base of the furnace. During heating, the forced gas circulation narrows the temperature difference between the heating elements and the parts, as well as the temperature difference between the top and bottom of the furnace, to accelerate the start of the hold period. After the hold period, flow devices in the furnace circulate one portion of the gas to the outside of the thermal barrier while the other portion is circulated to the inside.

These devices give optimal control over the flow of the gas inside the furnace and inside the pressure vessel, and by increasing the water flow in the internally cooled liner for better cooling of the thin-walls, true URC™ conditions are achieved. Fig. 3 shows a time-temperature curve with programmed cooling rates of 100°C/minute. The pressure is controlled and kept at 103 MPa through the full cooling phase. After cooling, the temperature is allowed to increase again to heat treat the material and allow for optimal grain growth, before the final cooling of the parts is done. The pressure is maintained during the whole cycle at 103 MPa, which aids the results for achieving optimal grain size.

Cycle times

Considerably lower cycle times can be achieved with Uniform Rapid Cooling, URC™. The obvious target is to increase productivity, and therefore lower part costs, by decreasing processing costs and depreciation time of the investment. Cooling rates of the gas can be up to and over 1000°C/minute, which is then called Uniform

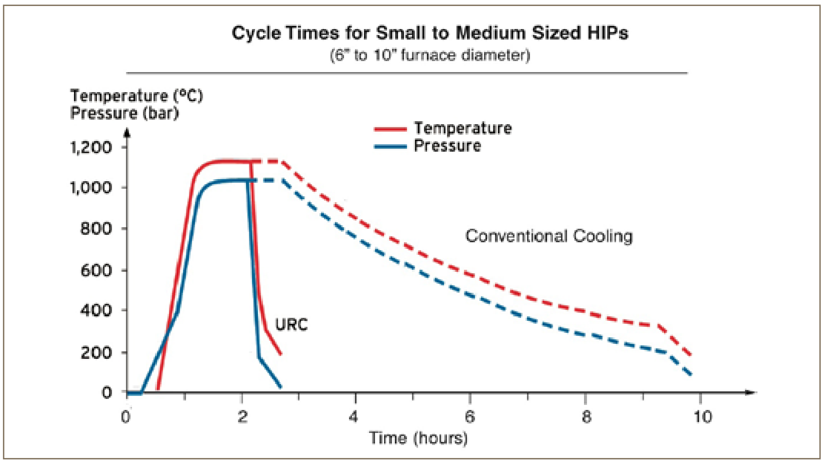

Fig. 4 HIP cycle times for natural cooling vs. uniform rapid cooling (URC™)

Rapid Quenching, URQ™. The cycle time can be lowered as much as 70% (Fig. 4). This means, that for a small to medium sized HIP-unit, three cycles per eight hour shift can be run instead of one cycle per shift with natural cooling.

Material properties

In many alloys, avoiding the detrimental phases such as the sigma phase, is crucial. By rapidly cooling parts down into the safe region of the phase diagram no detectable levels of these phases can be measured.

A more profound effect of the rapid cooling system can be seen when combining Figs. 3 and 4. By controlling the pressure and temperature independently, grain growth and grain size can be optimised and parts therefore have an improved metallurgy.

What also comes out from these illustrations is that parts can be heat treated in the same furnace, thereby eliminating separate handling and additional steps for heat treatment, such as heating and subsequent quenching in water, oil or salt baths. This lowers the total capital investment, as well as the running costs.

As previously explained, the increase in density of parts processed using HIP does not just affect their apparent density, but also their fatigue performance, ductility and toughness. Fig. 5 shows the results of tests undertaken on a range of popular MIM alloys. The greatest improvement was seen in the least dense starting material, the sintered Invar. The least improvement was recorded in the sintered 17-4 PH stainless steel, which started with the highest density. It can be expected that below 92% theoretical density there would be problems with open porosity.

When densification is done via HIP for MIM/PIM parts, internal pores in the material are closed (Fig. 6). One additional advantage with densification is that a decrease in the variation of different material properties, such as impact strength and tensile strength, is achieved. This gives the user an advantage when trying to predict the life time of the parts used.

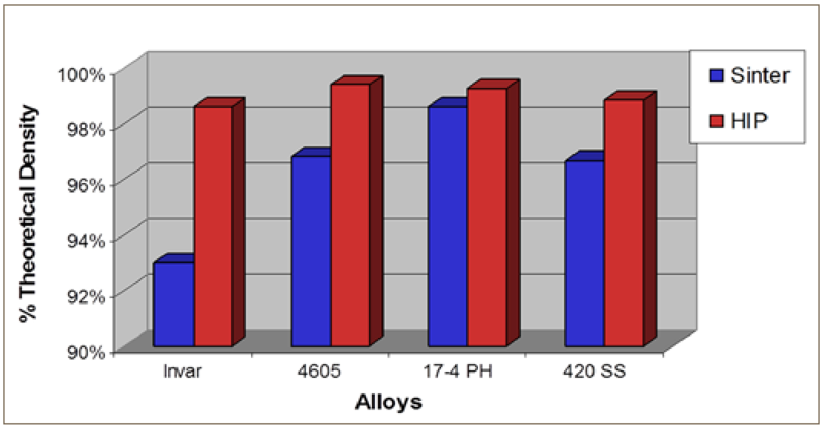

Fig. 5 Typical density comparisons for sintered and HIPed MIM products, measured with a helium gas pycnometer. The HIPed values are combined for all HIP temperatures (courtesy Bodycote)

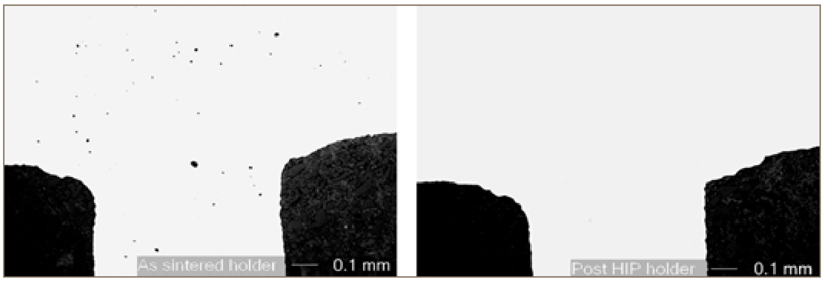

Fig. 6 Typical 17-4 PH stainless steel MIM component; un-etched microstructure shows isolated porosity as sintered (left) that has been eliminated after HIP processing (right), (original magnification 100x)

Fig. 7 A Compact HIP model for up to 24 kg / 55 lbs per cycle

Summary

The possibilities, with the latest developments, to control material properties and increase productivity with HIP technology have been shown and discussed in this paper. Productivity is increased three-fold by using Uniform Rapid Cooling, URC™. With this increase, and new techniques to increase the cooling rates up to and over 1000°C/minute, material properties can be optimised by Uniform Rapid Quenching, URQ™.

Contact

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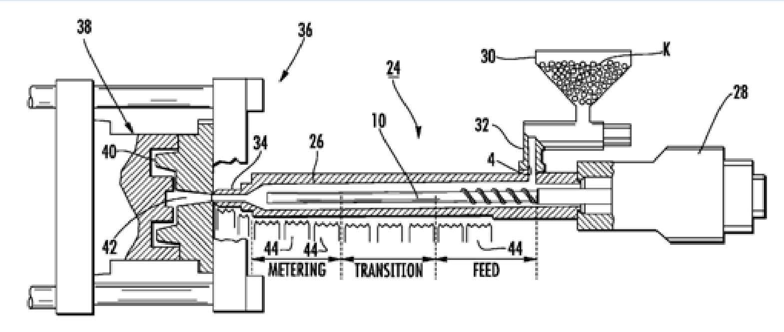
Global PIM Patents

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

US 2010276104 (A1)
SCREW DESIGN AND METHOD FOR METAL INJECTION MOULDING
Publication date: 2010-11-04
Inventor(s): K McCullough, Cool Options Inc, USA

This patent describes a method of processing metal, metallic alloys, and metal matrix composites in a plastics injection moulding machine. The method includes the steps of providing a plastics injection moulding machine having a screw. A step of removing the screw is included in the method. A step of replacing the screw with a modified screw configured and arranged for processing metals is also included. Alternatively, a step of removing the flights from the middle portion of the original screw shaft may be included.



CN 101745634 (A)
HIGH-PERFORMANCE METAL INJECTION MOULDING MATERIAL
Publication date: 2010-06-23
Inventor(s): Zuchun Ji et al, Wujiang Mingyang New Material, China

The invention discloses a high-performance metal injection moulding material, which comprises the following components in percentage by weight: 1.5 to 2.5% of nickel power, 1.0 to 2.5% of molybdenum powder, 1.5 to 2.5% of vanadium powder, 0.8 to 1.2% of carbon power and 92 to 95% of ferrous powder.

The components are mixed by powder mixing equipment into uniform alloy powder which is pelletized into the high-performance metal injection moulding material, and then the high-performance metal injection moulding material is delivered to metal powder injection moulding equipment to be produced into mechanical parts which are subjected to degreasing and sintering to be produced into part billets. The part produced in such a way has the advantages of high strength, great hardness and good wearing resistance, and the service life of equipment is prolonged when the part is used in the mechanical equipment.

KR 101000702 (B1)
BINDER FOR METAL INJECTION MOULDING
Publication date: 2010-12-10
Inventor(s): Jung Min Ho et al, Kyerim Metal Co Ltd, Korea

A binder for metal powder injection moulding is provided to improve degradation while ensuring excellent miscibility and mouldability and to reduce processes.

This patent describes a binder for metal powder injection moulding that comprises of the steps: (S1) injecting 80~95 wt% metal powder and 5~20 wt% binder, mixing and heating them at 200°C; (S2) providing the mixture to an injection machine and shaping the mixture at 100°C; (S3) removing a binder including moulding products using a hexane solvent at 35~40°C for 5~8 hours; and (S4) sintering the defatted moulding at 1340~1380°C for 120 minutes in an argon gas atmosphere.

WO 2010124398 (A1)
A METHOD FOR CO-PROCESSING COMPONENTS IN A METAL INJECTION MOULDING 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 of moulding a first component from a first feedstock comprising a first material powder and a first binder, then moulding a second component from a second feedstock comprising a second material powder and a second binder.

The first component and the second component are placed in physical communication with each other in order to form an assembled component. Removal of the first binder and the second binder from the assembled component and performing a sintering operation on the assembled component is done so as to bond the first component and the second component together.

Injection moulding simulation: New developments offer rewards for the PIM industry

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For many years 3D injection moulding simulation with SIGMASOFT® has been known as an efficient tool for reducing time to market in the MIM industry. In the meantime, software development advanced further to continuously increase the quality of results, in particular regarding rheological predictions such as jetting, flow front propagation and filling pressure. This paper summarises the basic requirements for successful MIM simulation and focuses on the benefits of advanced rheology models. Based on a bilateral project with an industrial partner, a simulation standard appropriate for MIM is defined and experimentally verified. Two results are interesting: firstly, the excellent match between experimental and simulated results prove the benefit of the continued software development and impressively document the current "state of the art" in MIM simulation. Secondly, the all-dominant role of material data becomes evident. Simulation reduces time to market significantly, but requires precise material data, which are not yet widely available.

The value of injection moulding simulation has been proven for many decades in the thermoplastic industry, and in the rubber industry since the millennium. It is interesting to remember how simulation started in these segments and to compare this to the current situation in the MIM industry.

During the mid 1980's computational power became high enough so that polymer flow could be simulated in shell-type models based on the well-known Hele-Shaw equations. It took days, sometimes even longer, to generate an appropriate midplane model required to start the simulation, which then took further time. This major effort was still worthwhile because now it was possible to understand plastics flow in a cavity and took knowledge-based decisions, for example regarding the selection of injection point locations. Iteration loops at mould try-out could be saved and therefore also saved significant amounts of money. Driven by this success more and more details of the moulding process were studied using simulation. The limitations became obvious, so additional, improved models had to be developed and implemented, for example to simulate compressible flow in the packing phase. This process is ongoing, even today the result quality of injection moulding simulation for thermoplastic applications is still improving further by adding more physics to the models and reducing simplifications. In a globalised world, companies have to drive moulding processes and applications to their limits. Thus, simulating more details reliably and as early as possible in the design process makes the difference after all.

The same story started at the new millennium in the rubber industry. Computational power was now sufficient to introduce 3D injection moulding simulation. The rubber industry was the first to buy into the new technology. Even when filling simulations took a week or even longer, the benefit was enormous. Applying the design and process integrated simulation approach of SIGMASOFT®, companies were able to reduce mould trials from several weeks to half a day.

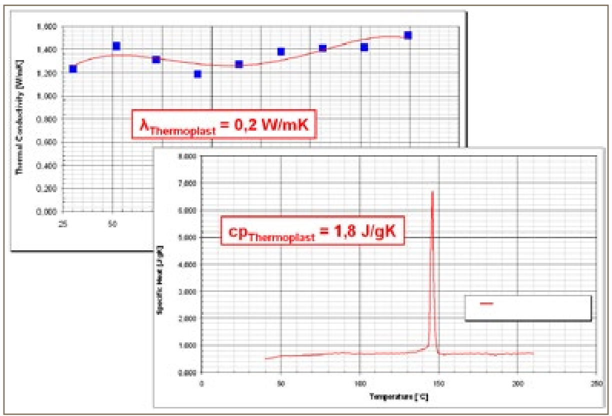

Fig. 1 MIM: Typical thermal conductivity and heat capacity as function of temperature

All the industries, moulders, designers, the respective raw material suppliers and the software vendors, had to play their specific role in these success stories. The software vendors developed robust simulation tools, easy to handle for the engineers (part-, mould-, process-). The material suppliers started to use standardised measures and to provide the material data required for any simulation. The moulders and mould designers applied the simulation to their applications and processes. They improved designs, reduced try-out phases and increased output. And finally the designer gained security and trust in the polymer materials. So they started to use this material group more and more. Collaboration is the key to success.

Today the worldwide acceptance of the 3D design and process integrated injection moulding simulation approach of SIGMASOFT® is growing enormously in the rubber and thermoplastic industries. More and more companies realise that both the injection mould and the moulding process are key factors

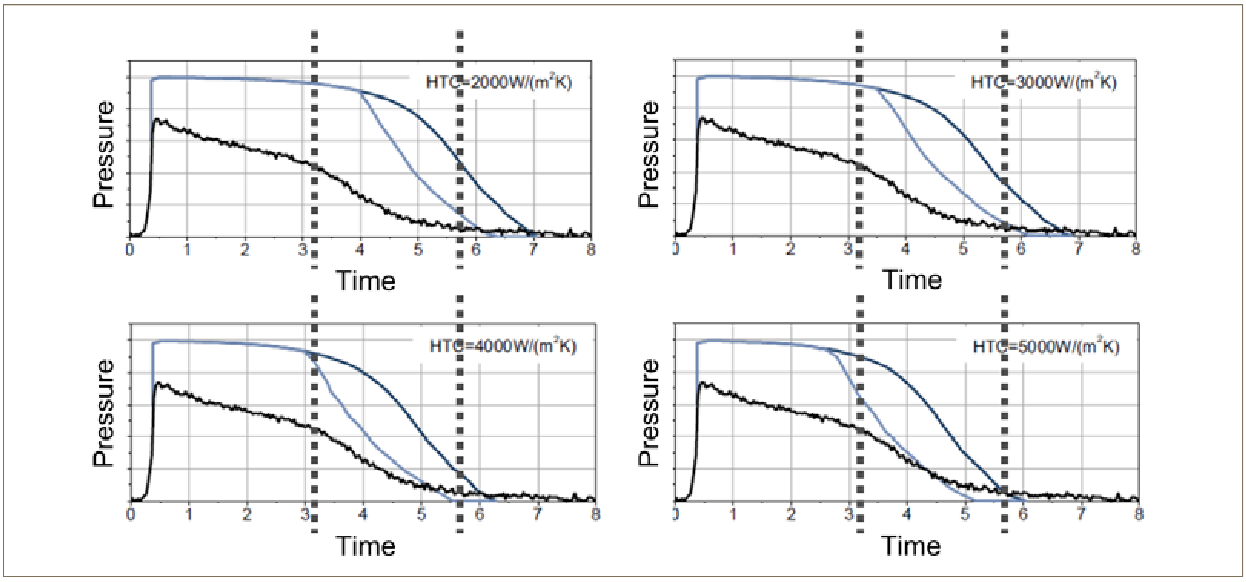

Fig. 2 Determination of htc-value based on comparison of measured (black) to simulated pressure curves (blue)

for their technical and economic success. They want to further understand what is going on inside, to identify inefficiencies and technical issues as early as possible in the product development process. While conventional simulation can help part designers to some extent, it fails to provide results for the subsequent development phases until production. Therefore conventional tools have today reached their limits and the integrated simulation approach of SIGMASOFT® offers totally new perspectives on processes and products, especially for MIM applications.

Injection moulding simulation is now ready to provide reliable answers to the MIM-industry [1]. Can the story repeat itself a third time?

MIM: Introducing injection moulding simulation

When a MIM company starts with injection moulding simulation today it is a foresighted, innovative decision and a courageous one. Injection moulding simulation is not yet perceived as a standard tool for MIM. Almost no material data are available, no guidelines about which material models should be used and how a simulation is setup appropriately. Even the measurement methods to acquire appropriate material data are not yet standardised. All these issues obviously make the companies still feel insecure and hesitant about buying into this technology, even when some innovators are already using it successfully.

This paper pursues two objectives. Firstly, to highlight a procedure on how to methodically introduce MIM simulation. This case could be guidance for companies interested in MIM simulation. Secondly, it discusses the need for material data and required measurement standards. This automatically leads to the question who will provide MIM material data to the industry in the future?

The question regarding material data arises immediately when a company starts to think about injection moulding simulation. Today, no common material databases exist for MIM. Some feedstock suppliers are measuring data for selected feedstocks and provide them to their customers upon request. In addition, some institutes provide feedstock characterisation as a service. When a feedstock is given to a lab for characterisation the next question arises: what kind of measurement device is recommended and which data range should the measurements cover? Data measurements are considered to be expensive. Thus companies, feedstock compounders and simulation users reduce it to a minimum. However what is the actual baseline; the minimum required data to enable reliable simulations? Today no guidelines are available to answer this question. Therefore companies either decide not to use simulation at all, or take over the standards available for thermoplastics injection moulding simulation. Both approaches lead to a dead end: not using simulation at all results in a limited perspective on processes and applications, which can easily mean stagnation and loss of competitiveness. Staying with thermoplastic standards is not promising, because MIM is different.

Looking at the thermo-physical data of thermal conductivity and heat capacity, the difference compared to thermoplastic polymers become obvious (Fig. 1). MIM feedstocks have a thermal conductivity about an order of magnitude higher compared to typical thermoplastics due to the metal powder, and a heat capacity easily a factor of three lower. That means MIM feedstocks carry only a very low amount of heat and loose it immediately to the mould. Processing becomes very temperature sensitive; much more sensitive than for pure thermoplastics.

Consequently MIM design rules have to be different compared to the thermoplastics standard. Specific MIM guidelines are required for product design, gate design, mould layout and processing and thus the requirements for simulation. Taking over simulation standards from thermoplastic applications seems to be obvious, but it actually is misleading. Fig. 2 shows experimental (black line) and simulated (light and dark blue lines) pressure curves within the packing phase. By interpreting the pressure curves within the packing phase one can understand the solidification process of the feedstock. Each diagram represents for one simulation experiment in which the heat transfer coefficient between feedstock and neighbouring mould components was varied. The heat transfer coefficient (htc) is a simulation parameter which has to be adjusted once for a material group due to model and thermo-physical properties. The general offset between measured and simulated curves is explained by a different setting of the switch over point, which has no influence on the interpretation done here. The shape of the curves is interesting for this experiment, not the exact values. The measured curve is taken from a pressure sensor positioned close to the gate at the cavity. The two curves from simulation are taken from exactly the same position close to the gate but from different locations cross the wall thickness.

There is a fundamental difference in the message of simulated pressures compared to measured curves. The real life pressure sensor always plots an integral pressure value representative for the whole wall thickness at the location of the sensor. In comparison, the simulation gives different values across the wall thickness depending on local temperature, viscosity and shear etc., and *not* an integral representation. Regarding the simulated curves, this means that the dark blue curve is the simulation output at the middle of the flow channel at the pressure sensor position and the light blue curve is the corresponding output at the mould surface. Looking at the measured pressure curve, two characteristic changes in the overall shape become obvious (marked by dotted horizontal lines). The first one at 3.2 s, where the pressure sensor looses perfect contact with the feedstock due to material solidification and shrinkage. The second one at 5.7 s, where the signal is lost completely, so the solidification is almost complete.

In the simulation, the htc value was changed from 2000 W/(m²K) to 5000 W/(m²K). The experiment with 4000 W/(m²K) gives the best results. All the other experiments detect the characteristic changes either too late or too early. In the left bottom diagram (4000 W/(m²K)) the first point is detected perfectly by the light blue curve close to the mould surface. Also the final loss of the signal is determined accurately - in the middle of the two simulated curves. It has to be concluded that the accurate htc value for the simulated feedstock is 4000 W/(m²K). The standard value for thermoplastics applications is however 800 W/(m²K), five times less. This clearly underlines the importance of a MIM specific development of injection moulding simulation and additionally the importance of an accurate simulation of the thermal phenomenon inside the mould.

Conventional injection moulding simulation knows nothing about the actual mould components. Due to the thermal sensitivity of MIM feedstocks described above, the actual mould design has a significant impact on processability, cycle times and green part quality. Simulation has to take this information into account and has to calculate the interaction of feedstock with the thermally relevant components of the mould. Otherwise the simulation results are not relevant. Process integrated injection moulding simulation with SIGMASOFT® provides exactly these functionalities. It clearly shows the heat exchange between feedstock and mould and the consequences regarding viscosity, flowability, mould design and tempering layout. This MIM specific accuracy comes together with an easy to use graphical user interface and a unique, automatic meshing method. Working with simulation has never been more efficient.

Understanding this challenge, the aim of the underlying study was to systematically determine a simulation baseline appropriate for MIM applications and feedstocks. Thus, feedstocks were rheologically characterised, injection moulding experiments were done and statistically interpreted (DOE). In parallel, all experiments were cross-simulated with SIGMASOFT® and different material models (rheology) investigated regarding their predictability. The study demonstrates which material measurements are required and how to use the simulation to predict the filling and the solidification phase reliably.

Understanding rheology and how to model it

Rheology is always the key factor determining product quality and processability. The focus of the study was therefore on the measurement and modelling of viscosity as input for the simulation. Fig. 3 shows viscosity measurements for one feedstock. For confidentiality reasons all actually measured data had to be transferred into a schematic form for this paper. For different temperatures, measurements with a capillary rheometer (HKR) were performed to determine the viscosity at higher shear rates (2 1/s - 2000 1/s) and additionally measurements with a Controlled Shear Stress Rheometer (CSS) for the viscosity at lower shear rates (10^{-3} 1/s - 2 1/s). The viscosity curves show different slopes in both shear rate ranges and no Newtonian plateau could be detected.

The easiest model to describe shear thinning behaviour is the First Order model (yellow curve). Historically it was the first model used for injection moulding simulation and it is still good, when no Newtonian plateau is evident in the measurements. However the First Order can only model one slope and the fit can be either adjusted to the HKR measurements or adjusted to the CSS data.

The standard rheological model for thermoplastics is the Cross WLF model (red curve). This model effectively predicts the shear thinning region (HKR data) however it always predicts a Newtonian plateau. Therefore, the Cross WLF also cannot be used to fit the CSS and the HKR data. This is again a strong indicator for not transferring thermoplastic standards to MIM applications.

Finally only the Cross WLF model with Herschel-Bulkley extension (green curve) is able to model the viscosity over the

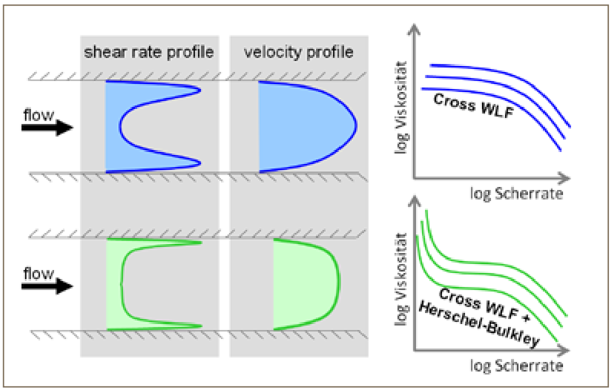

Fig. 4 Flow profile differences for the Cross WLF and the Cross WLF + Herschel-Bulkley Model

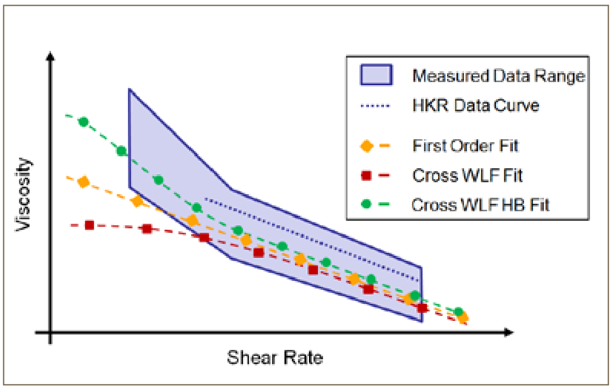

Fig. 3 Measured viscosity data range compared to model predictions

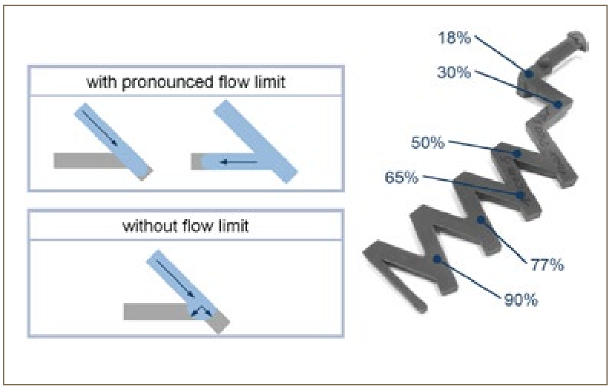

Fig. 5 Viscosity Measurements – Fit with CrossWLF + Herschel-Bulkley Model

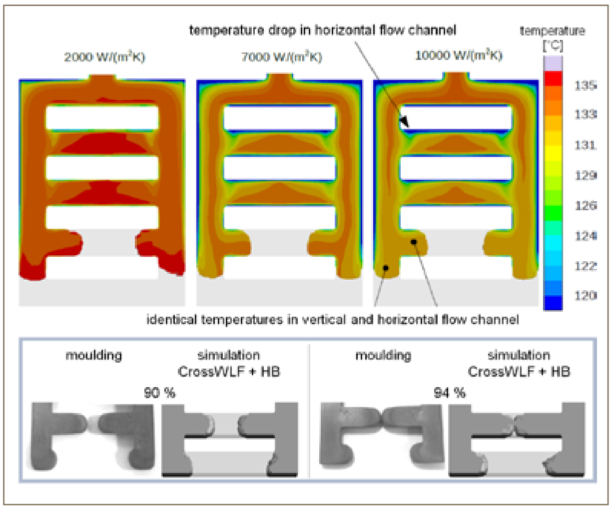

Fig. 6 Viscosity model validation: ladder mould

whole measured shear rate range. Consequently this is the most promising model to simulate filling and solidification of MIM applications. This model predicts the measured strong viscosity increase at very low shear rates. Due to this increase the feedstock behaves, in this shear rate range, more like a solid material than a polymer melt. Often people think that these low shear rates never occur in actual mouldings. If, however, one thinks about the shear rate distribution across a flow channel it becomes obvious that very low shear rates occur everywhere in the runner, gate and cavity. For typical thermoplastic melts this shear rate range does not influence the cavity filling and the corresponding simulation predictions significantly, but for MIM it becomes a dominating phenomenon.

Fig. 4 explains these underlying effects schematically and shows the differences in the flow profile comparing classical Cross WLF models (sequence at the top) appropriate for thermoplastics and the SIGMASOFT® Cross WLF model with Herschel-Bulkley extension (sequence at the bottom).

Due to the limiting shear stress (flow limit, also known as yield stress) the viscosity in the middle of the flow channel (very low shear rates) increases significantly. The result is a flattened velocity profile, well known as plug flow. Here, the whole shear load is concentrated at a very small layer close to the mould wall and the feedstock is "slipping" at this layer of low viscosity, causing typical jetting phenomena.

Fig. 5 shows one of the experimental moulds used to compare simulations and practical mouldings. This zig-zag mould was used to determine the importance of a limiting shear stress (flow limit) for the considered feedstocks and additional parameters in the filling and solidification phase.

Two approaches were used to compare predicted flow front positions with actually moulded short shots:

1. The simulation was done based on a fill time and the resulting flow front positions are compared to short shots. This is the usual procedure for comparing simulations with reality. It turned out that all three rheology models predicted the short shots reasonably well. So where is the need for advanced rheology models?
2. The drawback of approach 1 is that the actual moulding processes are significantly different between simulations and reality. A simulation based on a fill time (or a flow rate) will fill the cavity completely anyway, and only the fill isochrones can be compared to mouldings. This approach is often applied in industry but it compares apples and oranges. In reality, a reduced volume is injected to deliberately underfill the cavity. In order to really compare the two processes, and underfill the cavity in simulation too, a second run of simulations was performed. Now the maximum injection pressure monitored from the actual short shot mouldings was used as a start condition to fill the cavity in simulation. The First Order model and the standard Cross WLF model still filled the cavity completely. So the predictability of these models is way off for MIM. Only the Cross WLF model with Herschel-Bulkley extension was able to actually predict the short shots. Even in the simulation, the feedstock stopped flowing into the cavity due to the strong increase of viscosity at low shear rates.

Based on the findings of this first set of experimental and simulative experiments a second application was investigated to verify the results (Fig. 6). The Cross WLF with Herschel Bulkley extension provided excellent results regarding filling pattern and filling pressure. This improved predictability for MIM was also, in parallel, confirmed by several cases at other companies.

Both moulds (zig-zag and ladder) were used to investigate and determine additional simulation parameters such as the heat transfer coefficient (Fig. 2) and wall slip. Fig. 6 shows the influence of the heat transfer coefficient [W/m²K] on the temperature results. By comparing measured pressure curves with simulated ones, heat transfer coefficient and wall slip properties were determined to complete the simulation baseline.

Conclusions

Conventional injection moulding simulation, well-known from thermoplastics, is not sufficient for MIM applications. Simulation has to be adapted to the characteristic behaviour of MIM feedstocks and advanced rheology models are specifically required to predict cavity filling, pressure loss and feedstock solidification accurately. The powder content not only influences thermo-physical properties but also rheology in a dominant way, changing the flow profile from a typical fountain flow towards a plug flow. The advanced Cross WLF with Herschel-Bulkley extension therefore provides significantly improved results regarding both the filling and solidification phases.

Viscosity measurements with standard capillary rheometers are equally not sufficient for MIM applications. The very low shear rate regime occurs everywhere in the flow channel and has a substantial influence on the predicted filling pattern, jetting effects, pressure loss, packing efficiency and feedstock solidification. A combination of shear stress controlled measurements (low shear rates) and capillary rheometer measurements (high shear rates) are therefore required.

Finally, it can be concluded that the feedstock characterisation methods that are already available, and a measurement standard, can be defined at least for rheological characterisation. Now it is time for the industry to discuss and decide how the characterisation costs have to be shared and the data distributed. Material data can be a marketing tool for the MIM industry. The thermoplastic industry is the best example for this.

Successful injection simulation is commercially available for MIM today. However the development mission is not yet completed; one just has to think, for example, about pvt-data for multi-polymer binder systems and the powder-binder-separation effects. There is still a lot to do until the final sinter warpage can be reliably predicted.

References

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Fabrication of titanium implants with a gradient in porosity by 2-Component-MIM

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2-Component-Metal Injection Moulding (2-C-MIM) was investigated for the fabrication of net-shaped porous titanium implants for bone replacement. A gradient in porosity was obtained by the combination of feedstocks with and without space holder particles (NaCl, particle fraction 355-500 μm). The addition of a space holder promotes the formation of functional pores in the titanium structure, which support mechanical interlocking with the surrounding bone tissue, due to bone ingrowth into the implant structure. In this work, feedstocks specially tailored to the 2-C-MIM application were developed. Special boundary conditions regarding the employment of space holder particles in the feedstock and feedstock flowing behaviour had to be taken into account. Sintered titanium spinal implants with a gradient in porosity are shown, demonstrating the potential of this technique for biomedical application.

In the plastics industry, the combination of two or more materials often enables optimal properties in the finished part. The shaping technologies involved require specially constructed injection moulding machines, in which at least two injection units are used. The process has been generally named "2-Component-Injection Moulding " or "Co-Injection Moulding" [1] and essentially consists of the injection of one component after the other, in which part of the mould is moved or turned after the injection of the first component.

The transfer of the "2-Component Injection Moulding" technology to metal or ceramic powders has attracted much interest in the last few years, and parts combining both materials have been realised recently [2-7]. When only metal powders are used, the process has been often named 2-Component-Metal Injection Moulding (2-C-MIM).

Near-net-shape production of highly porous NiTi components by MIM in combination with the space holder method (SHM) was described previously [8]. By using this technique, the metal powder together with the space holder and the binder are homogenised in a kneader. The green parts are injection moulded and the space holder is removed after the first stage of debinding by dissolution in a water bath. The parts are then thermally debound and sintered. The SHM guarantees a well defined pore size distribution with total porosities up to 70%. Different pore sizes and shapes in the range of 100-500 μm can be achieved using suitable NaCl space holder particles. NaCl has proved to show a sufficient thermal and mechanical stability for injection moulding processing.

The space holder method (SHM) has already been successfully used for the production of parts with a gradient in porosity by established powder metallurgical routes, where shaping is based on pressing with subsequent machining in the unsintered state (green machining) [9]. An example is the prototype of a dental implant, recently developed at Forschungszentrum Jülich, which had a porous coating over the area which would be in contact with the bone [10]. Here, the outer layer is applied by cold isostatic pressing with subsequent green machining. In 2007, the company Synthes, Switzerland, launched a spine implant onto the market, which is used as an interbody fusion in the case of a complete replacement of the lumbar disc [11]. So far, this implant is produced based on the green machining technology developed in Jülich. The implant consists of a low porous part, which is responsible for the mechanical stability when positioning the implant during surgery and a high porous part with a porosity in the range of 60-65%, which enables the fixation of the adjacent vertebrae bones by bony tissue ingrowth. Near-net-shape production of this implant by 2-C-MIM would be exceptionally promising, especially due to cost effectiveness in the case of large scale production.

For the 2-C-MIM application with space holder, the viscosity of the employed binder system was already shown to be of great importance [12, 13]. Binder systems with very low viscosity tend to separate from the powder during injection moulding, bringing difficulties to the process.

In this work, different binder systems were evaluated for the 2-C-MIM application with space holder for the fabrication of parts with a gradient in porosity. Hereby, the influence of binder system viscosity was investigated. Prototypes of a titanium spinal implant with a gradient in porosity were produced using the 2-C-MIM technique in combination with the space holder method with selected feedstocks.

Experimental

Preliminary tests with warm pressing in a heated die were carried out before the injection moulding trials, in order to evaluate the potential of the materials investigated for the 2-C-MIM application. The flowing behaviour of feedstocks was afterwards evaluated in a capillary viscosimeter and the solids loading of the feedstocks was optimised. Finally, the results were transferred to 2-C-MIM.

Powders	Manufacturer	Particle size distribution (µm)		
		d_{10}	d_{50}	d_{90}
Ti	TLS Technik	7.9	19.5	37.3
NaCl	Merck	171.9	347.7	611.8
Fe22Cr	H.C. Starck	18.0	33.6	42.6

Table 1 Starting powders used in this work

Component	Viscosity (Pa.s)
Wax	0.012
Paraffin	0.002
Stearic acid	0.005
Low-viscous PE	0.43
Medium-viscous PE	11.2
High-viscous PE	360

Table 2 Binder components used in this work and their viscosities at 150°C

Binder system	1st component	2nd component	3rd component
SB	60 % Wax	40 % low-viscous PE	-
BS 1	60 % Paraffin	40 % low-viscous PE	-
BS 2	60 % Paraffin	40 % medium viscous PE	-
BS 3	60 % Paraffin	40 % high-viscous PE	-
BS 4	55 % Paraffin	40 % high-viscous PE	5 Vol.% Stearic acid
BS 5	60 % Paraffin	35 % high-viscous PE	5 Vol.% Stearic acid

Table 3 Binder systems used in this work (All contents in Vol.%)

Feedstock	Solids loading Φ (vol %)	Binder content 100 – Φ (vol %)	Metal powder fraction of the solids loading (vol %)	Space holder fraction of the solids loading (vol %)
Ti SB	68	32	100	0
Ti SB SH	75	25	50	50
Ti BS 1	68	32	100	0
Ti BS 1 SH	75	25	50	50
Ti BS 2	68	32	100	0
Ti BS 2 SH	75	25	50	50
Ti BS 3	68	32	100	0
Ti BS 3 SH	75	25	50	50
Ti BS 4	64	36	100	0
Ti BS 4 SH	68	32	50	50
Ti BS 5 SH	72	28	50	50

Table 4 Composition of the feedstocks prepared with titanium powder

Table 1 summarises the characteristic properties of the starting materials. The gas atomised Ti-powder was used in combination with NaCl as a space holder. Fe22Cr-powder was only employed for characterisation of the flowing behaviour of the feedstock, due to the high demand on powder material for conducting the measurements. The particle size distribution of the Fe22Cr-powder is similar to the one of the Ti-powder, so that a comparison of both feedstocks regarding their rheological properties was reasonable.

The binder components used for the development of the binder systems, along with their respective viscosities, are listed in Table 2.

The binder systems used in the investigation are shown in Table 3. The ratio of binder components is varied systematically to study the influence of binder viscosity on injection behaviour. The binder system "SB" is the standard binder system used in previous works [8], which proved itself to be suitable for the space holder technique. The binder systems "BS 1", "BS 2" and "BS 3" are paraffin-based binder systems with different polyethylene qualities as second components. The addition of the additive stearic acid was investigated for the binder system "BS 4". For the binder systems SB, BS 1, BS 2 and BS 3, the ratio of the first to second component was 60 to 40 Vol.%, which was the ratio used in previous studies mentioned for the standard binder system. In binder systems BS 4 and BS 5, 5 % of the first or second component are replaced by stearic acid.

Feedstocks with the binder systems SB, BS 1, BS 2 and BS 3 without and with the space holder were prepared by mixing titanium powder material with the binder system in a Haake HKD-T 0.6D kneader. The materials were mixed at 150°C for 2 hours. The denomination of the feedstocks was "Ti" followed by the abbreviation of the binder system used. If a space holder was used, "SH" was used at the end of the denomination. The solids loading of the feedstocks is given in Table 4. The solids loading for these feedstocks was the same as used in previous studies [8] and was determined by tap density measurements. Feedstocks with the binder systems BS 4 and BS 5 were prepared in the same manner but in this case the solids content was determined after tests with a capillary viscometer.

After the preliminary study with the titanium feedstocks and samples, the promising feedstock candidates were selected and further studies with the the capillary viscometer were carried out. Feedstocks with Fe22Cr-powder were produced with the binder systems BS 2, BS 3 and BS 4. Here the denomination of the feedstock starts with "Fe". Hereby the solids content was varied, as shown in Table 5. This variation in solids content was done in order to evaluate the influence of the amount of powder particles on the flowing behaviour of the feedstocks and to determine an optimal binder content to be used at the injection moulding trials.

The viscosity of the binder components and binder systems was determined with the rheometer MCR 100 made by Physica with the cone-plate principle with a 0.048 mm distance between the plate and the substrate and 25 mm plate diameter. The samples were investigated in an up and down shear rate ramp from 0.1 to 1,000 s^{-1}.

The capillary tests were performed with a twin bore capillary rheometer Rosand RH 2200 (Malvern Instruments) at 150°C. Dies with 2.0 and 0.5 mm diameter were used for the tests with feedstocks with and without space holder particles, respectively. As the space holder particles have a maximal particle size of 500 µm, it was assumed that a diameter four times larger than the maximum particle size of the particles would be suitable for the experiments. The feedstock was fed into the bores and allowed to heat up for 15 minutes. After this heating up phase, the test was started. Feedstocks with a space holder were investigated from 50 to 4,000 s^{-1} and feedstocks without a space holder from 50 to 20,000 s^{-1}. The difference in maximal shear rate achieved is related to the respective die diameter.

Preliminary experiments were conducted to demonstrate the ability of sintering distortion-free parts with a functional gradient in porosity. In this case, a heatable die from P/O/Weber was used for the compaction of the feedstocks. Samples were pressed with the titanium feedstocks with and without a space holder at 150°C and 110 MPa in cylindrical shapes with 12 mm diameter and approximately 10 mm height.

An Arburg Allrounder 370 U 700-100/100 2-component injection moulding machine was used for the injection moulding process for the manufacturing of the spine implant prototype. A specially constructed 2-C-MIM mould is employed for the injection of the feedstocks. Fig. 1 shows the working principle of this mould for manufacturing of the spinal implant. The feedstock without a space holder is firstly injected by the horizontal injection unit, followed by the injection of the feedstock with a space holder by the vertical injection unit.

The injection parameters were: injection flow rate 6 cm^3/s, injection pressure 800 bar, compression pressure time 3.6 s and 4.15 cm^3 and 6.75 cm^3 charging volume for the feedstocks with and without a space holder, respectively.

When the standard binder system SB was used, the obtained parts were then debound via wicking in Al_2O_3 sand at 150°C for 10 hours. Remaining wicking sand was removed with a scalpel. All the other samples (with the paraffin-based binder systems) were debound via solvent extraction in n-hexane for 24 hours at 50°C. After the first stage of debinding, interconnected pores are formed, which allow the subsequent removal of the NaCl space holder by dissolution in water bath at 60°C for 48 hours.

The sintering was performed in a Thermal Technology 121212 WM furnace, at 1300°C for 3 hours under vacuum of less than 10^{-3} Pa.

Results

Preliminary tests

Preliminary tests with warm-pressed samples with feedstocks with different binder systems already offer an impression about the potential of the materials for the production of parts with a gradient in porosity. Feedstocks with 50 Vol.% space holder particles and feedstocks without a space holder were pressed one after another. Hereby, the materials investigated were the feedstocks with the binder systems SB, BS 1, BS 2 and BS 3. After debinding and desalination, the parts were sintered. Their cross-section and typical microstructure are shown in Fig. 2.

The parts did not show significant distortion after sintering. The samples exhibited less than 1% standard deviation in diameter shrinkage, even after the gradient in porosity. The interface between the porous and dense parts, deriving from feedstocks with and without space holder particles, is very well defined.

After confirming the potential of the investigated feedstocks for the production of samples with a gradient in porosity, the rheology of feedstocks and binders was investigated by rotational and capillary viscosimetry (Table 6). The objective here was to choose the feedstocks which are best suitable for the 2-C-MIM application.

The viscosity of the SB and BS 1 binder systems are in the same range and the values are much lower than the viscosity of the BS 2 and BS 3 systems. In the case of binder system BS 3, the viscosity is clearly enhanced compared to the others.

After investigating the viscosity of the binder systems, the viscosity of the feedstocks was investigated with the capillary viscometer. The feedstocks without a space holder (Ti SB, Ti BS 1, Ti BS 2 and Ti BS 3) were investigated first.

After feeding the feedstock in the bore, the feedstocks Ti SB and Ti BS 1 showed binder-powder separation during the heating up phase.

Feedstock	Solids loading Φ (vol %)	Binder content 100 –Φ (vol %)	Metal powder fraction of the solids loading (vol %)	Space holder fraction of the solids loading (vol %)
Fe BS 2	50/60	50/40	100	0
Fe BS 3	50/60/64/68	50/40/36/32	100	0
Fe BS 4	50/60/64	50/40/36	100	0
Fe BS 4 SH	50/60/68	50/40/32	50	50

Table 5 Composition of the feedstocks prepared with Fe22Cr-powder

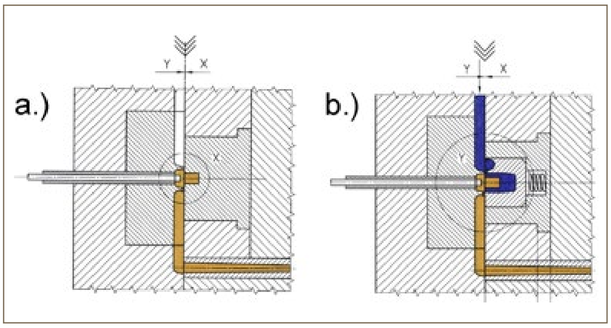

Fig. 1 Schematic drawing of the 2-C-MIM mould. a) Injection of the feedstock without space holder. b) Injection of the feedstock with space holder

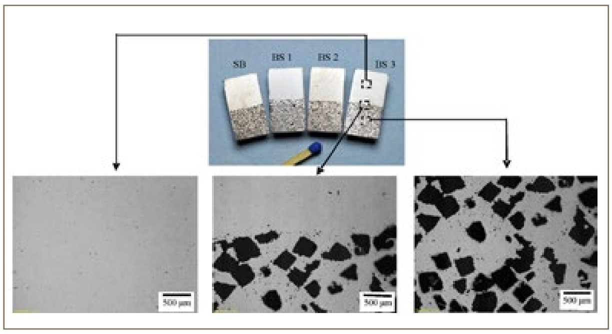

Fig. 2 Pictures of titanium samples with a gradient in porosity produced with different binder systems, sintered at 1300°C for 3h. The microstructure of the sample with the BS 3 binder system is shown as an example

	SB	BS 1	BS 2	BS 3
Binder viscosity (Pa.s)	0.15	0.04	0.53	12.4

Table 6 Viscosities of the investigated binder systems at 150°C and 100 s^{-1}

Binder material with powder particles in suspension flowed out of the capillary. When the test was started, the capillary was clogged by the remaining Ti-particles and no measurement could be carried out. Further studies at the capillary rheometer with these feedstocks were discontinued. Their use for the 2-C-MIM application seems to be unsuitable, especially if used in the vertical injection unit.

No binder-powder separation was observed during the heating up phase for the feedstocks Ti BS 2 and Ti BS 3. Nevertheless, when the measurement was started, the feedstock started to flow but stopped before reaching 1000 s^{-1} shear rate in both cases. It was presumed that the optimum powder content of the feedstocks

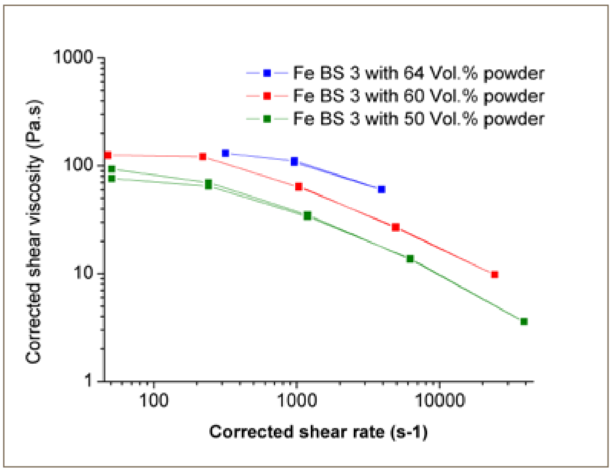

Fig. 3 Shear viscosity versus shear rate for feedstocks Fe BS 3 with different powder contents

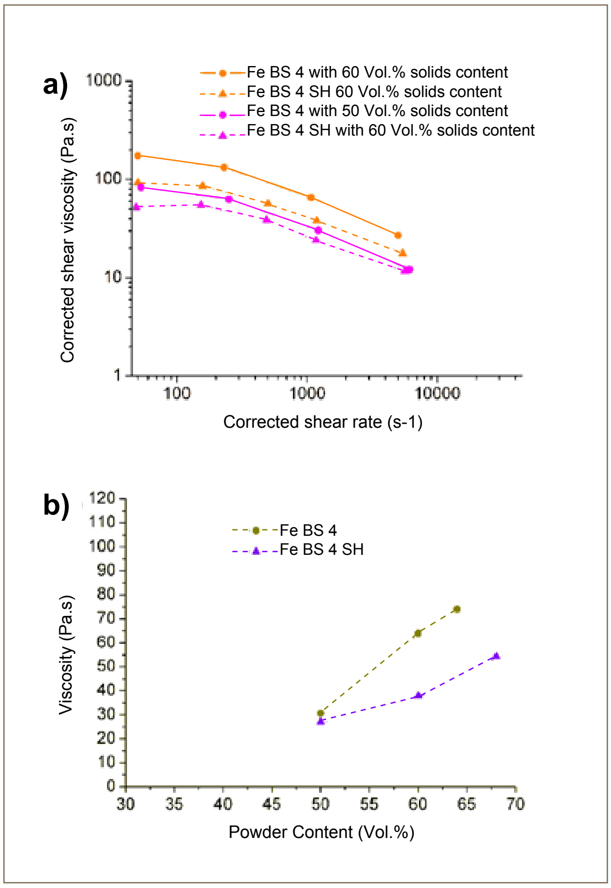

Fig. 4 a) Viscosity of feedstocks with and without space holder particles versus shear rate. b) Viscosity of feedstocks with and without space holder particles versus solids content at the same shear rate (γ = 1000 s⁻¹)

must be found for transferring the results of warm pressing to the 2-C-MIM process. Therefore, the solids loading of the feedstocks were varied for further investigation. As mentioned, this study was done using Fe22Cr powder. The feedstocks Fe BS 2 and Fe BS 3 were produced with 50 and 60 Vol.% powder. Here, two different behaviours were observed when heating up the feedstocks in the equipment bore. The feedstock Fe BS 2 flowed considerably out of the capillary during the heating up phase, whereas a negligible flow was observed for Fe BS 3.

As it is not desirable that the feedstock flows during the heating up phase, as the use in the vertical injection unit would be affected, further tests with the feedstock Fe BS 2 were discontinued. The investigation was further carried out with the Fe BS 3 feedstock, where the solids loading was further varied.

The shear viscosity versus the shear rate curves are shown in Fig. 3 for different solids loadings:

As expected, the viscosity of the feedstock increases with increasing solids loading, as described in the literature [14]. The materials exhibit shear thinning behaviour, as known also for other PIM-feedstocks [14].

The viscometer encountered difficulties in measuring the viscosity at 4000 s^{-1} shear rate, as the maximal force of the rheometer was reached. An attempt was made to measure the viscosity of the feedstock with 68 Vol.% powder, but in this case no measurement was possible. As the test was started after the heating up phase, the capillary was clogged. This is an indication that the critical point of the mixture was reached [14].

After confirming the potential of the method, feedstocks for 2-C-MIM are submitted to investigation of the flowing behaviour by capillary measurements. Hereby, the feedstock solids content and the influence of the space holder particles can be investigated. The viscosity of feedstocks with different solids contents, with and without space holder particles is shown in Fig. 4.

As expected, the viscosity increases with increasing solids content for feedstocks with and without space holder particles, as shown in Fig. 4a. When feedstocks with and without space holder particles are compared, it is clear that the addition of space holder particles decreases the viscosity of the feedstock. Feedstocks with space holder particles exhibit lower viscosities when compared to feedstocks without a space holder with the same binder system and same solids content. This effect is attributed to the difference in particle size between the space holder and metal powder particles (< 45 µm and 355-500 µm, respectively). Lower viscosities of mixtures with bimodal particle size distribution, as compared to single solid component suspensions are described in the literature [15].

Another effect of the addition of space holder particles to the feedstock is seen in Fig. 4b, where feedstock viscosity is shown as a function of the solids loading. The viscosity of feedstocks with various solids loadings was measured up to the critical point of the mixture. For the feedstock only with metal particles, the maximal solids loading reached (64 Vol.% powder) is lower when compared to the feedstock with the space holder (68 Vol.% powder). This behaviour is due to the bimodal distribution in particle size of feedstocks with a space holder, which contributes to a denser particle packing as compared to the feedstock without space holder. As a consequence, feedstocks with space holder particles have a lower demand on binder material and a higher maximal solids loading.

The solids loading of the feedstock with a space holder was further increased by reducing the amount of the high-viscous polyethylene material in the binder system composition (BS 5). By employing the BS 5 binder system composition, it was possible to further increase the solids loading to 72 Vol.% powder, which is advantageous for decreasing the microporosity of the Ti struts after sintering.

Transfer to 2-C-MIM

The careful examination of feedstock flowing behaviour and solids loading makes the combination of feedstocks with and without space holder particles possible. For injection moulding trials, the feedstocks Ti BS 4 and Ti BS 5 SH with respectively 64 and 72 Vol.% solids were used.

By employing feedstocks with and without space holder particles, the production of parts with a gradient in porosity becomes possible.

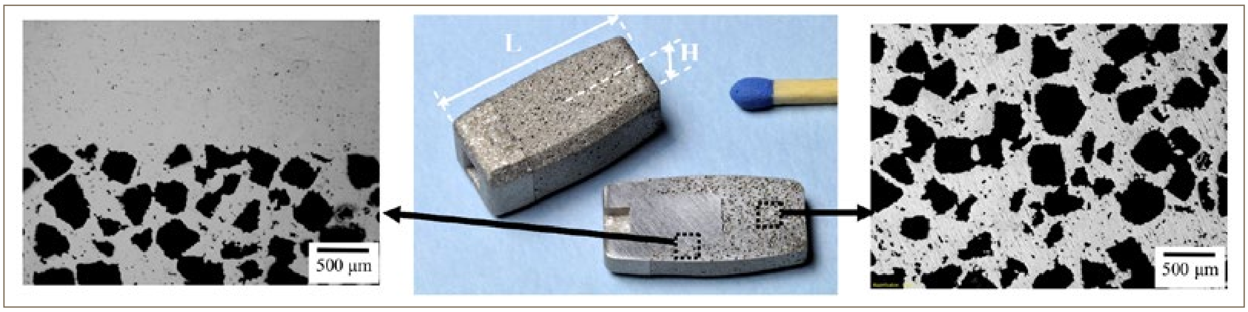

Fig. 5 Sintered titanium spinal implant and its cross-section, produced with feedstocks with and without a space holder. Parts were produced with the automated 2-C-MIM mould and sintered at 1300°C for 3 h

Prototypes of a titanium spinal implant with a gradient in porosity were produced in this way.

Fig. 5 shows the spinal implant prototype and its cross-section. The shrinkage in length and height (indicated by the letters L and H in the figure) are 13.3 ± 0.6 and 16.4 ± 0.6 %, respectively (n=4). The microstructure of the part is also shown in Fig. 5.

The dense and the porous areas of the implant are clearly visible in the cross-section of the part. The microstructure indicates a very well defined interface between porous and dense areas. Although good interconnectivity at the interface is normally present, some points of slight delamination were detected. Future works to overcome the problem focus on a redesign of the 2-C-MIM mould as well as optimisation of sintering and injection moulding parameters.

Conclusion

Warm-pressing is employed as a means of preliminary evaluation of potential feedstocks for 2-C-MIM. Sintering of distortion-free parts with a functional gradient in porosity was demonstrated sucessfully. The flowing behaviour of feedstocks with and without a space holder is investigated by capillary rheometry. The viscosity of feedstocks with space holder particles is lower as compared to the feedstock with only metal particles, at a given solids loading. Due to the higher packing density of metal powder-space holder-mixtures, feedstocks with a space holder have to be processed with higher solids loading. For 2-C-MIM trials, specially developed feedstocks which meet the application requirements are developed. The production of spinal implants with a gradient in porosity is made possible by the combination of feedstocks with and without a space holder in a specially constructed 2-C-MIM mould. The implant prototype is sintered without significant distortion, while showing a well defined dense-porous interface. Such kind of implant is attractive due to combining improved mechanical properties with optimised implant fixation by bone-ingrowth into the porous structure. The production of titanium implants by 2-C-MIM enables cost reduction in the case of large-scale production when compared to established processing routes such as pressing and green machining.

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Feedstock development for Powder Injection Moulding of Zirconium Silicate

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The interest in Zirconium Silicate $ZrSiO_4$ (zircon) arises from its wide availability and excellent properties. Due to its hardness, good corrosion resistance, high melting point, low thermal expansion and chemical inertness, $ZrSiO_4$ can be used in many different applications. Powder Injection Moulding is a suitable alternative for ceramic processing. In this paper, a complete PIM process has been developed for $ZrSiO_4$ using micrometer sized powders. Three different binders systems have been investigated: conventional LDPE-PW, HDPE-PEG and an alternative CAB-PEG system. Process parameters for different feedstocks have been evaluated and compared. The behaviour of binders using PEG as an additive is comparable to those using PW, with the benefit of a non harmful solvent. There is evidence of improved compatibility between binder components in the CAB-PEG system leading to higher sinter properties. It has been demonstrated that, for $ZrSiO_4$ contents up to 60 vol.%, injection moulding can be accomplished at low debinding rates.

Zirconium Silicate ($ZrSiO_4$), commonly known as zircon, is a ceramic material which can be found in the form of natural sand minerals or be synthetically produced. Among its unique thermo-physical properties, zircon offers a very low and uniform thermal expansion coefficient compared with typical structural ceramics ($4.1\cdot10^{-6}$/ºC from room temperature to 1400ºC) and a low thermal conductivity (5.1 W/m·ºC at room temperature and 3.5 W/m·ºC at 1000ºC) [1]. Both properties result in an outstanding thermal shock resistance. Zircon's strength retention at high temperatures is significant and its mechanical properties remain good up to 1400ºC for fully densified zircon. Moreover, Zircon exhibits excellent chemical and corrosion resistance against acids, glass melts, molten metal alloys and slag [2].

All these properties make zircon a very suitable candidate for refractory applications and make this material a potential candidate for structural applications as well. It is extensively used in a wide range of applications such as a construction material in glass tanks, in iron and steel production, in energy technology, as moulds and cores in precision investment casting or as protective coatings of steel-moulding tools.

Most of the processes for zircon powder consolidation found in the literature are based on press and sintering, with the work concerning hot isostatic pressing (HIP) being of relevance [1, 3]. However, there are few works which describe the processing of zircon by powder injection moulding. N. Schlechtriemen *et al.* describe a process for the consolidation of synthetic zircon by reaction sintering of silica and zirconia powder mixtures [4]. To the best of the authors' knowledge, there are no publications using the PIM technique applied to natural zircon powders from raw treated sand. The unique characteristics of PIM technology may lead to interesting advantages for zircon as a competitive material for structural purposes.

The present work carries out a study of the application of different binder systems commonly used in the PIM of mineral zircon powder. The study particularly focuses on the advantages of using a PEG based binder compared to conventional polyolefin based binder systems. Despite possessing exceptional characteristics for PIM, and being one of the most economic alternatives, polyolefin based binder systems are controversial since they use toxic and hazardous solvents during one stage of the debinding. Harmless PEG based binders exhibit good binder properties and an environmental and economic advantage when solvent debinding in water [5]. However, PEG blends commonly combined with a polyolefin polymer seem to be less compatible causing poorer homogeneity and green properties. The use of the natural derived CAB studied in this work, presents better compatibility with PEG [6] and it is proposed as a potential alternative to polyolefin binder systems.

Experimental procedure

Materials and feedstock development

Zircon powder used in this study (average size of 1.604 µm) was supplied by GUZMÁN GLOBAL S.L. (Spain). Its chemical composition and powder characterisation are given in Table 1 and Table 2 respectively. The particle size distribution curve obtained

	ZrO_2	SiO_2	Al_2O_3	TiO_2	Fe_2O_3
Composition (wt.%)	66	33.59	0.25	0.11	0.05

Table 1 Chemical composition (wt.%) of $ZrSiO_4$ powder

Properties	Zircon
Morphology	angular
Density (g•cm^{-3})	4.58
Tap density (%real)	32.45%
Apparent density (%real)	21.07%

Table 2 Properties of $ZrSiO_4$ powder

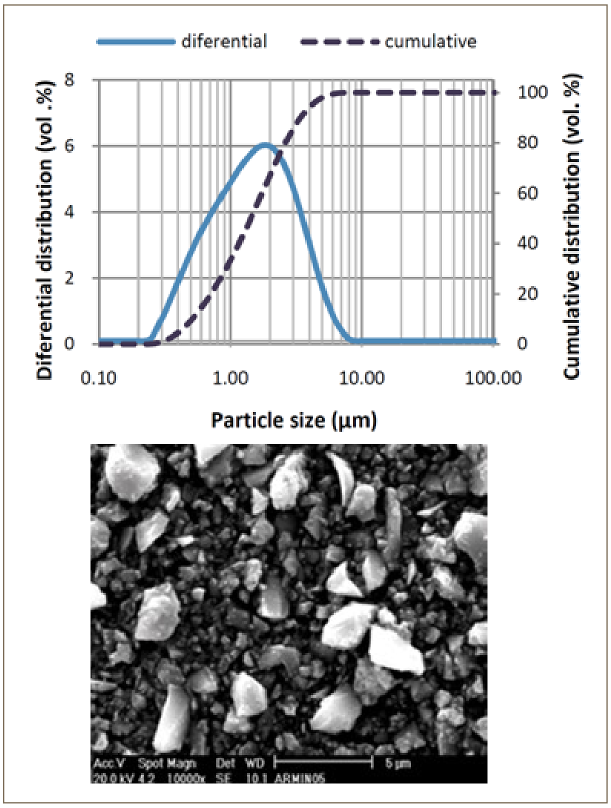

Fig. 1 Particle size distribution obtained by laser diffraction (top) and SEM image of zircon powder (lower)

by laser diffraction (Malvern 2000E equipment) and a micrograph of the zircon powder are shown in Fig. 1.

Three different binder systems were studied. Two conventional polyolefin based binders LDPE-PW and HDPE-PEG were selected in order to evaluate advantages of using a binder based on acetate-butyrate of cellulose (CAB) and PEG. Table 3 details the selected systems.

Blends were prepared in a torque rheometer ThermoHaake, model Haake Rheocord 252p, with varying solid loadings from 50% to 60% in volume of the powder for each feedstock. The details of the selected feedstock compositions are presented in Table 3. In the case of feedstock 1 (F1) and feedstock 3 (F3), a solid loading of 55 vol.% was chosen as the optimum solid loading, whereas 60 vol.% of powder was selected for feedstock 2 (F2). The mixing process was carried out at a temperature of 160ºC and a rotors speed of 40rpm for 60 min, since at this time duration homogeneity of the feedstock was observed. After granulation, an injection step was performed in a transfer injection moulding machine AB. Maximum pressure was set at 1 MPa and held for 11 seconds. Mould temperature varied from 40ºC, in the case of CAB-PEG feedstock to 100ºC for the polyolefin based binders.

Feedstock	Solid loading	Binder	Supplier	Tag
Feedstock 1	55 vol.%	47% LDPE	Dow Panreac	F1
		48%PW	Sigma Aldrich	
		5% SA		
Feedstock 2	60 vol.%	30% CAB 551	Eastman	F2
		60% PEG20k	Sigma Aldrich	
		5% PEG10k	Sigma Aldrich	
		5% PEG4k	Sigma Aldrich	
Feedstock 3	55 vol.%	55% HDPE	Dow	F3
		40% PEG	Sigma Aldrich	
		5% SA	Sigma Aldrich	

Table 3 Feedstock composition (vol.%)

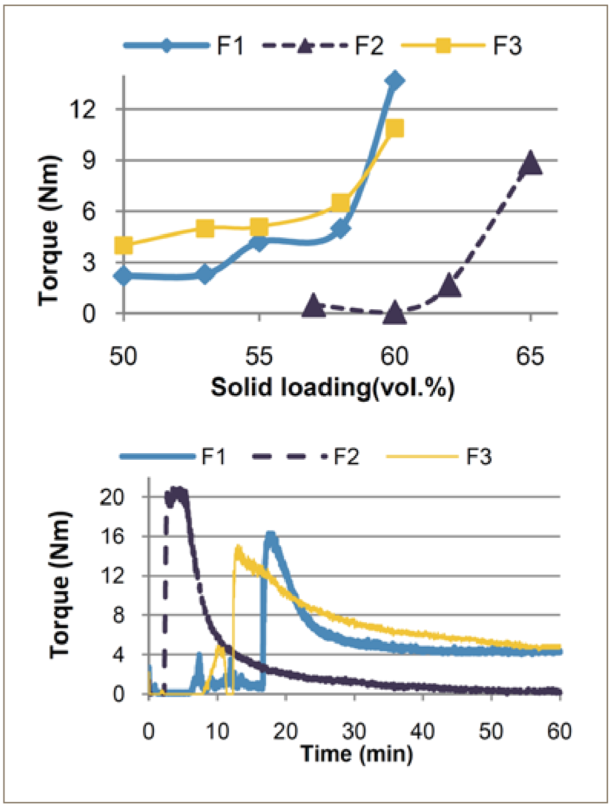

Fig. 2 Torque value at the steady state versus solidf loading (top), and torque curves for the three studied feedstocks obtained through torque reometry at 160ºC for 60 min (bottom)

Debinding and sintering

A two-step debinding process was carried out in this study. Firstly, solvent debinding was performed at 60ºC for 6h by immersion of F1 samples in hexane. The feedstocks F2 and F3 were subjected to a water-solvent debinding at the same temperature and time as F1. The selected heating profile during thermal debinding in air followed three different stages at 300, 475 and 550 ºC with holding times of 1h at each temperature. The debinding rate was set at 2ºC·min^{-1} for the entire process.

After complete binder removal, samples were sintered at 1500ºC for 1h and 3h in air in order to evaluate the influence of sintering time on final properties. The heating and cooling rate was 10ºC/min during the sintering cycle.

Characterisation

Thermogravimetric analysis (TGA) was performed in a Perkin-Elmer equipment, model STA6000, at a heating rate of 10ºC·min^{-1} in air in order to determine the decomposition temperatures of the binder components. After debinding, the carbon content is evaluated using conventional LECO CS-200 equipment.

The as-sintered samples were tested on a three-point bending machine; model Microtest, equipped with a 1kN load cell. A crosshead speed of 0.5 mm·s^{-1} was applied. Scanning electron microscopy (SEM Philips XL30) was used in order to investigate moulded and solvent-debound samples as well as fracture surfaces.

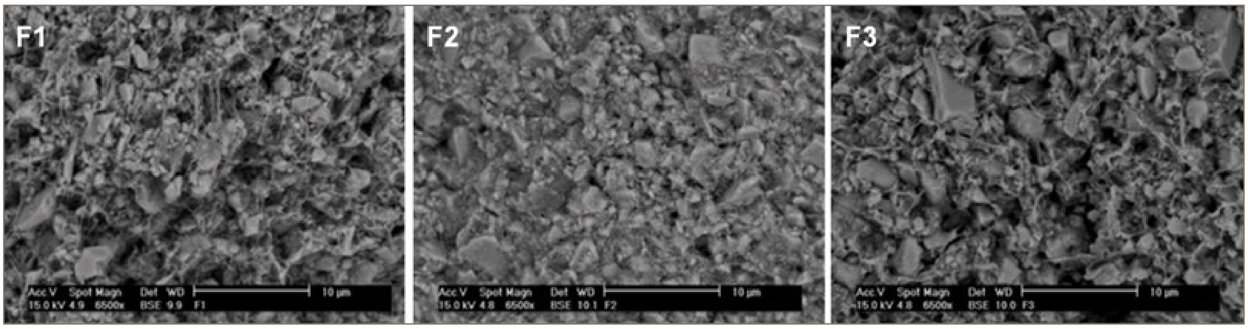

Fig. 3 SEM micrographs of specimens after injection at scale 10 µm and 6500x: (left) F1 based on LDPE and PW, (centre) F2 based on PEG and CAB and (right) F3 based on HDPE and PEG

Results and discussion

Feedstock characterisation

The main concern during feedstock production is to obtain a homogenous feedstock. Uniformity of the feedstock can be measured by means of torque reometry [7]. Fig. 2 shows torque values measured at the steady state versus solid loading for each feedstock. Critical solid loading was achieved at 60 vol.% in the case of F1 and F3. However, F2, based on the CAB-PEG mixture, allowed a higher volume fraction of powder (65 vol.%) due to lower viscosity of the mixture. This could reveal a better compatibility of the feedstock components.

It was previously determined that a good injection moulding process usually requires a torque value below 4Nm for the model of injection machine used in this study [8]. Therefore, powder fraction was limited to 55 vol.% for F1 and F3, whereas F2 contained 60 vol.% of solid loading. For these solid loadings, torque values versus time are given in Fig. 2 for each feedstock at 160ºC. Homogeneity of the feedstocks was achieved after 40 min for all cases.

After injection, feedstock homogeneity was also studied through density measurements and SEM characterisation. An inhomogeneous feedstock may result in density gradients and lead to distortion during consequent steps of the PIM process. Table 4 shows green density values of each feedstock. For F2, experimental values are consistent with theoretical ones calculated with the rule of the mixes, indicating uniform particle-binder distribution. Nevertheless, F1 and F3 experimental values partly differ from theoretical ones.

SEM micrographs of the feedstocks illustrate these differences. Fig. 3 shows an as-moulded sample of F2 where the binder is evenly distributed throughout the sample, and higher magnifications do not show any density segregation. However, density gradients are more noticeable for F1 and F3, which explains the larger deviation in green density measurements. In this case, binder separation is most probably promoted by either lower compatibility, or worse, wetting behavior between the powder and binder.

It is important to point out that even though powder did not have ideal characteristics for its implementation in MIM technology (shape and tap density), as-moulded parts presented perfect shape retention. In the case of F2, the presence of PEG, with low molecular weight and better powder-binder compatibility, significantly reduced the viscosity of the feedstock and made it more suitable for injection at higher solid loadings.

Debinding

During solvent debinding, more than 75% of soluble component (PW and PEG) was successfully removed after 6 hours for all cases without formation of cracks or swelling. More efficient elimination was achieved in the case of F2, from which 95% of the PEG was extracted even though higher powder loading was present. SEM characterisation showed homogenous elimination at both regions

Green density ($g \cdot cm^{-3}$)	F1	F2	F3
Picnometer value	3.01	3.23	3.11
Theoretical value	2.93	3.23	2.99

Table 4 Comparison of experimental and theoretical green density values for each feedstock

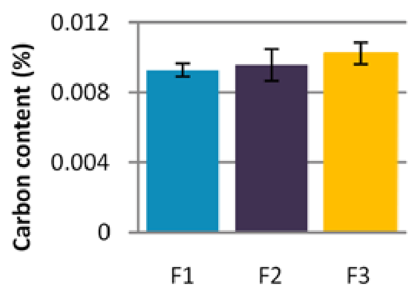

Fig. 4 Carbon content (%) after debinding at 2ºC/min

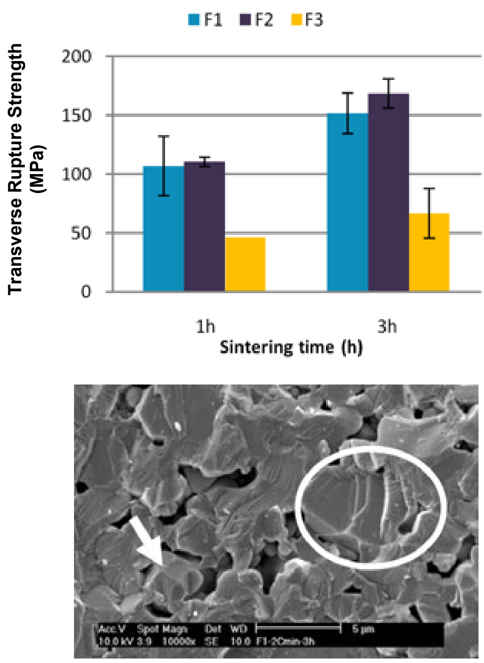

Fig. 5 Comparison of transverse rupture strength (TRS) values of the as-sintered samples of each feedstock after 1h and 3h of sintering (top) and SEM micrograph of F1-3h sintering specimen showing main failure mechanisms (bottom)

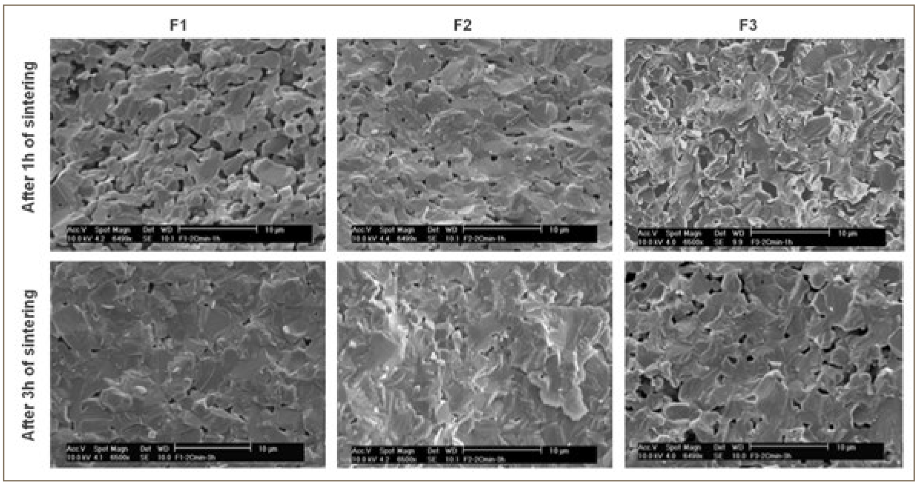

Fig. 6 SEM micrographs of as-sintered specimens of: (left) F1 based on LDPE and PW, (centre) F2 based on PEG and CAB and (right) F3 based on HDPE and PEG

close to the edge and the core of the specimen indicating good accessibility of the solvent as well as efficient diffusion of the soluble component.

Applying a heating rate of 2ºC·min^{-1} during thermal debinding did not result in any dimensional distortion or defects. Fig. 4 shows carbon content results of samples after debinding. There is no remarkable difference among them, although F3 shows the highest levels. This result confirms complete CAB removal after debinding.

Mechanical properties

The results from bending experiments on the as-sintered samples are given in Fig. 5. It can be observed that transverse rupture strength (TRS) increases with increasing sintering time in all cases. There is evidence of improved TRS of F2 compared to conventional polyolefin based binders.

This fact is especially true when compared to F3. Observed density gradients and higher viscosity of the feedstock may explain the detrimental effect on final properties of this sample. Besides, higher solid loadings in F2 also favour densification of the compact. The maximum value is achieved after 3h of sintering.

Maximum TRS (F2) is comparable to hot-pressed zircon samples at 1500ºC from the literature. Increasing sintering temperature up to 1600ºC leads to TRS enhancement from 150 MPa to 320 MPa for pure zircon, although impurities may decrease these values [3, 10].

The microstructural evolution of fracture surfaces at different sintering times are shown in Fig. 6. After 1h of sintering at 1500ºC, F2 specimen exhibited the greatest sinter neck growth. F1 and F3 seem to have less developed inter-particle connections and higher residual porosity, whereas F2 shows smaller rounded pores.

Increasing sintering time results in better developed inter-particle connections and, therefore, higher densification in all cases which is in agreement with the TRS results shown in Fig. 5. F2 exhibits almost full density, consistent with the maximum TRS value. Observed lower TRS of F3 is related to higher residual porosity even after 3h of sintering. Cleavage facets and intergranular fracture are predominantly the main failure mechanisms for all specimens (Fig. 5).

Conclusions

The present study carries out the optimisation of a PIM process for mineral zircon powder. Alternatively to conventional polyolefin binder, this study has focused on the development of an economic and efficient binder based on water-soluble PEG and naturally derived CAB. This combination resulted in rapid homogenisation of the feedstock in the presence of higher solid loading, compared to the prevailing polyolefin based binder. There is evidence of better compatibility between feedstock components that improves rheological behavior of the mix. Despite the characteristics of zircon powder, which are considerate inappropriate for PIM, the low viscosity of the feedstock favored zircon particle motions and facilitated injection of the feedstock at 60 vol.% of solid loading. A combination of solvent and thermal debinding resulted in gradual binder removal and comparable elimination efficiencies to conventional binders. Moreover, F2 provides the highest TRS after 3h of sintering in air at 1500ºC. Thereafter, improvement in final sinter density was achieved when the binder system was composed of CAB and PEG.

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