Vol. 11 No. 3 SEPTEMBER 2017

FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

POWDER INJECTION MOULDING INTERNATIONAL

in this issue

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Atomisation technology at PSI

POWDERMET2017 technical reports

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INOVAR COMMUNICATIONS

POWDER INJECTION MOULDING INTERNATIONAL

For the metal, ceramic and carbide injection moulding industries

Taking on the digital industrial revolution

MIM has never featured in the media as much as it has in the last six months. Unfortunately, its appearance has primarily been as a passing reference in news stories about a more in-vogue technology, Additive Manufacturing.

Binder-based metal AM processes borrow heavily from MIM technology to the extent that they are now becoming referred to as 'MIM-like'. Not only do they share the debinding and sintering processes used in MIM, but more often than not they also share the same powder specification, referred to in some instances as 'MIM cut'.

The question that many are now asking is if the new generation of 'MIM-like' AM processes will compete with MIM. To a certain extent this is inevitable, particularly for lower volume production runs. MIM's Achilles' heel has always been the high costs of tooling and development time.

Reducing the costs associated with MIM part development is one way that MIM can defend itself against those looking to encroach on its territory. As we discover in this issue, process simulation is one tool that can speed up MIM part development, reducing cost and risk by enabling the identification of issues very early on in a part's development cycle.

One can, of course, also choose to see the rapid commercialisation of MIM-like technologies as an opportunity to give MIM the promotion and exposure that it never benefited from in its early, pre-internet years. As more people discover AM, the MIM industry needs to ensure that they also learn about what MIM can offer as an advanced, net-shape manufacturing process with an excellent track record of growth and innovation.

Nick Williams,
Managing Director & Editor

Cover image
A 17-4 PH canard made for UTC Aerospace Systems and Raytheon by Dynacast (see p. 34)
(Courtesy MPIF)

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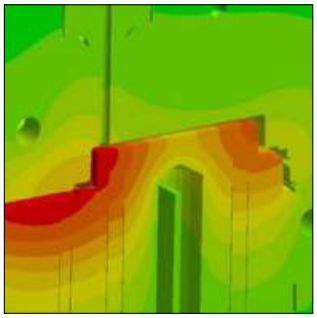

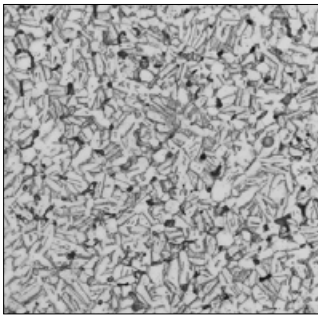

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In this issue

49 MIM in the digital age: Simulation technologies to help the industry compete in the 21st century

It is a fact that the vast majority of defects in MIM components occur during injection moulding. The latest moulding simulation technology not only anticipates these defects before tooling costs are incurred, but can also be used to automatically generate optimised tool and runner designs by analysing multiple variations of gate location and type. Dr Götz Hartmann, MAGMA GmbH, and Timo Gebauer, SIGMA GmbH, explain how simulation technologies can today be used to enhance new MIM part development.

59 PSI Ltd: Celebrating thirty years of innovation in gas atomiser production

Phoenix Scientific Industries Ltd (PSI) was founded thirty years ago with the goal of developing gas atomised metal powders for the then emerging Hot Isostatic Pressing (HIP) industry. Since then, the company has continuously developed its atomising technology in response to the evolving application areas for gas atomised metal powders, notably in the Metal Injection Moulding industry and the more recently developing Additive Manufacturing processes. Bill Hopkins discusses the company's story and comments on a recent move into metal powder production.

67 POWDERMET2017: Advances in the processing of 430L stainless steel, Ti6Al4V and nickel-base superalloy CM247LC

Two sessions at the POWDERMET2017 International Conference on Powder Metallurgy & Particulate Materials, Las Vegas, June 13-16 2017, focused on developments in materials processed by MIM. In this report Dr David Whittaker reviews three papers that cover advances in the processing and properties of MIM 430L, the microstructural control of MIM Ti6Al4V and the influence of heat treatment on MIM nickel-base superalloy CM247LC.

81 POWDERMET2017: Innovative technologies support the control of moulding and sintering

In our second report from the POWDERMET2017 International Conference on Powder Metallurgy & Particulate Materials, Dr David Whittaker reviews three papers that outlined the development of aids to support the control of PIM processing. These specifically addressed the mouldability of low pressure PIM feedstocks, the influence of feedstock properties on PIM of Lead Zirconate Titanate (PZT) and the evaluation of a ceramic temperature monitoring device for sintering furnaces.

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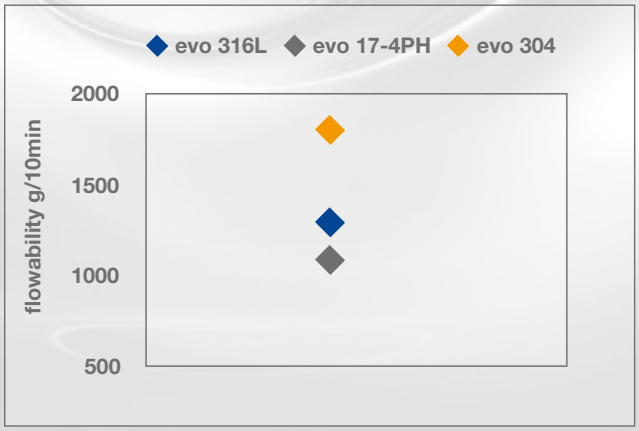

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Industry News

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

Ultrafuse 316LX: BASF's MIM technology adapted for Additive Manufacturing

BASF SE, the global leader in feedstock production for Metal Injection Moulding, has taken a step into the world of metal Additive Manufacturing with the launch of Ultrafuse 316LX for use in Fused Filament Fabrication (FFF) systems. The material has been designed for the production of complex metal components.

Ultrafuse 316LX is metal-polymer composite filament with a non-slip surface, allowing its application in any bowden or direct drive extruder. Its high flexibility allows it to be funnelled through complex idler pulleys as well as guide roller filament transportation systems. Once formed, the parts undergo the same debinding and sintering process as used for parts produced using BASF's Catamold® feedstock for Metal Injection Moulding. MIM technology has been in commercial use worldwide since the late 1980s.

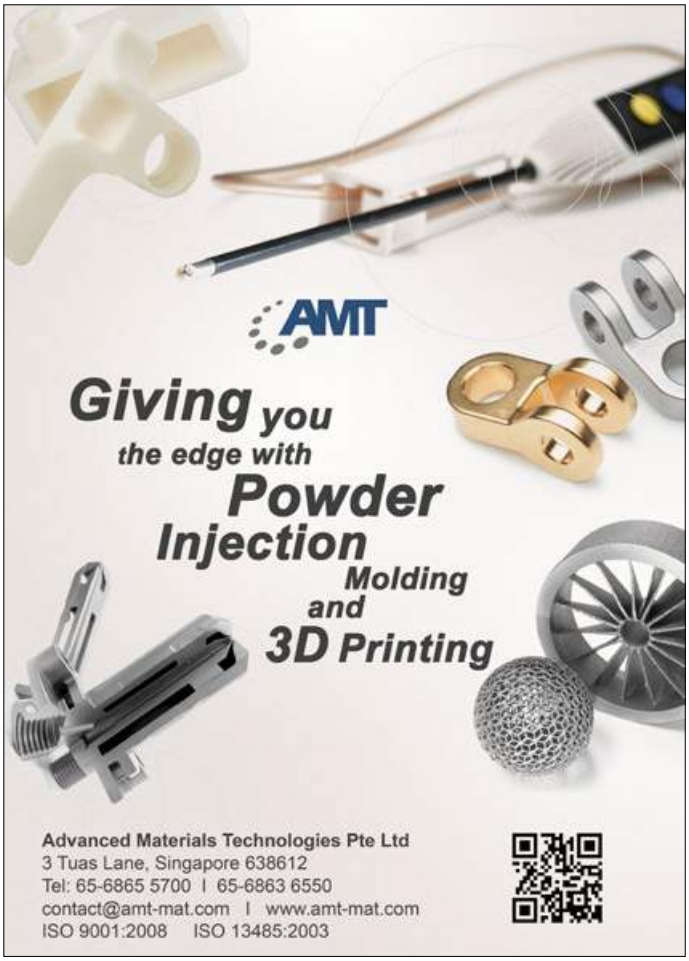
In this process, catalytic debinding removes polymer binder from the part and sintering in pure hydrogen or a vacuum results in a finished metal component that is close to full density. The whole process is said to be faster and less expensive than offered by existing Powder Bed Fusion (PBF) systems.

Ultrafuse 316LX is available in 1.75 and 2.85 mm diameter filaments. According to BASF, no changes to the FFF hardware are required to process the material. Currently only a 316L stainless steel option exists, but BASF states that other metal options will be developed.

The filament is said to be suited to a broad range of applications for functional prototyping and small series production. BASF lists various applications including watches, decorative parts, medical equipment and parts for the food and chemical industry.

www.basf.com

BASF'S Ultrafuse 316LX is suited to a broad range of applications for functional prototyping and small series production (Courtesy BASF)



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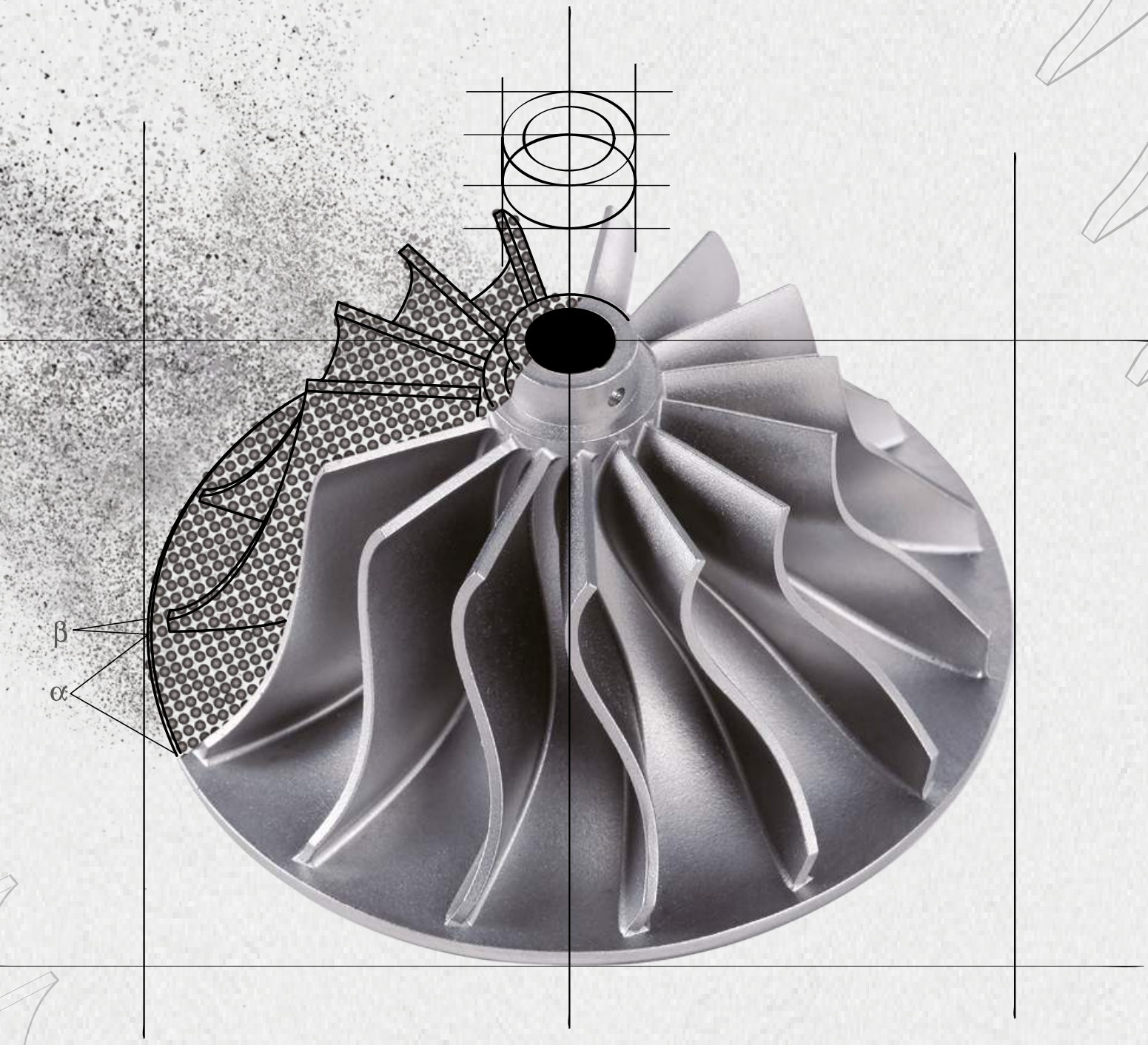

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State of the MIM industry in North America

According to MPIF President Patrick McGeehan, the US MIM sector saw a sales increase of about 10% in 2016 to an estimated range of $350-400 million. In his presentation during the Opening General Session of POWDERMET2017, Las Vegas, USA, June 13-16, 2017, McGeehan also estimated that MIM grade powders consumed in the US increased by at least 10% to a range of 1,365,000–1,745,700 kg (3–3.85 million lb).

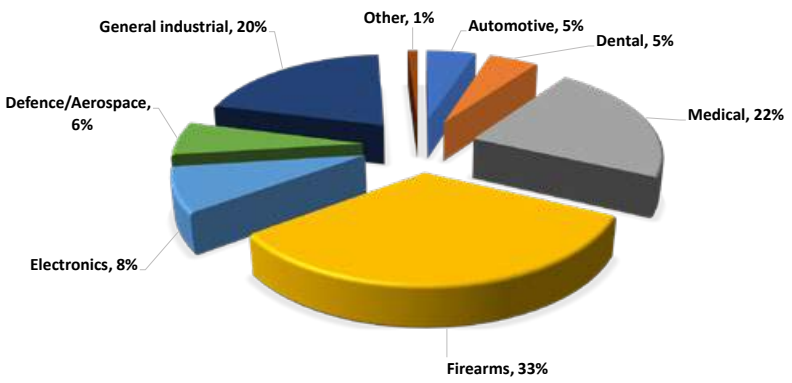
The US MIM industry includes between twenty-five and thirty commercial parts makers, plus fifteen to eighteen captive operations making primarily medical, dental and firearms components for their own products. It was stated that in the US, stainless steels and low-alloy steels continue to dominate the MIM materials mix, representing an estimated 83% of powders consumed. Other MIM materials include soft magnetic materials, tungsten alloys, titanium, tool steels, Inconel 625 and 718 and tungsten carbide. McGeehan stated that traditional MIM markets remain steady, except for a significant decline in the firearms market beginning in early 2017.

The MPIF's 2017 Pulse Survey among members of the Metal Injection Molding Association (MIMA) shows the market breakdown of North American MIM parts shipped by weight in 2016. The results point to continued sales increases, with 89% of Metal Injection Molding Association (MIMA) respondents projecting sales increases during 2017. It was also stated that Hot Isostatic Pressing (HIP) industry companies gained in 2016 based on a pick-up in demand from the aerospace and aircraft engine (commercial and military) markets. Demand for densification services for the MIM and AM sectors remains strong and will continue to grow.

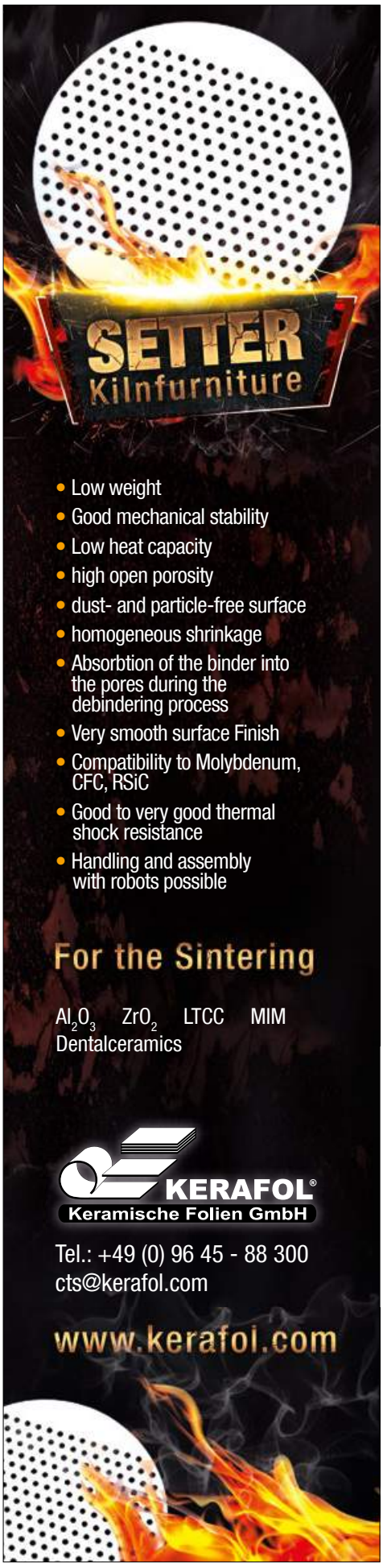
McGeehan summarised by stating that, with signals from the national economy still flashing good news, the broader Powder Metallurgy industry's general outlook for 2017 should be satisfactory, with this being especially true for the MIM and metal AM sectors. "PM companies are well-positioned to meet the challenges ahead with realistic expectations, careful planning and R&D investments. Opportunities within the industry are plentiful. PM's positive history of reinventing itself by adopting new technologies and entering new markets will certainly support continued growth in the years ahead."

www.mpif.org
www.mimaweb.org

2016 North American MIM market (by weight)



The North American MIM Market, by weight, in 2016 (Data MIMA Industry Pulse Survey / MPIF)



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Award winning MIM auto part helps to reduce emissions

In June this year Phillips-Medisize Corporation, along with its customer Delphi, received the 2017 Metal Powder Industries Federation (MPIF) Grand Prize Award in the Automotive Engine category for its metal injection moulded four-slot fuel valve seat part. The part is used in the Multec3.5 compressed natural gas (CNG) fuel injector, which can be found in several small-engine and automotive applications, including aftermarket CNG conversions for trucks and cars, and aims to help contribute to a reduction in greenhouse-gas emissions.

Matt Jennings, Phillips-Medisize Chairman and CEO, stated, "We are honoured to accept this award with Delphi. The customer was responding to the market's need for a low cost, low pressure, port fuel injection (PFI) injector and our MIM technology made it possible. Awards like this, along with our continued growth, support our commitment to expanding our Menomonie, Wisconsin facility last year."

"Phillips-Medisize was a key supplier development partner for the Delphi CNG injector and we were pleased with their technical expertise and manufacturing capability. This enabled us to produce a key injector component that met our design vision and performance expectations," stated Geoffrey Scott, Engineering Manager, Delphi.

Phillips-Medisize Corporation, along with its customer Delphi, received an MPIF Grand Prize for these Metal Injection Moulded four-slot fuel valve seat parts

Bill Welch, Phillips-Medisize's Chief Technology Officer, explained, "Our MIM technology allowed for a complex seat design that could be net-shape fabricated, while eliminating the need for expensive secondary machining. It also provided additional benefits, such as moulding intricate lip edge features, stainless steel material and seat sealing features that are uniform and consistent. Without MIM, the part would have had to be completely redesigned. There is no other way to achieve the lip seal surface and the cost of a multiple piece assembly/entire fuel injector would have been exponentially higher."

Phillips-Medisize's 4650 m^2 (50,000 ft^2) MIM facility houses four continuous debinding and sintering furnaces, multiple batch furnaces and dedicated moulding equipment, as well as a fully staffed and equipped metallurgical laboratory. The company states that it is common for MIM to produce parts at 50% less than the cost of CNC machining or investment casting.

At the same time, stated Phillips, the true value of MIM comes from its ability to produce parts with complex shapes, superior strength and excellent surface finish in combination with low- to high-volume manufacturing capability. With more than twenty years of experience in MIM technology, Phillips-Medisize reports that it produces parts from a variety of materials, without the need for machining, for nearly every market.

www.phillipsmedisize.com | www.delphi.com

A complete listing of all MIM-related winners in the 2017 Metal Powder Industries Federation (MPIF) Powder Metallurgy Design Excellence Awards can be found on page 34.

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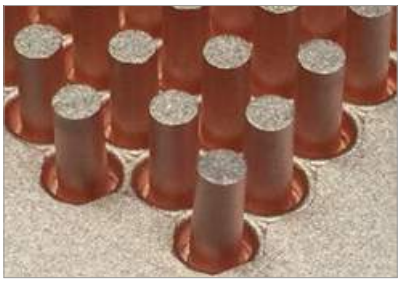
Taiwanese MIM producers seize opportunities provided by electric vehicles

A number of Taiwanese Metal Injection Moulded notebook and IT component suppliers have begun taking advantage of the opportunities provided for MIM producers in the electric vehicle industry, according to a report by Taiwan-based newspaper *Digitimes*.

A key opportunity for MIM producers in the electric vehicle market is the manufacture of Insulated-Gate Bipolar Transistor (IGBT) cold plates by MIM. These devices are typically used in high-end liquid cooling systems, particularly those with limited thermal dissipation space, such as Electric Vehicle/Hybrid Electric Vehicle power modules. These systems generally have a compact structure with little thermal dissipation space and thus have difficulties dissipating heat generated. MIM technology can be used to achieve a complex geometric design that provides both high thermal performance and low pressure drop and also enables the production of thin-walled metal components for better heat dissipation.

MIM faces some competition from forging in this arena, with Jentech, Taoyuan, Taiwan, reporting the certification of its forged IGBT power modules in the first quarter. However, MIM offers significant advantages over forging for the production of small, complex electrical parts, the demand for which is expected to grow with the rise in electric vehicle manufacture.

Tesla has reportedly been working with Taiwan's electronics manufacturers for products such as motherboards. According to *Digitimes*, Taipei-based Pegatron Technology is rumoured to have landed central computer motherboard orders from Tesla for its Tesla Model 3, but Pegatron declined to comment on its clients or orders. The Summer 2017 issue of *PIM International* (Vol. 11 No.2), reviewed the development of MIM IGBT power modules as part of the report: 'APMA 2017: MIM strengthens its competitiveness with novel technologies and new applications'.



*U-cup Fin™ designed by Amulaire Thermal Technology for an IGBT power module (*PIM International, *Vol. 11 No. 2, June 2017)*

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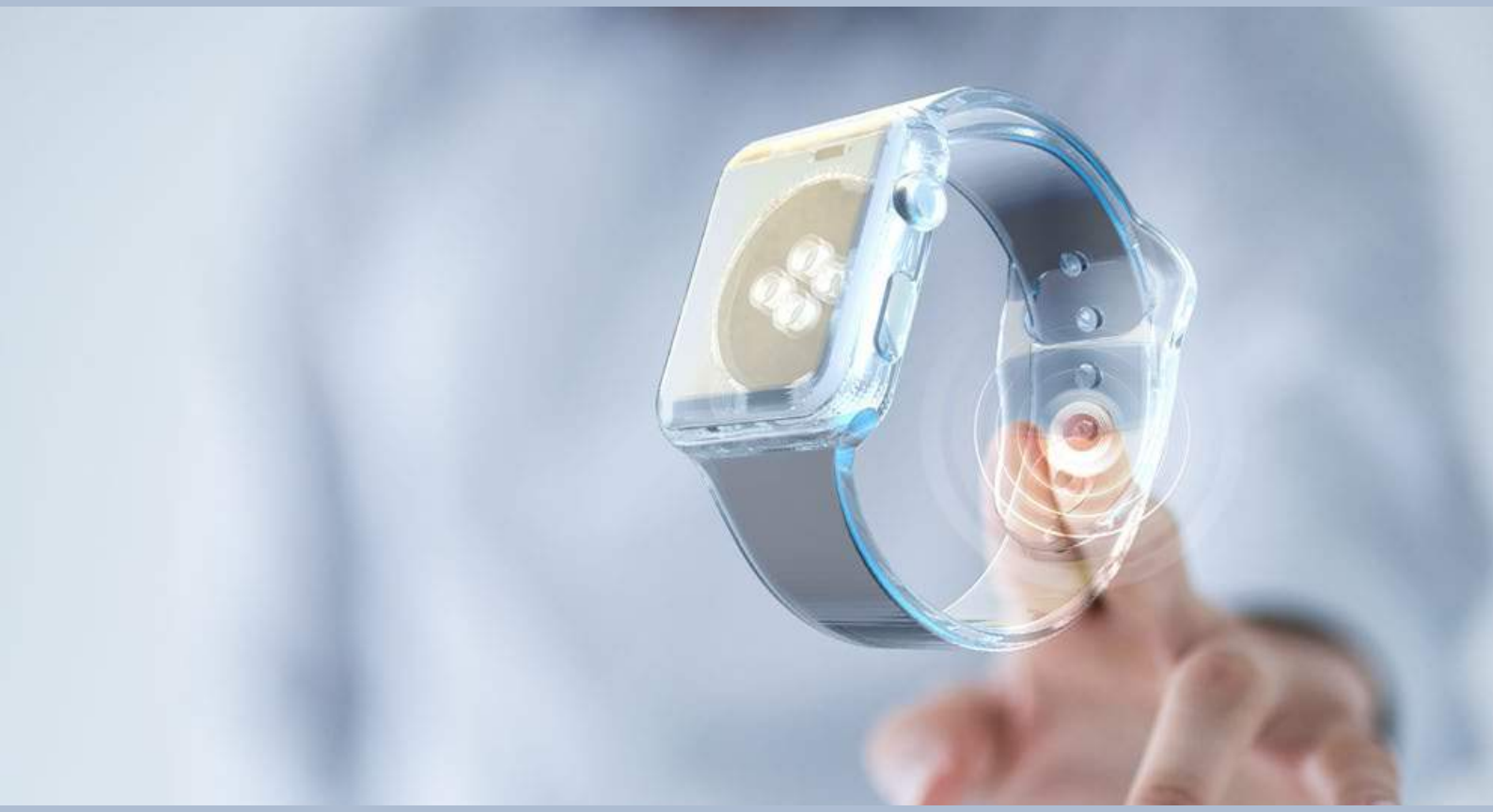

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Japanese MIM producers anticipate growth following another poor year

The Japan Powder Metallurgy Association (JPMA) has reported that sales of MIM parts decreased slightly again in 2016 to reach ¥10.26 billion ($93.8 million), compared to ¥10.54 billion ($99.5 million) in the previous year. A high point of Japanese MIM production was in 2010, when sales of MIM parts reached ¥11.952 billion ($112.8 million) (Fig. 1). This data is based on the JPMA's latest annual survey of twenty-one leading MIM parts producers in Japan, including JPMA members and non-members. However, respondents to the JPMA survey also indicated that they expected sales of MIM parts to grow by a yearly rate of 5-6% in the future.

A breakdown of the application areas for MIM in Japan showed that the Industrial Machine segment remained the largest single sector with a 26.7% market share, up slightly on the previous year's 23.5%, followed by medical appliances with a 19.9% share of the market, up from 19.3% in 2015 (Fig. 2). The automotive sector saw its market share decrease to 16.9% against the previous year's 17.4%, with the motorcycle sector down to 3% from the previous year's 6.3%. The Information Technology sector's market share was shown to be down slightly at 9.1% compared with 10.7% the previous year.

Stainless steel powders still dominate the market for materials used in MIM parts in Japan, with 66.8% of the market share (2008: 54.2%) (Fig. 3). The JPMA reported that stainless steels, ferrous based magnetic materials and Fe-Ni steels currently account for over 85% of total powder demand for MIM in Japan. According to the JPMA, positive growth was seen in the demand for materials for use in the production of wrist watches, which saw their market share grow from 3.2% in 2015 to 3.9% in 2016.

www.jpma.gr.jp

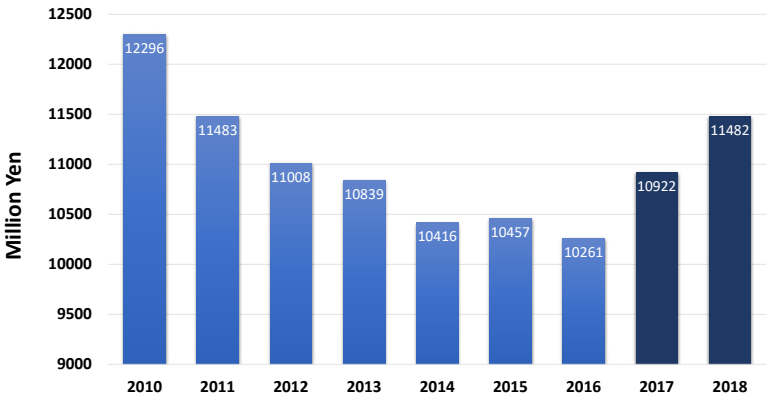

Fig. 1 Sales of MIM products in Japan. FY2010-2016 are based on results; FY2017-2018 are estimated (Data JPMA)

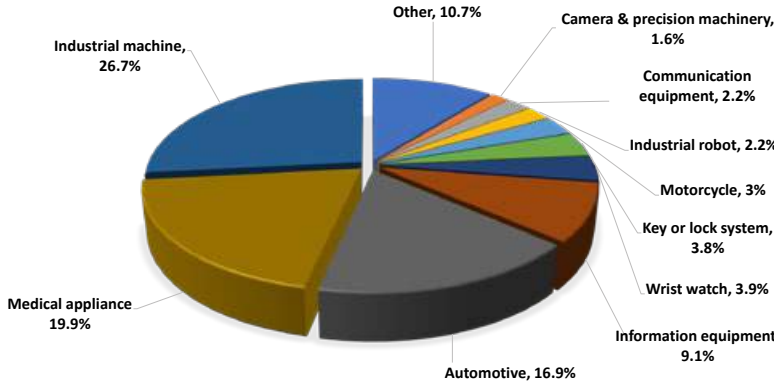

Fig. 2 MIM markets in Japan for 2016. The industrial machine and medical appliance sectors both saw an increase in overall share in 2016 (Data JPMA)

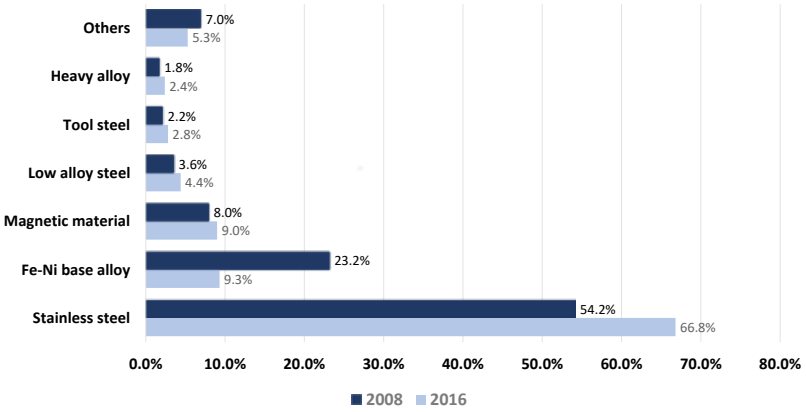

Fig. 3 Usage of MIM materials in Japan, comparing 2016 with 2008. A significant shift to stainless steels is visible (Data JPMA)

The June 2017 issue of *PIM International* features the report "MIM in Asia: A story of continuing success", available as a free download from our website.

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Rapid growth and new opportunities for India's MIM industry

India's economy registered 8.0% growth in 2016 and is expected to grow by 7.4% this year. This growth has been seen across all major market segments, from automotive to textile and industrial equipment, tools and consumer goods.

Ravi Bollina, CEO at consultancy firm LechTech Materials Pvt. Ltd, told *PIM International*, "This scenario has presented the ideal conditions for entrepreneurs and businesses to consider MIM as their manufacturing operations grow. This is reflected in the fact that the number of MIM companies now operating in India has risen from six to fourteen, more than doubling in the past four years."

The revenues of India's MIM companies has increased from $50 million in 2014 to $150 million in 2016, a threefold increase in three years. "Many of the newer firms which have become operational have seen robust growth and some reached breakeven within two years of setup," commented Bollina. India's MIM firms include Indo-MIM, Meeturaj Industries, Meta MIM, VDR Metals, Phoenix Metals, Kalyani Technoforge, WFB Industries, JJ Ortho, Modern Ortho, MD Metalline, MIM Components and Carborundum. It was also suggested that international manufacturers are considering establishing MIM production in India. Of the MIM operations which have begun production in the past three years, LechTech has been a key consulting and engineering partner in establishing these facilities to global standards. The company expects to bring another two facilities on-line by the start of the next business year.

Almost 90% of India's MIM production is currently exported to Europe, Japan and the US, however this balance is expected to shift. Domestically, the defence, automotive and textile sectors in India are expected to help push the MIM industry to higher revenues in the coming years. "The icing on the cake will be when the 3C sector, especially smartphone and tablet makers, expand their Indian production. Apple has already announced its intention to manufacture and assemble its iPhones in India. Samsung, along with Indian phone makers, already have some assembly lines in India."

The supporting infrastructure for MIM in India is currently rather weak, stated Bollina. Established global manufacturers of furnaces and moulding machines import into India. Powders and feedstocks are also imported. "This ecosystem needs to develop at a faster rate for MIM to progress even more rapidly. Skilled manpower is available in India and it is not an issue to setup and run a MIM firm. However, training in MIM and awareness needs to increase for further penetration of MIM in the local industrial base," concluded Bollina.

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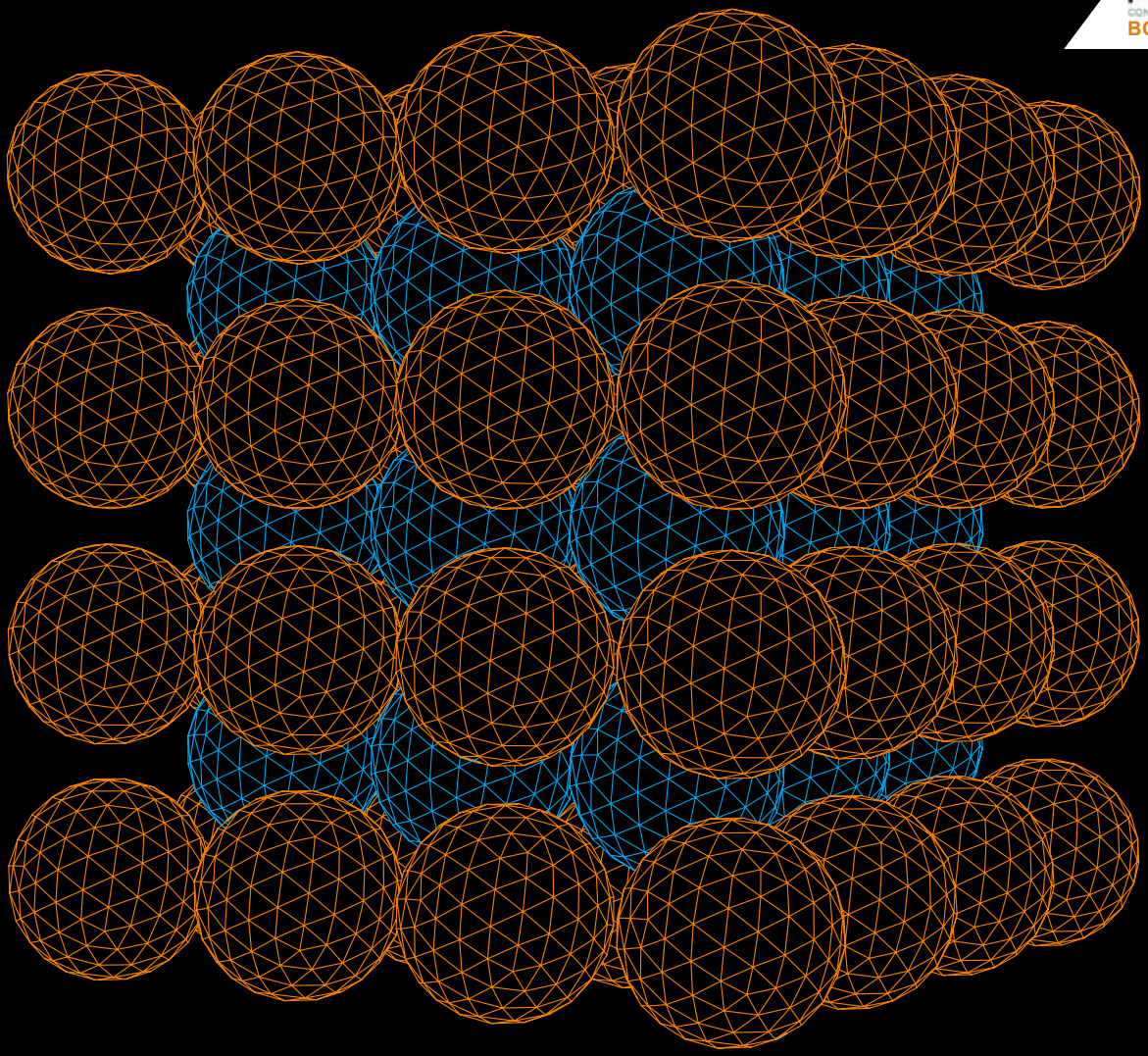

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International MIM producer ARC Group Worldwide announces Drew Kelley as new interim CEO

ARC Group Worldwide, Inc, Deland, Florida, USA, a key global provider of Metal Injection Moulding and metal Additive Manufacturing solutions, has announced the appointment of Drew M Kelley to Interim Chief Executive Officer and Board Member. Kelley replaces Jason T Young, who is leaving his position with the company and the board to pursue other interests.

Kelley has served as ARC's CFO since October 2013. Prior to joining the company, he was an investment banker and equity research analyst. "I appreciate the confidence the Board has placed in me and look forward to working with the entire ARC organisation as we establish and implement initiatives designed to improve operational efficiency, increase financial profitability and create a stronger balance sheet," he stated.

"Due to poor decisions and execution by the company over the past several months, ARC will evaluate all aspects of our business and consider, where appropriate, non-cash write-offs in order to put these matters behind us as we enter our new fiscal year this July. To that end, we have already initiated a robust review of our cost structure across all business units and have, with the board's approval, completed operational adjustments and other measures necessary to achieve these established objectives. Overall, I am confident this process will create a more fiscally disciplined company, while at the same time improving service to our customers and strengthening our leading role within the advanced manufacturing sector." Alan G Quasha, ARC's Chairman of the Board, commented, "We thank Mr Young for his service as CEO of ARC and wish him success as he pursues other opportunities. In connection with these leadership changes, the company will refocus its efforts to build upon its core capabilities of MIM and metal AM. I am confident that through these initiatives we can improve the Company's profitability and cash flow generation, as well as reduce our debt obligations to our targeted ratio of two times debt to EBITDA."

www.arcgroupworldwide.com

An MPIF award winning MIM part produced by ARC Group Worldwide (Courtesy MPIF)

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India's Phoenix Advanced Materials targets international growth

Phoenix Advanced Materials is an Indian producer of MIM and CIM components based in the city of Anand, in the western state of Gujarat. Established in 2012, the company today operates out of a 25,000 ft^2 facility and the company states that it has steadily been investing in both infrastructure and R&D. Ruchik Chandegra, CEO of Phoenix Advanced Materials, told *PIM International*, "MIM and CIM are our core manufacturing technologies and we have achieved considerable success providing high-quality products to customers. With state-of-the-art facilities and fully integrated technologies, we are now a one-stop resource for the conceptualisation, design and manufacture of complex precision components and sub-assemblies." Phoenix's customers include Tata Industries, Godrej & Boyce, Indian Defence Agencies and a number of other leading manufacturers in India. The company is, however, actively pursuing international markets. "In 2016, following a period of domestic growth, we began our sales expansion by venturing into the European market. Today, we have established business in Europe and plan to further expand our sales to North America and other international regions," stated Chandegra.

Phoenix uses two different feedstock systems, a proprietary polymer-based system and a feedstock from Germany's PolyMIM GmbH. "Our in-house polymer feedstock system is used for our CIM production, whilst we use the PolyMIM feedstock for MIM." The MIM division manufactures components primarily from low alloy steels, high alloy steels, copper, nickel and aluminium alloys. The CIM division manufactures components from a range of ceramic powders including pure alumina and zirconia, alumina-toughened zirconia, zirconia toughened alumina, aluminium nitride and silicon nitride. The company also offers high precision micro components as well as standard MIM and CIM components.

At present the company has a manufacturing capacity of 2 tons per month and has successfully crossed the annual sales mark of $1.5 million with a team of 75 employees. The company's MIM division has two debinding furnaces with two batch sintering furnaces. Six injection moulding machines are reported to be in operation, along with nine micro injection moulding machines. The CIM division also operates two debinding and two sintering furnaces. Chandegra stated, "We plan to invest further to expand manufacturing to twice the capacity by the end of 2018. We aim to be a reliable and preferred global solutions supplier and provider to customers across various segments of the precision engineering industry."

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Hagen Symposium to focus on 'Powder Metallurgy – Key to Mobility'

The 36th Hagen Symposium on Powder Metallurgy, to be organised by the Fachverband Pulvermetallurgie (FPM) in Hagen, Germany, November 30 - December 1, 2017, will discuss the use of PM as a key manufacturing technology in the automotive and aerospace sectors.

In addition to presentations on developments in sintered steels and fully dense powder forged components for conventional fuel-efficient vehicles with internal combustion engines, there will also be a focus on functional components for electric vehicles and developments in metal powder-based superalloys and light metal alloys used in the aerospace sector. The MIM of recycled NdFeB powder to produce permanent magnets will also be covered.

www.pulvermetallurgie.com

Smith Metal Products adds titanium MIM capabilities

Smith Metal Products, Center City, Minnesota, USA, has added titanium Metal Injection Moulding (TiMIM) capabilities to its portfolio of materials for Metal Injection Moulding. The company's existing materials portfolio includes stainless steels and ceramics.

TiMIM enables the production of complex titanium parts in a single operation and in higher volumes than possible by the conventional machining of titanium components. By adding TiMIM capabilities, Smith Metal Products states that it has opened up a new, wider range of component applications for the company, particularly for high strength-to-weight ratio parts. TiMIM part features include undercuts and varying wall thicknesses up to 3 mm (.125"). They also accept many surface finishes including anodising and electropolishing and can be finish machined if necessary.

"We are excited to have taken delivery of dedicated titanium Metal Injection Molding equipment," stated Todd Jensen, General Manager of Smith Metal Products. "Our Employee Owner-Operators are now trained on the equipment and process. We have received successful TiMIM moulding verification."

www.smithmetals.com

Smith's TiMIM capability will enable the company to produce complex titanium parts in a single operation (Courtesy Smith Metal Products)

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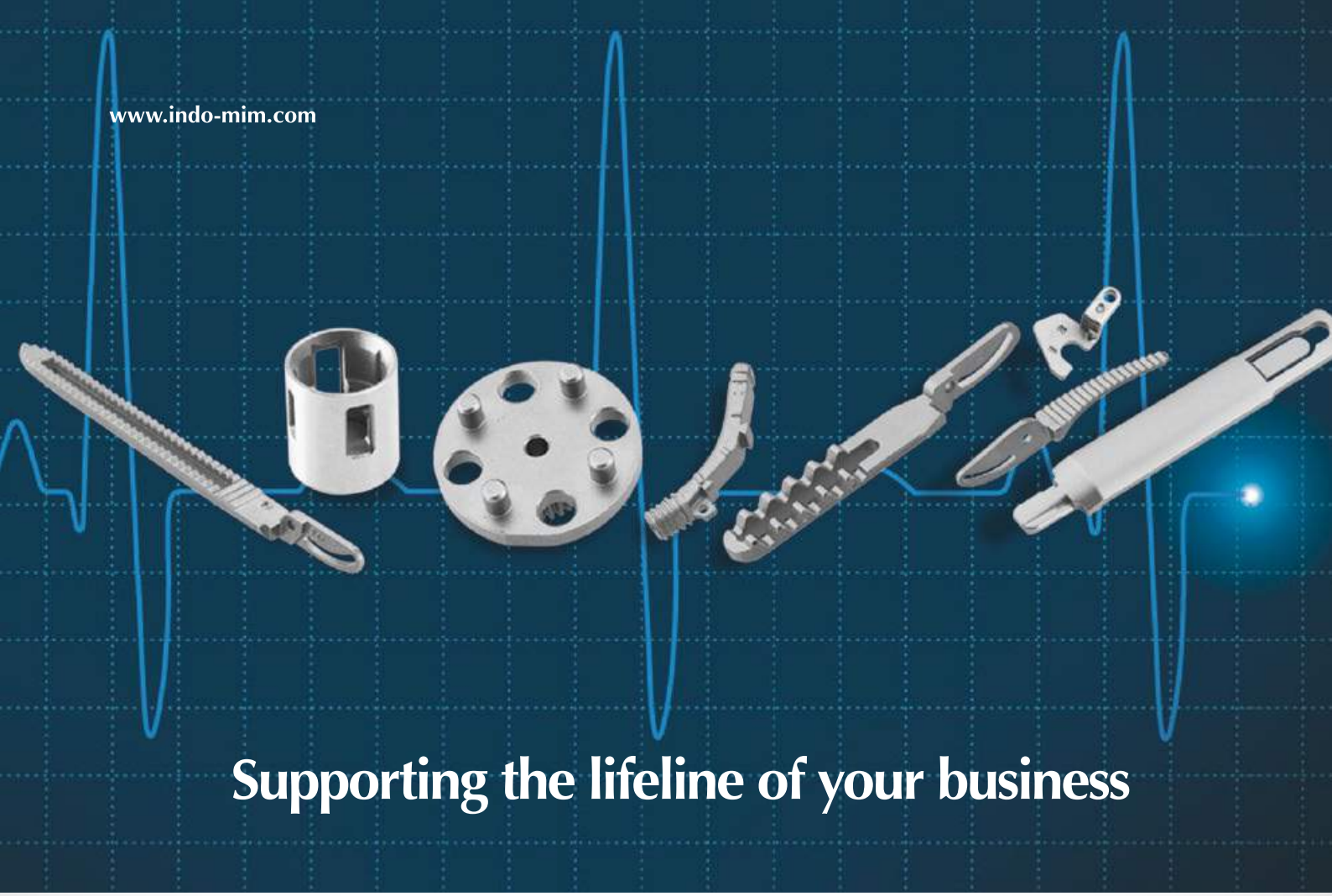
www.indo-mim.com



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Mikrotechnik HIRT and Wittmann Battenfeld develop 6-axis cell for MIM micro parts

Mikrotechnik HIRT (MTH), Schramberg, Germany, is becoming increasingly well established as a specialist in small components, micro parts and hybrid parts by injection moulding. To produce these parts, which are primarily made from metal or plastic, the company works closely with Wittmann Battenfeld, a leader in the manufacture of injection moulding machines, robots and peripheral equipment.

Most recently, the two companies collaborated on the installation of a MicroPower 15/10 machine from Wittmann Battenfeld with 150 kN clamping force and designed specifically for the production of micro parts by plastic and Metal Injection Moulding. MTH and Wittmann Battenfeld worked together to develop the machine, originally a 5-axis injection moulding machine, into a 6-axis production cell. According to MTH, this modification has enabled parts with unscrewing functions, helical micro cogwheels and shafts with complex profiles to be manufactured to the highest precision on one machine.

The machine features a two-step screw-and-plunger injection unit with shot volumes ranging from 0.05-4 cm^3. A thermally homogeneous melt is injected via this system, enabling the production of high-quality parts with an extremely stable manufacturing process and short cycle times.

Following mould opening, the cavity element inside the mould is driven by a toothed belt that has been installed on the side of the mould to release the moulded part for ejection. The ejector then demoulds the part using a servo-electric drive, ready for a new cycle to begin as soon as the contour element is returned to its original position.

The unscrewing unit's operation is integrated entirely into the machine's UNILOG B6 control system, making it very easy for the user to address any conceivable unscrewing position with high precision. This makes

Franz Hirt, MTH, Wolfgang Straubinger and Martin Philipp-Pichler, Wittmann Battenfeld, in front of the MicroPower 15/10 (Courtesy Wittman Battenfeld)

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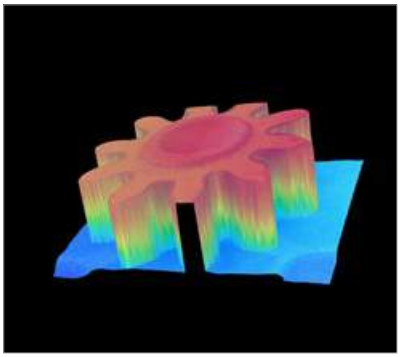
it possible, for example, to drive thread cores inside the mould in order to produce high-precision internal threads.

Franz Hirt, owner and Manager of MTH, has over forty years of injection moulding and stamping experience and designs Microtechnik's moulds in-house. Hirt's mould components have to be produced to a precision of +/- 5 µm, presenting a challenge in terms of manufacturing. According to Hirt, only few mould manufacturers are able to fulfil the stringent requirements imposed by his designs, with each of the suppliers selected using 100 µm tools for cutting. Even when suppliers are able to meet MTH's requirements, the initial success rate for new moulds is reported to be no more than 70%.

As a result of the stringent part specifications, the manufacture of the parts developed at MTH is only possible with certain injection moulding machines and the company believes that it has found a perfect partner through its collaboration with Wittmann Battenfeld. The company is now working on the development of micro thermoset processing in collaboration with a number of educational institutes and technology centres.

Drain tube compared with other objects to illustrate its size (Courtesy Microtechnik HIRT)

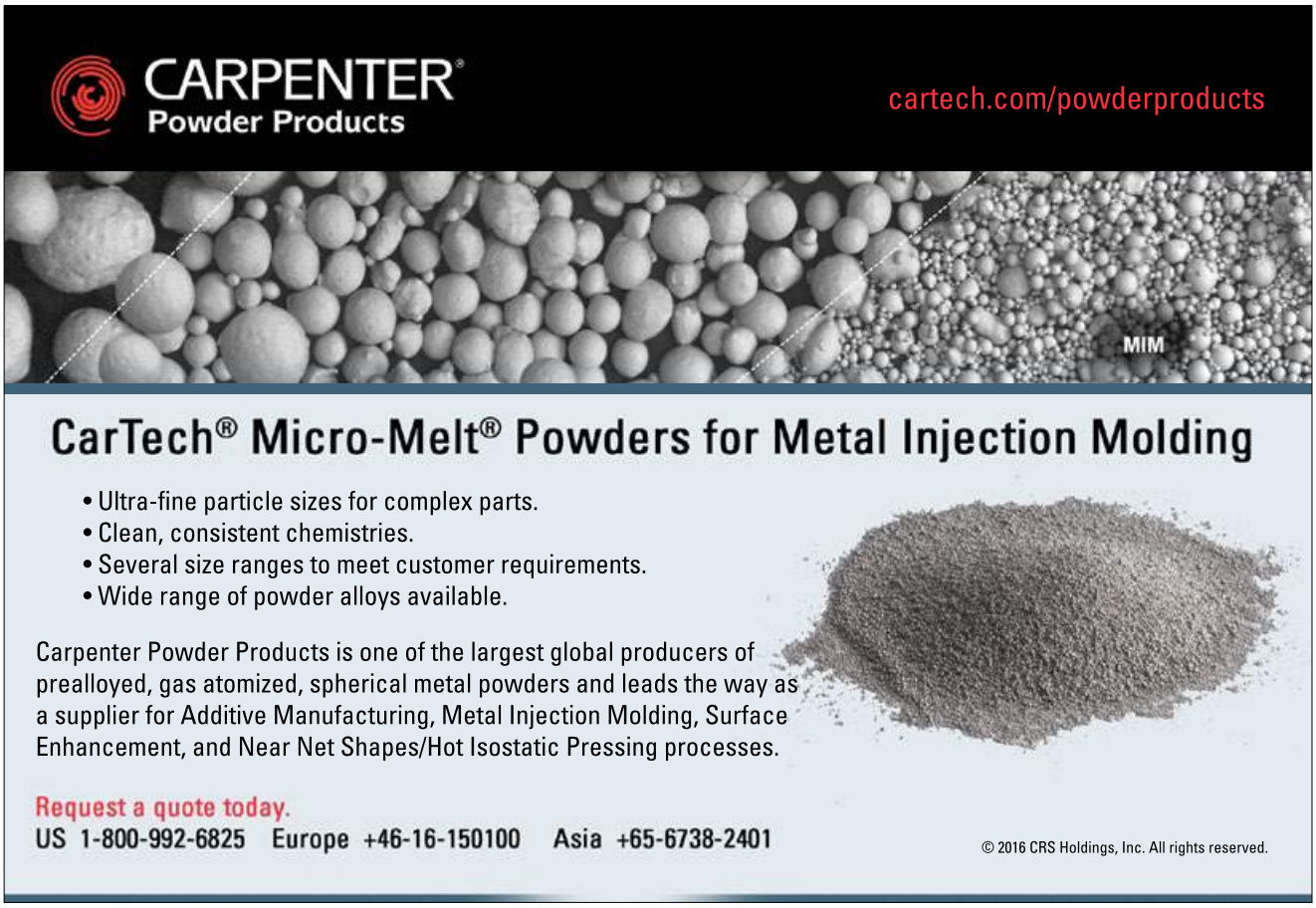


3D scan of a micro component (Courtesy Mikrotechnik HIRT)

MTH has stated that it is on a course of expansion and will continue to develop as a specialist in its field through the development of projects in injection moulding, including Metal Injection Moulding, which will provide micro parts for a number of industries.

The continuing trend toward miniaturisation and nanotechnology suggests that the market for micro parts will continue to grow, especially in the medical, electronic, communications, satellite and automotive markets.

www.mt-hirt.com
www.wittmann-group.com



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Ipsen reports strong furnace demand and announces management changes

Ipsen USA, Cherry Valley, Illinois, USA, reports that it shipped fifteen atmosphere and vacuum furnaces to global customers in the second quarter 2017. The company delivered its equipment to China, Hong Kong, India, Japan, Saudi Arabia and the US. Vacuum furnaces for the MIM industry are included in this number.

The company has announced several strategic organisational changes aimed at strengthening its position as a leading global provider of integrated heat treatment and sintering solutions. As part of the planned changes, Geoffrey Somary, current CEO of Ipsen USA and COO of Ipsen Group, will leave his USA position and focus solely on his position at the head of the group.

"We are in an exciting period of growth for Ipsen, which has been built on both innovation and exceeding customer expectations," commented Somary. "The Ipsen Group focus ensures that we continue to strengthen Ipsen performance in all global markets, delivering the same quality and performance anywhere in the world."

Jake Hamid will also leave his position as COO of Ipsen USA, moving into the position of Director of Global Product Development and Manufacturing for Ipsen Group, while Patrick McKenna will be promoted to President & CEO of Ipsen USA, taking responsibility for all US entity functions including Sales, Engineering, Operations, Service, Finance and Human Resources.

McKenna commented, "My focus from day one will be on delivering the highest quality products and services to our customers and ensuring that the established Ipsen performance culture continues far into the future."

www.ipsenusa.com

Titanium USA 2017 technical programme published

The technical programme for Titanium USA 2017, organised by the International Titanium Association (ITA), has been published and lists a wide range of topics including sessions on metal powder based-processes.

Taking place in Miami, Florida, USA, from October 8-11, 2017, the event aims to offer visitors insight into the current state of the industry as well as networking opportunities for titanium producers, OEMs, distributors, fabricators and vendors who offer products and services to the titanium community.

Over the past decade, attendance at the Titanium Conference has doubled, drawing delegates from more than thirty countries. Alongside the four day conference will be an exhibition featuring almost 100 international exhibitors.

www.titanium.org

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MIM producer Thortex acquired by Avalign Technologies

Avalign Technologies, headquartered in Bannockburn, Illinois, USA, and a portfolio company of Arlington Capital Partners, has announced that it has completed the acquisition of Metal Injection Moulding producer Thortex.

Based in Portland, Oregon, USA, Thortex is a leading provider of proprietary porous coatings and metal injection moulded components for the orthopaedic and aerospace markets, as well as precision manufacturing solutions for the medical device market. It is one of the few companies in the world that is able to provide porous coating solutions for titanium and cobalt-chrome orthopaedic implants.

Thortex provides a full suite of manufacturing and engineering technologies that, in addition to Metal Injection Moulding, include CNC machining, finishing and assembly to medical device OEMs.

Avalign Technologies also announced the acquisition of Millennium Surgical, Narberth, Pennsylvania, USA. Millennium is a provider of specialty surgical instruments which is focused on delivering difficult-to-find instruments through innovative e-cataloguing, web-based marketing and technical sales support. The company offers more than 17,000 branded SKUs across a variety of clinical areas including neurosurgery, ophthalmics and orthopaedics to a diverse array of hospital and ambulatory surgical centre customers.

Matt Altman, a Managing Partner at Arlington Capital, stated, "The incorporation of Thortex and Millennium into the Avalign platform adds new and proprietary capabilities which enhance Avalign's strategic position in the marketplace. The increased scale and breadth of services and technologies provided by these acquisitions will accrue to the benefit of Avalign's customers and bolster Avalign's already impressive growth."

"Thortex and Millennium nicely complement and expand the manufacturing technologies and services that Avalign offers and provide us with the ability to continue gaining share with our customers," stated Forrest Whittaker, CEO of Avalign Technologies. "We are excited to welcome the Thortex and Millennium teams to the Avalign family and thankful for Arlington's support in consummating these highly strategic acquisitions."

Malcolm Little, a Principal at Arlington Capital, added, "The proprietary technologies developed by both Thortex and Millennium represent truly unique offerings in the market. We are pleased to complete these acquisitions and continue to pursue additional opportunities to strategically scale our business."

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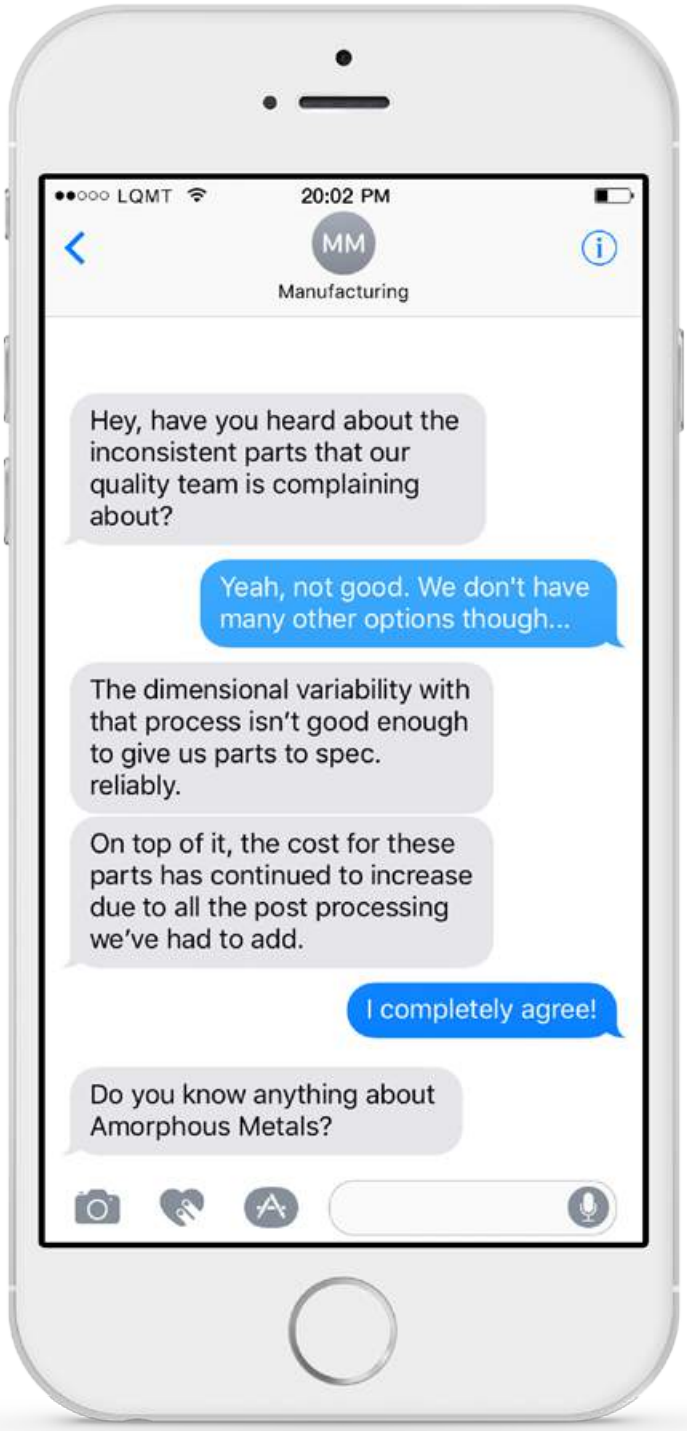

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Ceramitec 2018 to host 93rd Meeting of DKG & Symposium on High Performance Ceramics

The 93rd Annual Meeting of the German Ceramic Society (DKG) & Symposium on High Performance Ceramics will run parallel to ceramitec 2018, April 10-13, 2018, Munich, Germany.

The symposium is targeted at experts from the ceramics industry as well as those in the areas of teaching and research and development. Young scientists, students and doctoral students, in particular, are invited to present results from their qualifying papers and current projects. Ceramic Injection Moulding and Powder Injection Moulding are expected to feature strongly in the programme.

Speakers will have the opportunity to be entered into the Walter Hennecke Lecture Competition at the Exhibition Forum. This offers a valuable chance for young professionals of the ceramics sector to present themselves to a wide international trade audience.

One of the core topics of the symposium will be digitalisation and Industry 4.0. A number of international, highly-qualified speakers will discuss the changes and challenges that the ceramics industry can expect to face in the coming decades, such as changing component manufacture, the tracking of material life cycles to optimise production processes and minimise raw material consumption, and the necessary reduction of power consumption.

Marion Lintl, ceramitec's Exhibition Manager, responsible within Messe München for the DKG Meeting, stated, "The DKG Annual Meeting & Symposium has been held simultaneously with ceramitec for quite some time now. We are expecting some 300 participants. We are very much looking forward to it because this means that the entire ceramics family will get together under the roof of Messe München. Thus, we can offer young talents an international platform for presenting the results of their research, discovering new trends and having in-depth discussions with experts."

Submissions for the 93rd Meeting of DKG & Symposium on High Performance Ceramics will close on December 31, 2017.

www.ceramitec.com

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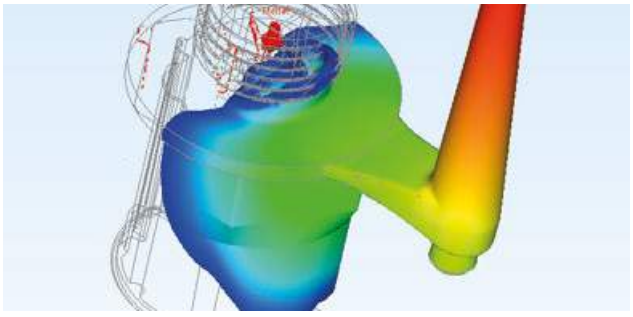

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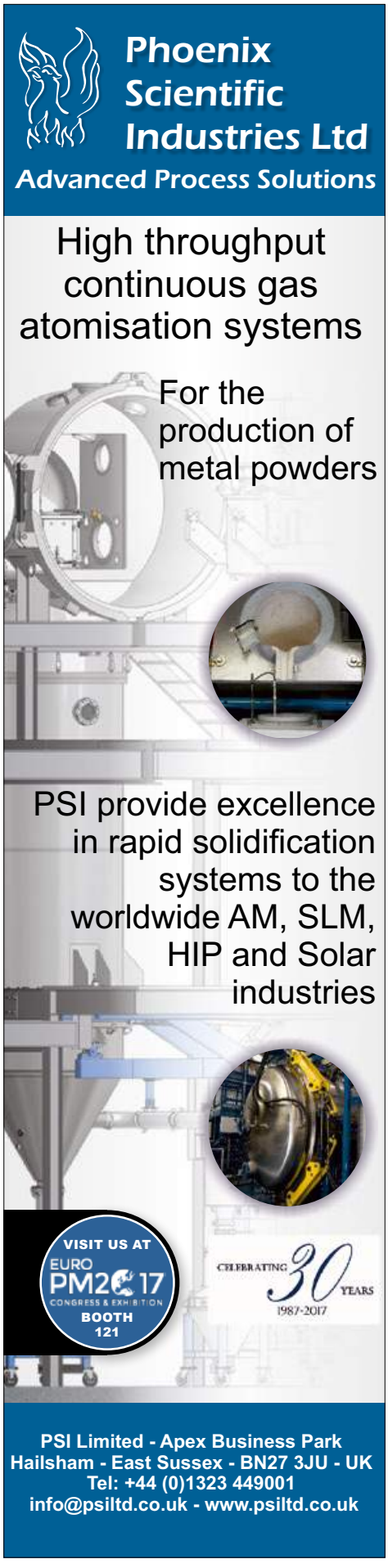

Carpenter Technology appoints Tom Houck as Plant Manager

Carpenter Technology Corporation, Philadelphia, Pennsylvania, USA, has appointed Tom Houck as Plant Manager of its new superalloy powder plant in Athens, Alabama, USA.

Houck brings over twenty years of experience in the MIM and PM industry, including operational expertise in lean manufacturing. In addition to his role at Carpenter, he serves as President of the Metal Injection Moulding Association (MIMA) and is a member of the MPIF's Board of Governors.

Prior to joining Carpenter, Houck spent seven years as Vice President of leading MIM specialist ARC Group Worldwide and eight years as Director of Operations US/Asia at Metaldyne. Carpenter Technology Corporation is a key producer and distributor of premium speciality alloys, including titanium alloys, powder metals, stainless steels, alloy steels and tool steels.

Carpenter recently announced its financial results for the fourth quarter and fiscal year ended June 30, 2017. Fiscal year 2017 net sales were $1,797.6 million, down slightly from $1,813.4 million reported in 2016. However, total net income for the year was reported at $47 million, up from the $11.3 million total for 2016.

www.cartech.com

formnext 2017: 100 new exhibitors from 22 countries announced, conference expanded

Now in its third year, 'formnext powered by tct' looks poised to continue its impressive growth path. Taking place from November 14-17, in Frankfurt, Germany, more than 20,000 m^2 of exhibition space has been booked by more than 290 exhibitors. formnext 2017 will for the first time occupy two levels of Messe Frankfurt's Hall 3. The organisers stated that industry leaders are positioned at strategic locations on both levels to ensure a balanced flow of visitors.

"In an exciting and dynamic market, formnext continues to grow apace and underscore its status as the leading international conference and exhibition for Additive Manufacturing and the next generation of intelligent production solutions," stated Sascha F Wenzler, Head of Division for formnext at event organiser Mesago Messe Frankfurt GmbH.

One of the pillars supporting the growth of formnext 2017 is its large number of new exhibitors. This year, a hundred companies from twenty-two countries have registered to exhibit at formnext for the first time. Leading the way are those from Germany, China, Austria, France, the Netherlands and the United States. A large number of metal powder suppliers are exhibiting, along with metal-powder based parts makers and AM technology providers.

Conference expands

The parallel formnext powered by tct conference brings together leading industry minds on each day of the exhibition. For the first time this year, it will cover the latest developments in Additive Manufacturing in two parallel sessions. The sectors covered will include aerospace, healthcare, automotive, heavy industry and the tool making sector.

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Taisei Kogyo highlights nanoscale porous MIM products and improved surface finishes

Japanese Metal Injection Moulding specialist Taisei Kogyo Co. Ltd., based in Osaka, has recently highlighted two product application areas that rely on some unique features of the company's proprietary μ-MIM technology.

The first of these application areas relates to the development of nanoscale porous metal products. Specific surface area is an essential design parameter, for example in electrode or cooling parts, since specific surface area per unit volume affects performance. High specific surface area is a feature of porous metal.

Taisei Kogyo has carried out joint research with Tohoku University Advanced Institute for Materials Research (AIMR) and has established a mass production process for bimodal porous metals with a pore structure consisting of micrometre and nanometre scale pores (Fig. 1). Using this process, a specific surface area of 100 m^2/g has been achieved.

The second application area relates to achieving fine surface finishes in μ-MIM parts. A satin or matte finish is generally considered to be the standard finish of MIM product surfaces; however, if smaller or finer metal powders are used, smoother surfaces can be realised.

According to the company, feedstock preparation, injection moulding and sintering deformation control are all made more challenging with finer metal powders. Despite these considerations, the company has successfully developed its μ-MIM technology to utilise single-μm or sub-μm metal powder in mass production. Using this process, the surface finish parameters achieved can be as low as Ra=1 μm and Rz=7 μm.

www.taisei-kogyo.com

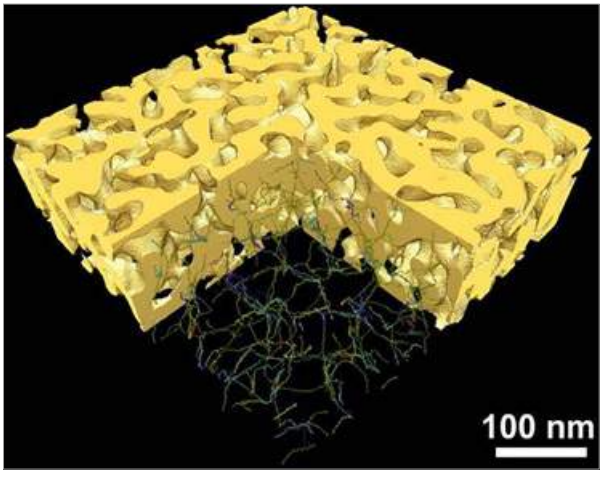

Fig. 1 Image of bimodal porous metal having specific surface area of 100 m^2/g

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Arburg invests in new Lossburg training centre

Arburg GmbH & Co KG celebrated the start of groundwork for its new Training Centre in Lossburg, Germany, this summer, with a foundation stone-laying ceremony on July 13. The new multi-storey building will provide 13,700 m^2 of usable space and represents an investment of several tens of millions of euros for the company. This will bring the total usable space at the Lossburg site to 180,000 m^2.

Prior to the foundation stone-laying ceremony, Managing Partner Michael Hehl, responsible for plant development, commented on the significance of this additional new building, stating, "This Training Centre is a clear sign of our commitment to Lossburg as a production location and demonstrates our long-term, goal-oriented strategy." The centre is just the latest addition at Arburg. Since 2007 it has also built a customer centre, an assembly hall, multi-storey car park and trade-fair logistics hall.

The construction of a multi-storey Training Centre means that existing and prospective Arburg customers will have an even better environment in which to learn and train. Explaining the importance of the project, Hehl stated, "In addition to our high-quality machines, which are used throughout the world, our customers traditionally value our first-class services. This also includes our broad range of training courses relating to machines, application technology and service. We have trained tens of thousands of specialists over the past decades - and demand continues to grow."

The ground floor of the new building will offer space for around fifteen Allrounder injection moulding machines for practical training. Twelve rooms will be available on the first floor for training customers on theoretical points. The next two floors will be open plan offices for administrative staff. Finally, the fourth floor will house Arburg's company health management services. A new staff entrance is also being built.

In architectural terms, the Training Centre will resemble the Customer Centre inaugurated in 2009. In addition to functionality and aesthetics, environmental protection and careful management of resources and energy will be a focus. In this context, double-glazed facades meet with the latest requirements of the EnEV energy-saving regulations and tried and tested energy saving concepts are used for building air temperature control. Surplus heat from production is used to keep the outdoor areas in front of the building ice-free.

www.arburg.com

Artist's impression: the new Arburg Training Centre (Photo: ARBURG / Schmelzle + Partner)

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MPIF award winning parts highlight MIM's growth

The MIM industry once again accounted for a significant proportion of the winning parts in the Metal Powder Industry Federation's (MPIF) 2017 Powder Metallurgy Design Excellence Awards competition. The winners were announced during the POWDERMET2017 International Conference on Powder Metallurgy and Particulate Materials, Las Vegas, USA, June 13-16, 2017. These parts demonstrate MIM's ability to deliver complex, high-performance and cost-saving solutions for end-users in a diverse range of end-user sectors, from automotive and aerospace to medical devices and firearms.

Fig. 1 A four-slot MIM valve seat made for Delphi

Fig. 2 A 17-4 PH canard made for UTC Aerospace Systems and Raytheon Company

Fig. 3 Components used in a Grohe shower system

Grand Prize Winners

Automotive/Engine: Phillips-Medisize

The Grand Prize in the Automotive-Engine category went to Phillips-Medisize, Menomonie, Wisconsin, USA, for a four-slot fuel valve seat made for Delphi. The MIM part goes into the Multec3.5 compressed natural gas (CNG) fuel injector that satisfies the market's need for a low-cost, low-pressure port fuel injector. It is currently used by several small-engine and automotive applications, including aftermarket CNG conversions for trucks and cars, helping contribute to a reduction in greenhouse-gas emissions.

Aerospace/Military: Dynacast Portland

The Grand Prize in the Aerospace/Military category was won by Dynacast Portland, Wilsonville, Oregon, USA, for a 17-4 PH canard made for UTC Aerospace Systems and Raytheon Company. The stainless steel part is used on the Talon, an add-on guidance and control package that transforms a legacy 2.75-inch Hydra-70 unguided rocket into a low-cost, precision-guided weapon. Three canards on each Talon act as the primary flight control surfaces. The MIM canard underwent a stringent qualification process.

Hardware/Appliance: Indo-MIM Pvt. Ltd

The Grand Prize in the Hardware/Appliance category was awarded to Indo-MIM Pvt. Ltd, India, for three MIM parts; an upper stop ring, a stop ring and a stop sleeve. The parts are for Grohe, Germany and go into the valve of a bath shower temperature controller unit. Made of 316L stainless steel, all three complex parts are fabricated close to net shape and special ceramic setters are employed for enhanced shape retention during sintering.

Medical/Dental: ARC Group Worldwide

The Grand Prize in the Medical/Dental category went to ARC Group Worldwide, Longmont, Colorado, USA, for a MIM surgical keel punch made for Paragon Medical. The part functions as a broach to remove bone during knee surgery. Made from 17-4 PH stainless steel, the part is moulded and sintered to net shape with no additional coining, machining or other post-processing to alter its shape.

Fig. 4 MIM surgical knee device components

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Fig. 5 MIM shock absorber components used in the Chevrolet Camaro sports car

Fig. 6 A MIM 4140 low-alloy steel latch made for Sig Sauer Inc

Awards of Distinction

Automotive/Chassis: Indo-MIM Pvt. Ltd
The Award of Distinction in the Automotive-Chassis category went to Indo-MIM Pvt. Ltd. for a MIM-4605 low-alloy steel top plate and check shim stop made for its customer Multimatic Dynamic Suspensions. The mating parts go into shock absorbers on the Chevrolet Camaro sports car. The Metal Injection Moulded design provided increased repeatability and accuracy, providing an estimated 25% cost savings.

Aerospace/Military: ARC Group Worldwide
An Award of Distinction in the Aerospace/Military category was presented to US MIM producer ARC Group Worldwide for a 4140 low-alloy steel latch made for Sig Sauer Inc., Newington, New Hampshire. The internal latch drives a subassembly for the telescoping feature of the collapsible stock on MCX and MPX rifles. The part was specifically designed for Metal Injection Moulding as it could not be economically made using any other fabrication method.

continued overleaf...

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Fig. 7 MIM parts used on an MDR rifle made by Deserttech

Hand Tools/Recreation: Indo-MIM Pvt. Ltd

An Award of Distinction in the Hand Tools/Recreation category went to Indo-MIM Pvt. Ltd. for a set of Metal Injection Moulded parts, including a barrel block, gas block, bolt catch, 7.62 NATO mag conversion bar, ejector retainer and extractor link. These are used in an MDR rifle made by Deserttech, Salt Lake City, Utah, USA. The MIM-designed parts replaced ones that were machined and reduced the cost by 30% while manufacturing lead-time was cut in half.

www.mpif.org

Call for Papers issued for POWDERMET2018 San Antonio

The Metal Powder Industries Federation (MPIF) has issued a Call for Papers for POWDERMET2018, the International Conference on Powder Metallurgy and Particulate Materials, taking place June 17-20, 2018 in San Antonio, USA. Both oral and poster presentations are requested for inclusion in the technical programme. The deadline for submission of abstracts is November 3, 2017.

The MPIF states that all submissions should be original and unpublished work addressing recent advances in the full spectrum of PM and materials technologies. In addition to the regular technical sessions and posters, several special interest programmes are being organised. Presentations in these programmes are intended for invited presentations only. POWDERMET2018 will include an international exhibition focused on the PM, PIM, particulate materials and metal AM industries.

www.powdermet2018.org

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PyroGenesis develops new plasma atomisation process for MIM cut powders

PyroGenesis Additive, a division of PyroGenesis Canada Inc., Montreal, Canada, reports that it has developed a new plasma-based process to produce metal powders which will enable MIM cut powder production at higher volumes. According to the company, the new process may have a greater impact on the powder production market than its original plasma atomisation technology, developed in 2001.

The company stated that whilst 'MIM cut' is a particularly small metal powder size traditionally used for Metal Injection Moulding and usually features particle sizes between 5-20 microns, it has in recent years become increasingly used in Binder Jet Additive Manufacturing systems. Among those companies using MIM cut powders for metal AM are Desktop Metal and ExOne. These processes are often referred to as 'MIM-like'.

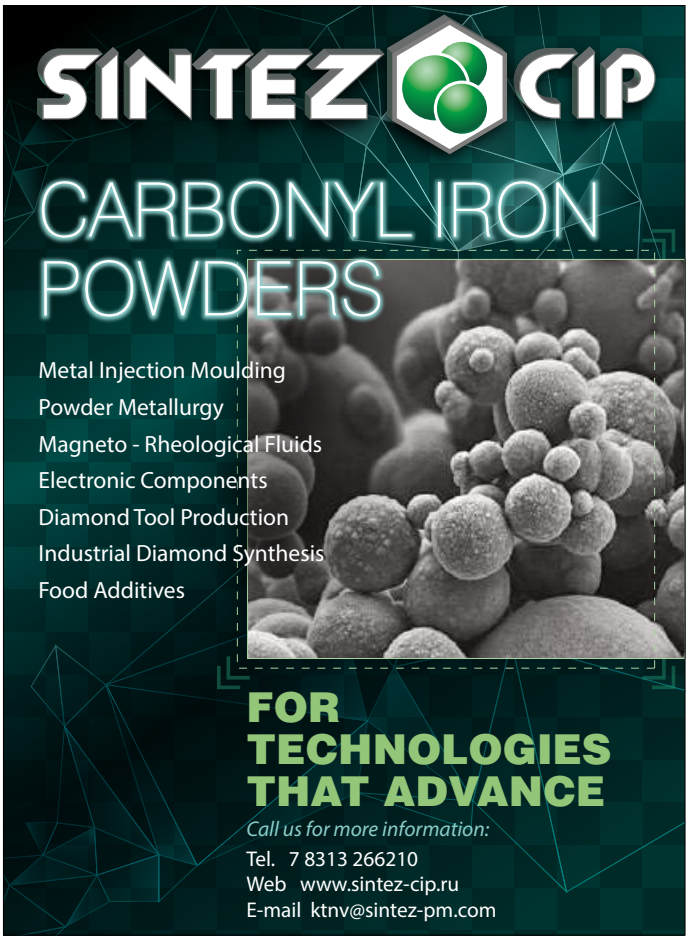
PyroGenesis had previously produced powders for Electron Beam Melting (EBM) Additive Manufacturing and Laser Sintering, while MIM cut has until recently been considered an undesirable by-product of the company's standard plasma atomisation process, which produces powders in the 15-106 micron range.

Peter Pascali, President and CEO of PyroGenesis, stated, "Several months ago, the company was approached by a number of companies who were interested in MIM cut titanium powder, whereby it became apparent to us that the appetite for this ultra-fine powder was significant. As a result, we decided to make adjustments to our plasma atomisation technology in order to try and shift the particle size distribution towards the low end of the spectrum and produce powders in the range required."

According to Pascali, the new plasma atomisation process gives the company significant control over powder sizes produced and offers higher powder production rates at lower cost. "MIM cut powders can now be produced in very large quantities with little-to-no waste," he stated, "thereby growing with and enabling those requiring ultra-fine powder and meeting their strategic growth needs. We believe this breakthrough is, if not more significant, then at least as significant as our original plasma atomisation technology," he added. "We believe we have not even scraped the surface of what this new process can do with regard to production rates and powder quality."

According to Pascali, PyroGenesis Additive now produces MIM cut titanium powder Grade 5, the grade currently requested for use in Binder Jet AM. The new process also has the potential to produce MIM cut titanium powder at Grade 23, the highest grade titanium powder, he reported, though this is not yet confirmed.

www.pyrogenesis.com



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Reducing the embrittlement effect of binder contamination in MIM Ti Alloys

During the Metal Injection Moulding process, titanium tends to react with carbon from the polymeric binder and carbides can be formed if the carbon solubility of the alloy is exceeded, leading to embrittlement. This is a critical issue for β-titanium alloys, where β-phase stabilising elements such as V, Mo and Nb decrease the carbon solubility of the Ti-matrix. To overcome this issue, Thomas Ebel and colleagues at Helmholtz-Zentrum Geesthacht, Germany, Montanuniversität Leoben, Austria, Southwest Petroleum University and Hunan University, China, have studied a number of different approaches to limit carbide precipitation in a Ti-22wt.%Nb alloy processed by MIM. The results of their research to date were published in *Powder Metallurgy* (Vol. 60, No. 3, July 2017, 157-166).

The authors stated that the uptake of carbon in Ti-Nb alloys is influenced by the type and percentage of binder as well as by processing conditions such as sweep gas flow, sintering set-up and the furnace actually used. Their experience shows that even the total number and size of the parts being processed and possible contamination of the furnace through previous runs appear to affect the final contamination. Thus, they investigated four different approaches to reduce carbide precipitation and embrittlement of the Ti alloy. These included:

1. Performing heat treatments after sintering
2. Adding potentially carbon-binding elements to form other and smaller carbides than Ti_2C
3. Adding grain refiners to form smaller and more distributed carbides in the microstructure
4. Using alloy modification to increase carbon solubility.

Five types of elemental metal powder mixtures were used to produce the following compositions (all in wt.%):

- Ti–22Nb
- Ti–22Nb–10Zr
- Ti–22Nb–0.5B
- Ti–22Nb–10Zr–0.3B
- Ti–22Nb–10Zr–0.5B

Two feedstocks, containing 10 wt.% and 8 wt.% respectively of a binder consisting of 35 wt.% polyethylene vinyl acetate, 60 wt.% paraffin wax and 5 wt.% stearic acid, were used to produce dog bone shaped tensile test specimens and also cylindrical specimens for microstructure analysis. The paraffin wax binder in the injection moulded parts was removed by solvent debinding

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in hexane at 40°C for 900 min, and thermal debinding was used to remove the remaining binder. Sintering was done in high vacuum at 1500°C for 4 h. The authors reported on the following:

- Effect of different heat treatments on as sintered Ti–22Nb samples.
- Ti–22Nb–10Zr samples were checked by TEM and EDS to establish if other carbides than Ti_2C can be found.
- Effect of boron addition on grain refinement and carbide reduction in Ti–22Nb–0.5B.
- XRD measurements were performed to investigate a possible induced change in the lattice parameter of the titanium matrix by the addition of Zr. This study was performed on Ti–22Nb and Ti–22Nb–10Zr specimens.
- Combinations of adding boron and zirconium to form the compositions Ti–22Nb–10Zr–0.3B and Ti–22Nb–10Zr–0.5B and the effect of applying heat treatments on both of these alloys.

Table 1 lists the grain size, porosity, carbide area fraction and total carbon content for each of the titanium alloys, and Fig. 1 shows two micrographs of the two boron-containing alloys, revealing that only few carbides remain visible when compared to Ti-22Nb. However, the agglomeration of borides and the rather high porosity prove that further improvement in the manufacturing process is necessary. It can be assumed that, for example, using pre-alloyed powder will lead to significantly more homogeneous microstructures and higher sintered density.

The authors concluded by stating that both heat treatment and alloy modifications can be used to reduce the amount of carbides in titanium alloys, especially β-titanium alloys. They proposed a ranking of their results as follows:

- Quenching or fast cooling is the most effective way for carbide reduction; however, it is difficult to realise for commercial products.
- Heat treatment around 650°C is effective in resolving carbides formed during cooling and can be performed after sintering in large batches of MIM samples.
- Addition of Zr increases the carbon solubility and lowers the carbide precipitation starting temperature, which reduces the amount and size of carbides. Furthermore, strength is increased and grain size reduced.
- Grain refinement by boron addition has a significant effect on carbide formation, especially in combination with Zr addition.
- Trials to bind carbon to other elements were not as yet successful.

Through combinations of these methods, a typical value of 3.3% carbides can be reduced to 0.7% using a heat-treated Ti–22Nb–10Zr–0.5B alloy. However, the effect on mechanical properties has to be investigated in future studies.

www.tandfonline.com/loi/ypom20

	Ti–22Nb	Ti–22Nb–10Zr	Ti–22Nb–10Zr–0.3B	Ti–22Nb–10Zr–0.5B
Grain size (μm)	284 ± 148	234 ± 109	168 ± 78	146 ± 65
Porosity (%)	4.72 ± 0.11	5.90 ± 0.26	7.87 ± 0.08	8.39 ± 0.24
Carbides (area%)	3.34	2.58	0.86	0.84
Carbon (wt.%)	0.18	0.16	0.06	0.08

Table 1 Results of grain size, porosity, carbide area fraction and total carbon content measurements on Ti-22Nb containing Zr And B additions. (From paper by T Ebel et al, Powder Metallurgy, Vol. 60, No. 3, 2017, 157-166)

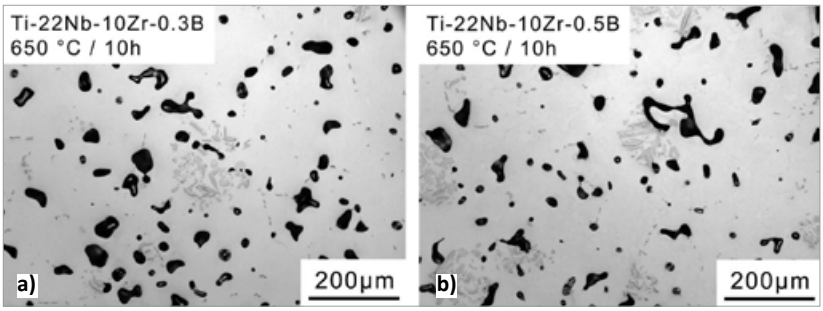

Fig. 1 Microstructures of heat-treated (650°C/10 h) Zr- and B-containing specimens; (a) Ti–22Nb–10Zr-0.3B and (b) Ti–22Nb–10Zr–0.5B. Agglomerations of borides and small carbides, mainly at the grain boundaries, are visible. (From paper by T Ebel et al, Powder Metallurgy, Vol. 60, No. 3, 2017, 157-166)

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BASF develops tungsten heavy metal feedstock for MIM

BASF SE, Ludwigshafen, Germany, has reported on the development of a Catamold® tungsten heavy metal feedstock capable of being processed on an industrial scale and having the essential requirements of 18,000 kg/m^3 density or higher and non-magnetic behaviour. Oliver Löber and colleagues presented the results of their development work at the 19th Plansee Seminar held in Reutte, Tirol, Austria, May 29 - June 2, 2017.

Löber stated that the challenges posed in the development and production of the tungsten heavy metal alloy feedstock system included the sustainable attainment of the specific sintered density as well as being able to use BASF's well established catalytic debinding process, designated Catamold, for mass production of MIM heavy metal components. Key to the successful injection moulding of Catamold feedstocks is the flowability, commonly described in the Melt Flow Index (MFI). The MFI describes the amount of material flowing through a defined nozzle over a fixed period of time. The optimised feedstock also needs to allow even the finest sections and structures to be moulded whilst meeting the necessary shrinkage characteristics. Löber stated that shrinkage is described by the so-called Oversizing Factor which the toolmaker has to take into account in order to meet the final part dimensions after processing.

	Sintered Density kg/m³	Oversizing Factor	Melt Flow Index g/10 min
Min.	18,000	1.2436	800
Max.	18,300	1.2524	2400

Table 1 Target feedstock characteristics for tungsten heavy alloy developed by BASF. (From paper by O Löber et al, 'Development and industrialisation of a tungsten heavy alloy feedstock for MIM based on BASF's Catamold technology', published in Proceedings of 19th Plansee Seminar, 2017)

	W, %	Ni, %	Cu, %	Co, %
Min.	95.4	2.6	0.4	0.4
Max.	96.6	3.0	0.7	0.7

Table 2 Composition of the tungsten heavy alloy feedstock developed for MIM (From paper by O Löber et al, 'Development and industrialisation of a tungsten heavy alloy feedstock for MIM based on BASF's Catamold technology', published in Proceedings of 19th Plansee Seminar, 2017)

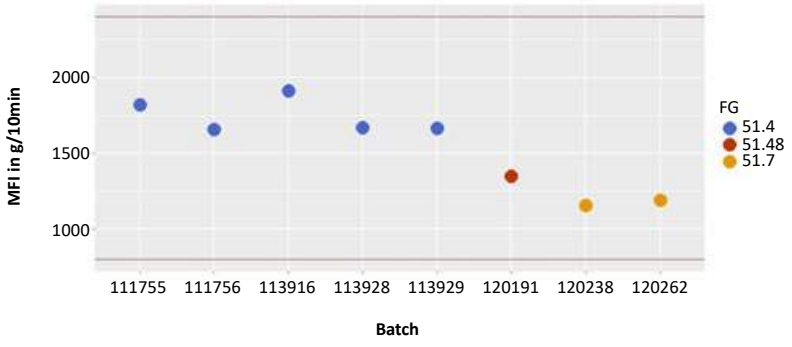

Fig. 1 Influence of powder loading of tungsten heavy alloy powder on melt flow index (From paper by O Löber et al, 'Development and industrialisation of a tungsten heavy alloy feedstock for MIM based on BASF's Catamold technology', published in Proceedings of 19th Plansee Seminar, 2017)

Table 1 shows the target characteristics of the tungsten heavy alloy feedstock for MIM applications, and Table 2 shows the composition of the tungsten heavy alloy using commercially available metal powders. Particle sizes (d50) used was 5.7 µm for tungsten, 10.4 µm for nickel and 15 µm for copper. The small addition of copper was found to make the alloy sufficiently non-magnetic.

To prepare the feedstock Löber stated that the metal powders were mixed with BASF's standard polyoxymethylene (POM) based binder, which has the proven ability for fast, catalytic debinding of the moulded parts. Tungsten heavy alloy powder loading was 51.4 to 51.7 vol.% and Fig. 1 shows the dramatic drop in flowability with an increase in powder loading of just 0.3 vol.%. One of the key features of the Catamold feedstock is its rapid debinding speed compared with wax-based binder feedstock using solvent debinding. Fig. 2 (left) shows the catalytic debinding chemistry reaction when introducing very small amounts of acidic atmosphere (nitric acid) to depolymerise the polyoxymethylene (POM) in a nitrogen purged furnace. The evolving formaldehyde gas is burned off using a torch at the outlet of the furnace making the catalytic process clean and safe. Fig. 2 (right) compares debinding time using Catamold feedstock and other wax-based feedstock which are solvent debound.

Tests indicated that the optimum weight loss during catalytic debinding of tungsten heavy metal alloy in a small furnace load was reached at an acid flow of 12 to 15 ml/ h, a nitrogen

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purge of 500 l/h and temperature of 145°C. A debinding time of ten hours was sufficient to achieve good debinding results. Löber stated that this is somewhat longer than normally found for catalytic debinding stainless steels and low alloy steels, which he attributed to the fineness of the W heavy alloy powder mixture used.

Further tests showed that the loading of the 40 l furnace used could be increased to around 3 kg of product, which is equivalent to a fully loaded furnace. This indicates that when debinding is done in a continuous furnace, which typically shows a better gas flow pattern in the debinding zone, the debinding of the tungsten heavy alloy MIM parts will be even more efficient for high volume production.

Löber reported that using a powder loading of 51.7 vol.% resulted in sintered parts which met both the shrinkage (dimensions) and density specifications, and that, by raising the soak (holding) time to 1 h at sintering temperature of 1430°C, the sintered density could be increased to 97% of TD as seen in Fig. 3. The increased soak time also had a very positive influence on both tensile strength (>850 MPa) and elongation (>9%) properties. Löber stated that increasing soak time also gives the option of decreasing sintering temperature, thereby minimising possible part distortion.

www.catamold.de

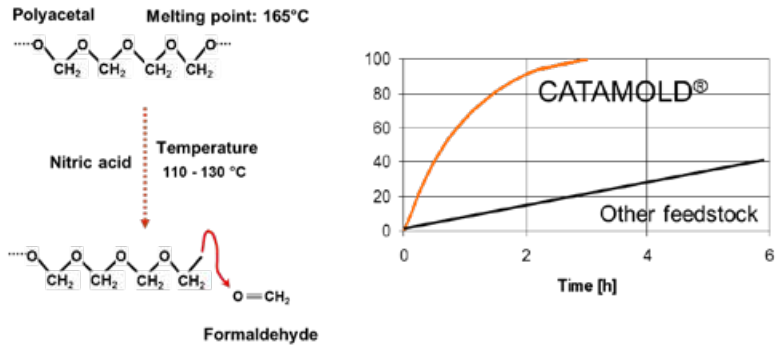

Fig. 2 (left) Schematic of the chemistry of the catalytic debinding process, (right) comparison of debinding speed of catalytic debinding with solvent debinding. (From paper by O Löber et al, 'Development and industrialisation of a tungsten heavy alloy feedstock for MIM based on BASF's Catamold technology', published in Proceedings of 19th Plansee Seminar, 2017)

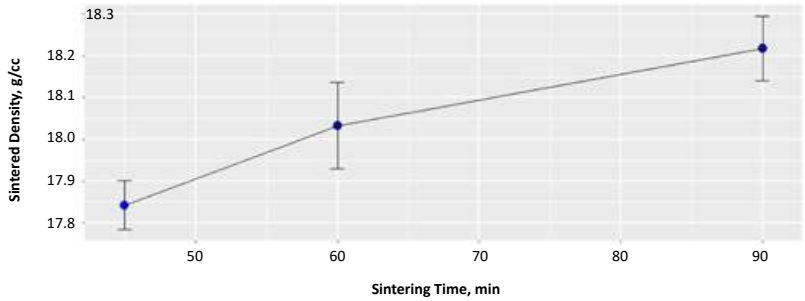

Fig. 3 Sintered density of tungsten heavy alloy as a function of sintering time at 1430°C (From paper by O Löber et al, 'Development and industrialisation of a tungsten heavy alloy feedstock for MIM based on BASF's Catamold technology', published in Proceedings of 19th Plansee Seminar, 2017)

Metal Powder Industries Federation elects officers

John F Sweet, President & CEO of FMS Corporation, Minneapolis, Minnesota, USA, has been elected as the 29th President of the Metal Powder Industries Federation (MPIF), succeeding Patrick J McGeehan, Ametek Inc., Eighty Four, Pennsylvania, USA. His two-year term will take effect at the conclusion of the Federation's annual Powder Metallurgy (PM) Management Summit and 73rd Annual MPIF Business Meeting, October 21–24, 2017, in Naples, Florida, USA.

One of the MPIF's six associations will also install a new President following the summit. Dax Whitehouse, NetShape Technologies, Floyds Knobs, Indiana, has been elected President of the Powder Metallurgy Parts Association (PMPA). Meanwhile, Dean Howard, PMT, North American Höganäs, Hollsopple, Pennsylvania, was re-elected to serve a second term as President of the Metal Powder Producers Association (MPPA). Tom Houck, Carpenter Technology, Bridgeville, Pennsylvania, will serve a second term as President of the Metal Injection Molding Association (MIMA).

Sweet has worked for FMS Corporation for 27 years, sustaining a family tradition as a third-generation entrepreneur. He most recently served as President of the PMPA and has served the association and MPIF actively for many years. Sweet received MPIF's Distinguished Service to PM Award at POWDERMET2017. He has been a member of APMI International for 29 years, is a certified Level I Powder Metallurgy Technologist and serves on the APMI Board of Directors. Sweet has also been an elected trustee of the Center for PM Technology. He earned his BS in materials engineering from Drexel University.

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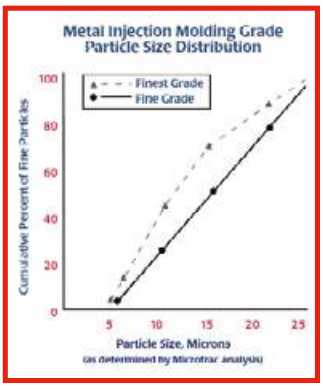
Stainless Steel Powders for MIM, Additive Manufacturing, & Other Fine Powder Applications



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Porous copper structures produced by PIM and space holder technique

A number of processes have been developed over the past two decades for the manufacture of micro copper structures used in heat pipes because of their good thermal conductance and capillary force properties. However, the processes used such as machining or lithography have relatively low production rates. H Cho and S J Park, at Pohang University of Science and Technology in Korea, recently reported on research to mass produce micro copper heat pipes using Powder Injection Moulding at the World PM2016 Congress [as reviewed in *Powder Injection Moulding International*, Vol. 11, No 1, March 2017, pp 86-87], stating that PIM is a promising route for producing micro copper heat pipes because of its ability to achieve the required total pipe thickness of less than 0.6 mm with a wall thickness of around 0.1 mm ~ 0.2 mm. The researchers have subsequently reported on additional research work to produce porous copper heat pipes using a combination of Powder Injection Moulding technology and space holder technology in a paper presented at the 19th Plansee Seminar held in Reutte, Tirol, Austria, May 29-June 2, 2017.

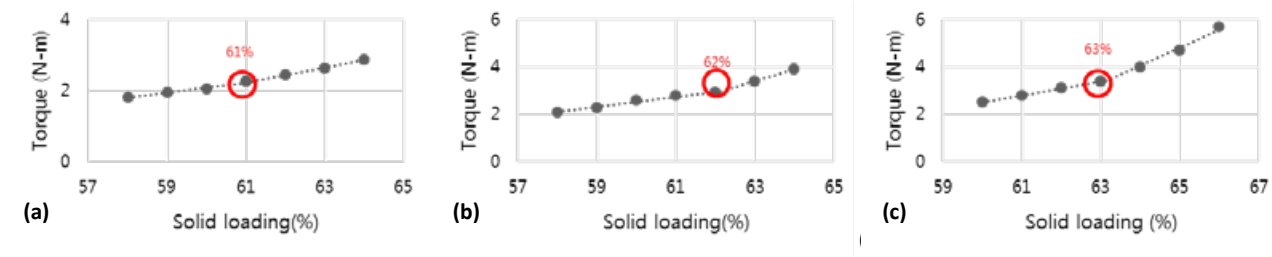

Fig. 1 Torque rheometer results using (a) 50 vol.% , (b) 60 vol.%, and 70% vol.% of NaCl space holder respectively (From paper by H Cho, S J Park, 'Fabrication of metallic porous structure by PIM process and space holder technique', published in the Proceedings of the 19th Plansee Seminar, 2017)

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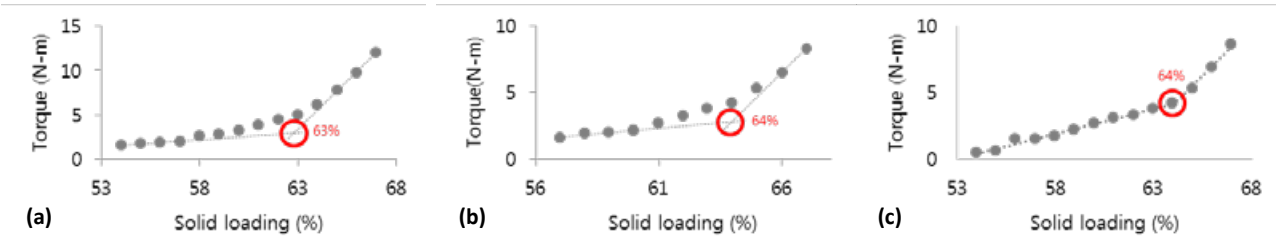

Fig. 2 Torque rheometer results for different PMMA space holder particle sizes (a) 4.7 µm, (b) 26.7 µm and (c) 71.0 µm (From paper by H Cho, S J Park, 'Fabrication of metallic porous structure by PIM process and space holder technique', published in the Proceedings of the 19th Plansee Seminar, 2017)

The authors stated that complex shaped porous copper structures can be produced using the space holder technique where the space holder content and size can be easily controlled. In the present paper they reported on their studies of the use of both sodium chloride (NaCl) and polymethyl methacrylate (PMMA) powders as space holder materials. The respective space holder powders are mixed with pure copper powder, and this mixture is in turn mixed with a wax-based binder system to produce the feedstock for powder injection moulding. The space holder material is removed after injection moulding to create the empty spaces in the porous copper structure.

The copper powder used in the research had a spherical shape and a particle size distribution (d50) of 4.73 µm. The particle size of the NaCl powder was >200 µm and the powder had a polygonal shape. Three different spherical shaped PMMA powders were used having particle sizes of 4.7 µm, 26.7 µm and 71.0 µm. The wax based binder was made up from paraffin wax, polypropylene, polyethylene and stearic acid. When NaCl space holder material was used the total powder loading in the feedstock, including copper powder, was 50 vol.%, 60 vol.% and 70 vol.%. The torque rheometer results for the different solid loadings are shown in Fig. 1. When PMMA space holder material was used each experiment was carried out with 70 vol.% of space holder content. The measured critical solid loadings for the two larger particle sizes was the same at 64%, whereas when the finer (4.7 µm) PMMA powder was used, the measured critical solid loading was 63%. Fig. 2 gives the torque rheometer results for the different PMMA particle sizes.

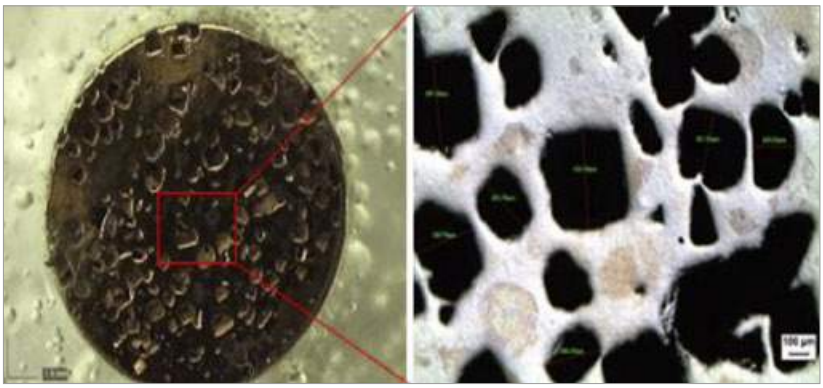

Fig. 3 Sintered porous copper specimen using NaCl space holder with its hexagonal pore shapes (From paper by H Cho, S J Park, 'Fabrication of metallic porous structure by PIM process and space holder technique', published in the Proceedings of the 19th Plansee Seminar, 2017)

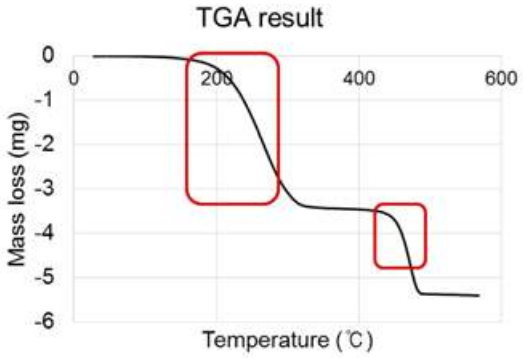

Fig. 4 Thermogravimetric analysis (TGA) results of mass loss measurement (From paper by H Cho, S J Park, 'Fabrication of metallic porous structure by PIM process and space holder technique', published in the Proceedings of the 19th Plansee Seminar, 2017)

The final PIM feedstock was prepared with 59% of solid loading by mixing in a twin extruder type mixer at 160°C. This was injection moulded in a cylindrical mould cavity measuring 8 mm in diameter and 13 mm in length. Sintering was carried out in hydrogen atmosphere at 700°C for 30 min to produce the porous copper structure using the NaCl space holder powder resulting in porosity measured at 46% and with polygonal pore shape (Fig. 3). Thermogravimetric analysis (TGA) was used to measure mass loss, as shown in Fig. 4.

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Co-Powder Injection Moulding of titanium alloy and hydroxyapatite for orthopaedic applications

Hydroxyapatite (HA) is an inorganic material with chemical properties identical to natural bone, but because of its poor mechanical strength it is unsuitable on its own for load bearing orthopaedic applications. Titanium alloys have good mechanical properties and corrosion resistance, but a higher Young's modulus compared to human bone, which could lead to stress shielding. F M Salleh and colleagues at the Universiti Kebangsaan Malaysia, in Selangor, have reported on their work to combine Ti-6Al-4V alloys with hydroxyapatite (HA) using a co-Powder Injection Moulding (co-PIM) process in a paper published in *Procedia Engineering*, (Vol. 184, 2017, pp. 334-343). Their work to-date has resulted in the development of a successful framework for a (co-PIM) process to produce Ti-6Al-4V/HA samples having a combination of good biological and mechanical properties.

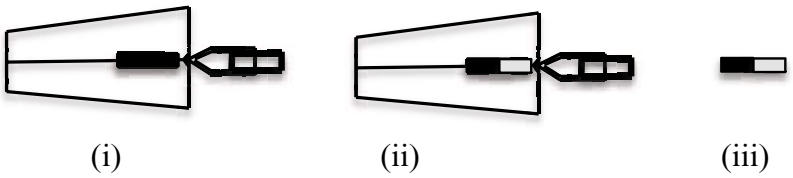

Fig. 1 Co-injection of Ti-6Al-4V/HA (i) Ti-6Al-4V is injected into the mould (ii) HA overmoulds half of Ti-6Al-4V part (iii) co-injected part (From paper by F M Salleh et al, Procedia Engineering Vol. 184, 2017, 334-343)

The authors state that the key to the success of the co-PIM process is the bonding mechanism of the two materials and their initial aim was to investigate the selection of polymer binders that would help optimise the bonding properties of the moulded green parts. The average particle sizes of the Ti-6Al-4V powders and the HA powders used were 19.6 µm and 5.31 µm respectively. Two feedstocks were tested using a binder system comprising 60 wt.% polyethylene (PE) and 40 wt.% palm stearate (PS). PE is the backbone polymer which holds the green part together and PS acts as the second backbone soft polymer that improves injection moulding. The authors

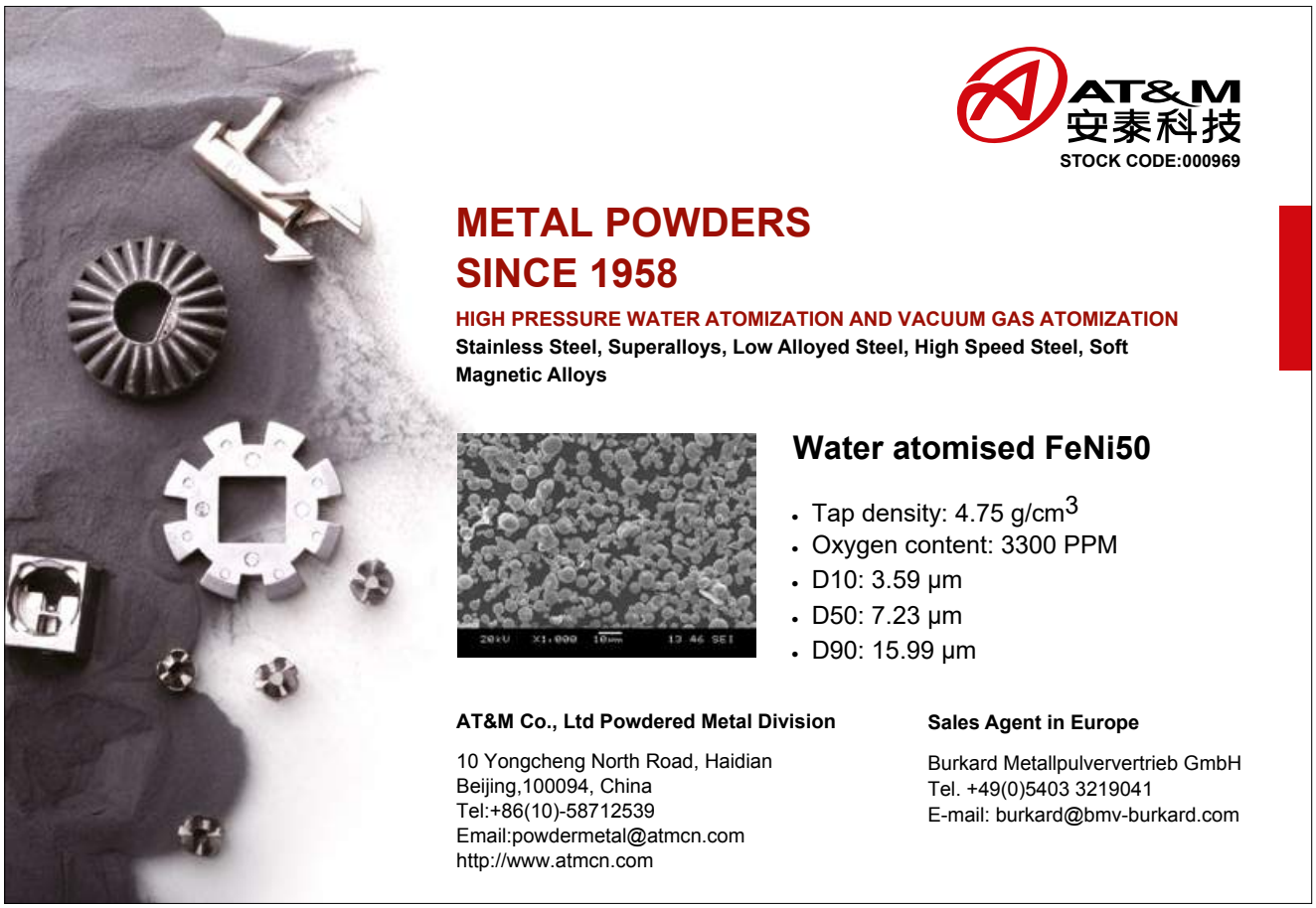

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Fig. 2 Co-injected Ti/HA green test parts (From paper by F M Salleh et al, Procedia Engineering Vol. 184, 2017, 334-343)

carried out TGA tests to obtain the melting temperatures for the binder both during mixing of the feedstock and during solvent debinding.

Powder loading in the feedstock was 68, 69 and 70 vol.% for Ti-6Al-4V and 56 vol.% for the HA powder. The mixing process started with melting the PE to which were added the Ti-6Al-4V powder and PS progressively. The same procedure was used for mixing the HA powder. Co-Powder Injection Moulding was done by first injecting the Ti-6Al-4V feedstock into the mould, then removing the moulded part, cutting in half and replacing one half back into the mould. The HA feedstock was then injection moulded over the half Ti-6Al-4V compact. Injection moulding parameters included: feedstock melt temperature of 150°C, mould temperature of 90°C, injection pressure of 10 bar and with 10 s holding time. These parameters were successful for both the Ti-6Al-4V and the HA feedstock. Fig. 1 shows the co-injection moulding sequence where (i) Ti-6Al-4V is injected into the mould; (ii) HA overmoulds half of the Ti-6Al-4V part. Fig. 2 shows the co-injected Ti-6Al-4V/ HA green test bars and Fig. 3 shows SEM micrographs for co-PIM green parts (a) 70 vol.%Ti/56 vol.% HA, (b) 69 vol.%Ti/56 vol.% HA and (c) 68 vol.%Ti/56 vol.% HA. The 70 vol.%Ti/56 vol.% HA combination showed the highest density (2.72 g/ cm^3) and bonding strength of 2.279 MPa and this composition has been selected for further research into the debinding and sintering stages of the co-PIM 70 vol.%Ti/56 vol.% HA material.

www.journals.elsevier.com/procedia-engineering

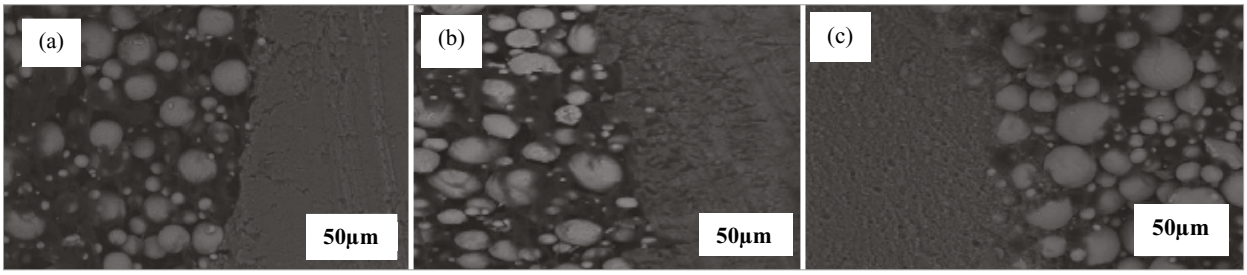

Fig. 3 SEM micrographs of Co-injected green part of Ti-6Al-4V /HA (a) 70.vol% Ti/56vol.% HA, (b) 69 vol.%Ti/56 vol.% HA (c) 68.vol % Ti/56 vol.% HA (From paper by F M Salleh et al, Procedia Engineering Vol. 184, 2017, 334-343)

Euro PM2017: Special Interest Seminar to focus on MIM for aerospace applications

A Special Interest Seminar at the Euro PM2017 Congress and Exhibition, Milan, Italy, October 1-5, 2017, will focus on MIM for aerospace applications. Chaired by Prof Dr -Ing Frank Petzoldt (Fraunhofer IFAM, Germany)
and Keith Murray (Sandvik Osprey Ltd, United Kingdom), the seminar will approach the special demands of the aerospace industry on MIM technology from a fresh angle, highlighting the viewpoints of a MIM part producer and two leading European aircraft engine manufacturers. Challenges such as new high-temperature materials and low volume production will be addressed.

The seminar will also give an outlook on what is required from the end-users' point of view to increase the portfolio of parts that can be produced for aerospace applications in the future. There will be time for discussion among the audience and with the speakers.

Confirmed presentations at the time of publication are:

- MIM For Aero-Engine Parts – Challenges and Opportunities
  Dr.-Ing. Enrico Daenicke (Rolls Royce, Germany)

- Challenges of Introducing MIM Parts in Aerospace
  Dr Sébastien Richard (Safran, France)
- MIM Components for Aerospace Actuation Systems
  Dr Pedro Rodriguez (MIMTECH Alfa, Spain)

Session 29 is scheduled for Wednesday October 4, 14.15 - 15.45.

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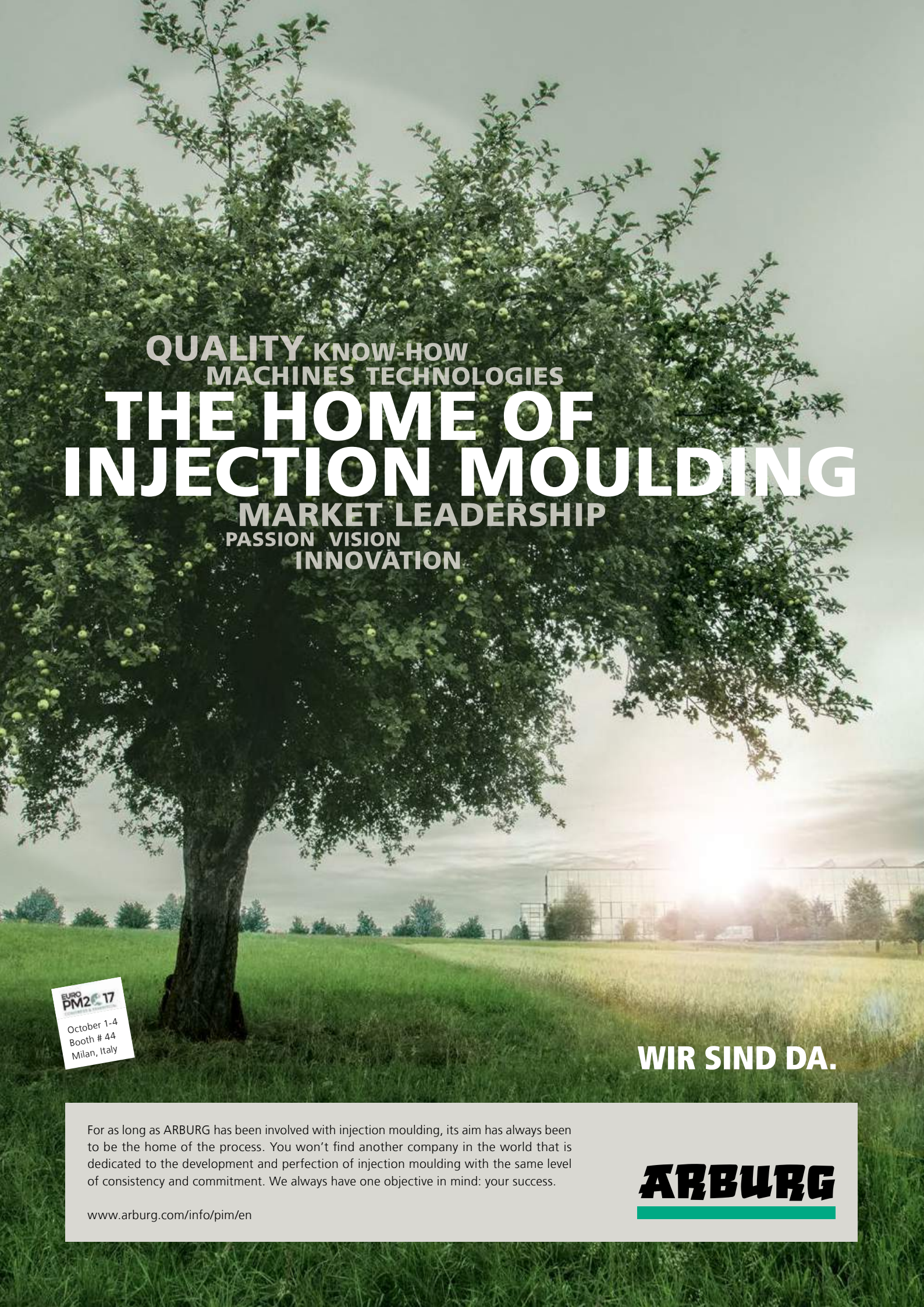

For as long as ARBURG has been involved with injection moulding, its aim has always been to be the home of the process. You won't find another company in the world that is dedicated to the development and perfection of injection moulding with the same level of consistency and commitment. We always have one objective in mind: your success.

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MIM in the digital age: Simulation technologies to help the industry compete in the 21st century

It is a fact that the vast majority of defects in MIM components occur during injection moulding. The latest moulding simulation technology not only anticipates these defects before tooling costs are incurred, but can also be used to automatically generate optimised tool and runner designs by analysing multiple variations of gate location and type. Dr Götz Hartmann, MAGMA GmbH, and Timo Gebauer, SIGMA GmbH, explain how simulation technologies can today be used to enhance new MIM part development.

At this summer's Metal Injection Moulding Short Course, organised by the European Powder Metallurgy Association (EPMA) in Ludwigshafen, Germany, leading European part producers, materials suppliers and equipment producers discussed a wide range of issues affecting the industry. These inevitably included concerns regarding cost-competitiveness and challenges from competing technologies.

Metal Additive Manufacturing is one technology that is now positioning itself as a viable alternative to MIM for lower production volumes. The latest binder-based AM processes openly borrow much from MIM, particularly in relation to binder materials, powders, debinding and sintering. An ever growing list of these 'MIM-like' Additive Manufacturing companies includes Desktop Metal, Xjet, Markforged, ExOne and Digital Metal. These companies are frequently in the headlines and openly embrace the opportunities presented by the new age of digital manufacturing.

So how can Metal Injection Moulding position itself as a competitive, dynamic 21st century technology? Following SIGMA's presentation at the seminar, *PIM International*'s Editor, Nick Williams, commented to us, "It amazes me how, while some in our industry see the benefits of simulation technology, others believe that relying on experience and trial and error is the natural way when it comes to developing MIM parts."

Whilst the conventional way of developing MIM components, tinted with traditional notions of the toolmaker's craft, may have worked to-date, perhaps the digital

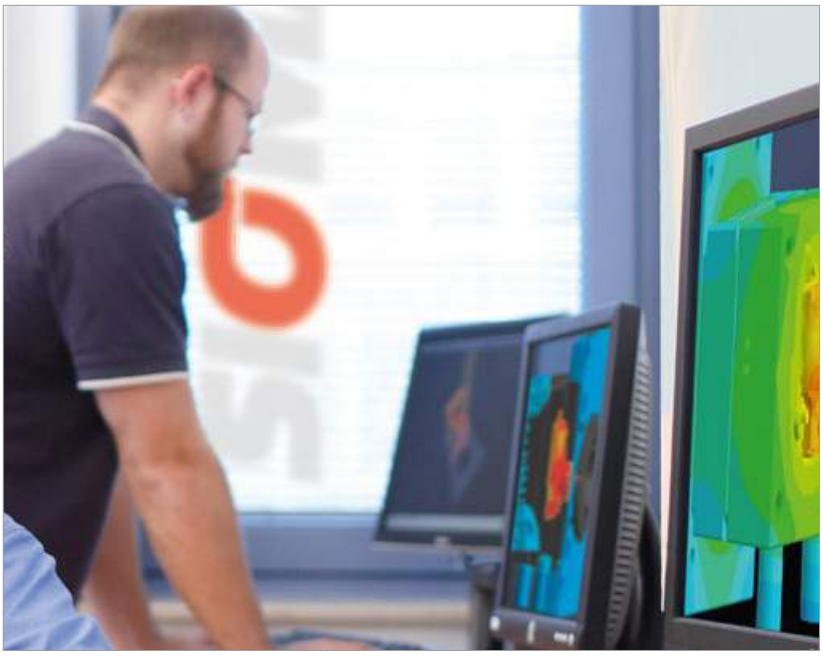

Fig. 1 PIM simulation runs underway at SIGMA, Aachen, Germany

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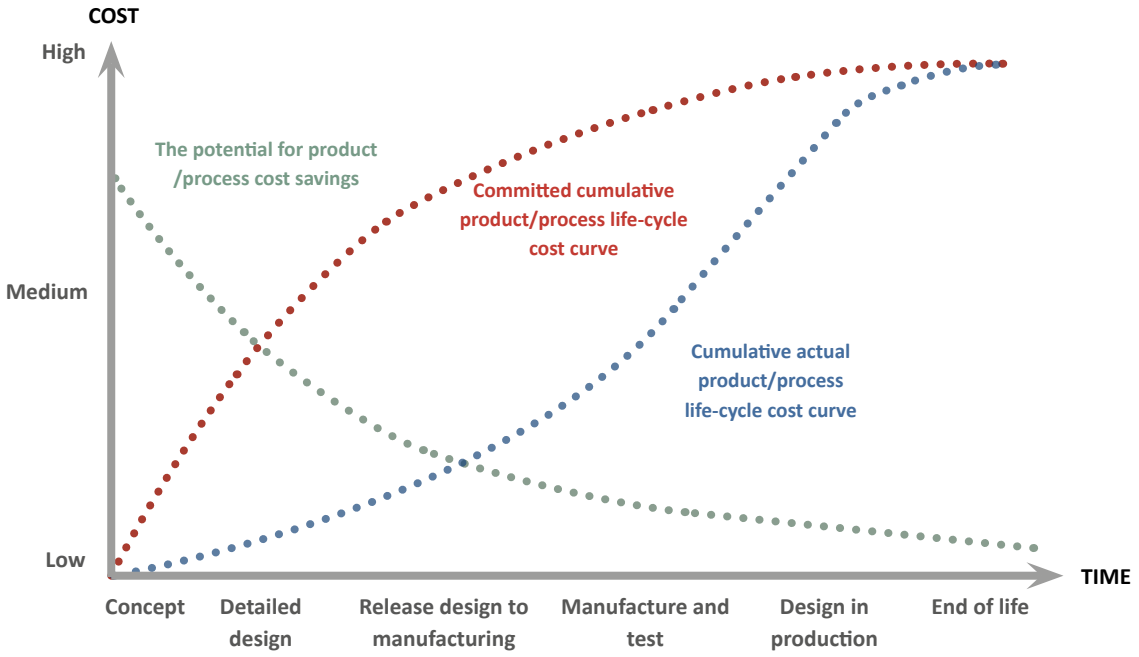

Fig. 2 The potential to influence part development and production costs across the life cycle of a part

manufacturing revolution that is upon us will require a re-evaluation of how the MIM industry's profile can be enhanced.

This article will show how simulation reduces both component development time and the lifetime cost of a component. What must also be considered is how the adoption of more advanced component development tools can change the perception of a technology amongst end-users. The viability of designs can be established at an early stage, risk and costs can be reduced, development speeded up and, as a result, PIM might just be more strongly positioned to compete with the emerging generation of manufacturing processes.

About cost responsibility and front loading

Powder Injection Moulding has been in use since the 1920s, when the first alumina insulators for spark plugs were produced using the technology. Sixty years prior, development and production engineers had already realised that around 70% of the responsibility for the later costs of a component's production rests on the shoulders of its designers. Google 'product life-cycle costs' and you will see hundreds of graphs from hundreds of publications, academic lessons or production handbooks showing the same correlation. One example is shown in Fig. 2.

Many of these publications describe the facts as given, but some also discuss some of the methods which can be used to reduce production costs, based on the knowledge and acceptance of these functional relationships. In these you will find key terms such as 'simultaneous engineering' and 'front loading'.

As large-scale production of Metal Injection Moulded components took off in the 1980s, a range of new procurement approaches, first suggested for the automotive industry by Ignatio Lopez, were coming into play. These approaches structured business-to-business cooperation along the product life cycle into many small and easy to control pieces. As a result, approaches such as 'simultaneous engineering' and 'front loading' were pushed to the background of the component design and production process.

Even now, with a few exceptions, close collaboration between component designer and manufacturer is difficult and rare. Perhaps the following statement is a little exaggerated, but with some truth: The product life cycle, from design to series production, is cut into sections in the hands of many departments of many companies, each of them chasing their own interests such as budgets, time schedules, costs, profits and quality of their delivery.

However, even if each party involved in this step-by-step process does their best, huge potentials for improving the overall process chain will be missed. This is not only frustrating for a good engineer, but sub-optimal for business and administrative issues such as time to market or cost/profit.

It should be absolutely clear that, if there is no responsibility for the technical and economic performance

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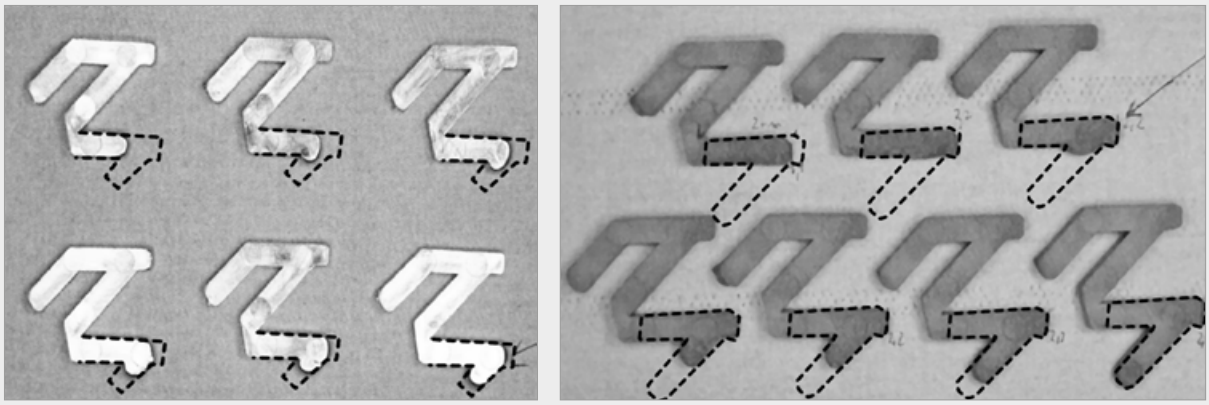

Fig. 3 In comparison to thermoplastics (left), the typical flow behaviour of MIM feedstocks (right) shows free jetting even at low flow velocities, due to high viscosities at low shear rates

of the total process chain or the product life, both the technical and economic potentials of a component will not be leveraged.

The value of the Virtual Molding approach

It was not just MIM that came to fruition in the 1980s. New computer simulation technologies targeting industrial production processes also came onto the market. Following the first numerical simulation technologies, which examined a part's performance under load and were positioned very close to part design, these simulation programs added manufacturing process engineers to their user community. The programs covered processes such as metal casting, polymer injection moulding, stamping and welding and became well established from the mid-1990s.

The value proposition now existed to use simulation to predict future manufacturing process issues at the design phase. This value is immense, because it is the value of looking into the future. The price for doing this today is, in reality, quite low compared to the alternative; the cost of the most detailed and complex Virtual Molding test run might be up to €1,500 and can be done before design freeze. On the other hand, a trial and error procedure for a MIM part, made up of the production of a test series (after design freeze, tool layout and manufacturing), detailed inspection of each part, evaluation of problems occurring and improvement measures, change of runner, gates and tempering of the mould and production of another test series costs at least ten times more and takes much more time.

The cost difference between a Virtual Molding approach and a real test series is hard fact. Other soft facts to add to this are that the design process using integrated Virtual Molding becomes much more effective because it delivers component designs for robust manufacturing; the necessity of going back after the design freeze is reduced to a minimum, and a Virtual Molding test run is much more informative and makes it far easier to make efficient improvement measures than the production of a run of test parts. All this leads to immense cost savings.

MIM processing is complex but predictable

The Virtual Molding of MIM processes means the prediction of what will happen during manufacturing by mathematical, physical, thermodynamic and thermo-mechanical models. There are always two prerequisites for the modelling of processes such as MIM.

On the one side are the mathematical descriptions of the phenomena, which usually occur during manufacturing – as far as they are understood. On the other side is the massive depth of knowledge that exists about numerical simulation techniques for the handling of three dimensional coupled differential equations of complex 3D transient problems.

Short cut models, such as two-dimensional flow simulations for thermoplastics, are insufficient to predict the complex material and heat flow of the MIM processes. This

"The value proposition now existed to use simulation to predict future manufacturing issues at the design phase. This value is immense, because it is the value of looking into the future"

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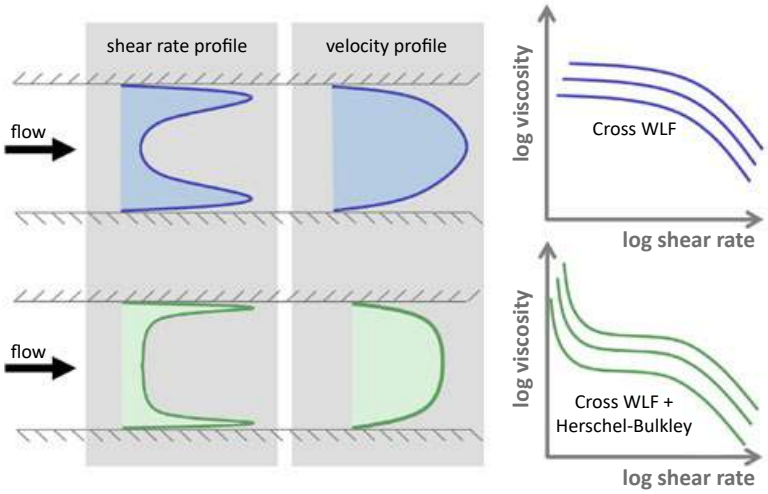

Fig. 4 The Herschel Buckley extension to the Cross WLF model helps to describe the particular MIM feedstock flow behaviour at low flow rates [2]

article will present the state-of-the-art for MIM Virtual Moulding, including extended flow models and models for the thermal balance of the whole mould, including all tempering devices.

Flow modelling

Because their viscosity is in the same order of magnitude, liquid metals flow like water. Semi-solid metals, thermoplastics, concrete and ketchup flow like blood, which is 'thicker than water' because its viscosity also depends on shear rates. MIM feedstocks, however, behave in an even more complex manner. At low flow rates in particular, MIM feedstocks behave more paste-like than liquid, even in comparison to thermoplastics (Fig. 3).

While thermoplastic flow can be modelled using, for example, the Cross WLF model, which combines a model of shear rate dependency with temperature dependency, MIM feedstocks need correction at low flow and low shear rates. Of the few proposed models, a Cross WLF including a Herschel Buckley approach [1] seems to describe the particular flow behaviour of MIM feedstocks quite well (Fig. 4).

Furthermore, metal powder tends to separate from the polymer binder because of collision effects and shear gradients in the feedstock during mould filling (Fig. 5). The resulting metal particle concentrations in the green part have a negative impact on the total process chain, from debinding through to sintering to the finished part.

Unfortunately, locally different metal powder concentrations create a back coupling effect of local powder contents on local viscosities, thus increasing the complexity of flow effect modelling even more. The modelling of back coupled effects normally leads to longer calculation times, which is why particularly efficient physical models and numerical algorithms are required.

The models and algorithms used in the MIM module of SIGMA's Virtual Molding technology to represent the above mentioned back coupling effects increase calculation time by just 10-15% and show flow and separation effects as they are observed in reality (Simulations 1 and 2, available to view via link).

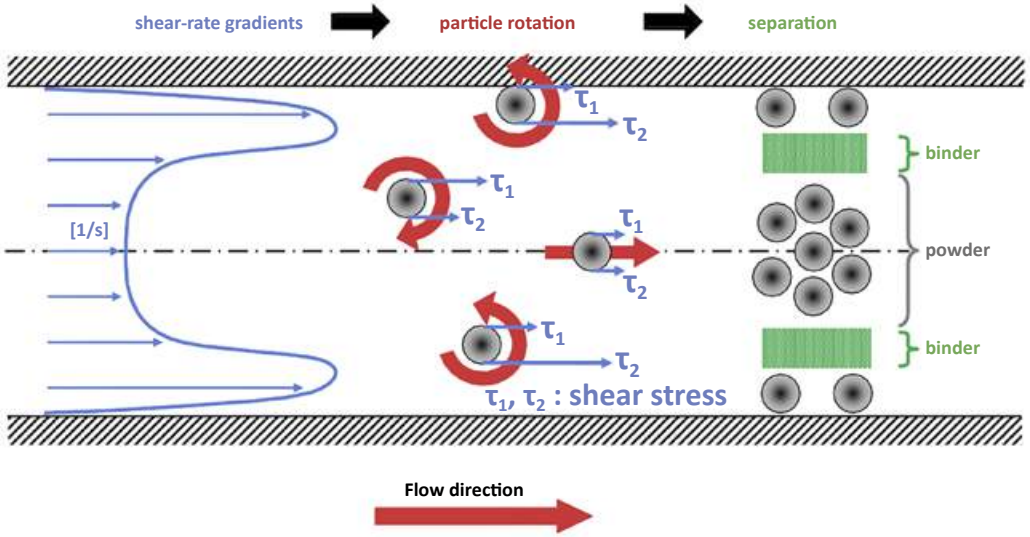

Fig. 5 The shear rate gradients in a MIM feedstock flow lead to separation or segregation of the particles [3]

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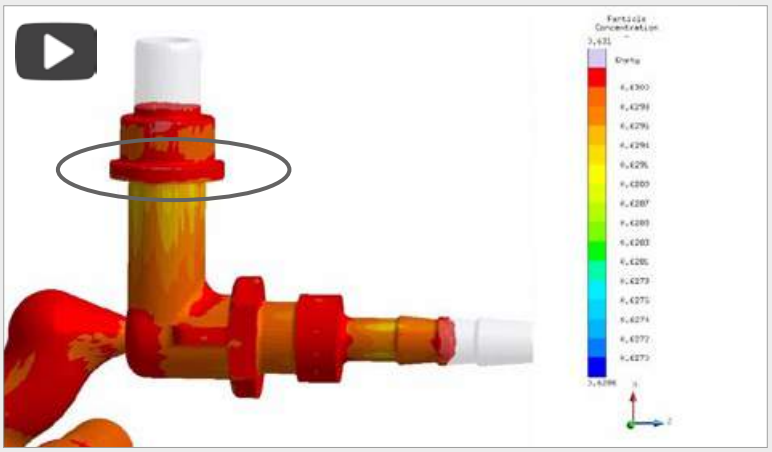
Heat transfer modelling

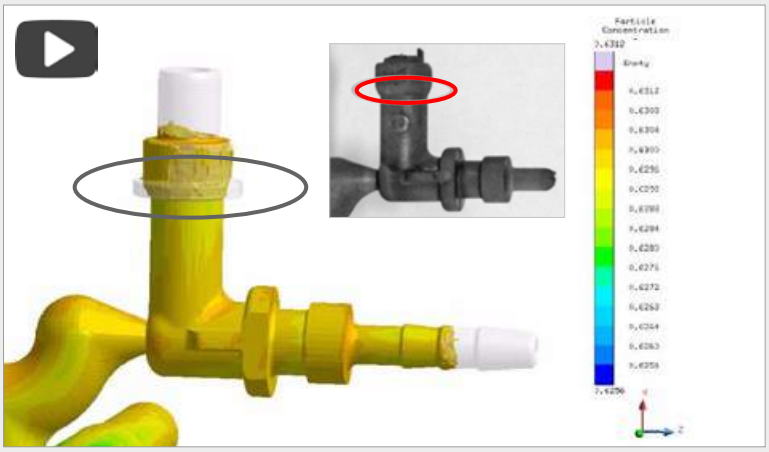
Because of their high solid metal content, MIM feedstocks cool down very differently to thermoplastics. In comparison to thermoplastics, MIM feedstocks have a thermal conductivity that is six times higher, while the thermal conductivity of a 316 steel is about fifty times higher. On the other hand, the volume-related heat content of steel MIM feedstock is about three times higher than aluminium MIM feedstocks, in the same order of magnitude compared to thermoplastics (Fig. 6).

All this means that the MIM process is much more sensitive to thermal balance than thermoplastic injection moulding. Tool materials and temperatures, cooling and heating procedures, cycle timing, etc, are all critical to process stability and product quality, whether measured in terms of local mechanical properties, surface finish or residual stress and distortion.

The main issues for modelling all of the thermal balance aspects of the moulding stage of the MIM process are not necessarily the physical models: Fourier's law for heat conduction within materials and across interfaces was postulated in 1822 and is still the backbone of today's analytic or numerical heat flow simulation technologies.



Simulation 1: A MIM part's filling pattern without back coupling, leading to fountain flow. Higher particle concentrations are in red. A more complex model (Video 2) is needed to get correct predictions (view video at www.pim-international.com/media/sigmasoft)



Simulation 2: This filling pattern, with back coupling, leads to jet flow and accurately replicates a mould filling issue, as shown in inset image (view video at www.pim-international.com/media/sigmasoft)

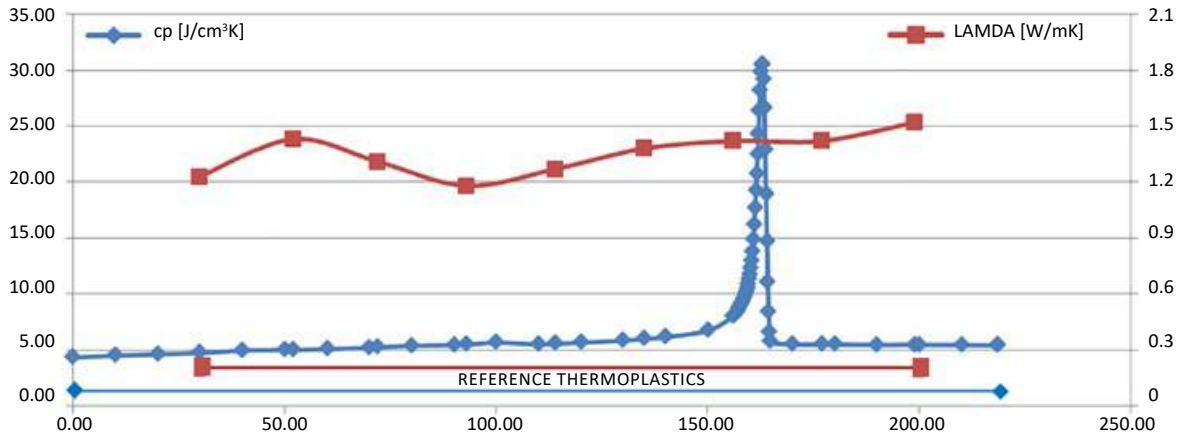


Fig. 6 Both specific heat cp and thermal conductivity LAMDA of MIM feedstocks (measured values) are quite different from thermoplastics (reference lines), thus making the MIM process much more dependent on the thermal balance of the mould

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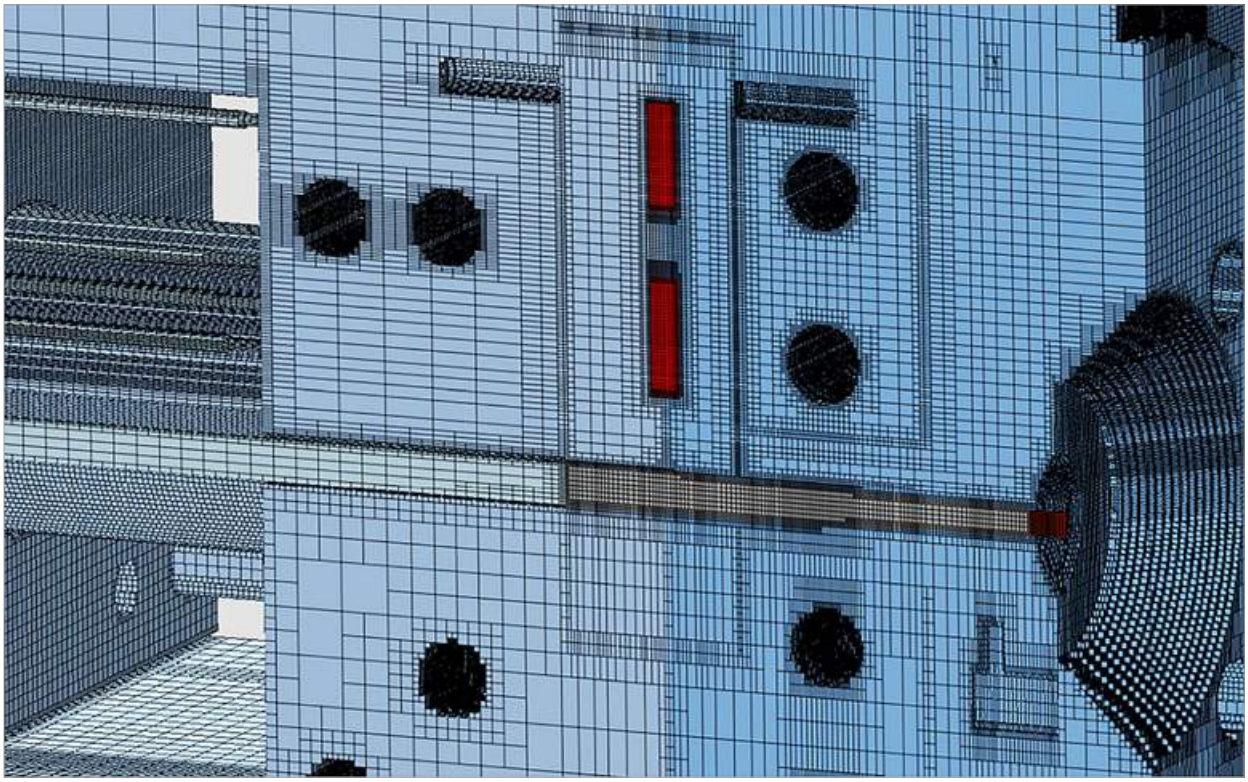

Fig. 7 The enmeshment of this MIM mould includes all components of the mould. The simulated thermal balance of the mould founds on heat conduction within components, heat transfer over interfaces between them and controlled heat sources and sinks

The first issue is that a MIM tool consists of hundreds of individual parts made from different materials. These can be located between tool steel sections, with tempering media and cooling or heating channels, etc, and there are hundreds of interfaces between these components, all with different heat transfer specifics. All these tool components, their materials and the interfaces between them must be represented in a geometric 3D model and in the enmeshment that is needed for the numerical solution of the 3D Fourier equations.

A reasonable mesh of a medium size MIM tool with average complexity easily requires 100 million volume elements or nodes (Fig. 7). Virtual Molding technology, with the ability to handle such complete moulds in an injection moulding process simulation, has been on the market for twenty years. Today, we can handle large, complex multicavity moulds with up to 350 million mesh elements on a normal engineering work station, while it takes a maximum of one to two hours from reading the CAD data of a mould and a part to start a simulation run.

"Virtual Molding technology, with the ability to handle such complete moulds in an injection moulding process simulation, has been on the market for twenty years"

A second issue is the rather large number of heat sources and heat sinks. The control of these can be time or temperature dependent, or even more complex with PI or PID controllers. The simulation of these aspects is state-of-the-art but is now possible with SIGMASOFT Virtual Molding. Simulation 3 shows the thermal balance of a mould during injection.

Recent activities by a number of groups (for example [4]) have proved how important and effective mould tempering is for MIM production. A fast-acting mould tempering system is the prerequisite for part quality, for example with regard to low segregation and low distortion, as well as for cost effective processes, for example with respect to cycle timing and energy efficiency.

A reliable virtual modelling technology, such as described above, is the prerequisite for the development of a reliable process in terms of part quality, productivity

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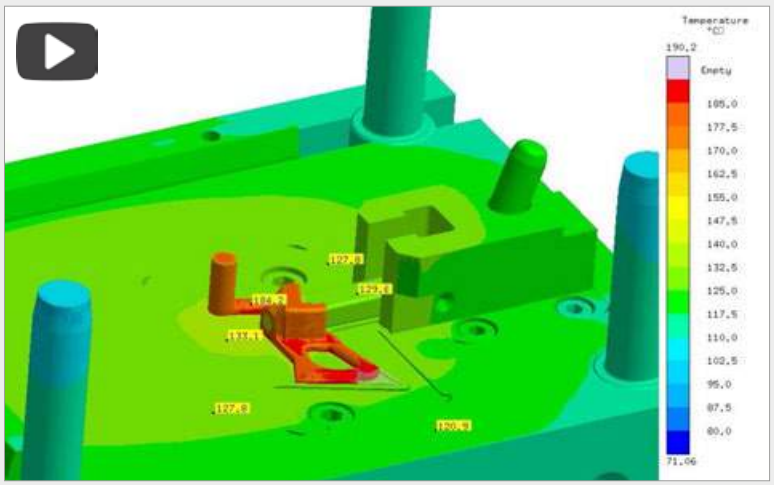
and resource sustainability. Simulations 4 and 5 compare the temperature stability of a MIM mould in the startup phase and during established production.

The old dream: Virtual Molding DOE and autonomous optimisation

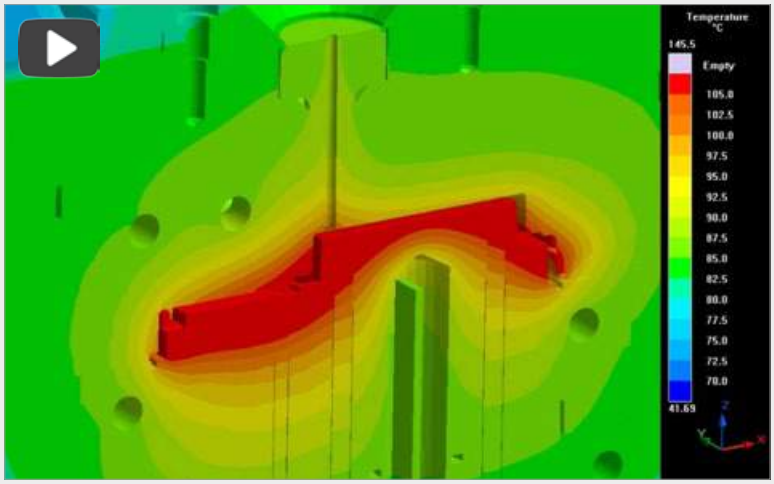
Thirty years ago, when process simulation technologies for the major metal and polymers shaping processes were first made available for industrial use, statements, such as "software which does not solve my problem right away makes no sense," or "if the software doesn't tell me what I have to do it is worthless," set some basic expectations of simulation technology that exist to this day. In the 1990s, users were taught that simulation remained part of a trial and error approach, but with reduced costs and risks. Simulation programs could therefore not deliver immediate solutions until they were coupled with DOE or autonomous optimisation methodologies.

Today, the coupling of Finite Element codes with optimisation algorithms for structural optimisation is state-of-the-art [5]. However, the computational optimisation of processes such as MIM is on another level; here, complex physics and thermodynamics meet complex geometries. Furthermore, many process parameter or tool design related constraints meet uncertain degrees of freedom. Nevertheless, over the last fifteen years some relatively easy to use but powerful DOE and optimisation tools for metal casting and polymer injection moulding were developed and leveraged to an everyday practical use for optimising casting and injection moulding processes [6].

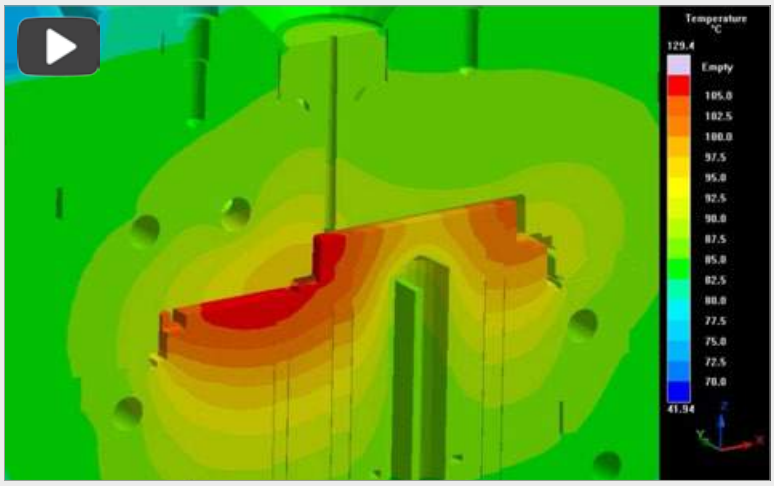
As previously mentioned, particle segregation during injection moulding is a common issue and MIM technology is, therefore, a good candidate for virtual process optimisation. For jewellery parts, such as the clasp of a wristband produced by MIM (Fig. 8), surface homogeneity is critical.



Simulation 3: The thermal balance of the injection moulding process in MIM: Mould filling and mold temperature simulations are combined (view video at www.pim-international.com/media/sigmasoft)



Simulation 4: Temperature development of a MIM mould over 25 cycles. The video is based on temperature just before injection images (view video at www.pim-international.com/media/sigmasoft)



Simulation 5: Temperature development of a MIM mould within an established production cycle. This reveals significantly higher process stability than in the initial 25 cycles (video at www.pim-international.com/media/sigmasoft)

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Fig. 8 The gate area of this MIM wristband clasp is equipped with a MeltFlipper® like section to homogenise shear rates

It is clear, even without going through any process simulation, that process parameters such as flow velocity influence surface homogeneity. The functional chain goes from flow velocity over shear rate gradients, inhomogeneous viscosities and particle segregation visible as surface defects at the polished part. Shape parameters such as part and runner/gate design have some influence on particle segregation, because they are responsible for altering shear gradients and local viscosities.

A DOE, set up to evaluate the effect of these process and design parameters on the surface quality of the MIM part, consequently consists of a reasonable number of variants (Fig. 9). This DOE is made up with twenty gate design variants and four different flow velocities during cavity filling, resulting in a total of eighty variants. The statistical evaluation of the DOE shows that the deviation of the particle concentration strongly depends on flow velocity: high velocities result in high shear gradients, lead to inhomogeneous velocities of low values and finally promote powder segregation. On the other hand, there is no clear tendency with regards to the gate design, but the Pareto set clearly identifies one variant with a minimum deviation of the particle concentration.

As result of the automatically running DOE, a gate design variant providing the lowest possible segregation – out of the variants of the design space – can be identified within the gate area of the tool (Fig. 10).

The effort for these virtual experiments, where eighty possible gate design and process parameter variants were tested, was in total 1½ man days. This means that the total costs for this method to find relative optimal MIM processing conditions are within one or two orders of magnitude lower than producing the usual three to five test series of parts prior to beginning series production.

Conclusions

Computer simulations provide predictions and, generally speaking, some kind of view into the future. The value of predictions can be extremely high: if, for example, lottery numbers were to be predictable, the pay-off could be the weekly jackpot. However, there are numerous conditions, all of which have to be fulfilled, if a prediction is to pay off in reality.

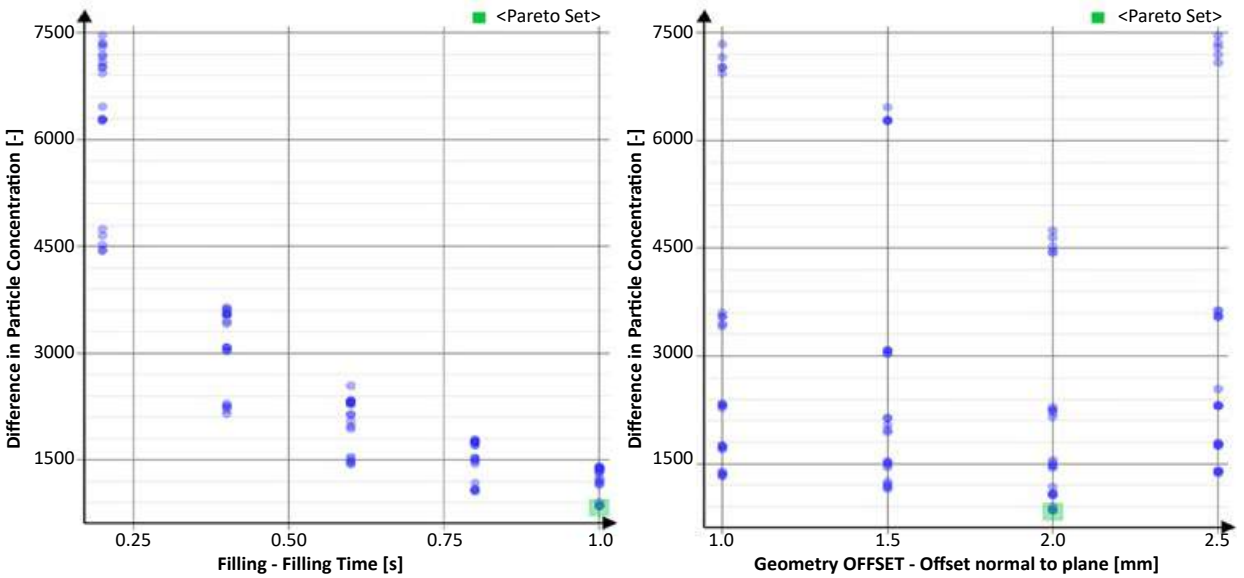

Fig. 9 Dependence of surface quality. Shown here are the local differences of particle concentrations from cavity filling time and 40 different gate designs

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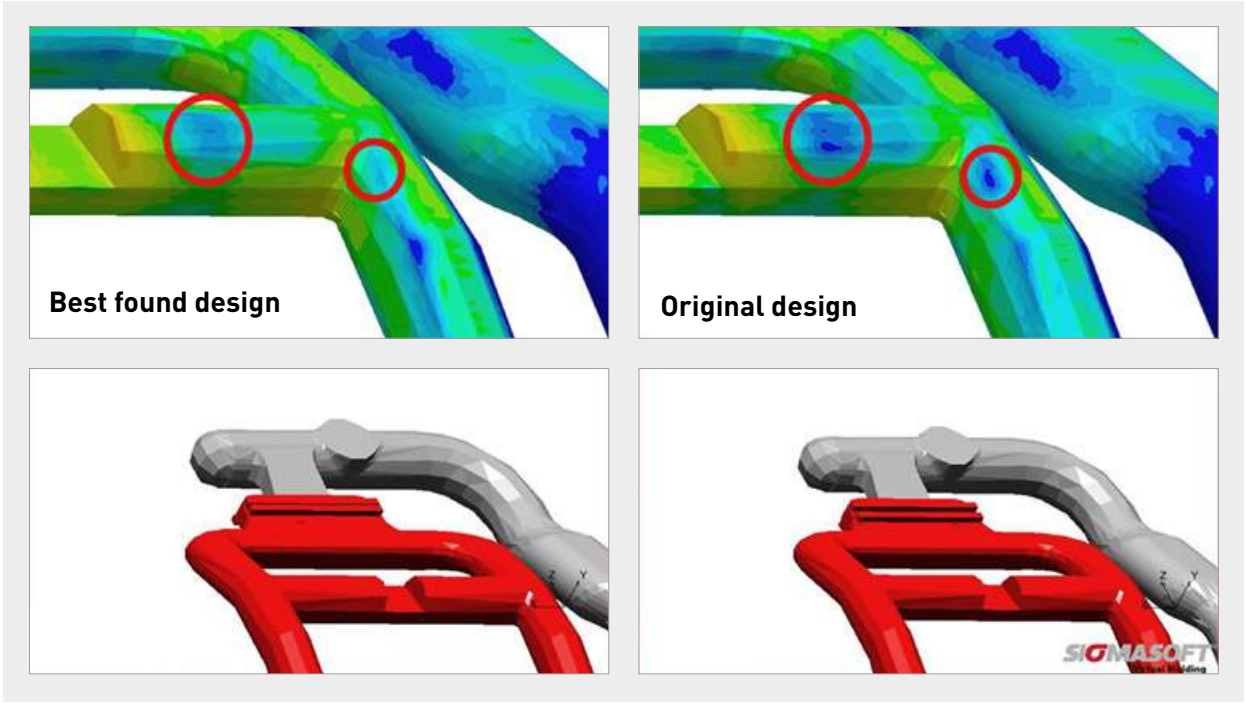

Fig. 10 Improvements in particle homogeneity achieved by evaluating different variants of the gate design with a virtual DOE. The blue area in the original design shows low particle density and would have resulted in surface imperfections during polishing the buckle

Firstly, the occurrence which is being predicted has to follow some known rules. This makes lottery number prediction improbable, but an accurate weather prediction probable; when known rules, in the format of mathematical equations, are applied to certain boundary conditions, some occurrences can be predicted – provided that the rules, their translation into mathematical expressions and the evaluation schemes are all correct. Even then, such predictions have no value right away.

The prediction of MIM manufacturing occurrences by means of Virtual Molding copes with the same principle. The physics and thermodynamics of MIM, as with many other technical processes, will probably never be completely understood. However, the models that have been presented allow a reasonably reliable prediction of success in terms of the identification of critical occurrences in MIM manufacturing.

Simplified models, when more realistic ones are available, can lead to wrong predictions, while state-of-the-art models such as those mentioned here can offer more reliable predictions. The high level of reliable Virtual Molding technologies can help to avoid problems and lead to quality improvements or cost savings if integrated into the MIM component development, tooling and manufacturing chain.

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PSI Ltd: Celebrating thirty years of innovation in gas atomiser production

Phoenix Scientific Industries Ltd (PSI) was founded thirty years ago with the goal of developing gas atomised metal powders for the then emerging Hot Isostatic Pressing (HIP) industry. Since then, the company has continuously developed its atomising technology in response to the evolving application areas for gas atomised metal powders, notably in the Metal Injection Moulding industry and the more recently developing Additive Manufacturing processes. Bill Hopkins discusses the company's story and comments on a recent move into metal powder production.

PSI's founding mission was to convert the theories of materials processing into engineering plants for the production of advanced materials. The company now produces machines for atomising metal to powder, PVD systems for turbine blade coatings, melt spinners for magnetic materials and CVD furnaces for ceramic fibre-reinforced composites. However, the company is chiefly known for its work in metal atomisation and, in particular, for its high performance gas atomisers for the production of quality, fine and spherical metal powders.

Formed in 1987 by Bill and Jan Hopkins and backed by Lucius Cary of venture capital company Oxford Technology, the company has grown from building small research machines for universities to supplying multi-million dollar production systems for large corporations. Owing to the founders' previous international business experience and in recognition of the fact that such a specialist business would need to address the wider world market, the management team addressed this challenge from the outset. Today, the company has installed systems in twenty one countries on all seven continents with the exception of Antarctica, a market still proving hard to crack.

"Thirty years ago the Powder Metallurgy market was dominated, at least in terms of volume, by irregular water-atomised steel powders for automotive components and tungsten carbide for machining tools, both materials being suited to the press and sinter technology then available," Bill Hopkins told *PIM International*. "Spherical and irregular powders of the type made, respectively, by gas and water atomisation were used in thermal spray technologies

Fig. 1 A Hermiga Mini atomiser Model 75/3 VI

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Fig. 2 A view of a production scale Hermiga atomiser at PSI Ltd. This system has been specifically designed for ease of cleaning between different material runs

for wear and corrosion resistant coatings. The application that attracted our attention was their use in Hot Isostatic Pressing [HIP]. Previously, we had worked in metals R&D and, being tasked with developing new alloys and components especially suited to the HIP process, had noticed the limited availability of and high price premium commanded by the very pure [low oxide] gas atomised spherical powders required for HIP."

The ability to produce large, near-net-shape critical components such as aero engine turbine discs from powder offered an exciting alternative to the costly and cumbersome cast and forging route used conventionally. However, the catastrophic in-flight failure of a PM disc, which was attributed to uncertain powder quality, emphasised the need for a step change in the approach to the manufacture of gas atomised powder if PM was to be used in critical engineering applications.

Close-coupled gas atomisation

At around the same time Nick Grant, a professor at MIT, had developed the concept of 'close-coupled' gas atomisation. Until then, metal powder had been produced by generating a free-falling stream of molten metal from a furnace and aiming jets of water or gas in the general direction of that stream. If inert gas was used and the melt was 'clean' in terms of gas and refractory inclusions, this was a perfectly good way of making spherical metal powder suitable for HIP.

This process was, however, inherently inefficient in terms of usage of the kinetic energy of the atomising fluid, and produced only coarse (mainly above 50 µm) powder, while the industry wanted finer powder for a range of new applications. In close-coupled gas atomisation, the atomising gas is aimed directly at the end of the ceramic tube from which the melt emerges. This enables the energy of the gas stream to be absorbed by the melt stream much more readily, greatly increasing fine powder yields. In some cases, the powder distribution can contain more than half of its weight less than 15 µm in diameter; and this was what the newly emerging Metal Injection Moulding industry required. If the technology was to flourish, however, costs needed to be reduced.

Bill Hopkins stated, "These requirements defined the type of atomiser that PSI decided to build. Clean melts were required as this cleanliness would translate into clean powder and therefore vacuum processing of melt was required. When not operating under vacuum the whole process needed to operate under inert gas or nitrogen and take place in a highly polished stainless steel process plant more typical of the pharmaceutical industry than that found in the fume-filled caverns of the foundry industry. To produce the fine powder required by the MIM industry, close-coupled atomising nozzles had to be developed that were both efficient and reliable."

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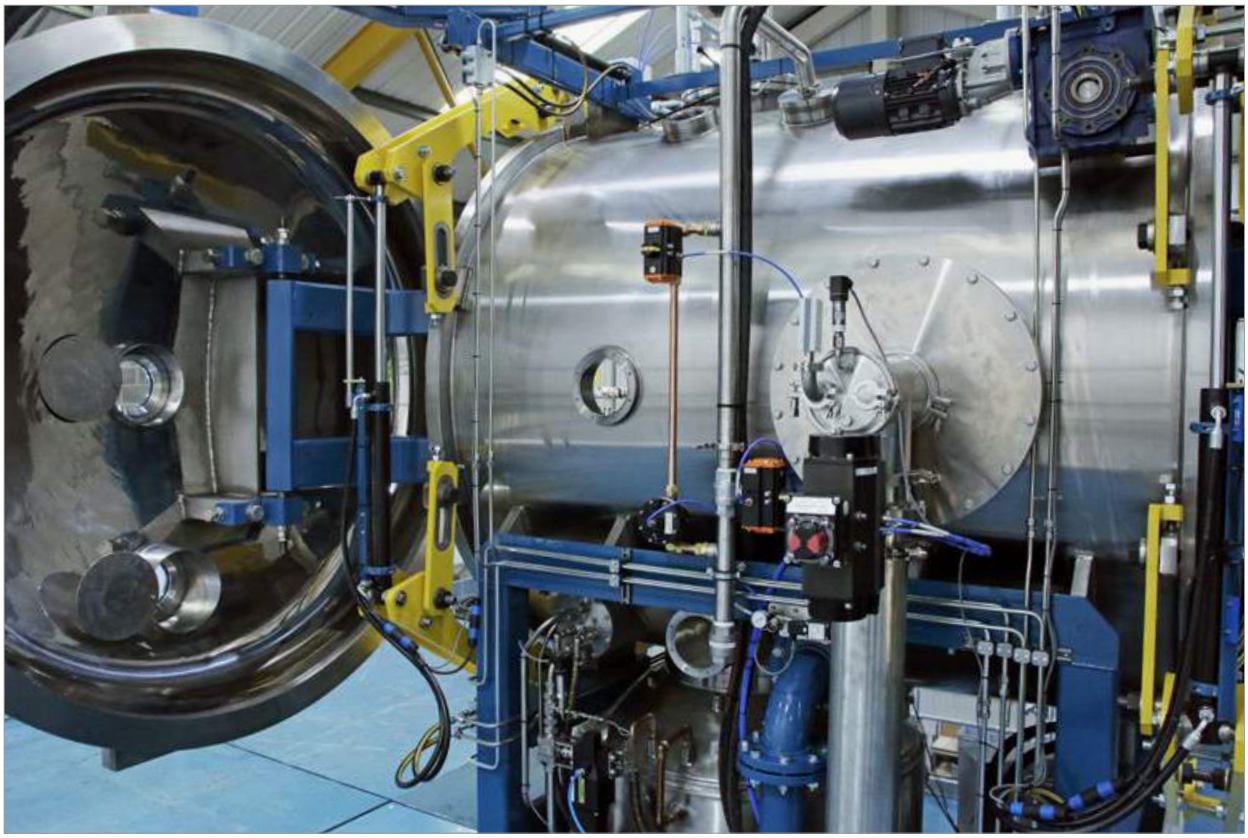

Fig. 3 Rapid cooling of melt chamber for tundish refurbishment

Atomiser development

PSI's first atomiser, the Hermiga Mini (Fig. 1), produced a few kilograms of powder and was intended to be used by universities and in the research departments of engineering companies for R&D into fine powder applications. "A sales visit to Japan had uncovered a market demand for these R&D atomisers to be accommodated within the four metre repeat distance between floors in a typical office block, because factory floor space in Japan was so expensive due to the high price of land," stated Hopkins. "The challenge, therefore, was to build an atomiser that would fit a small office space. Typically, atomisers need to be tall to give the powder particles plenty of time to cool down as they descend downwards in the process chamber. In addressing this challenge, the first generation of Hermiga Minis was born."

The standard Hermiga range of vacuum/inert gas atomisers has developed over the years and now includes production-scale units capable of outputs of over 2,000 tonnes per year, whilst still retaining the performance and quality capabilities of the smaller R&D and pilot-scale atomisers (Fig. 2). Now that these capabilities are considered standard throughout the range, recent development activity at PSI has been focused on the capital expenditure and operational expenditure considerations of the company's atomiser users.

"Having satisfied themselves of the powder quality characteristics, many companies are understandably concerned about their return on investment and the unit cost of 'in-size' powder," explained Hopkins. "Detailed engineering discussions are thus carried out to ensure that PSI can achieve the 'sweet spot' in atomiser specification to match the customer's downstream production requirements." Over the last decade PSI has therefore devoted much of its resources to the development of features designed to achieve these objectives.

"Detailed engineering discussions are carried out to ensure that PSI can achieve the 'sweet spot' in atomiser specification to match the customer's downstream production requirements"

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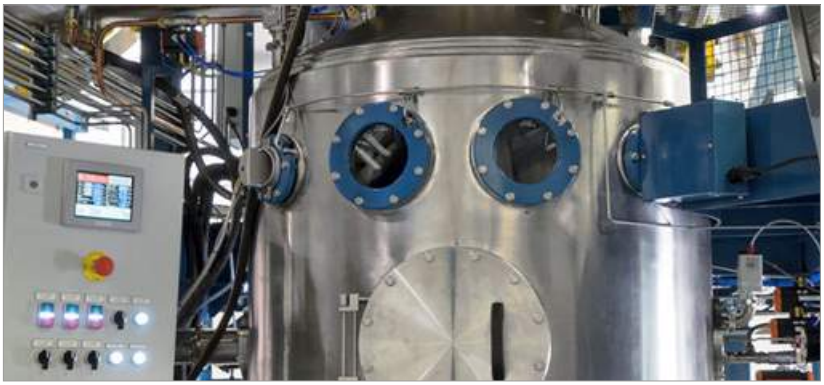

Fig. 4 A close-up view of the production-scale Hermiga system

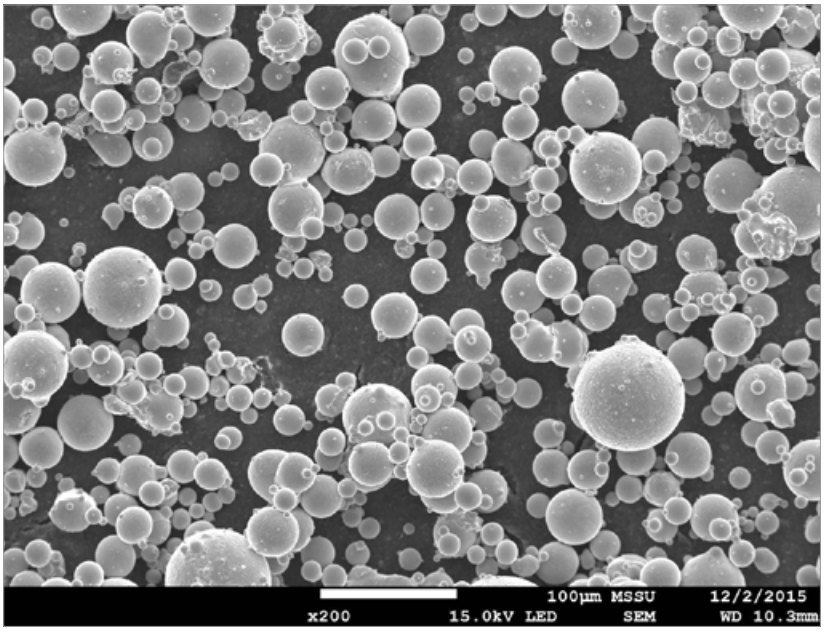

Fig. 5 An SEM of Inconel 718 powder manufactured by MPP. IN718 is a nickel based heat resistant alloy that offers good tensile, fatigue, creep and rupture strength at temperatures up to 700°C

The use of heated atomising gas

When the atomising gas is heated to, for example, 500°C, median particle size may be reduced from 20 µm to 15 µm in a steel powder. This may be of no great import to a manufacturer of powder intended for Additive Manufacturing or HIP, but it transforms the economics for a MIM powder producer.

Fast turnaround

"Most atomisers are still built for batch production, the comparison being ingot production of steel compared to Concast," stated Hopkins. "Considering that the atomising stage of the machine cycle of load-melt preparation – atomise – cool down may only represent 25% of the total cycle time, it is essential to turn the unit around quickly."

To this end, systems have been developed to ensure both a fast change of consumable refractories in tundish design and the rapid cooling of collected powder and chamber internals to allow operator access to unload product and prepare the next melt (Fig. 3).

Fast cleaning

The ability to quickly and thoroughly clean the complete system between compositional changes is essential when a company is looking to get maximum value from its investment. Rapid access to the atomising and melt chambers in the Hermiga units is facilitated by a hydraulic clamping system.

Tandem melting systems

The objective of tandem melting systems is to keep the capital intensive part of the atomiser system fed with molten metal for the highest proportion of the duty cycle as is possible. The use of alternatively pouring melting furnaces whilst maintaining the vacuum and inert gas environment improves production economics markedly, stated Bill Hopkins.

Cold crucible titanium powder production

Over the last few years PSI has invested heavily in 'cold crucible' technology, meaning the ability to melt and atomise reactive metals such as titanium. Cold crucible or Induction Skull Melting (ISM) - the preparation of the melt in water-cooled copper crucibles - is technically complex but feasible, and several atomisers based on this technology are now in operation.

"We believe that the only viable method of producing quality titanium powder at a price to enable its use outside of aerospace and into the automotive and medical sectors is by the use of low-cost titanium feedstocks and cold crucible techniques to prepare and refine melts in preparation for atomisation," stated Bill Hopkins.

"In the past five years, Additive Manufacturing has grown at a rapid rate in the field of titanium and it has become apparent that CP and '6/4' grades of titanium will need modification to alloy compositions to meet the special solidification characteristics encountered in AM processes. To this end we have developed cold crucible equipment to enable the production of novel titanium alloys by the continuous casting and

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Alloy family	Specific alloys that are manufactured
Cobalt alloys	CoCr (Stellites), CoCrMo, CoCrNi
Copper alloys	Brazing alloys, CuAlFe, CuInGa, CuInGaSe, CuNi, CuSn, CuSnTi
Iron based alloys	FeCoCrNi, FeCr, FeCrNiMo, FeMn, FeCrNiNb
MCrAlY alloys	M = Co or Ni or Co,Ni
Nickel alloys	Fe64Ni36, INVAR, CuNi, NiCrAl, NiCrFe, NiCrSi
Nickel superalloys	IN625, IN690, IN718, Haynes 230
Precious metal alloys	Au, Ag, Pd, Pt
Steels	17-4PH (precipitation hardening), maraging steels

Table 1 A list that demonstrates the diversity of gas atomised powders that MPP produces. The majority of alloys listed are based on ASTM standards, however these can also be provided in accordance with other regional standards

extraction of circular billets. This meets the demand for meltstock of novel compositions that the primary titanium manufacturers are not currently ready to address."

A move into metal powder production

A sister company to PSI, Metal Powder and Process Ltd (MPP), was recently formed with the purpose of developing and producing novel powder alloys for demanding applications such as the Inconel 718 powder shown in Fig. 5. This is done in collaboration with industry partners in order to develop PM applications as far down the value chain as possible towards the finished product.

MPP benefits from its access to PSI's atomising technology, coupled with its thirty year history in metal powder production, including several hundred alloy compositions over the whole metallurgical spectrum, for a wide range of industry sectors. This places the company in a strong position to address world demand for new PM applications requiring high purity spherical metal powders. Table 1 lists a small selection of the gas atomised powders that MPP produces. Bill Hopkins stated, "We work closely with clients to develop and produce powders that meet their specific requirements, adding significant value to their business."

Industry collaboration

PSI is actively involved in a number of collaborative research projects that address the need to develop new advanced metal powders and innovative production processes, as well as building a knowledge base on how these powders perform in today's advanced applications.

Engineering Powders UK (ENGPOW)

PSI is the lead partner in a thirty month, £1.3 million, collaborative research programme to develop aluminium gas atomised powders via Vacuum Induction Melting (VIM) with novel surface coating/heat treatment technology. The technology will be used in the manufacture of high-value aluminium and magnesium aerospace parts. Powders will additionally be modified/optimised so that they are more suitable for Additive Manufacturing processes.

PSI's Business Development Manager, Dr Gordon Kerr, stated, "Research into similar powders has recently started in the USA and many technical challenges remain. The powders must be further developed and proven, and a reliable commercial supply established. In this project, the UK has the chance to establish a global lead in engineered powders for a number of downstream processes."

"With Innovate UK investment, PSI will take advantage of its thirty years' experience in powders to position itself as a global supplier of advanced thermally treated powders." PSI is partnering with BAE Systems, TWI and Alphatek Hyperformance Coatings Ltd. BAE Systems estimates the project outcomes could save the UK taxpayer >£100 million per annum after eight years.

MAPP: bridging the gaps in current knowledge

Manufacture using Advanced Powder Processes (MAPP) is a new £20 million project funded by the Engineering and Physical Science Research Council (EPSRC) within its Future Manufacturing Hub. MPP and PSI have been selected as one of the seventeen industrial partners in this programme.

Dr Kerr stated, "Recent advances in powder based materials and processes are amongst the most exciting we have ever witnessed in manufacturing and such technologies are at the forefront of a new industrial revolution. Yet challenges remain to realising their full potential: problems associated with emerging manufacturing processes and new materials often only become evident as we move from the lab scale to pre-production. The underpinning fundamental material science research is very often conducted separately to the manufacturing process research. MAPP will bridge this gap and address this challenge."

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A focused and collaborative research agenda on emerging powder based manufacturing technologies including Spark Plasma Sintering, Freeze Casting, Inkjet Printing, Spray Forming, and Laser, Electron Beam and Indirect Additive Manufacturing will be undertaken. The goal of the project is to deliver on the promise of powder-based manufacturing processes to provide low energy, low cost and low waste high value manufacturing routes and products to secure UK manufacturing productivity and growth.

Research will cover a wide range of engineering materials where powder processing has the clear potential to drive disruptive growth – including advanced ceramics, polymers, alloys and materials for energy storage – but where common problems must be addressed. The problems to be addressed include the dynamic nature of the conditions these processes impose during manufacture makes their control challenging; the influence of process conditions on the particulate feed-stocks and their morphology is not understood; the variability of behaviours that emerge as a result of the interplay between dynamic process conditions and highly active powders.

PSI and TiPOW

PSI is also a key partner in a three year, £3.1 million, collaborative research programme to develop titanium powder specifically formulated and blended to meet the needs of AM aerospace component producers.

The programme, called TiPOW (Titanium Powder for Net-Shape Component Manufacture), involves developing the techniques and equipment to produce the powder consistently, in quantity and at a lower price than currently available in the material supply chain. A variety of low cost materials are being investigated as potential sources of feedstock.

The TiPOW programme is backed by the UK's Aerospace Technology Institute (ATI) and the country's innovation agency, Innovate UK. Consortium partners include GKN Aerospace, Metalysis and the University of Leeds. As programme leader, GKN's Aerospace business will also draw on the expertise of GKN Powder Metallurgy, a world-leading supplier of metal powders and precision engineered components.

Although the inspiration for the programme was low-cost Ti 6/4 grades, the technology will apply equally to more complex titanium alloys and the clean melting techniques will be of interest to users of superalloy powders, nickel-titanium shape memory alloys and indeed any metal powders which are required to be ceramic inclusion-free.

Outlook

Over its thirty year history, PSI has always held the belief that to fail to innovate is to perish in a world of ever faster technology dissemination. Its answer has been to devote much of its resources as possible to R&D - whether internally, single-client or collaboratively, in the UK or internationally.

"Many companies throughout the world are investigating different technologies to produce titanium powders from a variety of different feedstock inputs. The source of these inputs is varied and comes from a number of different production processes. We have shown that the cold crucible processing route has a refining effect with regards to removing unwanted impurities in the feedstock materials."

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"We have shown that the cold crucible processing route has a refining effect with regards to removing unwanted impurities in the feedstock materials"

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POWDERMET2017: Advances in the processing of 430L stainless steel, Ti6Al4V and nickel-base superalloy CM247LC

Two sessions at the POWDERMET2017 International Conference on Powder Metallurgy & Particulate Materials, Las Vegas, USA, June 13-16, 2017, focused on developments in materials processed by Metal Injection Moulding. In the following report Dr David Whittaker reviews three papers that cover advances in the processing and properties of MIM 430L, the microstructural control of MIM Ti6Al4V and the influence of heat treatment on MIM nickel-base superalloy CM247LC.

Processing and properties of MIM 430L(Nb) made by pre-alloy and master alloy routes

A paper from Martin Kearns, Keith Murray, Paul Davies and Mary Kate Johnson (Sandvik Osprey Ltd, UK) and Viacheslav Ryabinin and Erainy Gonzalez (TCK S.A., Dominican Republic) reported on the processing and properties of MIM 430L(Nb) made by the pre-alloy (PA) and master alloy (MA) routes [1].

These authors have published a series of papers on the potential advantages of adopting a master alloy approach in combination with carbonyl iron powder (CIP) as opposed to a pre-alloy powder route. Previous studies relating to low alloy steels and 420 and 440C stainless steels had shown that the MA route offered benefits in terms of higher sintered density and UTS and much reduced MIM part distortion, compared with parts made with PA powders.

This new study had pursued similar comparisons for the ferritic stainless steel 430L with and without a Nb addition, with the objectives of characterising the materials' sintering behaviour in nitrogen and hydrogen atmospheres and examining the relationships between microstructure and mechanical and corrosion properties.

430L stainless steel is an alloy that combines good corrosion resistance with good formability and ductility in the wrought form. It is a ferritic,

Fig. 1 Delegates at the POWDERMET2017 opening session (Courtesy MPIF)

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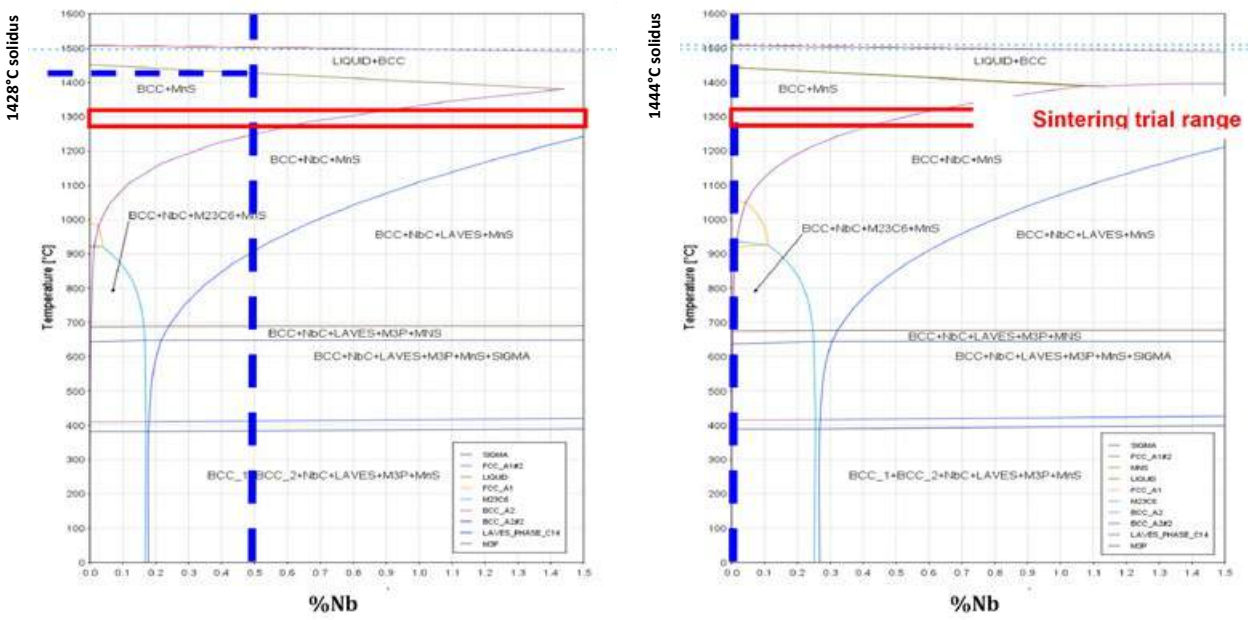

Fig. 2 Phase stability in 430L showing the influence of Nb level. Red construction lines relate to the reported study [1]

non-hardenable stainless steel with excellent surface finish and resistance to acidic and atmospheric corrosion in particular. Like most ferritic stainless steels, 430L is not susceptible to chloride-induced stress corrosion cracking and, at elevated temperature, it forms an adherent oxide scale that provides protection up to 800°C in dry air. It is therefore also used in the automotive industry for exhaust systems.

Slow cooling of stainless steels in the range 500-800°C can lead to formation of chromium carbides at grain boundaries. To combat this problem of sensitisation in stainless steels, high temperature carbide/nitride formers, such as Ti or Nb, are added in levels that are in excess relative to C and N in order to fix these elements in the form of carbo-nitrides. In the nitrogen atomised powders used in this study, the nitrogen level is far higher than the carbon level and the combination of N and Nb is recognised as enhancing creep resistance of stainless steels.

The magnetic properties of 430L are attractive for use in the construction of electric motors (armatures, pole pieces) and magnetic sensors. Magnetic power losses are dependent on the grain structure and state of recrystallisation of the microstructure. The ability to control grain size with the use of Nb additions gives 430L(Nb) soft magnetic properties that are of interest in the development of electromagnetic injection valves. Improving response time of fuel injection valves is a goal for the automotive industry in order to enhance engine efficiency and limit harmful emissions.

The choice of sintering temperatures in the reported study was guided by Thermocalc studies on the final alloy chemical compositions, with and without Nb. Fig. 2 shows the stability fields of different phases in each alloy as a function of temperature and Nb content. This indicates that, as the % Nb increases, the liquidus temperature decreases. It is known, however, that the activity of Nb is affected by the presence of oxygen and that a small amount of oxygen is picked up during inert gas atomising. The presence of oxygen reduces the activity of Nb leading to a small rise in liquidus temperature.

Two pre-alloy and two master alloy powder lots were produced for the study using Sandvik Osprey's proprietary inert gas atomisation process using nitrogen gas. Each of the as-atomised powders was air

Material	Lot #	Fe	Cr	Mn	Si	Nb	P	C	S	O	N
430L	16D1530	Bal	17.1	0.62	0.60	-	0.010	0.019	0.005	0.087	0.144
430L + Nb	16D1531	Bal	17.0	0.69	0.66	0.50	0.012	0.016	0.003	0.086	0.159
Fe50(LC)	16D1529	Bal	49.9	1.70	1.60	-	-	0.006	0.006	0.185	0.245
Fe50Cr4.5Nb	16D1951	Bal	50.2	0.92	1.46	4.56	0.019	0.019	0.009	0.166	0.093
CIP Grade BC	1063	Bal	-	-	-	-	-	0.033	0.001	0.154	0.003

Table 1 Chemical analyses of powders used in the reported study [1]

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Alloy	Fe	Cr	Mn	Si	Nb	P	C	S	O	N	X
430L PA	Bal	17.1	0.62	0.60	-	0.010	0.019	0.005	0.087	0.144	1.4/0
430L+Nb PA	Bal	17.03	0.69	0.66	0.5	0.012	0.016	0.003	0.086	0.159	1.5/0.5
430L MA	Bal	16.62	0.57	0.53	-	N/A	0.024	0.002	0.164	0.08	0.9/0
430L+Nb MA	Bal	16.66	0.46	0.51	0.61	0.003	0.025	0.003	0.162	0.06	0.76/0.61

Table 2 Chemical analyses of feedstock batches used in the study [1]

classified to produce a particle size distribution of 90%-22 µm, a distribution typically favoured by MIM parts makers. The chemical composition of each powder batch is shown in Table 1. Note that the MA powders, with higher Cr, show elevated oxygen levels.

Table 2 gives the chemical analyses of the four feedstock batches made from these input lots. The MA-based feedstocks were combined with CIP to achieve the final target composition shown. Included in Table 2 is a column headed X which shows an index of the degree of stabilisation of the alloy. The ratio X = (0.08+ 8x(%C +%N))/%Nb and, if this is less than 1.0, the alloy is stabilised, but, if X >1.0, the alloy is prone to sensitisation. It should be noted that, by this measure, none of the starting powders was fully stabilised.

Table 3 shows the particle size distributions of the different feedstock lots and their corresponding melt flow indices. Feedstock batches were produced by TCK using its proprietary binder formulation and aiming for a 17.4% shrinkage factor (the scale factor applied to the target final part dimensions in order to design the mould).

Alloy	Melt Flow Index	D_{10} µm	D_{50} µm	D_{90} µm
430L PA	477 g/10 min	5.04	12.05	21.41
430L+Nb PA	367 g/10 min	3.91	10.02	21.14
430L MA	187 g/10 min	3.22	7.59	16.31
430L+Nb MA	200 g/10 min	3.20	7.50	16.27

Table 3 Particle size distributions and MFIs of different feedstock batches [1]

The feedstocks were moulded to produce standard MIMA tensile and Charpy test specimens. Moulded green parts were subjected to an initial solvent debind followed by a thermal debind at 500°C for 3.5 h and were sintered at 1280-1320°C in a nitrogen or hydrogen atmosphere using the following cycle: 2°C /min ramp to 1000°C, 60 min hold, 2°C/min ramp to hold temperature, 60 min hold, furnace cool. Charpy bars were suspended both cantilever style (20 mm overhang) and across refractory supports (38 mm separation) in order to determine the extent of distortion as a function of powder make-up, chemical composition and sintering temperature.

As-sintered parts were tested to determine density, tensile properties and hardness. Table 4 shows the results from the sintering trials conducted with a N_2 sintering atmosphere. Results show that the density of parts sintered in N_2 at 1280°C is lower than that achieved at 1320°C and that, at an equivalent temperature, the PA variants achieve a higher density than the MA samples. All of the variants, however, showed good density levels (in excess of 97%) and the mechanical properties achieved were higher than the average values in previously published

Alloy	Sinter Temp,°C	Batch #	UTS MPa	0.2%PS MPa	%El	Density g/cm³	Density %TD	%C	%N	Delta %C Delta %N	VHN 10kg
430L PA	1280	16D1530	468	376	1.4	7.47	97.9	0.01	n/a	0.0, 0.0	209
	1320		444	362	1.5	7.51	98.4	0.005	0.14	-0.014, 0.0	179
430LNb PA	1280	16D1531	296	-	-	7.49	98.0	0.015	n/a	0.0, 0.0	180
	1320		430	303	1.5	7.58	99.1	0.001	0.17	-0.014, 0.0	165
430L MA	1280	16D1529	517	339	2.5	7.53	97.2	0.015	n/a	0.01,0.015	181
	1320		483	306	3.5	7.56	97.6	0.005	0.10	0.0, 0.015	170
430LNb MA	1280	16D1951	n/a	n/a	n/a	7.30	94.2	0.01	n/a	0.02, ?	163
	1320		n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a

Table 4 Properties of nitrogen-sintered (as-sintered) samples, density by pycnometry, %C & N as per finished parts [1]

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Alloy	Batch #	UTS, MPa	0.2%PS MPa	%El	Density g/cm³	Density %TD	%C	%N	Delta %C Delta %N	VHN 10 kg	HRB
430L PA	16D1530	398	245	22	7.58	99.3	0.00	N/A	0.02, ?	129	49
430LNbPA	16D1531	459	250	6	7.65	100.0	0.01	N/A	0.01, ?	140	67
430L MA	16D1529	381	212	19	7.68	99.0	0.01	N/A	0.02,?	134	55
430LNb MA	16D1951	414	236	11	7.71	99.5	0.01	N/A	0.02 ,?	140	65

Table 5 Tensile and hardness data for samples sintered at 1280°C in H_2 [1]

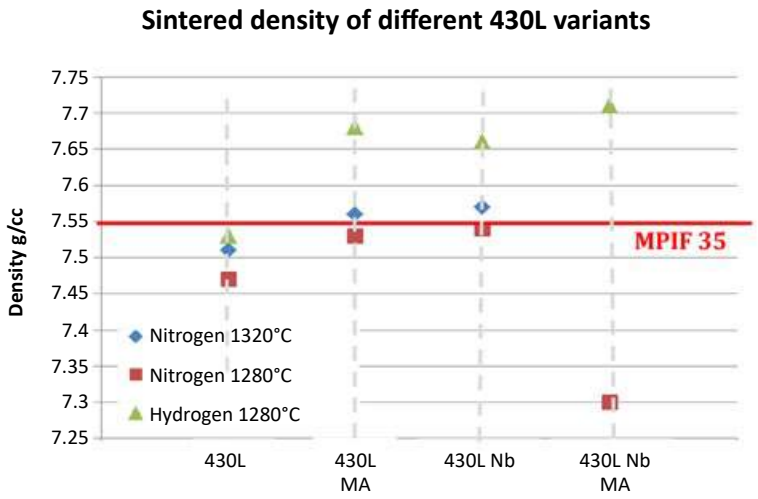

Fig. 3 As-sintered densities of different feedstock types vs sintering temperature and atmosphere [1]

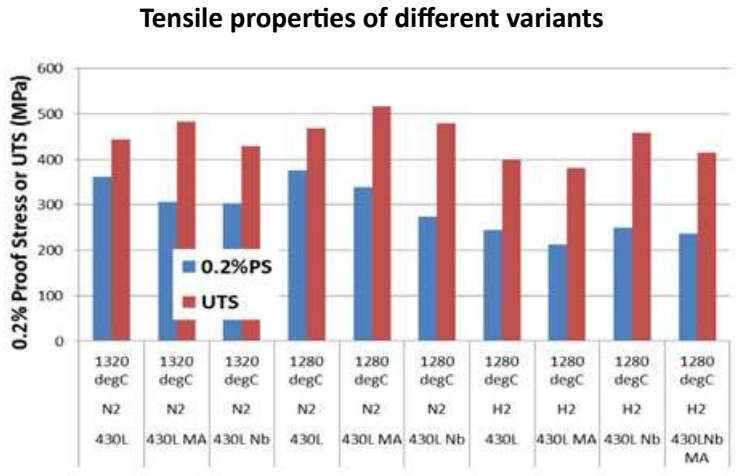

Fig. 4 Tensile properties of different 430L variants as a function of sintering temperature and atmosphere [1]

data for MIM-processed 430L. The UTS values may be compromised by the relatively low elongation to failure values, which were believed to be traceable to surface defects introduced during moulding/debinding. The proof strength levels achieved in the PA samples were higher than those achieved in the MA variants, although this appears to be offset by the higher ductility seen in the MA samples.

Table 5 shows corresponding data from samples sintered at 1280°C in H_2. In all cases, higher density was achieved, reaching full density for the 430LNb PA material. The proof strength levels achieved were significantly lower than the values achieved by sintering in N_2. Fig. 3 shows the trend in density for the four different feedstock types as a function of sintering temperature and sintering atmosphere. It is apparent that the alloys containing Nb achieved higher density, irrespective of process temperature and atmosphere, and that MA was superior to PA in this respect. It is also evident that sintering at higher temperature and using a hydrogen sintering atmosphere is advantageous in achieving the highest density.

Fig. 3 indicates that the MPIF 35 standard of 7.55 g/cm³ is not quite achieved with the 430L PA variant when sintered in N_2 at 1320°C, but comes close when sintered in hydrogen at 1280°C. Other variants readily exceed the threshold density when sintered in hydrogen at the same temperature. It is notable that the Nb addition to the PA variant appears to enhance densification quite markedly in hydrogen and this may be in part related to a reduction in the solidus temperature (Fig. 2).

Metallographic analysis indicated that, after sintering in H_2 at 1280°C, 430L PA showed marked intra-granular porosity compared with the MA variant and exhibited a coarser grain size. The 430L(Nb) variants displayed a similar trend in porosity between the PA and MA variants and also exhibited a darker etching microstructure with extensive fine intra-granular precipitation of Nb(C,N), which is absent in 430L. In 430L(Nb) MA, there was some evidence of a precipitate-free-zone

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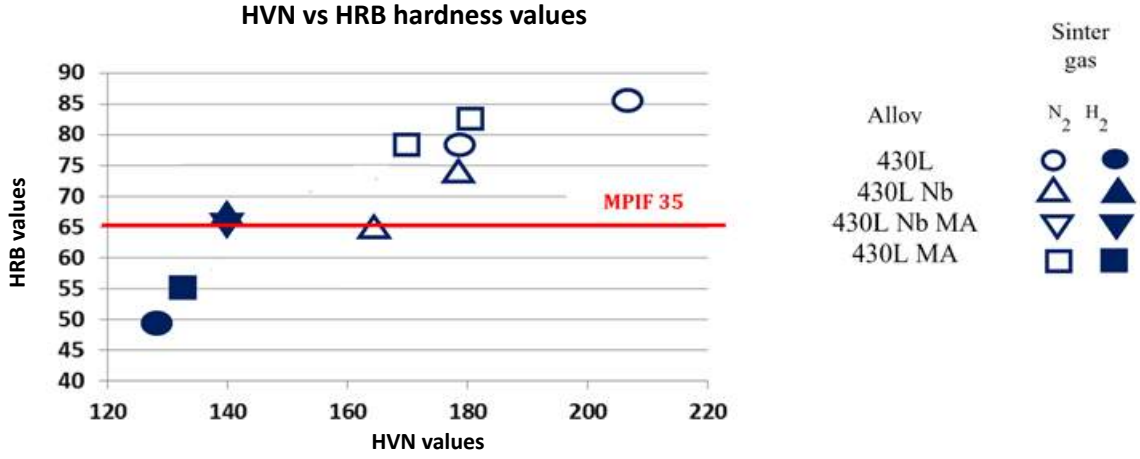

Fig. 5 Comparison of HVN and HRB hardness values measured on samples produced under different conditions [1]

(PFZ) around grain boundaries. The grain size of the PA and MA materials appeared to be similar in this instance. For samples sintered in N_2, 430L(Nb) MA had an inhomogeneous microstructure with concentrations of dark-etching material distributed in the microstructure. These were sites of prior master alloy particles that were still rich in Cr and Nb and were then preferentially nitrided during sintering. This was in contrast to the same material system when sintered in H_2 and confirmed the view that reduction of the stable oxide on the MA surface was a necessary precursor to inter-diffusion, sintering and densification.

The histogram in Fig. 4 compares the proof and ultimate tensile strengths of the different variants and it is clear that the N_2-sintered materials to the left of the figure display higher strength levels than the H_2-sintered materials to the right. This is clearly not relatable to differences in porosity, which would tend to enhance the H_2-sintered materials relative to N_2-sintered, but is believed to stem from nitride formation during sintering. Tables 4 and 5 show that carbon loss during sintering is small in all cases and the nitrogen level is stable when sintering in N_2. However, significant loss of nitrogen occurs when sintering in H_2, leading to lower mechanical properties.

The hardnesses of as-sintered materials (Rockwell B) and failed tensile test-pieces (HVN) were measured. Fig. 5 plots the different sets of hardness figures and there is a clear relationship between the two. It can be noted that, of the H_2-sintered variants, only the Nb-containing alloys can achieve the MPIF 35 standard of 65 HRB. Conversely, all of the N_2 -sintered variants meet or exceed this threshold.

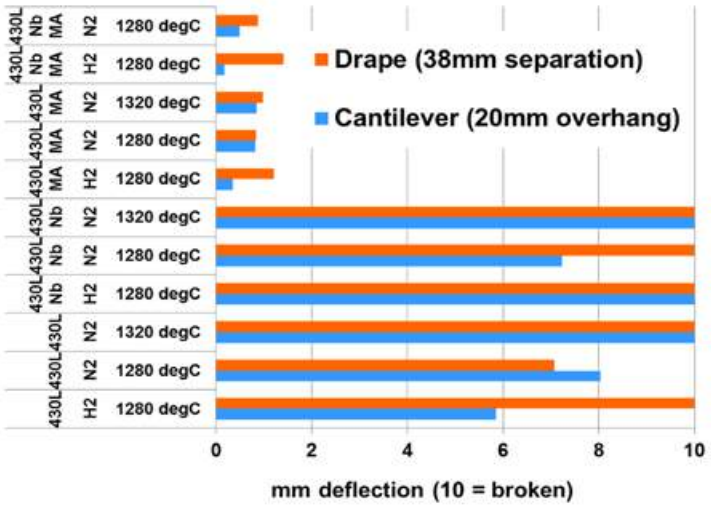

Fig. 6 Deflection of Drape and Cantilever Charpy specimens under different sintering conditions [1]

Fig. 6 shows the effect of different process variables on the distortions measured on cantilevered and suspended Charpy bar specimens. Maximum deflections were measured at the mid-point of draped bars and the tips of the cantilever specimens. It is apparent that the MA samples showed modest drape deflection of ~1 mm, while PA equivalents typically failed under the same conditions.

Salt spray corrosion testing showed early onset pitting on all variants sintered in N_2. Those with higher porosity showed more pitting and those containing Nb appeared no better than those without. It remains to be seen whether samples sintered in H_2 will show improved performance without the presence of Cr nitrides.

The grain size of 430L sintered in H_2 was finer when made by the MA route and it was notable that higher sintering temperature led to grain coarsening and more pronounced Precipitate-Free Zones (PFZs)

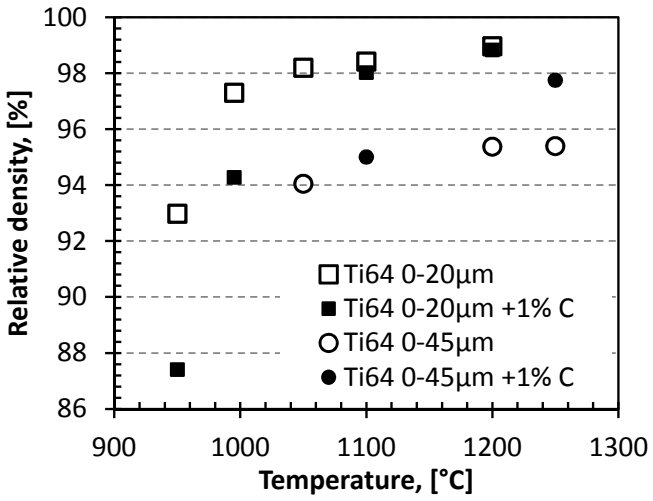

Fig. 7 The effect of sintering temperature on the relative density obtained with the various feedstocks [2]

adjacent to grain boundaries. Finest grain sizes were seen in N_2-sintered 430L(Nb) MA samples and coarsest in H_2-sintered 430L PA. The effect of grain size on hardness has been explored and can be defined by a Hall-Petch relationship.

Microstructure of MIM Ti6Al4V produced using fine metal powders

The second paper in this review, authored by Roger Pelletier and Louis-Philippe Lefebvre (National Research Council, Canada) and Jonathan Allaire (Advanced Powders and Coatings, Inc., Canada), turned attention to the MIM processing of the titanium alloy Ti6Al4V [2].

Titanium alloys possess a variety of attractive characteristics such as low density, high specific strength and excellent biocompatibility. Ti6Al4V is one of the most widely used titanium alloys, because of its remarkable mechanical properties that can be tailored for various applications by mechanical working and/or heat treatments.

Two of the key aspects that control the mechanical properties of MIM Ti6Al4V are its microstructure and its final density. In order to achieve adequate density with MIM processing, Ti6Al4V, which is an alpha-beta titanium alloy, has typically to be sintered between 1200 and 1350°C, well above the β-transus temperature (≈1000°C), leading to a coarse lamellar structure with reduced mechanical properties compared to its wrought equivalent. Therefore, refining the microstructure is a key to improved MIM Ti6Al4V mechanical properties. As one potential means of achieving this desired microstructural refinement, the reported study presented the effects of using a fine Ti6Al4V powder and a small carbon addition.

"For temperatures between 995°C and 1200°C, the use of a fine powder leads to a gain of around 2% in relative density. Relative densities close to 99% were achieved at 1200°C"

The metal powders used in the study were -45 µm and -20 µm pre-alloyed (PA) Ti6Al4V powders, produced by plasma atomisation by AP&C. The -45 µm powder is a typical MIM particle size and the -20 µm powder is considered to have a fine size. To produce Ti6Al4V reinforced with titanium carbides, a fine (-13 µm) graphite powder was added to the feedstock. The feedstocks used in this study had a wax-polymer binder and were prepared at a solid loading of 0.69.

Injection moulded specimens were solvent debound for 7 h in hexane at 60°C and then thermally debound up to 800°C in a box furnace under a purified argon atmosphere. Sintering was performed in a vacuum furnace under high vacuum (≈10^{-3} Pa).

Achieved density levels were reported as relative densities in comparison with the theoretical densities of the materials; 4.43 g/ cm^3 for Ti6Al4V and 4.45g/cm^3 for Ti6Al4V-1%C. A complete reaction between the carbon and the titanium to form titanium carbide (TiC) was assumed.

The effect of sintering temperature on relative density for materials made from fine and typical sized powder with and without a 1% carbon addition is shown in Fig. 7. For temperatures between 995°C and 1200°C, the use of a fine powder leads to a gain of around 2% in relative density. Relative densities close to 99% were achieved at 1200°C. The densification of the fine powder remained very good even at temperatures slightly below the beta-transus temperature (≈1000°C). On the other hand, the positive impact of a 1% carbon addition, observed with the typical powder and for a sintering temperature of 1250°C, is not observed with the fine powder. Nevertheless, at these relative density

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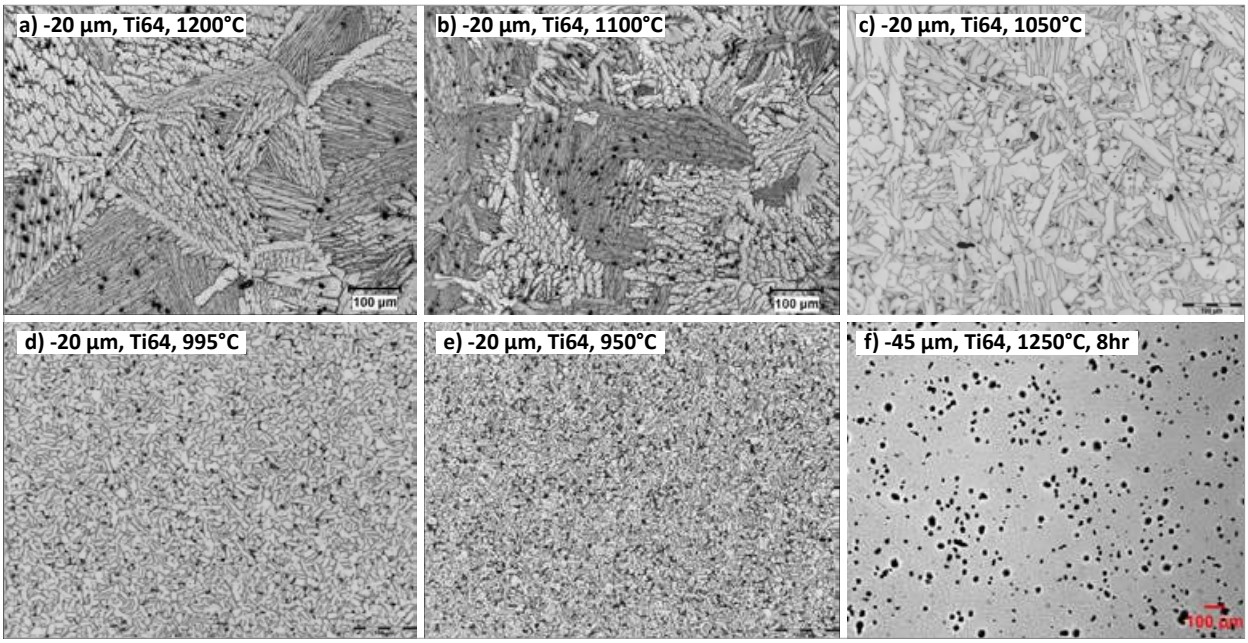

Fig. 8 Effect of sintering temperature on the microstructure obtained with a fine powder a) 1200°C, b) 1100°C, c) 1050°C, d) 995°C, e) 950°C and f) -45µm at 1250°C [2]

levels, it would be anticipated that the material made with fine powder and sintered at 995°C (and potentially also at 950°C) could be HIPed successfully. This opens the possibility of producing MIM Ti6Al4V parts fully dense without exposing them to temperatures above the beta-transus.

When using the fine powder, for sintering temperatures of 1100°C and above, it was found that the microstructure was typical of MIM Ti6Al4V, i.e. a relatively coarse microstructure composed of long acicular α-Ti grains surrounded by a thin layer of β-Ti. Residual pores resulting from incomplete densification could also be observed. For a sintering temperature around 1050°C, the microstructure began to change, as large α-Ti colonies were no longer observed (Fig. 8). For sintering temperatures close to or below the beta-transus temperature (≈1000°C), the microstructure became much finer (Fig. 9) . The α-Ti grains had a more nodular shape. The presence of some α-Ti grains during the last stage of sintering and the slower diffusion kinetics prevented the excessive growth of the β-Ti grains, leading to this significant change in microstructure.

The materials containing the 1% carbon addition had a much more refined microstructure, still composed of α-Ti grains surrounded by β-Ti grains, but with the former tending to be globular rather than acicular. The microstructure also contained a large number of rounded titanium carbide secondary phases. These carbides were uniformly distributed and no evidence of clustering was visible. The primary purpose of forming such a low content of titanium carbides is to impede the growth of β-Ti grains and therefore to refine the microstructure. Although the carbide precipitates had a very positive impact in limiting β-Ti grain growth, they did not enhance densification. This is especially true for sintering temperatures close to or below the beta-transus temperature, where individual Ti6Al4V particles could still be observed. Titanium carbide precipitates are very efficient in limiting the growth of the β-Ti

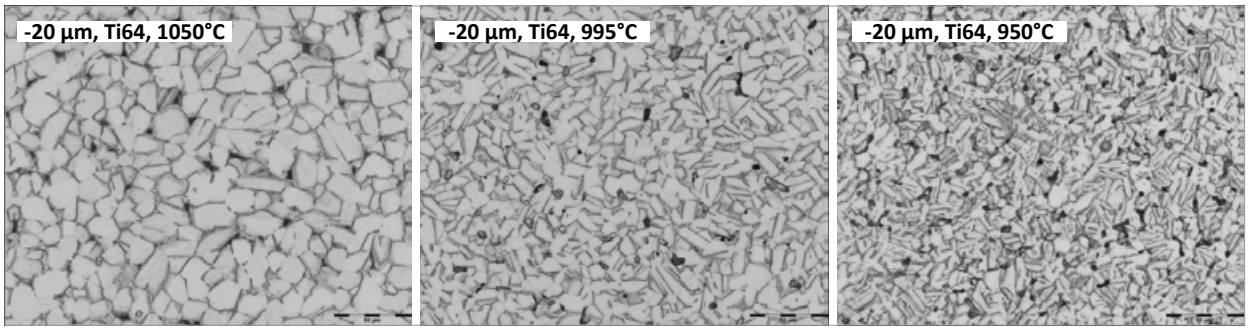

Fig. 9 Microstructures obtained with a fine powder when sintered at temperatures close to and below the beta-transus temperature a) 1050°C, b) 995°C and c) 950°C [2]

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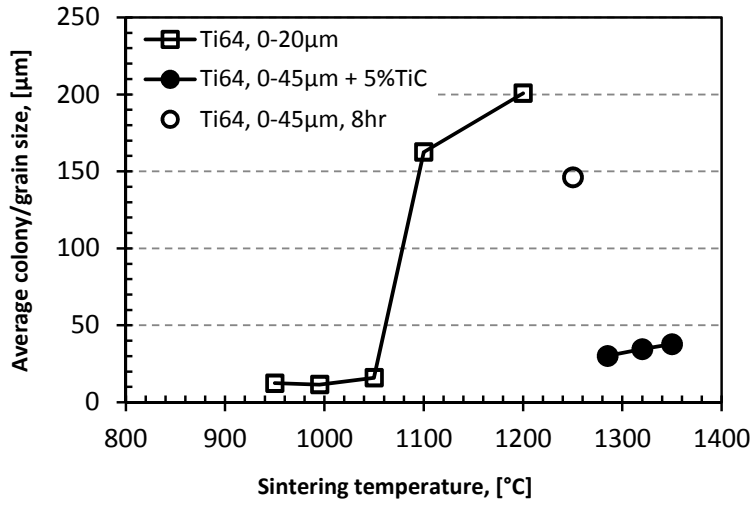

Fig. 10 Average α-Ti grain (colony) size obtained with various materials when sintered for 2 h. Results obtained with the -45 µm powder when sintered for 8 h is the reference condition [2]

grains, even at very high temperatures. In the microstructure obtained with a -45 µm powder when sintered for 2 h at 1320°C, only a fraction of the α-Ti grains were elongated and colonies were composed of, at most, a few α-Ti grains.

The average grain size of some of the samples was evaluated to illustrate the impact of using a fine powder and of adding 1% of carbon (or directly TiC) (Fig. 10). The size of α-Ti colonies for the reference condition (-45 µm powder sintered for 8 h) has been added for comparison. For the fine powder, it can be observed that the average grain size remained very small, up to a sintering temperature between 1050 and 1100°C. Below this transition temperature, the grain size was very close to the original D_{50} of the powder. At higher temperatures, the average grain size, or more accurately the average α-Ti colony size, increased very rapidly and surpassed that obtained with the coarser (-45 µm) powder. By comparing with the density results (Fig. 7), it can be seen that a good level of densification was reached before the size of the α-Ti colonies began to grow excessively. The addition of 1% of carbon, or 5% of TiC, in the feedstock was very efficient in keeping the α-Ti colonies small. The data shown (filled circles) demonstrate that they only marginally increased in size as compared with the original Ti6Al4V particles.

The titanium carbide grains had a similar final morphology, whether they resulted from the reaction between carbon particles and the titanium matrix or from the direct addition of titanium carbide in the feedstock. When a carbon addition was made to the feedstock, they appeared to form at relatively low temperatures by direct solid-solid reaction at the surface of the titanium particles. Fig. 11a shows an as-polished microstructure, in which the positions of the original titanium particles can be readily defined. Fig. 11b shows examples of titanium carbide grains, several of which are located at the sintering necks of adjacent titanium particles. Evidence of their presence in samples heated (debound) at 800°C cannot be observed metallographically in Fig. 11c, but XRD analyses performed on these samples suggested, however, that some titanium carbide had already formed.

Figs. 12 and 13 show the results of tensile testing of the materials based on the fine (-20 µm) powders sintered for 2 h at temperatures ranging from 950°C to 1250°C. On the basis of the high density and fine globular microstructure discussed earlier, it can be observed that the optimum levels of strength and ductility (above ASTM F1472) arose on sintering at 1050°C. Fig. 13 emphasises that, in relation to ductility, densification creates an increase of elongation with rising sintering temperature up to 1050°C, but, at higher sintering temperatures, coarsening of α colony size becomes the dominant microstructural influence that reduces elongation.

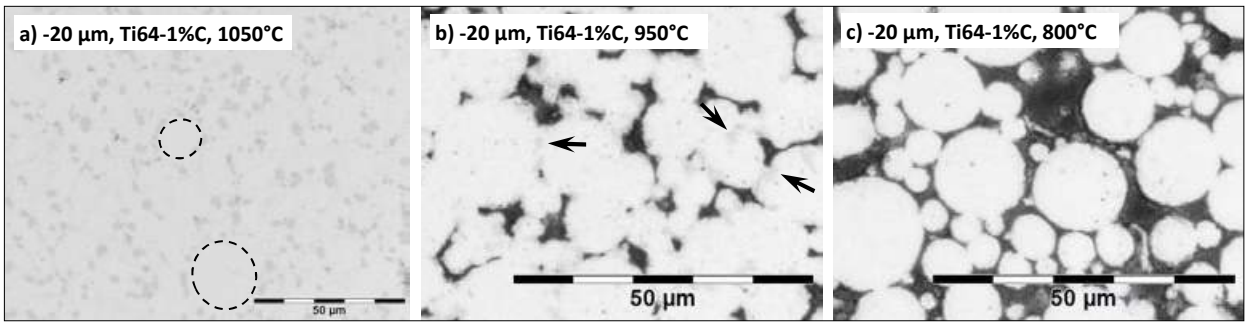

Fig. 11 Evolution of the microstructure as a function of temperature with respect to the formation of the titanium carbide grains on sintering at a) 1050°C, b) 950°C and c) 800°C [2]

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MIM of nickel-base superalloy CM247LC: Influence of heat treatment on microstructure and mechanical properties

The final paper in this review, presented by Andreas Meyer (Friedrich-Alexander Universitat, Germany) and co-authored by his colleague Robert Singer, Katharina Horke and Enrico Daenicke (Rolls-Royce Deutschland Ltd., Germany) and Sieglinde Muller and Ingolf Langer (Schunk Sintermetalltechnik GmbH, Germany), assessed the MIM processing of the nickel-base superalloy CM247LC [3].

Nickel-base superalloys offer excellent creep resistance, fatigue strength and corrosion and oxidation resistance and are consequently widely used for aerospace, power generation and automotive high temperature applications. Nickel-base CM247LC is a superalloy derived from the MAR-M-247 composition, specifically designed for directionally solidified (DS) blade and vane applications. It is a γ'-precipitation hardened superalloy with high aluminium and high refractory element (Ta, W, Mo) contents and demonstrates exceptional creep strength along with high oxidation resistance. The low carbon content improves the carbide microstructure, carbide stability and ductility.

Superalloys with high strength and high Al and Ti contents, such as CM247LC, cannot be forged and are not readily machined or cast. Therefore, the near-net-shape process of MIM is a promising alternative.

Over the past decade, the MIM capabilities of several nickel-base superalloys, such as Inconel 625, Inconel 713, Inconel 718, Nimonic 90, Udimet 720 and MAR-M-247, have been investigated. However, to date, little work has been published on the metal injection moulding of high strength/high Al and Ti content alloys, such as CM247LC. Previously reported studies of the material have shown promising results, i.e. good mechanical properties at room temperature as well as good high temperature oxidation behaviour. However, little information is available on high temperature mechanical properties including creep.

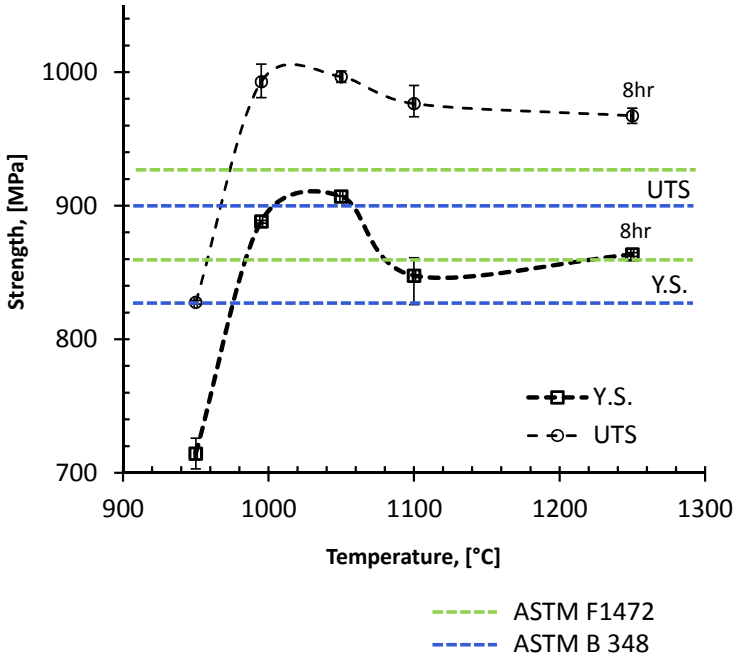

Fig. 12 The effect of sintering temperature on UTS and YS for -20 µm powders sintered for 2 h [2]

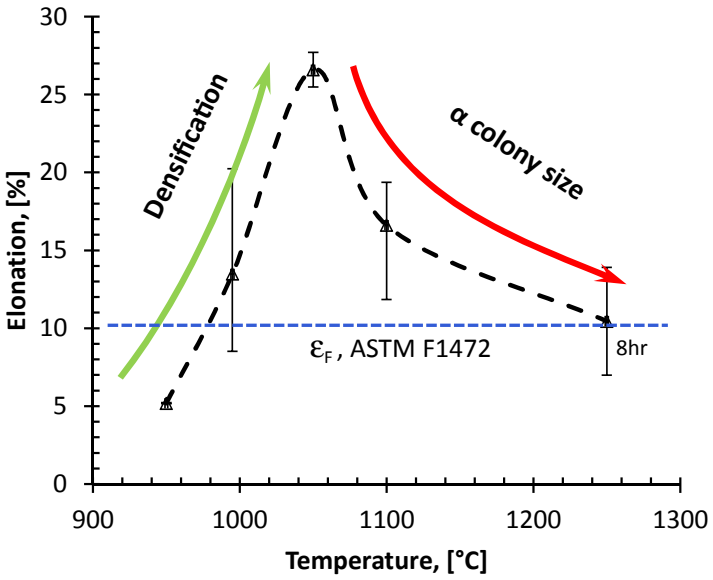

Fig. 13 The effect of sintering temperature on Tensile Elongation for -20 µm powders sintered for 2 h [2]

In this study, the influence of a heat treatment schedule, designed to improve the creep resistance of CM247LC produced by MIM, on microstructure and mechanical properties was presented.

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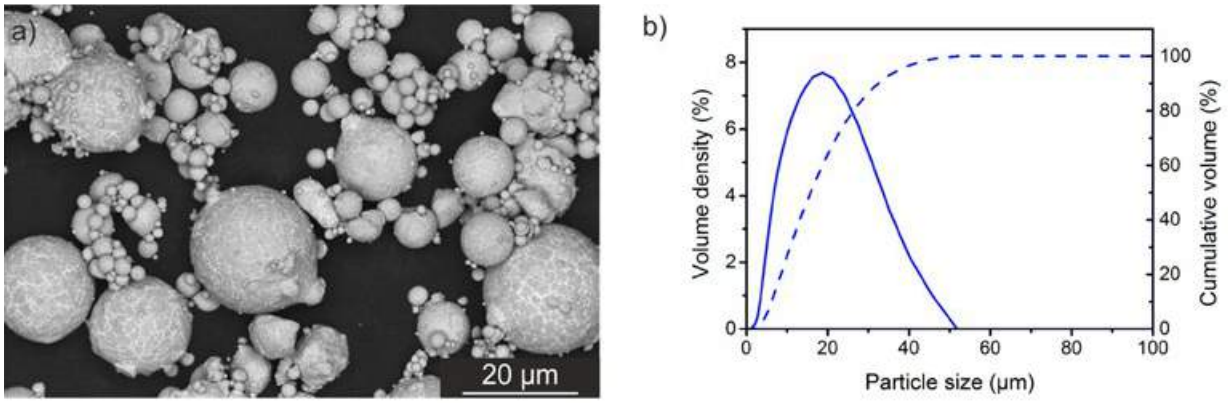

Fig. 14 a) SEM image of the CM247LC powder used for feedstock preparation, b) particle size distribution of the powder [3]

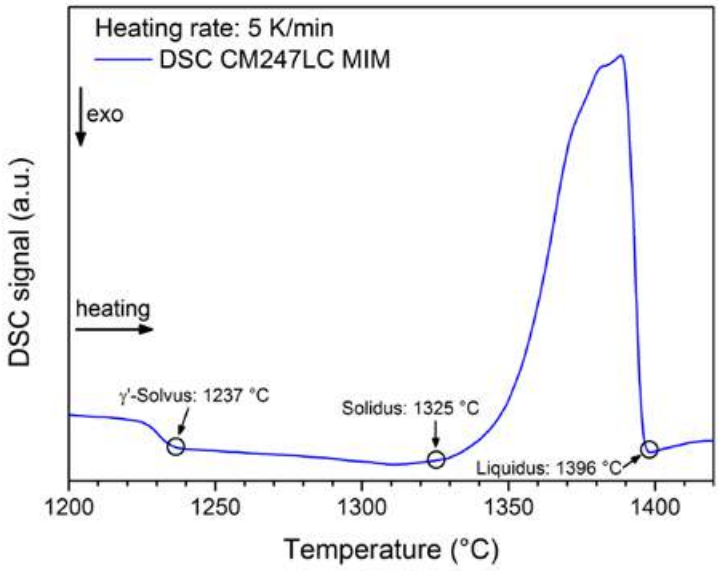

Fig. 15 DSC curve of as sintered CM247LC used for determining the phase transition temperatures of the alloy [3]

A pre-alloyed gas atomised CM247LC powder was used for feedstock preparation. The chemical composition of CM247LC, according to Cannon-Muskegon, is given in Table 6. Conventionally cast, hot isostatically pressed and heat treated CM247LC was used as reference material for tensile tests.

An SEM image, showing the powder morphology of the CM247LC powder, is given in Fig. 14a. The powder consisted mainly of spherical particles, though there were also some collided and irregularly shaped particles. Some small satellite particles were also present. The particle size distribution is shown in Fig. 14b. The D_{10}, D_{50}, D_{90} and D_{100} values are 6.2 µm, 16 µm, 33 µm and 52 µm, respectively. With reference to this particle morphology and size, the powder was considered to be well suited for processing by metal injection moulding.

In order to define the phase transition temperatures of the alloy and therefore find suitable heat treatment parameters, thermal characterisation of the material was performed by differential scanning calorimetry (DSC) measurements. The DSC results for as-sintered CM247LC are shown in Fig. 15. At 1237°C (2259°F), the peak for dissolution of the γ'-phase can be observed. The large endothermic peak is assigned to melting indicating a solidus temperature in the range from 1320°C to 1330°C (2408-2426°F) and a liquidus temperature of 1396°C (2545°F). Other phase transformations, such as carbide dissolution, were not observed. In order to guarantee complete γ'-dissolution without any incipient melting, 1260°C (2300°F) was chosen as the temperature for the solution heat treatment. The final two-step ageing was derived from Cannon Muskegon literature on the alloy CM247LC.

In the course of MIM processing, impurities, such as carbon and oxygen, may be picked up from the binder or the atmosphere during thermal debinding and sintering. For nickel-base superalloys produced by MIM, this impurity pick-up is generally undesirable, as it may be detrimental to mechanical properties and oxidation resistance. Carbon and oxygen are usually visible in the form of additional carbides and oxides, often at prior particle boundaries (PPBs).

The carbon, oxygen and nitrogen contents of the CM247LC powder, as sintered and heat treated samples are listed in Table 7. The carbon content of the sintered samples

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is 1332 ppm, corresponding to a pick-up of 750 ppm. The oxygen pick up is 300 ppm, leading to 552 ppm oxygen in the as-sintered condition. The heat treatment decreases the carbon content to about 1100 ppm, but increases the oxygen content further to 719 ppm. The nitrogen content of 190 ppm in the powder is slightly decreased by sintering and heat treatment. The measured pick-ups of carbon and oxygen are not considered to be sufficiently significant as to make the material unusable, but should ideally be reduced by further optimisation of thermal debinding and sintering.

Images of the as sintered CM247LC MIM microstructure are shown in Fig. 16. The amount of porosity, pore distribution and pore size can be seen in Fig. 16a. The overall porosity was 1.7%, while most of the pores had a size below 20 µm. The γ-γ'-microstructure can be seen in Fig. 16b. The γ'-particles within the grains had an almost cubic or somewhat irregular shape with a size of 0.5 to 1 µm. There were also some coarser blocky γ'-precipitates at the grain boundaries.

In addition, the microstructure of CM247LC MIM displayed many carbides and oxides. The carbides were Hf- and Ta-rich MC-carbides, while the oxides were mainly HfO_2. These carbides and oxides were precipitated within the grains, along the grain boundaries and often associated with pores. Preferred locations for the oxide and carbide particles were the prior particle boundaries, which were also the natural location for residual porosity.

Ni	Co	Cr	W	Al	Ta	Hf	Ti	Mo	B	C	Si	Zr
Bal.	9.2	8.1	9.5	5.6	3.2	1.4	0.7	0.5	0.015	0.07	< 0.03	0.015

Table 6 Chemical composition of the nickel-base alloy CM247LC in wt.% according to Cannon-Muskegon [3]

CM247LC	Carbon (ppm)	Oxygen (ppm)	Nitrogen (ppm)
powder	582 ± 1	251 ± 6	191 ± 26
as sintered	1332 ± 3	552 ±123	150 ± 5
heat treated	1090 ± 42	719 ± 149	133 ± 6

Table 7 Impurity contents of CM247LC powder, as sintered and heat treated samples [3]

The influence of the applied heat treatment of 20 h at 1300°C (2372°F) followed by 2 h at 1260°C (2300°F) and ageing on the microstructure was studied through electron backscatter diffraction (EBSD) maps. The average grain size in the as-sintered condition was 12 µm. The heat treatment led to grain growth and a resulting grain size of 21 µm. In both conditions, the grains were equiaxed, showing no preferred orientation. CM247LC Cast+HIP, which was used as reference material for tensile tests, had a much coarser grain size of 640 µm. The heat treatment led to a complete dissolution of the γ'-phase during the 1300°C and 1260°C (2372/2300°F) steps, followed by precipitation during cooling and subsequent ageing. Consequently, the γ'-particles were more homogenous in size and displayed a more regular morphology compared with the as-sintered condition. In the as-sintered condition, the γ'-particles had a size of around 800 nm. This particle size was reduced to 600 nm by the applied heat treatment.

Tensile testing was carried out for both material conditions at seven temperatures from 20°C to 900°C (68-1652°F). The 0.2% proof stress, ultimate tensile strength and plastic elongation to fracture are shown in Figs. 17a-c. The quoted values are normalised with respect to the values for CM247LC Cast+HIP at room temperature.

The 0.2% proof stress of CM247LC MIM and MIM+HT is comparable to the cast material up to a temperature

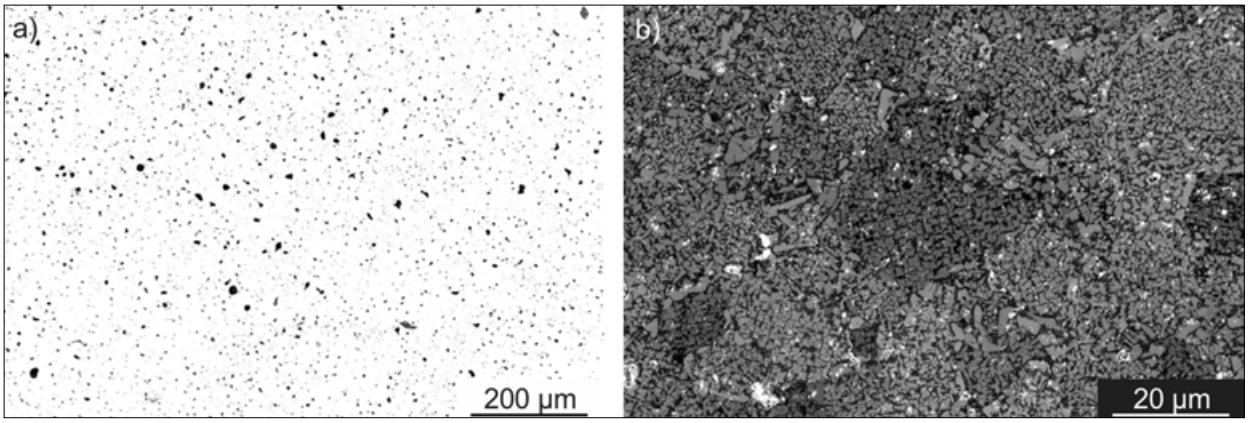

Fig. 16 a) Light optical microscope image showing the porosity of 1.7 % of CM247LC MIM in the as-sintered condition, b) SEM image showing γ-γ'-microstructure [3]

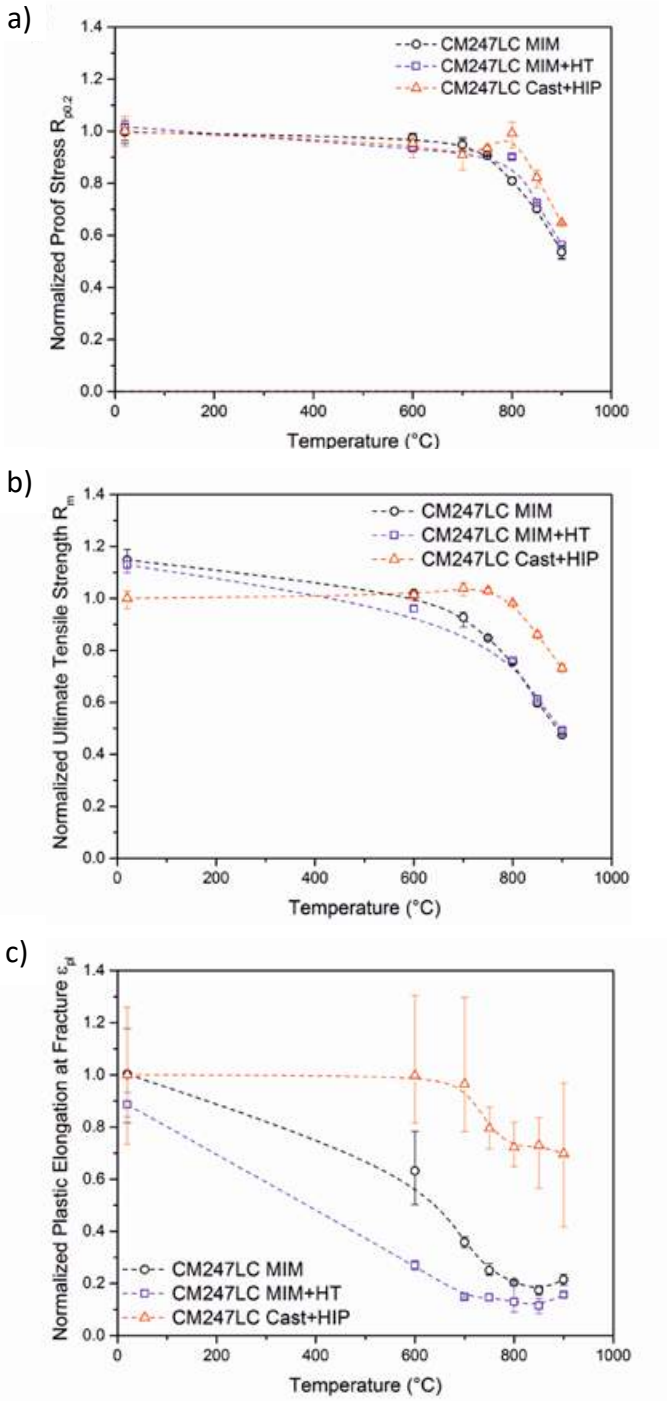

Fig. 17 Normalised a) 0.2% proof stress, b) ultimate tensile strength and c) plastic elongation at fracture of CM247LC MIM, MIM+HT and Cast+HIP. The values are normalised to the values of CM247LC Cast+HIP at room temperature [3]

of 700°C (1292°F). At higher temperatures, the MIM materials exhibit a somewhat lower 0.2% proof stress than the cast alloy (Fig. 17a).

The ultimate tensile strength of the two MIM materials at room temperature is 13-15% higher than the UTS of the reference cast material. At temperatures higher than 600°C (1112°F), the Cast+HIP material exhibits a higher UTS than MIM+HT. With reference to plastic elongation to fracture, a similar behaviour can be observed. At room temperature, the MIM materials and the cast material have a similar ductility, while, at higher temperatures, the cast material has a much higher elongation at fracture. The scatter for the Cast+HIP material is higher than for MIM.

The applied heat treatment had little influence on the 0.2% proof stress and ultimate tensile strength of CM24LC MIM at all temperatures. With regard to ductility, the heat treatment led to a reduced plastic elongation at fracture.

Analyses of fracture surfaces of CM247LC MIM and MIM+HT materials showed a mainly inter-granular fracture, indicating that the grain boundaries are much weaker than the grain boundaries of CM247LC Cast+HIP. It seems that prior particle boundaries (PPBs) with their high density of pores and carbides/oxides provide sites for easy crack nucleation. Further investigations are necessary for a better understanding of grain boundary strengthening and weakening effects for this kind of material.

The results of creep testing can be seen in Fig. 18. The heat treatment led to an increase of time to rupture by a factor of 5-6 for the different test temperatures and creep stress levels. An example of the increase in time to rupture is shown in Fig. 18a for a test condition of 900°C and 100 MPa (1652°F, 14.5 ksi).

All of the creep test results are shown in Fig. 18b in a Larson-Miller diagram. The Larson-Miller parameter P, including both test temperature and time to rupture, is a parameter giving an indication of creep resistance for a material and can be calculated with the equation, P = T(C+log(t)), where T is temperature (in Kelvin), t is time to rupture (in hours)and C is a constant (in this case, C = 20). A higher Larson-Miller parameter indicates better creep resistance.

The Larson-Miller curves of CM247LC MIM and MIM+HT show that the heat treatment leads to an improved creep resistance. The main reason for this is the coarser grain structure that leads to a slower creep deformation, also visible in Fig. 18a. Grain boundary sliding, superimposed

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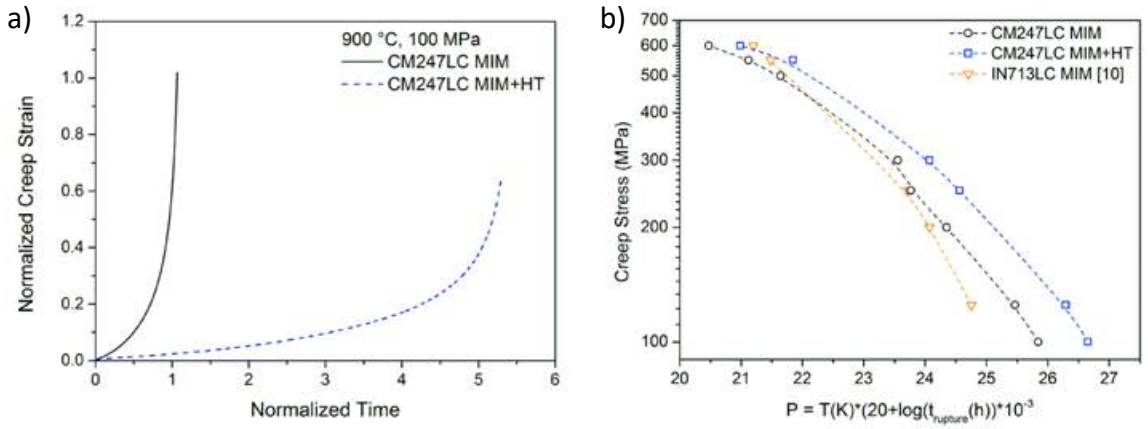

Fig. 18 a) Normalised creep strain-time-diagram for 900°C and 100 MPa (1652°F, 14.5 ksi) showing the influence of the heat treatment. b) Larson-Miller diagram of both CM247LC materials in comparison with IN713LC MIM alloy [3]

on dislocation creep, is retarded by the larger grain size. In addition, the homogeneous γ-γ' microstructure should also contribute to better creep properties. The creep resistance of both CM247LC MIM materials is comparable to IN713LC MIM at lower temperatures and higher stresses, while, at higher temperatures and lower stresses, CM247LC MIM exhibits a higher creep resistance.

The authors drew the following overall conclusions from this study:

1. The nickel-base superalloy CM247LC can be processed successfully via MIM and sintered to a relative density of more than 98% with moderate pick-up of impurities such as carbon and oxygen.
2. The tensile properties of MIM material at low and intermediate temperature are comparable to Cast+HIP material.
3. Ductility and creep resistance at high temperature are inferior for MIM, probably due to the existence of prior particle boundaries (PPBs) and small grain size, respectively.
4. The influence of a heat treatment on the microstructure and mechanical properties has been investigated. The applied heat treatment leads to grain coarsening and to a homogeneous γ-γ' microstructure. As a result, creep strength was improved. However, ductility was reduced by the heat treatment. It is assumed that coarsening of the PPB precipitation made crack opening easier.

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[2] Microstructure of MIM Ti6Al4V produced using fine metal powders, Pelletier R et al. As presented at the POWDERMET2017 International Conference on Powder Metallurgy & Particulate Materials, Las Vegas, USA, June 13-16, 2017, and published in the proceedings by the Metal Powder Industries Federation (MPIF).

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POWDERMET2018, the International Conference on Powder Metallurgy & Particulate Materials, will take place in San Antonio, Texas, USA, from June 17–20, 2018. A Call for Papers & Posters has been issued and the abstract submission deadline is November 3, 2017.
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POWDERMET2017: Innovative technologies support the control of moulding and sintering

In our second report from the POWDERMET2017 International Conference on Powder Metallurgy & Particulate Materials, Dr David Whittaker reviews three papers that outlined the development of aids to support the control of PIM processing. These specifically addressed the mouldability of low pressure PIM feedstocks, the influence of feedstock properties on PIM of Lead Zirconate Titanate (PZT) and the evaluation of a ceramic temperature monitoring device for sintering furnaces.

The mouldability of Low-Pressure Powder Injection Moulding feedstocks

A paper by Vincent Demers, Marwan Mohamed Elmajdoubi and Philippe Bocher (Ecole de Technologie Superieure, Montreal, Canada), discussed the characterisation of the mouldability of Low Pressure Powder Injection Moulding (LPIM) feedstocks, including the definition and calculation of a mouldability index [1].

Recent progress in feedstock formulations has generated new opportunities for the injection of low-viscosity (< 10 Pa·s) molten powder-binder mixtures (< 100°C) using low injection pressure (< 800 kPa). Initially used in ceramics forming, LPIM technology has also gained rapid attraction for the development of high value-added metallic parts in several industrial sectors. LPIM technology takes advantage of feedstocks of low viscosity in order to achieve high mouldability. A lower injection pressure directly minimises deformation of the mould and favours a laminar filling, resulting in defect-free parts.

The main variables influencing viscosity of powder-binder mixtures are the shear rate, temperature, solid loading, powder shape and size and binder composition. The mouldability index has been used in conventional Metal Injection Moulding or in the LPIM process to compare the moulding potential of feedstocks during injection. Initially developed for predicting the flow behaviour of polymer, the mouldability index is calculated from the combination

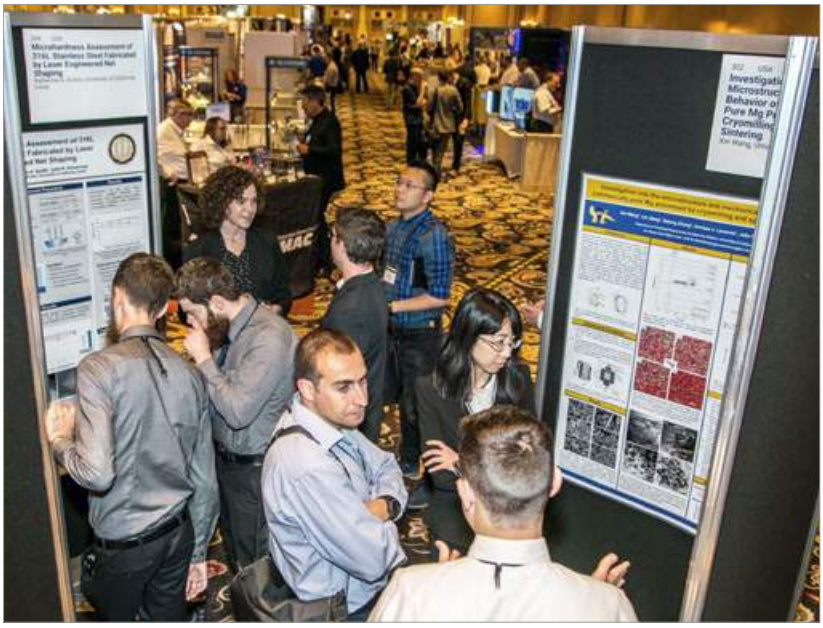

Fig. 1 A busy poster session at POWDERMET2017 (Courtesy MPIF)

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Feedstock #	Feedstock identification	Vol.% [%]				Melting point [°C]
		Metallic powder (MP)	Paraffin wax (PW)	Stearic acid (SA)	Ethylene vinyl acetate (EVA)	
Feedstock #1	48MP-52PW	48	52	-	-	54
Feedstock #2	48MP-46PW-1SA-5EVA	48	46	1	5	51
Feedstock #3	48MP-46PW-6SA	48	46	6	-	50
Feedstock #4	60MP-34PW-1SA-5EVA	60	34	1	5	51
Feedstock #5	60MP-34PW-6SA	60	34	6	-	50

Table 1 Nominal volume fractions and melting temperatures of the binders used for the feedstocks [1]

of rheological parameters such as viscosity, the shear rate sensitivity index and the activation energy for viscous flow of the feedstock.

The reported work in this study was aimed at comparing mouldability index results with real-scale injection results obtained with several typical feedstocks used in Low-Pressure Powder Injection Moulding.

The study utilised a water-atomised stainless steel 17-4PH powder with a typical near-spherical or ligament shape and an average particle size of 10 µm. Paraffin wax (PW), stearic acid (SA) and ethylene vinyl acetate (EVA) were used as the main binder constituent, the surfactant and the thickening agent, respectively. These polymer constituents were mixed with the metallic powder to form homogeneous feedstocks with the formulations given in Table 1. The solid loadings of feedstocks, determined at room temperature, were set at 48 and 60 vol.%. The lower solid loading (48 vol.%) is the maximum value needed to ensure the wettability of metallic powder with a single-binder constituent, while the higher solid loading (60 vol.%) represents a typical value for homogeneous powder-binder mixtures.

The viscosities of the feedstocks were measured with a rotational rheometer using a concentric cylinder and a Peltier temperature-controlled measuring system. The warm feedstocks were poured into the cylinder and then tested at shear rates ranging from 0.5 to 3500 s^{-1} at two temperatures (70 and 90°C). The rheological properties at low shear rate (1 s^{-1}) can be correlated with the feedstocks during mixing, the process dead time, or the cool down of the injected part, while the properties calculated at moderate to high shear rates (100-1000 s^{-1}) are useful for assessing the behaviour of the feedstock during the injection stage of the process.

Differential Scanning Calorimetry (DSC) was conducted to obtain the melting point of each constituent and each binder formulation (Fig. 2). These values correspond to the minimum temperatures required for the injection and melt rheology tests. The DSC tests were performed over a 20 to 100°C temperature range. Where a feedstock exhibited several melting points (i.e. more than one DSC peak), the feedstock melting point was determined from the last peak obtained during a heating cycle, as indicated by a black arrow in Fig. 2.

The DSC results for each binder constituent and for two typical feedstock formulations, shown in Fig. 2, confirm that the stearic acid (SA) and ethylene vinyl acetate (EVA) have a certain degree of solubility with the paraffin wax (PW). Adding 6 vol.% of SA to PW only slightly changes the mixture melting point of the pure PW binder. Adding 5 vol.% of EVA to a PW/SA mixture has less effect on the thermal profile or the melting point of the feedstock. SA and EVA can therefore be added to PW without inducing a significant increase in the feedstock melting temperature. Melting point values, varying from 50 to 54°C, were extracted from the DSC thermograms and are reported in Table 1. The minimum temperature for rheological and injection tests was therefore selected to be 70°C, in order to ensure that all feedstocks were in the molten state.

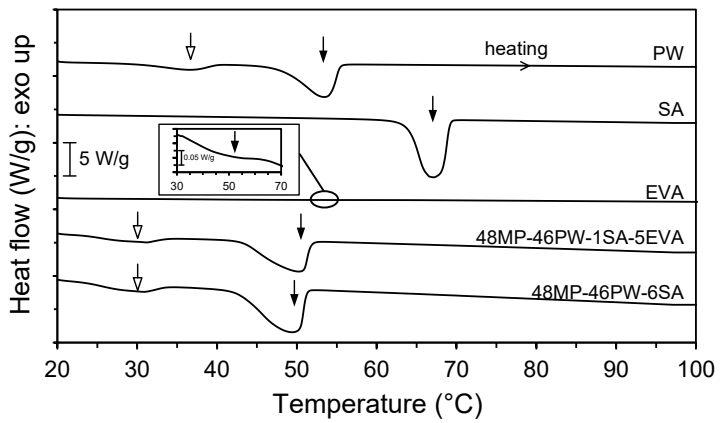

Fig. 2 DSC results for binder constituents (heating) [1]

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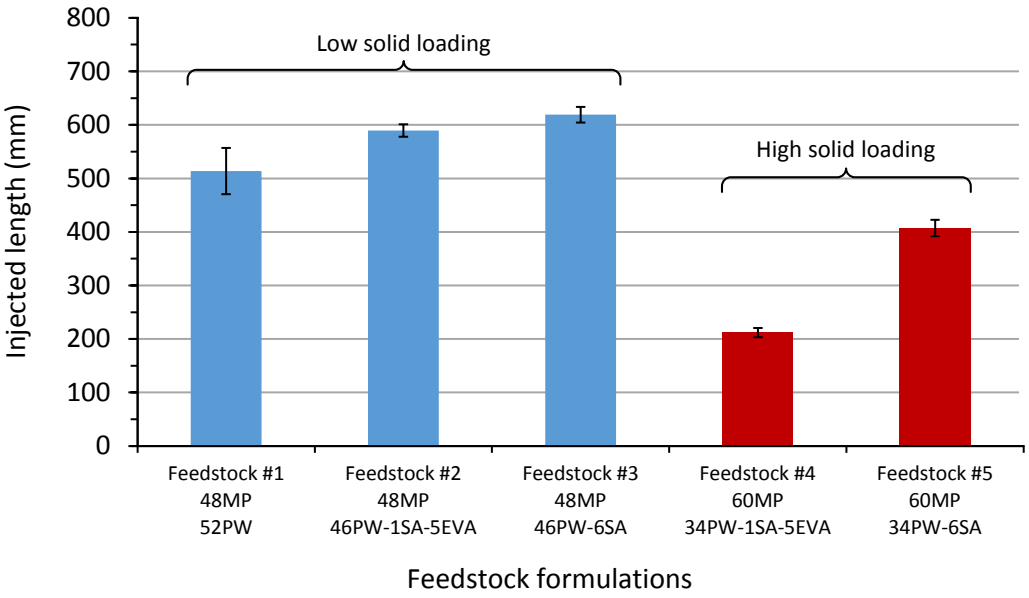

Fig. 3 Injected length of spiral specimens ($T_{feedstock}$ = 70°C; T_{mould} = 40°C; injection rate = 5.8 mm/s) [1]

Injections were performed using a laboratory injection press in which a new injection concept was developed to minimise segregation in low-viscosity feedstocks and to quantify the full mouldability potential of the LPIM process. Feedstocks were heated up to 70°C, blended using a planetary mixer at 10 rpm for 45 min under vacuum and then injected into a mould cavity designed to produce spiral specimens. Between injections, the feedstock remaining in the injection cylinder was returned to the container to be re-blended. The injection was performed using a controlled constant stroke (5.8 mm/s), while the pressure value was recorded using a load cell located in the injection axis. When the friction between the feedstock and mould walls became too high to allow the feedstock to flow, a sudden increase in pressure (up to 2.5 MPa) was measured and the injection piston was stopped. The green part was removed from the mould and the length of the injected part was measured.

The injected length values obtained for each feedstock formulation are presented in Fig. 3. The error bars indicate the standard deviations of the measurements obtained from each feedstock. At low solid loading, adding a mixture of SA-EVA or only SA to PW slightly increased the injection capacity of the simple PW feedstock by about 15% and 20%, respectively. At high solid loading, the feedstock containing SA and PW produced spiral parts two-fold longer than that injected using the mixture formulated with SA, EVA and PW. For similar binder formulations, an increase in solid loading from 48 to 60 vol.% resulted in a significant decrease in the injected length.

Feedstock 3 (48MP-46PW-6SA) exhibited the highest injected length value. The enhancement of the rheological properties of this feedstock was attributable to the surfactant effect of the stearic acid between the powder and the main wax-based binder constituent. The surfactant effect associated with the stearic acid constituent appears to be more pronounced for the higher solid loading feedstocks.

This confirms that the surfactant effect plays a more important role in the injection properties of feedstocks, when the inter-particle space is decreased. The poor repeatability of the measurements for feedstock 1 (48MP-52PW) confirms that the injection properties are low and not constant, compared with the other low solid loading feedstocks. This can be probably explained by the weak wettability potential of the paraffin wax constituent when used alone. This suggests that this simple mixture is not suitable for the LPIM process and should potentially be used with another surfactant agent.

"The enhancement of the rheological properties of this feedstock was attributable to the surfactant effect of the stearic acid between the powder and the main wax-based binder"

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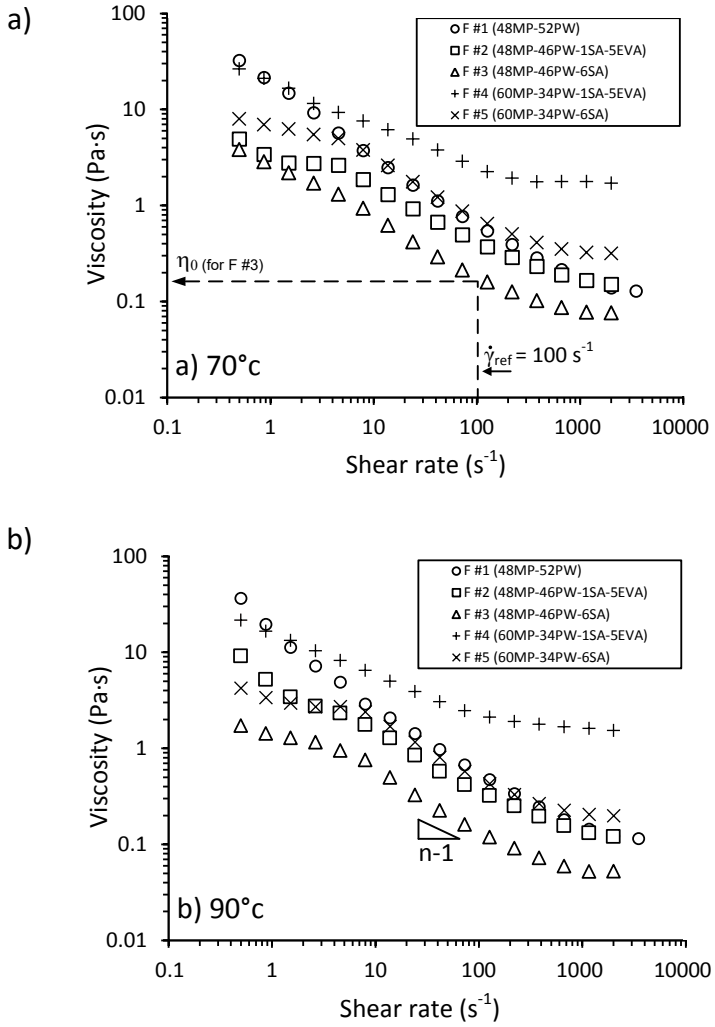

Fig. 4 Viscosity dependence on shear rate for feedstocks at (a) 70°C, and (b) 90°C [1]

Feedstock #	Feedstock	η_0 (Pa·s)	n	E (kJ/mol)
1	48MP-52PW	0.64	0.28	6.9
2	48MP-46PW-1SA-5EVA	0.43	0.44	7.9
3	48MP-46PW-6SA	0.18	0.39	14.7
4	60MP-34PW-1SA-5EVA	2.52	0.56	5.6
5	60MP-34PW-6SA	0.74	0.41	20.9

Table 2 Rheological parameters of feedstocks at 70°C [1]

The low mouldability properties of feedstock 4 (60MP-34PW-1SA-5EVA) are attributable to the increase in powder solid loading, combined with the thickening effect of the ethylene vinyl acetate in the wax-based binder constituent. The use of thickening agents and high solid loading content are often required to control the quality of MIM parts, by preventing powder-binder separation at the injection stage as well as by reducing heterogeneous shrinkage at the sintering stage.

The changes of viscosity with shear rate at 70 and 90°C for the different feedstocks are shown in Fig. 4. The viscosities of all feedstocks decrease as the shear rate increases, corresponding to the pseudo-plastic behaviour generally required for LPIM. These viscosity profiles clearly indicate that the viscosity values of LPIM feedstocks depend on solid loading and binder formulation.

The flowability of a feedstock is generally quantified by its viscosity. This value should be as low as possible in order to maximise mould filling capability and produce more complex shaped components. The viscosity values η_0 at a reference shear rate = 100 s^{-1} were extracted from Fig. 4 and summarised in Table 2 for each testing condition. In the study, an increase in the injected length was also observed with a decrease in viscosity. From a flowability perspective, the lowest viscosity value was obtained with feedstock number 3 (48MP-46PW-6SA).

At a given temperature, the dependence of the viscosity on shear rate for PIM feedstocks can be described by the power law equation, $\eta = K\dot{\gamma}^{n-1}$. Pseudo-plastic behaviour is characterised by a value of n (the shear rate sensitivity) below 1, where a low n value indicates that the feedstock viscosity is strongly dependent on the shear rate. The values of n were calculated from the $n-1$ slopes of the viscosity profiles presented in Fig. 4 and are summarised in Table 2, for each testing condition. The lowest value of the shear rate sensitivity index was associated with feedstock number 1 (48MP-52PW).

The decrease of the feedstock viscosity with the increase in temperature can be described by the Arrhenius equation.

$$\eta = \eta_{T0} \exp\left(\frac{E}{RT}\right)$$

where η is viscosity, η_{T0} is the viscosity at a reference temperature, E is the activation energy for viscous flow, R is the gas constant and T is the temperature.

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The value of the activation energy, E, is used to quantify the degree of the temperature sensitivity to the feedstock viscosity. Low values of E will minimise the possible changes in feedstock viscosity between the hot and cold zones in the injection press or in the mould. The values for the natural logarithm of the viscosity measured at the reference shear rate (see Fig. 4a) were plotted against $1/T$ in Fig. 5 to calculate E from the slope of each graph. The values of E are summarised in Table 2 for each feedstock. From a temperature sensitivity perspective, feedstock number 4 (60MP-34PW-1SA-5EVA) is the best candidate for injection in a relatively wide temperature range.

The ability of the feedstocks to be injected into the mould cavity was addressed by the quantification of the mouldability index α_{STV}, which, according to Weir's model, can be calculated using the equation

$$\alpha_{STV} = \frac{10^8}{\eta_0} \frac{|n-1|}{E/R}$$

where n is the shear rate sensitivity, E is the activation energy in the Arrhenius equation and R is the gas constant.

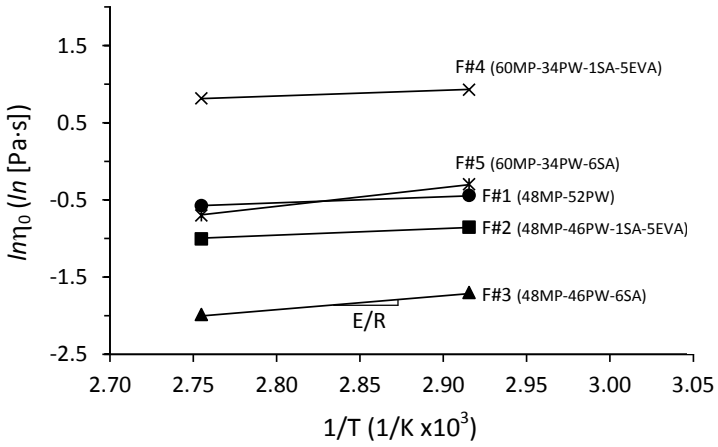

Fig. 5 Dependence of viscosity on temperature for different feedstock formulations [1]

The mouldability indices of feedstocks at 70°C, calculated from the rheological parameters are shown in Fig. 6. From a mouldability perspective and in the absence of injection problems such as feedstock jetting, significant residual stresses in green parts, or powder segregation, the best candidate feedstocks at low and high solid loadings are the mixtures containing paraffin wax and stearic acid.

As a means of validating the mouldability index model against experience in real scale injection, the injected length and mouldability index of each feedstock formulation were both normalised with respect to their maximum value and compared in Fig. 7. The maximum values were obtained with feedstock number 3 for the two mouldability measurement techniques. In general, the trend of the mouldability index values is in good agreement with the real scale injection results. However, the normalised values from the model do not perfectly correlate with the

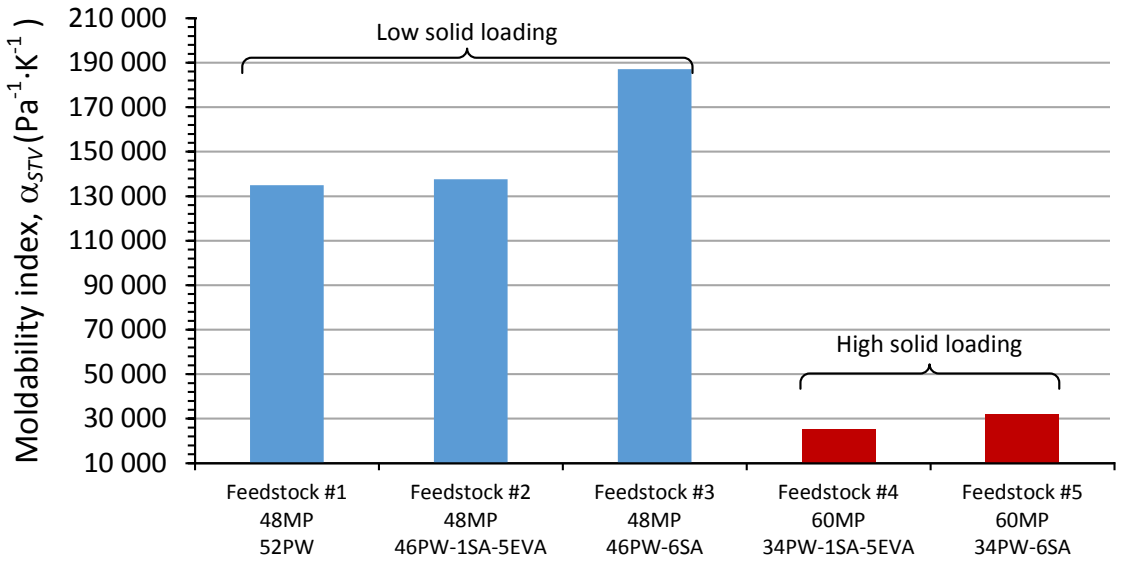

Fig. 6 Mouldability index (α_{STV}) of feedstocks at 70°C for viscosity values obtained at a shear rate of 100 s^{-1} [1]

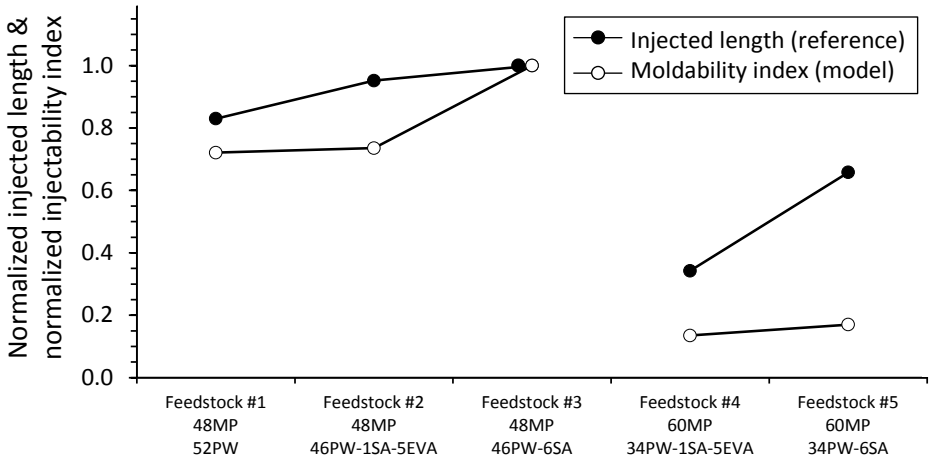

Fig. 7 Comparison of the injected length and mouldability index of feedstocks at 70°C for viscosity values obtained at a reference shear rate of 100 s^{-1} [1]

Particle size (µm)			Density (10^3 Kg/m^3)			Dielectric constant
(D_{10})	(D_{50})	(D_{90})	Apparent	Tap	Pycnometer	
0.31	0.52	1.01	1.62	2.54	7.98	3731

Table 3 PZT powder properties [2]

Powder vol %	Melt density (kg/m^3)	Solid density (kg/m^3)
0 (binder)	727	879
45	1950	2119

Eqn1 Powder mass fraction $X_p = \frac{\phi_p \rho_p}{\phi_p \rho_p + \phi_b \rho_b}$

Eqn2 Binder mass fraction $X_b = \frac{\phi_b \rho_b}{\phi_p \rho_p + \phi_b \rho_b}$

Eqn3 Inverse rule-of-mixtures $\frac{1}{\rho_c} = \frac{X_b}{\rho_b} + \frac{X_p}{\rho_p}$

Φ_p - volume fraction of powder, Φ_b - volume fraction of binder, Φ_c - volume fraction of composite, ρ_p - density of powder, ρ_b - density of binder, ρ_c - density of composite

Table 4 Solid and melt densities of feedstocks with 45 vol.% PZT powder [2]

normalised values of the injection length. The difference between injected length and mouldability index is relatively low at 48 vol.% solid loading (% error ~ 13-23%), but significantly higher at 60 vol.% solid loading (% error ~ 60-75%).

The difference observed between this simple model and the experimental injections suggests that the interactions between the feedstock and the mould, which are not taken into account in Weir's model (such as thermal exchange, friction, etc.), play an important role in the real scale injection results. The differences between the experimental observations and the mouldability indices can be also explained by the weight of each rheological parameter in Weir's model.

In this respect, the mouldability index could be calibrated in future work in order to develop a tool able to predict more precisely the mouldability of LPIM feedstocks.

Influence of feedstock property estimates and measurements on the PIM of lead zirconate titanate

A second paper in this session also concentrated on the injection stage of the PIM process. This paper was delivered by Bhushan Bandiwadekar (University of Louisville, USA) and was co-authored by Swathi Vunnam, Kunal Kate and Sundar Atre (also University of Louisville) and Joo Won Oh and Seong Jin Park (Pohang University of Science and Technology, Korea) and discussed the influence of feedstock properties on PIM of Lead Zirconate Titanate (PZT), with particular reference to their use in the numerical simulation of the injection process [2].

PZT has very high piezoelectric and dielectric properties and is widely used for transducer applications. Although there has been previous work on PIM of piezoelectric ceramics, the feature sizes have been higher than hundreds of microns. It is very difficult to produce high aspect ratio fine features of piezoelectric ceramics using PIM. However, injection moulding experiments are highly sensitive to feedstock properties and processing conditions. Mould flow computer

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simulation of traditional injection moulding is frequently used in process optimisation.

3D PIM simulation can be used to optimise the injection moulding, identifying key problems before doing the actual fabrication. Among the available mould flow simulation platforms, Moldex3D is a leading CAE product for the PIM industry. It can carry out in-depth simulation for a wide range of injection moulding processes and to optimise product designs and manufacturability.

The aim of the study reported in this paper was to validate the estimated material properties for 3-dimensional mould flow simulations, specifically for 45 vol.% PZT feedstock, through comparisons with experimental results. Subsequently, the effects of feedstock properties on mould filling behaviour and defect formation were studied using the Moldex3D simulation platform. The authors proposed that results from this study will provide a quantitative understanding of the influence of estimated feedstock properties on the injection moulding process.

Commercially available PZT was used for feedstock preparation. The morphology of the PZT powder was assessed by scanning electron microscopy. Fine powders with irregular shape and some amount of agglomeration were observed. Table 3 summarises the size and density values for the PZT powder. A multicomponent binder system, consisting of paraffin wax, polypropylene, polyethylene-g-maleic anhydride (L DPE-g-MA) and stearic acid (SA), was used in the study.

The rheological properties of the binder were measured using a capillary rheometer at different shear rates and temperatures. The temperatures were between the highest melting temperature and the lowest degradation temperature of the binder system. Torque rheometry was performed to determine the maximum packing density of the powder-polymer mixture. A continuous processor with twin shafts was used for continuous extrusion of PZT feedstocks. Thermogravimetric analysis (TGA) was performed on the extruded feedstocks under nitrogen flow in the temperature range 50-600°C with a heating rate of 20°C/ min in order to confirm the powder weight fraction in the feedstock. A Thermal Conductivity System was used to measure the thermal conductivity of the binder.

The experimentally determined binder physical properties were used to estimate properties of PZT powder-polymer mixtures with 45 vol % loading, using semi-empirical scaling rules that are available to estimate material properties. Tables 4 to 7 summarise the estimated material properties. The viscosity and PVT data required curve fitting to extract constants, required for mould filling simulations using Moldex3D software.

Specific heat measurements were conducted with the cooling rate being kept constant of 20°C/min. The specific heat of powder-polymer mixtures has been estimated by different mixing rules. In this study, a model that has been successfully applied to mixtures with high volume fraction fillers was used as shown in Equation 4 (Table 5). The parameter, A, is a correction factor assumed to be 0.2 for spherical particles. The specific heat values calculated at various temperatures are shown in Table 5.

Specific heat capacity, C_p (J/kg-K)		
Temperature (K)	Powder vol %	
	0 (Binder)	45
283	2077	632
322	3360	977
352	3840	807
368	4894	1515
377	4639	668
407	3484	680
443	2528	696
Thermal conductivity (W/m·K)		
Temperature (K)	Powder vol %	
	0 (Binder)	45
315	0.2	0.56
336	0.2	0.55
356	0.2	0.54
377	0.2	0.53
397	0.2	0.53
417	0.2	0.52
436	0.2	0.51

$$^{Eqn4}\ \text{Specific heat } C_{p_c} = \left[C_{p_b}X_b + C_{p_p}X_p\right] * \left[1 + A * X_b X_p\right]$$

$$^{Eqn5}\text{Thermal conductivity } \lambda_c = \lambda_b \phi_b + \lambda_p \phi_p$$

Table 5 Specific heat capacity and thermal conductivity values at various temperatures for feedstocks with 45 vol.% PZT powder [2]

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dual-domain Tait constants	Powder vol %	
	0 (Binder)	45
b_5, K	0	336
b_6,K/Pa	336	1.54×10^{-7}
b_{1m}, m^3/kg	1.5×10^{-7}	2.63×10^{-4}
b_{2m}, m^3/kg·K	1.3×10^{-3}	1.13×10^{-7}
b_{3m}, Pa	1.3×10^{-6}	2.24×10^{8}
b_{4m}, K^{-1}	1.3×10^{8}	3.04×10^{-4}
b_{1s}, m^3/kg	6.0×10^{-3}	2.52×10^{-4}
b_{2s}, m^3/kg·K	1.2×10^{-3}	1.24×10^{-7}
b_{3s}, Pa	8.6×10^{-7}	5.08×10^{8}
b_{4s}, K^{-1}	2.4×10^{8}	1.46×10^{-3}
b_7, m^3/kg	4.2×10^{-3}	1.20×10^{-5}
b_8, K^{-1}	8.5×10^{-5}	9.40×10^{-2}
b_9, Pa^{-1}	6.7×10^{-2}	2.05×10^{-8}

$$^{Eqn6}\ \text{Specific volume } v_c = X_p v_p^X + v_b(1 - X_f)$$

$$^{Eqn7}\ v(T,p) = v_o(T)\left[1 - C\ln\left(1 + \frac{p}{B(T)}\right) + v_t(T,p)\right]$$

$T > T_t$:

$$v_o = b_{1m} + b_{2m}(T - b_5), X$$

$$B(T) = b_{3m} e^{[-b_{4m}(T - b_5)]}, X$$

$$v_t(T,p) = 0X$$

$T < T_t$:

$$v_o = b_{1s} + b_{2s}(T - b_5),$$

$$B(T) = b_{3s} e^{[-b_{4s}(T - b_5]},$$

$$v_t(T,p) = b_7 e^{[b_8(T - b_5) - (b_9 p)]}$$

$v\ (T,p)$ - specific volume at a given temperature and pressure
v_o - specific volume at zero-gauge pressure, p - pressure,
C - constant, 0.0894
b_{1s}, b_{2s}, b_{3s}, b_{4s}, b_5, b_7, b_8, b_9 - curve-fitted coefficients
b_{1m}, b_{2m}, b_{3m}, b_{4m}, b_5, b_6 - curve-fitted coefficients

Table 6 Dual-domain Tait constants for feedstocks with 45 vol.% PZT powder [2]

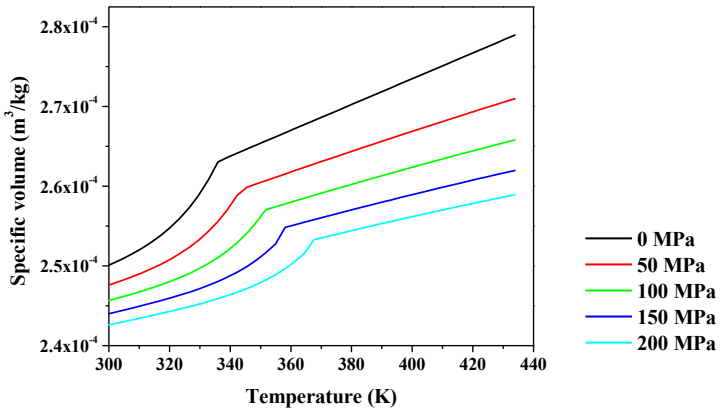

Fig. 8 PVT behaviour for 0, 100, and 200 MPa pressures for 45 vol.% PZT powder [2]

In this study, a general rule-of-mixtures model was used, as represented in Equation 5 (Table 5) to estimate the thermal conductivity. The estimated values of thermal conductivity at various temperatures are shown in Table 5.

The specific volume was calculated using the general rule-of-mixtures as shown in Equation 6 (Table 6). Fig. 8 shows the comparative plot of specific volumes at 0, 100 and 200 MPa pressure for 45 vol.% PZT powder. Additionally, to perform mould-filling simulations in Moldex3D software, the specific volume of the material needs to be represented in terms of fitted constants. A dual-domain Tait equation (Equation 7, Table 6) was used to extract these fitted constants using curve fitting for 0, 50, 100, 150 and 200 MPa pressure for 45 vol.% PZT powder. The values of these coefficients are summarised in Table 6.

A simplified Krieger-Dougherty viscosity model was used to estimate the viscosity values (Equation 8, Table 7). The Cross-WLF model was used to model the viscosity dependence of the powder-polymer mixture on shear rate (Equation 9, Table 7). The temperature dependence of viscosity of the PZT powder-polymer mixture was estimated using Equation 10 (Table 7). The values of the coefficients (T*, D1 and A1) were extracted by curve-fitting the estimated viscosity for 45 vol.% PZT powder at various shear rates and temperatures.

Fig. 9 shows the shear-rate dependence of viscosity for feedstocks with 45 vol.% PZT powder at 413, 420, 426 and 433 K. The zero-shear viscosity was estimated from the plateau region at low shear rate, while the power law index was obtained from the slope at higher shear rates. The curve-fitted WLF parameters n, τ^*, D1, T*, A1 and A2 were estimated for four different temperatures (413, 420, 426 and 433 K). The estimated values for n and τ^* for each temperature were then averaged. All the coefficients related to viscosity are summarised in Table 7.

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As a basis for validation of the numerical simulations, PIM experiments were performed using a micro-injection moulding machine. PIM experiments were performed at injection moulding conditions of 40 MPa injection pressure, melt temperature of 433 K, mould temperature of 318 K and injection velocity of 90 mm/s. Moldex3D software was used to perform PIM simulations for the same process conditions as the PIM experiments. A rectangular plate geometry was designed using Solidworks 2016 software and was imported into the Moldex3D R14.0 software and a runner and sprue system was added. Moldex3D Mesh Designer software was used to generate a mesh with 200,000 elements for the rectangular plate and exported to Moldex3D R14.0. Simulations were conducted for 45 vol.% PZT using the estimated material properties. The designed rectangular plate geometry is shown in Fig. 10(a) and the meshed plate with runner and sprue are shown in Fig. 10(b). Simulations were performed for fill-and-pack conditions.

The cavity dimensions and process conditions from the PIM experiments and estimated material properties were used to perform simulations. Experiments and simulations were carried out with estimated feedstock properties at different injection velocities and pressures to predict incomplete to complete mould filling behaviour of the 45 vol.% PZT feedstock. At an injection velocity of 90 mm/s and packing pressure of 400 kg/cm^2, a completely filled mould cavity was achieved. The PIM of completely filled 45% PZT base plate samples have an average cavity weight of 5.66 g. From Moldex3D simulations, the completely filled mould cavity weight was 5.12 g. The error in estimating cavity weight with respect to experiments was therefore around 10% using estimated properties.

Fig. 11 shows a simulation of progressive injection moulding behaviour inside the rectangular geometry and this was comparable to that observed in PIM experiments. The

Cross-WLF constants	Powder vol %	
	0 (Binder)	45
n	0	0.40
τ^*, Pa	0.40	15831
$D1$, $Pa{\cdot}s$	793	1.08×10^{16}
T^*, K	4.29×10^{23}	367.87
A_1	333.00	56.59
A_2, K	78.13	51.60

Eqn8 Viscosity of composite $\eta_c = \frac{\eta_b}{\left[1-\frac{\phi_p}{\phi_{max}}\right]^2}$

Eqn9 Melt viscosity $\eta = \frac{\eta_0}{1+\left(\frac{\eta_0 \gamma}{\tau^*}\right)^{1-n}}$

Eqn10 Zero shear viscosity $\eta_0 = D_1 exp\left[-\frac{A_1(T-T^*)}{A_2+(T-T^*)}\right]$

η_b - viscosity of binder, Φp - volume fraction of powder,
Φ_{max} - maximum volume fraction, γ - shear rate
τ*- critical stress level at the transition to shear thinning
n - power law index in the high shear rate regime
T - temperature, Tt - volumetric transition temperature
T*, D_1, A_1 - curve-fitted coefficients,
A_2 - WLF constant, 51.6 K

Table 7 Cross-WLF constants for feedstocks with 45 vol.% PZT powder [2]

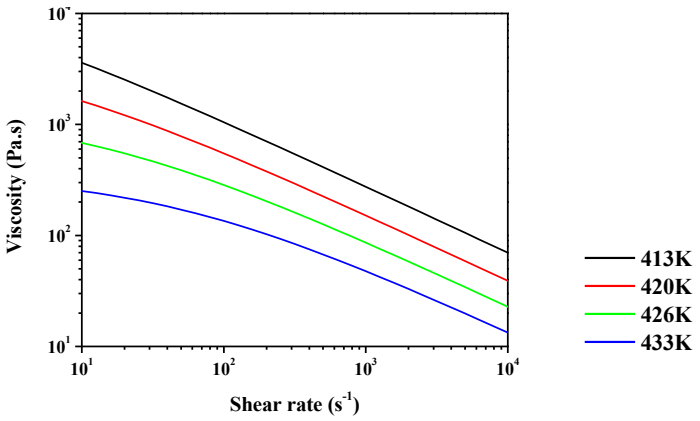

Fig. 9 Comparison of viscosity with shear rate at different temperatures for 45 vol % PZT powder [2]

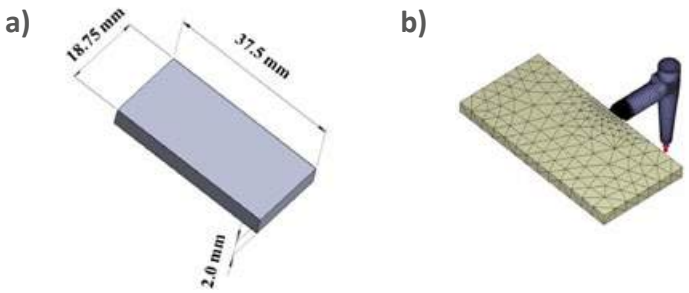

Fig. 10 Rectangular plate (a) Solidworks part file (b) Meshed with runner gate system in Moldex3D Designer [2]

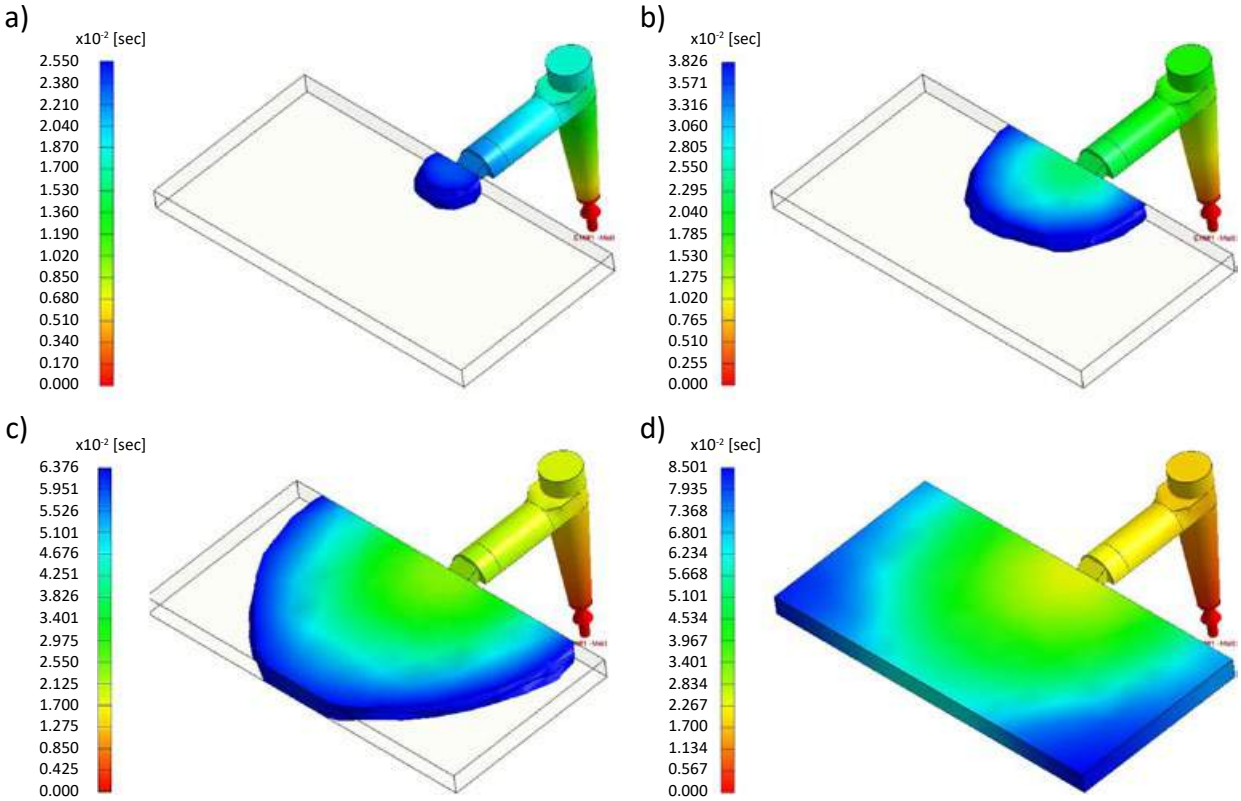

Fig. 11 Moldex3D simulation for 0.45% PZT base plate - 100% filled at injection velocity 90 mm/s (a) 30% filled in 0.025 s, (b) 45% filled in 0.038 s, (c) 75% filled in 0.064 s and (d) 100% filled in 0.085 s [2]

average volumetric shrinkage for the PIM experiment was 2%, compared with 5% in the Moldex3D simulations.

The authors' overall conclusion from this validation study was that using estimated material properties to perform PIM simulations can help reduce the number of trial-and-error experiments involved in designing new PIM feedstocks and PIM process parameters. Furthermore, such an approach can be utilised in selecting component geometry attributes to optimise the PIM process.

The in-situ characterisation and process control of temperature within a vacuum retort furnace using a non-electronic ceramic device

Finally, attention was switched to the control of the sintering furnace in a paper from Stefan Joens (Elnik Systems LLC, USA), Thomas McInnerney and James Litzinger (The Edward Orton Jr. Ceramic Foundation, USA) and Satyajit Banerjee (DSH Technologies LLC, USA). This paper related to the process control of temperature in vacuum retort furnaces using a non-electronic ceramic device [3].

In this context, the reported study was aimed at demonstrating the utilisation of TempTAB ceramic discs to carry out Temperature Uniformity Surveys (TUS) within a retort-based high temperature vacuum/partial pressure processing furnace.
The objective of the study was to demonstrate the proper usage and examine results in varying processing environments, while utilising Orton's TempTAB product along with an additional back up of Flexible Type K survey thermocouples (TCs), when applicable, to verify the data results from the ceramic tabs.

For many metal and ceramic processing technologies that include a process step in a furnace, temperature uniformity within the furnace must be regularly surveyed. The frequency of surveying is largely dependent on the type of equipment in use, the industry in which the furnace is being utilised to supply parts and its previous history of accuracy and reliability. Quarterly and semi-annual temperature uniformity surveys are fairly standard.

Orton's TempTAB is a temperature sensing ceramic disc that is used as a quality assurance tool and can be incorporated into any Statistical Process Control (SPC) programme. It is made with a small hole in the centre to allow it to be hung on wires, suspended in the load or simply placed flat within the load to be sintered.

TempTAB discs are used to monitor the amount of thermal energy absorbed into the sintered product, by converting the amount of solid state sintering shrinkage that the TempTAB undergoes and translating that shrinkage into an equivalent sintering temperature.

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Since shrinkage is a function of sintering of the powders, which is dependent on both temperature and time, Orton has taken both these factors into consideration in producing the conversion data tables for the TempTAB. In use, the operator chooses the hold time data that comes closest to their process's peak temperature hold time.

To ensure the highest accuracy, the data range used to convert the TempTAB shrinkage into temperature is chosen from the most linear area of the shrinkage curve. Once the furnace runs were complete, the TempTAB discs were measured using a desktop gauge.

Flexible Type K thermocouples were used at temperatures of 1200°and 1325°C. Testing at 1325°C required the use of a fibreglass insulation sleeve to ensure that there was no liquid phase formed between the Type K thermocouple sheath and the molybdenum retort. Fig. 12 shows the location of the thermocouples, stainless steel heat sinks and ceramic TempTABs during each trial run. The example in this figure shows thermocouples with the high temperature insulation sleeve.

To record the Type K survey thermocouples during the experiment, a data acquisition unit was used. All tests were performed at DSH Technologies LLC on an Elnik MIM3045T debind and sinter furnace, with 6-zone control and proprietary AccuTemp™ Control on all Type C control thermocouples. The tests performed to gather the various data for analysis are defined in Table 8. Testing was performed under pure vacuum conditions, as well as at 300 torr partial pressure (a standard pressure used in the MIM part production industry).

When adding a retort or any partial pressures into TUS testing, the results may vary due to the capability of the process gas to alter the heat loss properties. Additionally, it has the ability to negate the insulation properties of a hot zone, resulting in the heating elements having to compensate by demanding a higher percentage of power. The performed tests in this study helped to demonstrate the variations that might be experienced under these conditions. All survey thermocouples were positioned within stainless steel heat sinks, measuring 1" in diameter and ½" in height. All TABS and thermocouples were located at the standard 9-point configuration in accordance with AMS 2750 specifications.

1	1200°C	60,90 (mins)	Vacuum (with surveys)
2	1200°C	60 (mins)	400 mbar/N_2 (with surveys)
3	1325°C	60,90 (mins)	400 mbar/H_2 (with surveys)
4	1400°C	60,90 (mins)	400 mbar/H_2 (no surveys)

Table 8 Furnace conditions used to gather data for analysis [3]

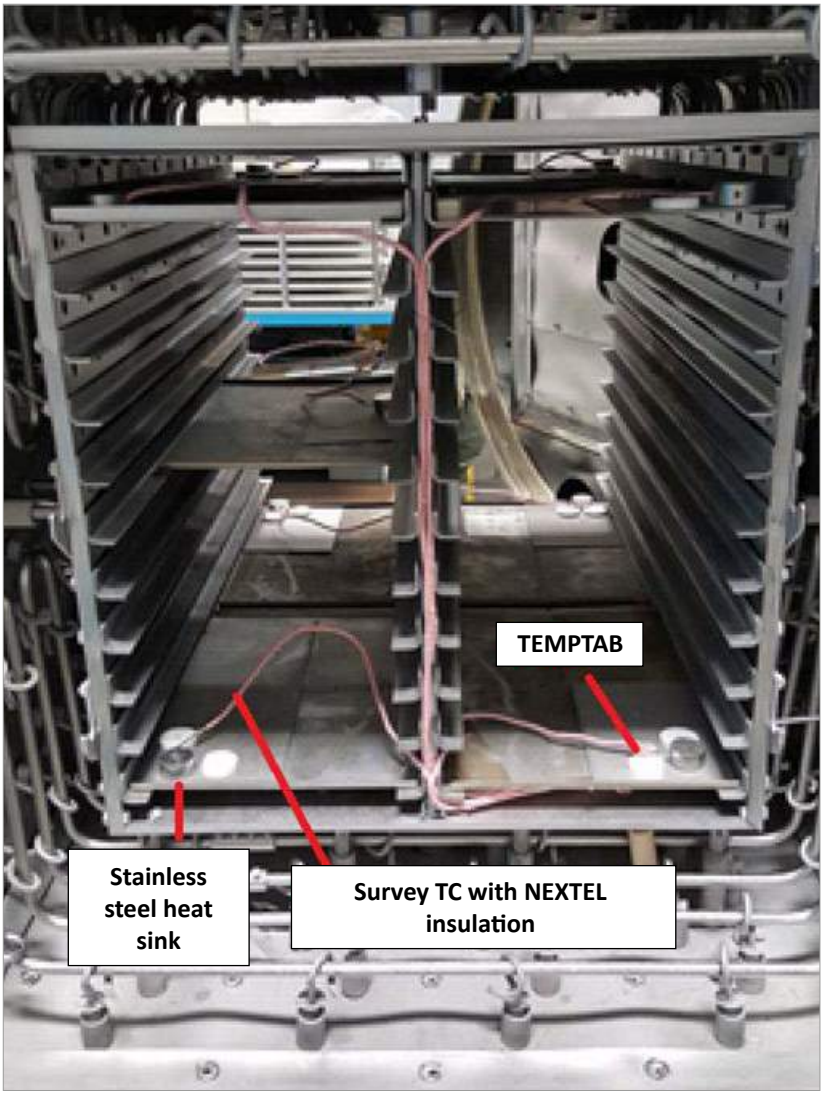

Fig. 12 Examples of the locations of survey thermocouples, stainless steel heat sinks and ceramic TempTABs within the furnace [3]

A Temperature Uniformity Survey (TUS), using established procedures and methods that fully meet the requirements of the specification, must be performed for a vacuum furnace to satisfy AMS 2750E and to allow for consistent and more accurate results of actual furnace capabilities. When performing a TUS, using a metal heat sink with metal

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thermocouples, the temperature that the thermocouple will achieve should be at or near the furnace set point and not increase the longer it sits at this temperature. When performing a TUS with TempTAB type ceramic discs, however, this is where the resulting shrinkage of the discs needs to be analysed properly. The discs that are near the outer walls of the retort will be at or near the set point temperature sooner than the discs that are located near the middle of the retort. The TempTABs are made from ceramic powders and will sinter at a slower rate when compared with most metallic powders. The ceramic continues to shrink the longer it is at the set point temperature. With ceramic powder discs, this is a concept known as heatwork, the combined effect of sintering time and temperature.

Another factor that alters the standard temperature uniformity survey testing procedure is when a process gas is introduced into the furnace chamber, which is then variably maintained at a partial pressure environment. For retort furnaces, where a sensor in the retort is used to control temperature, it is the temperature variation with respect to the sensor in the retort and not the furnace set point temperature that must be assessed.

Tests performed at 1200°C for 60 min under pure vacuum (1×10^{-3} mbar) resulted in all TempTABs displaying temperatures above 1200°C (ranging from 1203.1°C – 1209.7°C). The corresponding Type K survey thermocouples used in the same locations showed lower results, ranging from 1191°C to 1201°C. The same tests performed for 90 min showed different results. The TempTABs displayed a temperature range from 1207.5°C to 1215.5°C. However, the Type K survey thermocouples used in the same locations showed more consistent results, ranging from 1191°C to 1201°C. The differences in temperature obtained from the TempTABs compared with the survey thermocouples can be due to normal variations in scaling up from a laboratory furnace to a production furnace. By correcting the values read from the TempTAB to thermocouple values, the temperatures correlated well.

Tests performed at 1200°C for 60 min under a partial pressure of 400 mbar of nitrogen shared similar results. The TempTABs displayed temperatures ranging from 1200.9°C to 1210.4°C. The corresponding Type K survey thermocouples used in the same locations showed results ranging from 1195°C to 1203°C.

To demonstrate the effects that a gaseous environment and partial pressures have on overall uniformity within a process zone inside a retort, tests were performed at 1325°C for 60 min under a partial pressure of 400 mbar of hydrogen. Here, the TempTABs displayed temperatures ranging from 1326°C to 1338.7°C. The corresponding Type K survey thermocouples used in the same locations showed lower results, ranging from 1324°C to 1332°C. The same test performed for 90 min showed different results. The TempTABs displayed a temperature range from 1332.2°C to 1341.7°C. However, the Type K survey thermocouples used in the same locations showed more consistent results, ranging from 1324°C to 1331°C.

Tests performed at 1400°C for 60 min under a partial pressure of 400 mbar of hydrogen demonstrated better results. The TempTABs displayed a temperature range from 1394°C to 1403.3°C. There were no Type K survey thermocouples used to verify results due to material limitations at this temperature. The same tests performed for 90 min showed a similar increasing temperature result. The TempTABs displayed a temperature range from 1396.9°C to 1408.1°C.

The results from this study demonstrate that the TempTAB device can be used to assist with understanding the overall uniformity within a retort-based work zone. Tests performed at 1400°C showed the best overall uniformity of results for the TempTABs when comparing high and low values to the furnace set point. Testing at lower temperatures displayed larger deviations that might fall outside a typical acceptable +/- deviation band as commonly used for TUS testing in accordance with ASTM2750E specifications.

However, even bearing in mind this issue, it may be concluded from the results that the overall uniformity throughout the work zone of the tested equipment demonstrated an acceptable temperature deviation, when comparing the difference between all corners and the middle of the process zone. This is assessed by comparing the high and low results of the TempTABs with the temperature results of the middle positioned TempTAB.

The tests were performed in a furnace that was empty and only contained shelving for the 9 uniformity based locations. Centre temperatures measured by thermocouples demonstrated that the thermal transfer from outside to inside occurred in a matter of less than 10 min. In a furnace that is fully loaded, the heat transfer would take longer. Therefore, it is also common to see a slower ramp rate to assist with a more uniform transfer of heat throughout the process zone. Based on this thermal heat transfer principle and the results of this study, it may be inferred that a fully loaded furnace might generate data that would show a larger variation of temperature between the outside and inside of the process zone, when using only TempTABs. Further tests on this subject would lead to a clearer understanding of TempTAB behaviour in a fully loaded retort-based furnace.

Performing tests with a hold time of 60 min has demonstrated the best results, as compared with the data temperature chart from Orton. Additional hold time causes greater variation between all TempTABs located throughout the work zone. Tests, performed under a partial pressure of a gaseous environment, demonstrated a larger than expected and higher than desired variation of temperature uniformity when reading the TempTabs.

The authors drew the overall conclusion that the comparative data from the survey thermocouples,

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especially those within a partial pressure gaseous environment, demonstrate the need to develop an acceptable offset range to ensure that the TempTABs can offer a reasonable resemblance to the actual temperature involved at the measured location within the process zone. In most cases, a range of 6-10°C may be acceptable, in order to utilise TempTABs for truly accurate readings of temperatures within a retort-based processing furnace.

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POWDERMET2018, the International Conference on Powder Metallurgy & Particulate Materials, will take place in San Antonio, Texas, USA, from June 17–20, 2018. A Call for Papers & Posters has been issued and the abstract submission deadline is November 3, 2017.

www.powdermet2018.org

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www.europm2017.com

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November 12-16, San Diego, USA
www.ceramics.org

formnext powered by TCT 2017
November 14-17, Frankfurt, Germany
www.formnext.com

36th Hagen Symposium
November 30 - December 1, Hagen, Germany
www.pulvermetallurgie.com

Rapid + TCT
April 23-26, Fort Worth, USA
www.rapid3devent.com

POWDERMET2018 / AMPM
June 17-20, San Antonio, USA
www.powdermet2018.org

World PM2018
September 16-20, Beijing, China
www.worldpm2018.com

Euro PM18 Congress & Exhibition
October 14-18, Bilbao, Spain
www.europm2018.com

2018

MAPP First International Conference
January 30-31, Sheffield, United Kingdom
www.mapp.ac.uk/events/mapp-1st-international-conference

PM 18 International Conference
February 21-23, Navi Mumbai, India
www.pmai.in/pm18

MIM2018
March 5-7, Irvine, USA
www.mim2018.org

PM China 2018
March 25-27, Shanghai, China
www.cn-pmexpo.com

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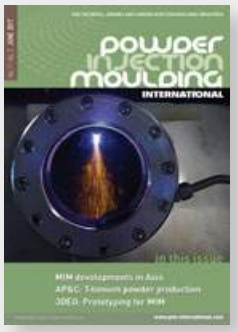

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