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FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

POWDER INJECTION MOULDING INTERNATIONAL

in this issue

Nippon Piston Ring
Ceramic Injection Moulding focus
Advanced PIM processes

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Powder Injection Moulding International

For the metal, ceramic and carbide injection moulding industries

The rise and rise of ceramics

As a class of materials, ceramics have never been as in-vogue as they are today. Ceramics have moved from solely functional precision components for a relatively narrow range of industrial applications to a new, elevated position in the world of advanced engineering, medical and luxury applications.

From Rado's first pioneering use of Ceramic Injection Moulding (CIM) three decades ago, there has been a steady rise in the use of ceramics which is based primarily on the material's unique feel, appearance and scratch resistance. Today, ceramic watch cases and components are commonplace across the market, on watches from entry level brands to the most exclusive luxury labels. As our use of smart devices continues to grow, ceramics will become an ever more attractive option, not just because of their ever-growing consumer appeal, but also their compatibility with new technologies such as wireless charging.

Ceramic components are also being developed for a new generation of technical applications, from heat management devices for LED lights to components for electric and hybrid vehicles. With its proven ability to deliver high volumes of complex, net-shape components, CIM is in a strong position to capitalise on these opportunities.

A further boost may also come from the rapid rise of ceramic Additive Manufacturing. The ability to prototype CIM-like components without the need for expensive tooling will inevitably increase accessibility to the technology. On the pages of this issue alone we report on the commercialisation of three different ceramic AM processes that could at once be invaluable tools for CIM producers or, of course, a threat.

Nick Williams,
Managing Director & Editor

Cover image
A CIM thermal insulation tube in the green, sintered and finished states (Courtesy Kläger Spritzguss GmbH & Co. KG, Germany)

March 2018

Powder Injection Moulding International

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

Morgan Advanced Materials expects surge in demand for CIM medical and dental implants

The Technical Ceramics business of Morgan Advanced Materials is expecting an imminent surge in demand for ceramic medical and dental components as a result of a recent change in legislation whereby some USA medical insurers now cover implantable devices. Morgan's proprietary zirconia grade is already ISO 13356 approved, ensuring full compliance with stringent regulations around the production of medical implements.

This includes applications such as surgical tips that are widely used in microwave ablation operations for cancer treatment, as well as implantable casings that can become part of a miniaturised bioelectronic device used in cranial and spinal implants. 'Metal free' dental implants are also widely acknowledged as the next phase in dental implant technology.

Morgan's Technical Ceramics division will take advantage of its continued investment in CIM technology to meet demand for complex parts such as implants to be produced in high volumes to tolerances as low as 0.5%. Compared with conventional methods for manufacturing engineering ceramic components, where tolerances are typically between 1 - 2%, the CIM approach eliminates the time and cost associated with additional machining of components, resulting in high tolerance parts in complex geometries with shorter lead times.

Many implants now incorporate electrical circuits as part of their design, adding further complexity to the supply chain due to the requirement for specialist brazing. The company reports that it has the capability to deliver ceramic to metal assemblies using molybdenum manganese (MoMn) brazing and metallised ceramics as well as active alloy brazing with Morgan Technical Ceramics' extensive range of active braze alloys.

Dr Yifei Zhang, Business Development Manager at Morgan Technical Ceramics, explained, "We are expecting a real increase in demand for medical implants and our integrated business model means we are well positioned to help accommodate future demand. Our long-standing relationship with the medical sector makes us the ideal partner for manufacturers of implants."

www.morgantechnicalceramics.com/medical

Dynacast becomes Form Technologies, new OptiMIM Metal Injection Moulding brand launched

Dynacast International Inc., headquartered in Charlotte, North Carolina, USA, recently announced that Dynacast and its associated brands has become a family of brands under a newly formed entity called Form Technologies. The new company will combine three precision component manufacturers, which are all regarded as leaders in their respective market segments: Dynacast for precision engineered die cast components, Signicast for investment casting and prototyping and OptiMIM, the company's new brand for Metal Injection Moulding.

Under Form Technologies, the companies will undergo a brand redesign for consistency across the platform. However, each company will maintain its own identity and continue with business as usual.

Form Technologies will be led by Simon Newman, Dynacast's Chairman and CEO, who stated, "As we continue to make strategic acquisitions that add processes and capabilities to our portfolio of companies, it became evident that we needed a larger, more inclusive brand that allowed us to expand our identity beyond a specific metal or process. With Form Technologies, we are better positioned for future acquisitions that fall within our same strategic vision of becoming the leading global manufacturer of precision components." Dynacast first announced that it had developed its own variant of the MIM process in 2013. This was followed in 2014 with the acquisition of US MIM producer Kinetics Climax, Inc.

www.optimim.com

Ipsen USA reports multiple furnace shipments

Ipsen USA, Cherry Valley, Illinois, USA, has reported a strong close to its financial year 2017. The company stated that it shipped fifteen furnaces in its fourth quarter 2017 to customers in eight US states, as well as in Asia and Europe. This includes customers in the Metal Injection Moulding and Additive Manufacturing industries.

The reported shipments included nine TITAN® vacuum furnaces, including three TITAN DS (debinding and sintering) units, two LT (low temperature) units and a number of H2 and H6-sized furnaces.

The company stated that some of the furnaces sold were shipped to repeat customers and one customer placed two furnace orders.

www.ipsenusa.com

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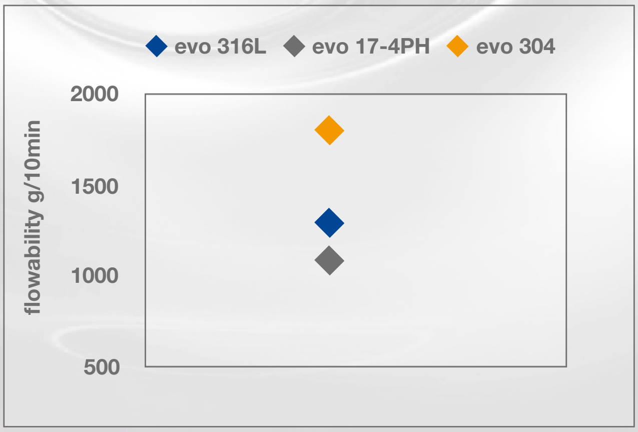


BASF
We create chemistry

Arburg's second international PIM conference scheduled for June

Arburg's second international Powder Injection Moulding conference is set to take place in Lossburg, Germany, from June 5-6, 2018. Invited participants from around the world will meet at the company's headquarters in order to examine current trends in the PIM industry.

Arburg's first international PIM conference was held in Lossburg in 2013 to celebrate fifty years of the company's involvement in PIM. This first event was praised for its distinctive international character, which provided the opportunity for 250 industry experts to network and conduct discussions as to the benefits and opportunities for the technology.

Commenting on the event's second iteration, Gerhard Böhm, Arburg's Managing Director Sales, stated, "We would now like to build on this success and hold the second major event of this kind for the international PIM world in June 2018."

On both days of the event, focused presentations will review the potential, technical options and future prospects for the PIM process, enabling companies to exchange their experiences of the industry. Experts from globally active companies and institutions in the US, Asia and Europe have been invited to present during the conference, including keynote presentations from:

- Professor Randall M German, San Diego State University, USA: 'The future of Powder Injection Moulding: Growth in materials versus applications'
- Professor Peng Yu, South University of Science and Technology of China: 'The current status of and outlook for the Asian PIM sector'
- Professor Frank Petzoldt, Fraunhofer IFAM, Germany: 'Current developments in MIM technology'

In addition, Arburg reports that speakers from international PIM part producers will report on practical application examples, while material manufacturers will present on the latest feedstock innovations. PIM specialists from Arburg itself are expected to present the company's latest innovations in areas including mould technology.

The event will also offer visitors the opportunity to view PIM part production in practice. In the Arburg Customer Center, injection moulding machines will produce a number of PIM components in the breaks between presentations, so that the participants can experience the process 'live'. During a company tour, visitors will also be able to view Arburg machines during their production and assembly.

www.arburg.com

The Customer Center in Lossburg will once again be the venue for Arburg's PIM conference (Photo: Arburg)

XJet to showcase ceramic NanoParticle Jetting at Ceramitec 2018

XJet Ltd., Rehovot, Israel, is set to showcase its ceramic Additive Manufacturing technology at Ceramitec 2018, Munich, Germany, April 10-13, 2018. The company's new Additive Manufacturing technology uses NanoParticle Jetting™ to enable the production of short-run ceramic parts with complex geometries, superfine detail and high density without using moulds.

Additive Manufacturing enables reduced product development timescales and costs, as well as a greater degree of design freedom. Using AM, complex parts are easier to produce and a greater range of design iterations is possible through the elimination of tooling and short production times.

XJet's Carmel 1400 AM System will run live on the stand throughout the show. Printing ultrafine layers of NanoParticle 'inks', users of the system are reported to be able to produce ceramic parts with the ease and versatility of inkjet printing. Hanan Gothait, CEO and Founder of XJet, stated, "We're very excited to see Ceramitec's reaction to a new game-changing manufacturing process."

"With a unique combination of detail, density and design freedom not previously afforded by traditional ceramics manufacturing, we think our new NanoParticle Jetting AM technology can have a significant positive impact on the industry," he continued. "At Ceramitec, our expert team will be poised to answer questions, we'll have lots of sample parts, plus visitors can take a 'deep-dive' tour of the technology with an augmented reality experience on the XJet stand."

Ceramic AM parts by XJet

Dror Danai, XJet CBO, added, "With our first installations already underway, it's an exciting time for XJet and also means we can talk about real-world applications at Ceramitec. Our first Carmel AM system is up and running at Oerlikon citim in Germany, whilst the second has shipped to the Youngstown Business Incubator (YBI) in the USA for imminent installation. Both sites are using ceramic material to produce parts for a variety of applications serving a plethora of industries."

www.xjet3d.com
www.ceramitec.com

Morgan invests in Lithoz ceramic Additive Manufacturing system

Morgan Advanced Materials, Windsor, UK, has made an investment in ceramic Additive Manufacturing with its acquisition of an AM system from Lithoz, Vienna, Austria. The system, which uses Lithoz's lithography-based Ceramic Manufacturing (LCM) technology, and is capable of processing high-performance ceramics, will be installed at Morgan's Global Centre of Excellence in Stourport-on-Severn, UK, which the company states is a global hub for the development of new structural ceramic materials and processes.

Parts developed at Morgan's Centre of Excellence are designed for application in sectors as diverse as aerospace, medical and energy. Using LCM technology, the company reports that it is possible to additively manufacture ceramic parts with the same properties as conventionally formed parts.

Mike Murray, CTO of Morgan Advanced Materials, explained, "Additive Manufacturing has the potential to set new standards for the manufacture of high-performance ceramics and we are excited to be increasing our exploration of the possibilities of the technology."

Johannes Homa, Lithoz CEO, stated, "Morgan is as a future-oriented company earning its success by pioneering technical innovations. We are grateful Morgan trusts in Lithoz manufacturing systems. Innovative enterprises and technology leaders such as Morgan have already internalised that technological advantages come incrementally by affording oneself to reconsider traditional thinking patterns."

"With its professional expertise Lithoz considers itself as a strong partner along the supply chain of AM of ceramics," he continued. "We are not just selling machines. We accompany our customers as partners by providing them with any necessary support they need - so they can benefit from technological virtues on an emerging market."

Morgan Advanced Materials plc is a global engineering company and is listed on the London Stock Exchange. It operates approximately eighty-five manufacturing sites across more than thirty countries, serves customers in more than one hundred countries and has expertise in Ceramic Injection Moulding. With this investment, Morgan stated that it is on the forefront of evaluating the opportunities arising from the AM of high-performance ceramics.

www.lithoz.com
www.morganadvancedmaterials.com

Metal Powder Products acquires NetShape

Metal Powder Products LLC ("MPP"), a Mill Point Capital portfolio company based in Westfield, Indiana, USA, has acquired NetShape Technologies Inc., a leading manufacturer and solutions provider of MIM and PM components headquartered in Floyds Knobs, Indiana.

Dennis McKeen, President and CEO of MPP, stated, "The combination of MPP and NetShape creates a global leader in powdered metal manufacturing. The combined company's increased scale and capabilities will provide our customers with unmatched quality and service."

"MPP and NetShape are a perfect fit from a business perspective," commented Dax Whitehouse, CEO of NetShape. "The two businesses strongly complement each other, and we look forward to building upon our respective strengths."

"The NetShape acquisition provides MPP with key strategic additions such as MIM capabilities and a Chinese manufacturing footprint," said Chuck Spears, Executive Chairman of MPP. "The combination reflects MPP's continued focus on investments that drive value for customers and enhance the company's culture of quality and reliability."

www.metalpowder.com | www.netshapetech.com

GKN confirms plans for sale of Powder Metallurgy business

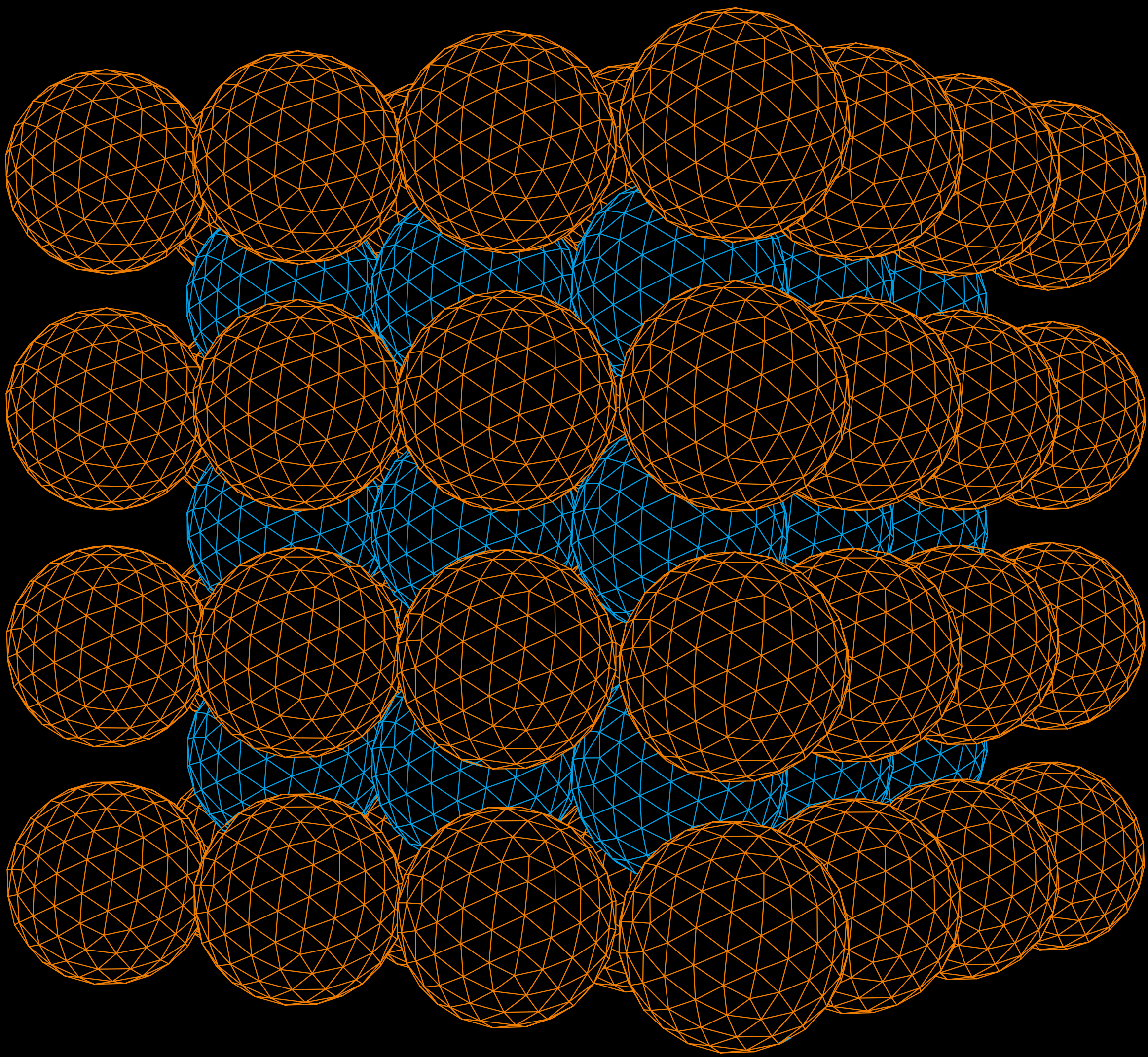
GKN plc has confirmed that it will look to divest GKN Powder Metallurgy, comprising GKN Sinter Metals and Hoeganaes, within the next 12-18 months as part of its new business strategy to transform the company, including the sale of non-core segments. GKN's PM division also includes a large Metal Injection Moulding operation in Radervormwald, Germany. The new strategy announcement comes in response to Melrose plc's widely-reported takeover bid in January 2018. After an initial proposal from Melrose to acquire GKN was rejected by GKN's Board, both companies have launched campaigns to convince GKN shareholders of their respective plans for the group.

GKN stated that there are three components to its new strategy: the company plans to deliver distinct strategies for different product segments through rigorous capital allocation and focused performance targets; establish a delivery culture based on greater accountability, capability and pace, supported by aligned incentives; and separate operationally now and formally when it maximises shareholder value – with the operational separation of GKN Aerospace and Driveline already underway.

As part of a plan to divest non-core segments, it will also look to sell GKN Driveline's Wheels, Cylinder Liners and Off-Highway Powertrain businesses, while growing Driveline China and developing its eDrive business. GKN Aero Additive Manufacturing was also identified as a product segment positioned for growth. The Board stated that it is targeting up to £2.5 billion cash return to shareholders over the next three years, with a significant portion of this expected to come from divestments executed in the first 12-18 months, including the sale of PM. The group will also enact a 'progressive dividend policy' targeting an average pay-out of 50% of free cash flow to its shareholders over the period of 2018 - 2020.

Anne Stevens, GKN's CEO, stated, "The new strategy brings clarity, accountability and focus to GKN's world-class businesses and will allow the group to attain world-class financial performance. GKN has great technologies and great people. We have strong market positions and have delivered good growth, with management revenues last year of over £10 billion." She added, "But too often we pursued growth at the expense of returns. This will no longer be the case. The new strategy brings discipline, both financial and operational. We expect to deliver £340 million of recurring annual cash benefit from the end of 2020".

www.gkn.com



Höganäs to acquire H.C. Starck Surface Technology & Ceramic Powders

Höganäs AB has signed an agreement to acquire H.C. Starck Group's Surface Technology & Ceramic Powders (STC) division. STC manufactures a wide variety of thermal spray powders and complementary coating technology materials, as well as ceramic and metal powders. The company has two production facilities in Germany and currently operates as a legally separated stand-alone division within the H.C. Starck Group. In addition to its extensive non-oxide ceramic powder portfolio for advanced ceramics and high-end applications, STC's atomised metal powders are targeted at a broad scope of innovative technologies including AM, HIP and MIM.

Fredrik Emilson, Höganäs CEO, stated, "STC's broad product portfolio and strong trademarks will expand our existing product portfolio and add significant product development capabilities and know-how to Höganäs. The acquisition of STC also enables us to get access to new customer segments within aerospace and adds a complementary geographic fit with STC's strong presence in Europe in addition to our strong geographical presence in Asia and the Americas".

"STC's extremely competent workforce, together with significant development capabilities, will enable us to support our customers more and help them bring new applications to the market. We look forward to welcoming our new colleagues to Höganäs and to collectively work towards achieving our targets and cement our position as the world's leading manufacturer of metal powders."

The closing of the transaction is expected during the first half of 2018 and is subject to the approval of relevant authorities.

www.hoganas.com

www.hcstarck.com

Arcam delists from Nasdaq Stockholm exchange

GE has announced that Arcam AB has now been delisted from the Nasdaq (Stockholm) exchange, a move which the company states will allow a more fulsome integration with GE. GE purchased controlling shares of Arcam in a public cash offer to tender all shares in late 2016. The company recently surpassed 90% ownership of Arcam AB shares, permitting initiation of the compulsory buy-out of the remaining shares in accordance with the Swedish Companies Act.

Based in Sweden, Arcam is the parent company of AP&C, a leading producer of advanced metal powders for MIM and AM with customers in the aerospace and orthopaedic industries. The last day of trading of Arcam stock was reported to be January 26, 2018.

www.ge.com/additive

Sandvik to build $25 million titanium and nickel metal powder plant

Sandvik AB, headquartered in Stockholm, Sweden, is investing approximately 200 million SEK ($25 million) in a new plant for the manufacturing of titanium and nickel fine metal powders. The new facility, within the business area of Sandvik Materials Technology (SMT), will be located in Sandviken, Sweden, close to the company's in-house titanium raw material supply and centre for Additive Manufacturing. The plant is expected to be operational in 2020.

According to the company, the investment will complement SMT's existing powder offering and strengthen Sandvik's position in the rapidly growing market for metal powders. Sandvik is a leading producer of fine metal powders and serves a number of Additive Manufacturing companies. Its stainless steel, nickel-based and cobalt-chromium alloy powders are manufactured in the United Kingdom and Sweden and sold across Europe, North America and Asia through the Sandvik Osprey brand.

With demand for metal powders expected to increase significantly in the coming years thanks in part to the growth of metal Additive Manufacturing, the company sees titanium and nickel-based alloys as key growth areas accounting for a significant portion of the metal powder market.

Annika Roos, Head of product area Powder at Sandvik Materials Technology, commented, "This investment is an enabler for future growth and means that we are expanding our metal powder offering to include virtually all alloy groups of relevance today. In addition, it will also support the overall Additive Manufacturing business at Sandvik."

"The metal powder segment and the Additive Manufacturing business are of increasingly strategic importance to us. This investment should be viewed as the latest evidence of our commitment to an area that we believe strongly in," added Göran Björkman, President of Sandvik Materials Technology.

www.home.sandvik

POWDERMET2018 heads to Texas

The Metal Powder Industries Federation (MPIF)'s POWDERMET conference and exhibition series is North America's largest Powder Metallurgy and Particulate Materials event. This year it takes place in San Antonio, Texas, USA, from June 17-20 and the conference will once again prominently feature the latest advances in Metal Injection Moulding research. Within the exhibition hall, leading supplies of MIM materials and equipment will be promoting their latest innovations.

www.mpif.org

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CeramTec showcases its ceramics for electromobility at eMove360°

CeramTec Group, Plochingen, Germany, showcased its solutions for electromobility at eMove360°, an international trade fair for Mobility 4.0. The event, which took place from October 17-19, 2017, in Munich, Germany, featured innovations in connected and autonomous driving, urban mobility and vehicle design. CeramTec demonstrated key ways in which ceramic materials are taking on a key role in innovative drive concepts. The company presented advanced ceramic components for use in fuel cells, systems for generating, transmitting, storing and distributing energy, and in power electronics and temperature management.

Ceramic materials are distinguished by their mechanical strength and tribological properties, as well as their electrical insulation and thermal conductivity. This enables ceramic components to be applied in areas in which traditional materials such as metals and plastics have limited suitability. Ceramics also offer resistance to changes in temperature, chemicals and corrosion. CeramTec processes a wide range of materials and combinations of properties, making it possible to offer customised solutions across the entire spectrum of e-mobility, including for automobiles, electric bicycles and electric motorbikes.

CeramTec uses piezoceramics in the production of very small ceramic components which, it states, are already widely used in the automotive industry. Piezoceramic parts generate an electrical charge when mechanically deformed, and are also capable of converting electrical signals into mechanical movement or vibration. As a result, they can be used in sensor and actuators, in power transducers or in intelligent engine management systems, among other applications. They also play an important role in safety applications such as distance sensors, parking aids and airbags.

CeramCool®, CeramTec's air or fluid heat-sinks, are used to enable drives in electric vehicles to deliver the highest possible electrical power. The challenge here lies in controlling this power on the smallest of spaces, consistently and reliably over long periods of time. The surrounding temperature plays an important role; CeramCool air or fluid heat-sinks present extremely low thermal resistance during cooling, while at the same time possessing electrical insulation. This is also reported to make CeramCool suitable for use in applications such as thermal management for batteries, voltage converters, drive control systems or braking energy recovery.

Acquisition by BC Partners

CeramTec Group was recently acquired by a consortium led by funds advised by private equity firm BC Partners. The consortium, in which the Public Sector Pension Investment Board and Ontario Teachers' Pension Plan both hold a stake, reached an agreement to acquire CeramTec Group from its current owner, Cinven.

Henri Steinmetz, CEO of CeramTec, commented on the transaction, "We are delighted to welcome the BC Partners-led consortium as our new owners. Over the past four years, we have invested substantially in our operations and our people. We have doubled our ceramic implant capacity in Marktredwitz, we have simplified the organisational set-up and we have created a leading platform in piezoceramics with a UK acquisition."

"In partnership with Cinven, we have started our journey from a German-centric technology leader towards a true global market leader. We are looking forward to continuing on this journey together with our new owners," he concluded.

Stefan Zuschke, Managing Partner of BC Partners, stated, "We believe CeramTec has great potential to achieve profitable and sustainable growth, both organically and through acquisitions, and we look forward to working together with the company's management team and its employees." The acquisition is subject to approval by anti-trust and foreign investment authorities.

www.ceramtec.com

2C-CIM and in-mould labelling combined for high temperature applications

In a publicly funded German Central Innovation Programme for SMEs (ZIM) project, Fraunhofer IKTS and M.K.S. Kunststoff-Spritzguss GmbH have developed a sensor part made with a combination of two component (2C) Ceramic Injection Moulding and in-mould labelling (IM), which pairs electrical conductive and electrical insulating ceramic materials.

Fraunhofer IKTS's Dr Tassilo Moritz and Johannes Abel stated that to achieve electrical conductivity, a $MoSi_2$ and SiC containing Si_3N_4 system was modified by exceeding the percolation limit of SiC and $MoSi_2$ phase. Electrical insulation was also achieved by underrunning the percolation limit, having a higher amount of non-conductive Si_3N_4 in the alloy. These materials are suitable for high temperature applications such as sensors for metal melts or heating devices. The key requirements for combining different materials for high temperature applications are to choose materials with the same coefficient of thermal expansion (CTE) for fatigue concerns and to adjust the same shrinking behaviour for co-sintering.

Custom-made spray dried powders were used to manufacture a thermoplastic feedstock for high pressure injection moulding. Additionally, for tape casting, a slurry made of the conductive powder was developed to fabricate thin tapes by

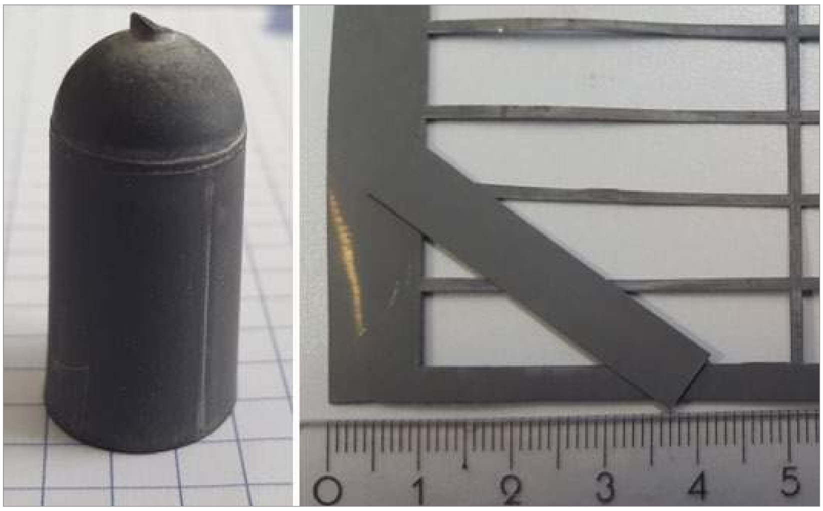


Fig. 1 The co-sintered sensor part (left) and laser cut Si_3N_4 / $MoSi_2$ green tape for in-mould labelling (right)

the doctor blade technique. After laser cutting, these tapes were used for injection moulding by insertion into the tool cavity prior to subsequent injection moulding of the feedstock. This thimble-like component consists of an electrical insulating cylinder with an electrical conductive hood. In the cylinder, an electrical conductive thin tape is implemented in an axial manner by in-mould labelling to connect the base of the cylinder with the hood (Fig. 1).

The tape was placed in the tool in such a way as to allow the feedstock's flow fronts to clamp the tape without wrinkling during injection. This prefabricated cylinder was then transferred into a second cavity to be covered with the conductive hood. After co-sintering, an electrical resistivity could be measured between base and hood (Fig. 2). The conductive regions are in red while the insulated cylinder is the grey transparent area. A Field Emission Scanning Electron Microscopy (FESEM) image of the microstructure in the triangle cylinder, tape and hood is shown in Fig. 3.

This study demonstrates the high flexibility of CIM by combining 2C-CIM with IM. The project was funded by the Federal Ministry for Economic Affairs and Energy, Germany, on the basis of a decision by the German Bundestag (KF2087364AG4 and KF2449902AG4).

www.ikts.fraunhofer.de | www.mks-gmbh.de

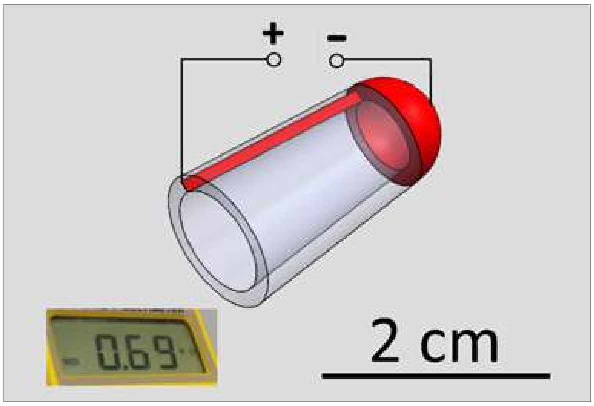


Fig. 2 CAD sketch of the sensor element, principle of resistance measurement

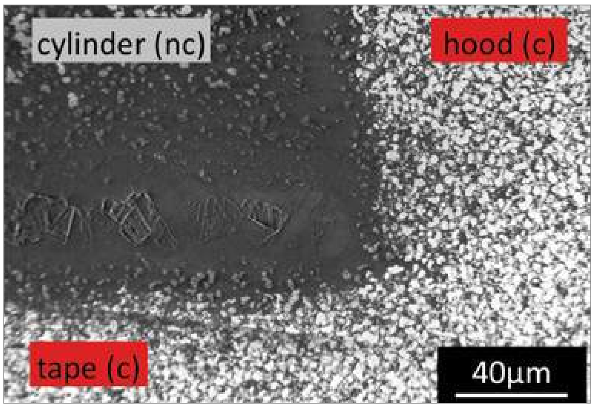


Fig. 3 In-lens FESEM image of the microstructure of the triangle cylinder, tape and hood (c=conductive, nc= non-conductive)

Malaysian team fabricates endoprosthetic hip stem using MIM

A recently completed research project undertaken by SIRIM Berhad (formerly the Standard and Industrial Research Institute of Malaysia) and the International Islamic University of Malaysia (IIUM) has successfully produced a prosthetic hip stem using Metal Injection Moulding.

The use of Co-based alloys for surgical applications is mainly related to orthopaedic prostheses for the knee, shoulder and hip as well as to fracture fixation devices. Joint endoprostheses (prosthetics which are implanted in the body) must meet extremely high requirements with regards to biocompatibility and corrosion resistance. CoCrMo has been widely used in endoprosthetics due to its mechanical properties, good wear and corrosion resistance and biocompatibility.

Dr Mohd Afian Omar, Principal Researcher, SIRIM Bhd, and colleagues are investigating the use of MIM for the production of femoral hip stem implants with high surface quality. To manufacture the new implants, the team used CoCrMo alloy metal powder with a median particle size of 15 µm and a binder consisting of palm stearin and poly ethylene, with a powder loading of 65 vol.%.

SIRIM Berhad's MIM prosthetic hip stem

A closer view of the MIM hip stem

According to the team, the hip stems were injection moulded using a vertical injection moulding machine with the nozzle temperature of 200°C. The hip stems were then debound using a combination of solvent extraction and thermal pyrolysis method, before sintering under vacuum at 1390°C. The results showed that the highest final sintered densities were about 98% of theoretical maximum value with good mechanical properties, including hardness of 42 HRC, elongation of 10% and UTS of 822 MPa, in line with standard ASTM F75.

To determine the potential toxicity and biocompatibility of the prosthetic, a cytotoxicity test of the hip stem was conducted, in which it was extracted for twenty-four hours in Minimum Essential Medium (MEM), and an extract prepared from the test material then placed on cell monolayers. The cells were examined for morphologic changes, malformation, degeneration and cytolysis to determine a toxicity score. The cell morphology showed very little change from that of the control specimen, indicating that no morphological abnormalities took place. Cell growth towards the material demonstrated good biocompatibility, particularly on longer incubation times, which also indicated good material-tissue integration. A biomechanical study and preclinical test are expected to be conducted soon.

Email: afian@sirim.my
www.sirim.my

Bodycote to expand European HIP capabilities

Bodycote's Hot Isostatic Pressing (HIP) facility at Sint Niklaas, Belgium, is awaiting delivery of a new 'Mega-HIP' unit which is expected to be operational by the end of 2018. According to the company, the new high-pressure, high-temperature Mega-HIP is Nadcap capable and is expected to aid the company in meeting growing demand from the European aerospace market over the next five years and beyond. This investment is expected to significantly increase Bodycote's Nadcap HIP capacity globally and follows an increase in Nadcap capable HIPing capacity which it completed in 2017. These recent investments were said to highlight the company's commitment to expanding its global HIP capacity to meet market requirements.

Bodycote operates the world's largest HIP equipment network and continues to invest in recognition of the growing demand for HIP technology. Having established its HIP expertise over several decades, Bodycote has over fifty HIP vessels of varying sizes in multiple locations. Its processing capabilities can reportedly accommodate components which are nominally up to 2 m diameter by 3.5 m high and weighing from 0.1 kg to over 30,000 kg. As well as standard quality and environmental accreditations, Bodycote's HIP facilities also hold ASTM and NORSOK accreditations. In addition to aerospace, Bodycote HIP serves clients around the world in markets as diverse as the medical, power generation, marine, nuclear, automotive and electronics industries, with both HIP services and its Powdermet® technologies.

The recently launched Powdermet technologies incorporate new, patent-pending techniques that combine Additive Manufacturing with well-established net shape and near net shape (NNS) techniques. This new hybrid technology is said to dramatically reduce the manufacturing time and production cost of a part compared to producing the same part using AM alone.

www.bodycote.com

A HIP vessel is lowered into place at one of Bodycote's facilities (Courtesy Bodycote)

EPMA's 2018 Powder Metallurgy Component Awards open for entries

The European Powder Metallurgy Association (EPMA)'s 2018 Powder Metallurgy Component Awards has opened to entries from EPMA members. The 2018 competition will consist of the following component categories:

- Additive Manufacturing
- Hot Isostatic Pressing
- Metal Injection Moulding
- PM structural (including hard materials and diamond tools parts)

Entries will be judged by a panel of independent industry experts from across Europe. According to the EPMA, judging will be on the grounds of the following criteria:

- To what extent is the PM component described in the entry expected to provide cost savings and/or improved quality?
- To what extent is the entry expected to stimulate further usage of PM materials and technology?
- How well is the entry prepared (description of component, inclusion of diagrams, photographs and other illustrations)?
- How does the component rate in terms of excellence in exploiting PM or in terms of novelty, surpassing borders or bringing new ideas into practice?

The deadline for entries is May 31, 2018. The awards will be presented during the Euro PM2018 Congress & Exhibition in Bilbao, Spain, October 14-18, 2018. All entries will be extensively marketed by the EPMA in print and online, providing an opportunity to promote the latest innovations in PM to an international audience.

www.epma.com ■

MAPP reports success of First International Conference

MAPP (EPSRC Future Manufacturing Hub in Manufacture using Advanced Powder Processes) has reported that its First International Conference was a success. Held in Sheffield, UK, from January 30-31, 2018, the conference is reported to have attracted some 180 delegates and speakers from America, France, Germany, Spain and the UK, from over thirty-five companies and twenty universities.

Taking place one year after MAPP's official launch, the event was targeted at bringing together experts in particulate and powder science across a wide range of manufacturing processes, with a particular focus on in-situ process and performance characterisation, advanced characterisation, and modelling, optimisation and control.

www.mapp.ac.uk ■

Tosoh boosts zirconia powder production capacity

Tosoh Corp., headquartered in Tokyo, Japan, is a global manufacturer of basic and fine chemicals, petro-chemicals, and specialty products covering a broad spectrum of materials including zirconia grinding and dispersion media, zirconia fine beads and powders, zirconia injection moulded components and zirconia-based feedstocks for Ceramic Injection Moulding.

For its first half 2018, the company reported consolidated net group sales of ¥391.7 billion ($3.5 billion), up 16.2%, from the same period a year earlier. Net sales by the Specialty Group, which includes the zirconia-based materials, increased by ¥9.6 billion, or 11.6%, to ¥91.9 billion ($827.6 million). In March 2016, the Specialty Group launched commercial production of zirconia powder at the Nanyo Complex's new zirconia manufacturing facilities in Shunan City, Yamaguchi Prefecture. This was followed by a similar launch at the company's Yokkaichi Complex in Yokkaichi City, Mie Prefecture, in April 2017, further boosting Tosoh's zirconia powder production capacity.

Tosoh produces a wide range of CIM parts from zirconia and yttria-stabilised zirconia for industrial and commercial applications such as fibre-optic connectors, electronic parts, grinding balls, etc. Recent advances at Tosoh are extending the market for zirconia powder to dental applications and decorative fashion accessories, including watch cases and bracelet parts.

www.tosoh.com

Setter plates for CIM and MIM sintering

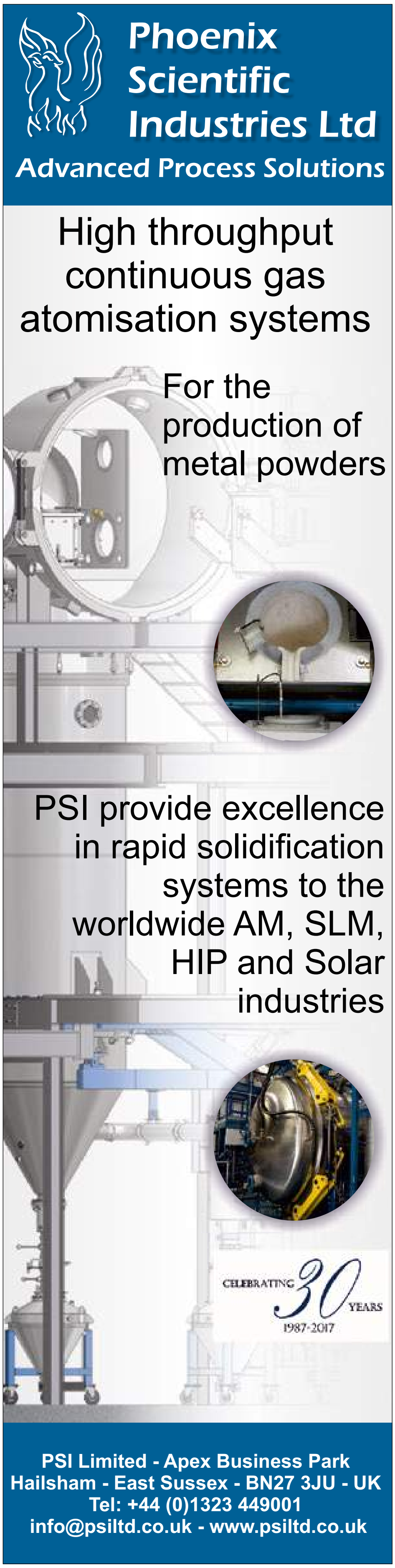
Sintering trays and setter plates help to optimise part arrangement and secure CIM and MIM parts in the sintering furnace in order to prevent undesirable deformations during sintering.

CeramTec GmbH, Marktredwitz, Germany, produces Al_2O_3 and AlN setter plates/sintering trays, which the company states feature low surface roughness for optimum gliding of the injection moulded parts when filling the plates, high thermal conductivity to ensure homogeneous thermal distribution within the parts being sintered, excellent shock resistance allowing for faster sintering cycles, and improved energy efficiency in the furnace.

Setter plates and fixtures manufactured by CeramTec GmbH

Sintering setter plates made from advanced Al_2O_3 and AlN ceramics also make the use of releasing agents or protective layers such as coatings obsolete, as there are no contact reactions with metals. Thus, they also have a long lifetime and do not require reconditioning. Both AlN and ultrapure alumina (> 99%) can be used in protective gas atmospheres and reducing atmospheres. They are also stable in reactive atmospheres and in hydrogen.

www.ceramtec.com



CNPC nears completion of facility for metal powder production

CNPC Powder Group is nearing completion of a new 30,000 m^2 facility in Anhui, China, that will provide a major increase in capacity for the production of metal powders. The company is reported to be on schedule to begin manufacturing powders in July 2018, with an annual capacity capable of reaching around 3,500 tons in 2019.

The facility is expected to house six metal powder production lines and will provide clients with services across the metal powder process chain, from alloy development to powder production and testing. The expansion will see CNPC strengthen its position as an international producer of materials for Metal Injection Moulding and Additive Manufacturing, adding to the company's extensive range of metal powders for a wide variety of applications.

Also housed on site will be CNPC Powder's new research and development facility, which it states is set to focus on three critical projects. Firstly, the team will explore new materials, looking at the production of new alloys for MIM and AM applications. Secondly, the team will investigate the optimisation of material production and cost reduction, which it hopes will lead to greater cost savings for clients. Finally, the facility will serve as a research hub for innovation in AM.

CNPC Powder is a family owned and operated business established in 1998, which specialises in the development and commercialisation of powdered metal materials. The company has invested in a number of expansions over the years to increase its capacity and secure its position in the market. From 2003-2005, three factories were established to serve a growing domestic market and in 2009 the Shanghai office was opened to enable exports of powders to international clients. In 2015 a North American subsidiary, CNPC Powder North America, was established to expand services in the Americas.

As a result of its North American expansion, CNPC Powder states that it has been able to establish connections with North and South American powder users as well as a growing presence in Europe. As the company develops new powders for advanced manufacturing processes such as Additive Manufacturing, the new facility and R&D Centre at Anhui will allow for continued support. "This facility is a step in line with our core values of long-term relationship building and developing a high-quality product for our clients," concluded Abigail Franco, CNPC's Marketing Director.

www.cnpcpowder.com

Arcast installs inert gas atomiser at Royce Translational Centre

Arcast Inc. has installed an inert gas atomiser at the new Royce Translational Centre (RTC) in Sheffield, Yorkshire, UK. The centre is part of the Sir Henry Royce Institute for Advanced Materials at the University of Sheffield and aims to evolve novel materials and processing techniques developed by research teams and make them accessible for trial by industry.

As part of this project, Arcast was chosen to supply an inert gas atomiser for the production of advanced alloy powders. The system is reportedly capable of producing large quantities of clean, spherical, fine metal powders based on titanium, iron, cobalt, nickel and many other alloys, and is complemented with a wide range of other advanced casting and Powder Metallurgy equipment. The RTC hopes to situate itself as an important centre for the advancement of technology in the field of Additive Manufacturing and will allow the full scope of materials science to be explored in this field from raw elemental metallurgy, through the Powder Metallurgy route to finished components, using the latest in powder production and AM techniques.

www.arcastinc.com

Arcast's inert gas atomiser (Courtesy Arcast)

Verder adds to testing capabilities

Dutch technology group Verder Group has acquired the majority of Qness GmbH, Golling, Austria, a manufacturer of hardness testing machines used in both industrial and research applications. With the acquisition, Verder stated that it extends the portfolio of its Scientific Division, adding a product line for hardness testing.

Qness's hardness testing machines are said to comply with recognised standards such as Brinell, Vickers, Knoop and Rockwell. The company develops and manufactures all products at its Golling headquarters, where it employs forty staff. According to Verder Group, the Qness portfolio complements the offering of fellow Verder Scientific company ATM GmbH. The existing Qness management team will remain in place.

www.verder.com

NSL Analytical receives scope expansion and renewal of ISO/IEC 17025 certification

Independent commercial testing laboratory NSL Analytical Services, Inc., Cleveland, Ohio, USA, has received scope expansion and renewed ISO/IEC 17025 accreditation for both its Metallurgical and Analytical Laboratories.
Additional specifications and methods have been adopted by NSL and received approval from ANAB. The scope expansion includes these added capabilities:

- ASTM B214 Sieve Testing for Sieve Analysis – Metal and other Powders
- ASTM B527 Tap Density for Metal Powders
- ASTM B213 Flow Rate for Metal Powders
- ASTM B212 Apparent Density for Free Flowing Metal Powders
- ASTM B417 Apparent Density for Non Free Flowing Metal Powders
- AMS 2750 Heat Treat of Specimens for Metallic Materials
- IEC 6221-7-2 Colorimetric Cr+6 on Polymers and Electronics for RoHS Testing
- IEC 62321-8 Phthalates for RoHS Testing
- SEM Qualitative Elemental Analysis, EDS Profile, Surface Morphology and Imaging with Hitachi Scanning Electron Microscope Model SU3500

"This accreditation recognises the testing competence of NSL in everything we do," commented Larry Somrack, NSL President. "NSL has been ISO/IEC 17025 accredited since 1999. Our commitment to quality and continuous improvement has made our accreditation of ISO/IEC 17025 a cornerstone of trust for our clients."

www.nslanalytical.com ■

EPMA launches 2018 Powder Metallurgy Thesis Competition

The European Powder Metallurgy Association (EPMA) has launched its 2018 Powder Metallurgy Thesis Competition at both Diploma/Masters and Doctorate/PhD levels.

The competition is open to all applicants who have graduated from a European university and who have had their theses approved during the 2015/2016, 2016/2017 and 2017/2018 academic years. The subject must be classified under the topic PM, including MIM. The prizes, sponsored by Höganäs AB, will be presented at the Euro PM2018 Congress & Exhibition in Bilbao, Spain, October 14 - 18. Winners will each receive a cheque for €750 in the Diploma Category and €1,000 for the Doctorate category as well as complementary congress registration. The thesis submission deadline is 25 April, 2018.

www.thesiscompetition.epma.com

APMI International names 2018 fellows

APMI International has named Stephen Mashl and Alberto Molinari as the recipients of its Fellow Award 2018. Established in 1998, the award recognises APMI members for significant contributions to the goals, purpose and mission of the organisation, as well as for a high level of expertise in the technology, practice or business of the industry. The 2018 Fellow Award recipients will receive Fellow status in an awards ceremony at POWDERMET2018 International Conference on Powder Metallurgy & Particulate Materials, taking place in San Antonio, USA, June 17-20.

Mashl, based at Michigan Technological University, USA, is a research professor of materials science and engineering and has worked in the PM industry for thirty years, working primarily in particulate materials, product and processes development. According to APMI, much of his career has been spent in industry, during which time he has reportedly developed process simulation models, worked to identify particle formation mechanisms and developed an integrated HIP plus solution heat treat process for the treatment of aluminium castings.

He is listed as co-inventor on several patents, with research appearing in more than fifty papers and publications, and has also served as technical reviewer for multiple journals. As an APMI member, Mashl has served as president of the Advanced Particulate Materials Association and as a director of the Isostatic Pressing Association. He formerly served as a member of the MPIF's Board of Governors and as a Fellow of ASM International.

Molinari is a professor of metallurgy the University of Trento, Italy, and according to the APMI is "one of the most active PM technology professors in the world." Over thirty-five years of research, fifteen of which as an APMI International member, Molinari has published 500 papers in international and national journals, as well as in conference proceedings, mostly on PM subjects.

He is reported to have contributed to the development of some low-alloy powders as well as the optimisation of several industrial processes. He has carried out extensive scientific work on three main subjects: high-energy milling and sintering of powders to produce nanostructured materials, deformation and fracture behaviour of porous materials and wear mechanisms of porous materials. In addition, he is currently said to be developing a modified theory of sintering, accounting for the effect of prior uniaxial compaction of parts, and is working on the experimental determination of constitutive models of metallic powders when uniaxially cold compacted.

www.apmiinternational.org

Rise of 'MIM-like' processes continues as GE reveals developments

The seemingly inexorable rise of 'MIM-like' Additive Manufacturing processes appears to be continuing as engineers at GE Global Research, GE Aviation and GE Additive report that they have successfully built and tested thirty different prototypes of a football-sized jet engine component in just twelve weeks. The prototypes were produced on GE Additive's H1 binder jet AM system, which it states is capable of manufacturing at speeds "ten times faster" than Powder Bed Fusion (PBF) and can be used to produce larger parts.

The part is destined for use in the LEAP jet engine, developed by CFM International, a joint venture of GE Aviation and Safran Aircraft Engines. The original part is said to have taken several years to develop using casting and other conventional manufacturing processes. Using binder jet AM, the engineers were able to design, build and test designs to the required heat and durability standards within a much shorter time-frame.

Compared with PBF manufacturing, binder jet AM consumes much less energy, making the technology more cost effective as well as environmentally sustainable. Arunkumar Natarajan, a senior scientist at GE Global Research and technical lead on the company's binder jet programme, explains: "Instead of firing high-power lasers over a bed of metal powder, we're depositing a binder glue like ink on paper." According to Natarajan, the speed and power of the technology mean it could disrupt the multi-billion-dollar casting industry.

GE is reported to have developed a special binder for its process, which Natarajan states is core to the project's success. "We're very excited about the binder jet concept, given the opportunity it provides for faster printing of more parts versus other additive and even conventional manufacturing techniques," stated Natarajan. "We already have successfully printed several complex metal test parts, using this advanced binder jet process."

GE's binder jet system is said to have been developed over just forty-seven days, with the express aim of "challenging casting." On the first announcement of the machine, Mohamed Ehteshami, Vice President and then-General Manager of GE Additive, stated, "We consume so much casting inside GE. Billions and billions of dollars – and we can disrupt this, not only for ourselves, but for everyone else. We will use this and we will sell this."

www.ge.com/additive
www.safran-group.com
www.cfmaeroengines.com

MIM specialist Shenzhen ElementPlus targets advanced materials and applications

Shenzhen ElementPlus Material Technology Co., Ltd. (EPMT), established in 2016, is located in China's most active MIM industry area – the Bao'an district of Shenzhen. Each year, hundreds of millions of MIM components, especially those used in consumer electronics, are supplied from here. Chris Gu, General Manager, re-established the company following extensive experience elsewhere in the MIM industry. Prepared with a wealth of market experience and a forward-looking business philosophy, she stated that the company has established a reputation for its advanced capabilities.

Dr Peng Yu, from Southern University of Science and Technology, serves as EPMT's Chief Technology Officer, bringing more than twenty years of academic and industrial experience in metallurgy and PIM, with over fifty technical papers in international journals and a number of proprietary technologies. As such there is strong support for the development of new materials and technologies.

Fig. 1 A military belt buckle, 316L stainless steel, weighing 58 g

Fig. 2 Catalytic debinding furnaces (left) and vacuum furnaces (right)

The company's technical team, with its R&D background, can effectively respond to customers' requirements for different material grades and develop optimised processes for part production. Upon receiving a customer's drawings, engineers review the project and provide a professional DFM report. Tooling is usually designed in-house but manufactured externally. Tooling design is assisted by simulation software to predict defects created during the injection moulding process, find the best position for the gate and ultimately reduce the risk and cost of later tooling changes. Initial component samples can be delivered in three to four weeks, including two weeks for tooling fabrication and one to two weeks for in-house MIM production.

Unless specified by the customer, the feedstocks used are manufactured in-house. Most are POM-based, with the exception of a few wax-based feedstocks for special materials that are not compatible with catalytic debinding processes. Five feedstock production units have been installed, including kneading machines and pelletisers, providing a daily production capacity over two tons. In 2017, over 130 tons of 316L stainless steel feedstocks were produced, most of which were used in the manufacturing of parts such as the military belt buckles shown in Fig. 1.

EPMT currently operates eight injection moulding machines, with clamping force ranging from 50 to 80 tons and screws of different diameters that are capable of producing sophisticated parts weighing from 0.3 g to 130 g. Each machine is also equipped with a robotic system to increase production efficiency.

The debinding facility includes four catalytic debinding furnaces and a solvent debinding unit. The catalytic debinding furnaces (Fig. 2 left) precisely control the debinding temperature and the flow of nitric acid that is pumped into the furnace, remove binders effectively without causing defects such as micro-cracks or distortion. Four vacuum furnaces with graphite heating elements (Fig. 2 right) are used to sinter most iron-based and stainless-steel products, as well as for simple heat

treatment operation. The company also has facilities for post-processing, including hydraulic coining machines, magnetic grinding machines and sand blasting machines.

Mobile phone frames

The company continues to push the limits of MIM technology. Its R&D team has significant experience in the fabrication of large structures and is pioneering the use of MIM for mobile phone frames. Frames with a length of 145 mm and a width of 70 mm have been successfully developed and manufactured in small batches. In combination with CNC machining and secondary processes such as polishing or PVD coating, the MIM frames offer excellent dimensional stability and the required high quality appearance. MIM, it was stated, can significantly reduce the machining time for mobile phone frames, therefore reducing fabrication costs. MIM is therefore regarded as viable technology to replace the all-machined frames.

Advanced MIM materials

In addition to conventional iron-based materials and stainless steels, MIM technologies for titanium alloys, tungsten alloys and copper alloys have also been developed and successfully applied to various products. TiMIM alloys with oxygen content consistently controlled to ~0.2% and elongation greater than 15% have been produced.

Fig. 3 TiMIM arms for augmented reality glasses

Fig. 4 MIM tungsten-copper alloy tensile bar and its microstructure

The material has been successfully applied to the fabrication of smart glass arms (Fig. 3). Technology for the MIM of low-cost Ti powder is also under development and is expected to be brought to market soon, significantly reducing the price of TiMIM parts and promoting their applications into further market sectors.

The MIM of tungsten-copper alloys (Fig. 4) has been successfully undertaken to produce thermal management components for a communication base station. After sintering, the tungsten-copper alloys have relative density higher than 98% and thermal conductivity greater than 170 w/m·k. In order to meet market requirements for hard, non-magnetic and corrosion-resistant materials, a high-nitrogen stainless steel has also been introduced. With nitrogen content substantially higher than the equilibrium concentration, this austenitic stainless steel has a hardness value higher than 280 HV10 and a permeability less than 1.01, having potential applications in electronic devices.

Contact: chris.gu@ep-pim.com

Chinese MIM powder producer LDNMT to increase production capacity to 5000 tons/year

China's Yingtan Longding New Materials & Technology Company Ltd (LDNMT) has rapidly grown to become a domestic market leader in atomised powder for Metal Injection Moulding. Established in 2010, the company uses advanced gas and water combined atomisation technologies to manufacture its powders.

Sales figures for 2017 were recently released, along with plans for increased production. Thanks to the strong demand for atomised powders in the global MIM market, it reported sales of 2,500 tons of MIM powder for the year. The company also announced that it would increase its production capacity to 5,000 tons per year through the construction of a new metal powder factory that will go into operation in 2019.

LDNMT states that since the start of 2014 its stainless steel powders have been well received on the international market, including in the Asia Pacific, Europe and the United States. The company provides a range of powders for metal injection moulded structural applications, as well as magnetic material powders and other alloy powders in a variety of particle sizes and tap densities, based on the demands of customers. Its product line includes 316L, 304L, 17-4PH, 4J29, F75, HK30, 420W, 440C, Fe2Ni, 4140 and FeSi.

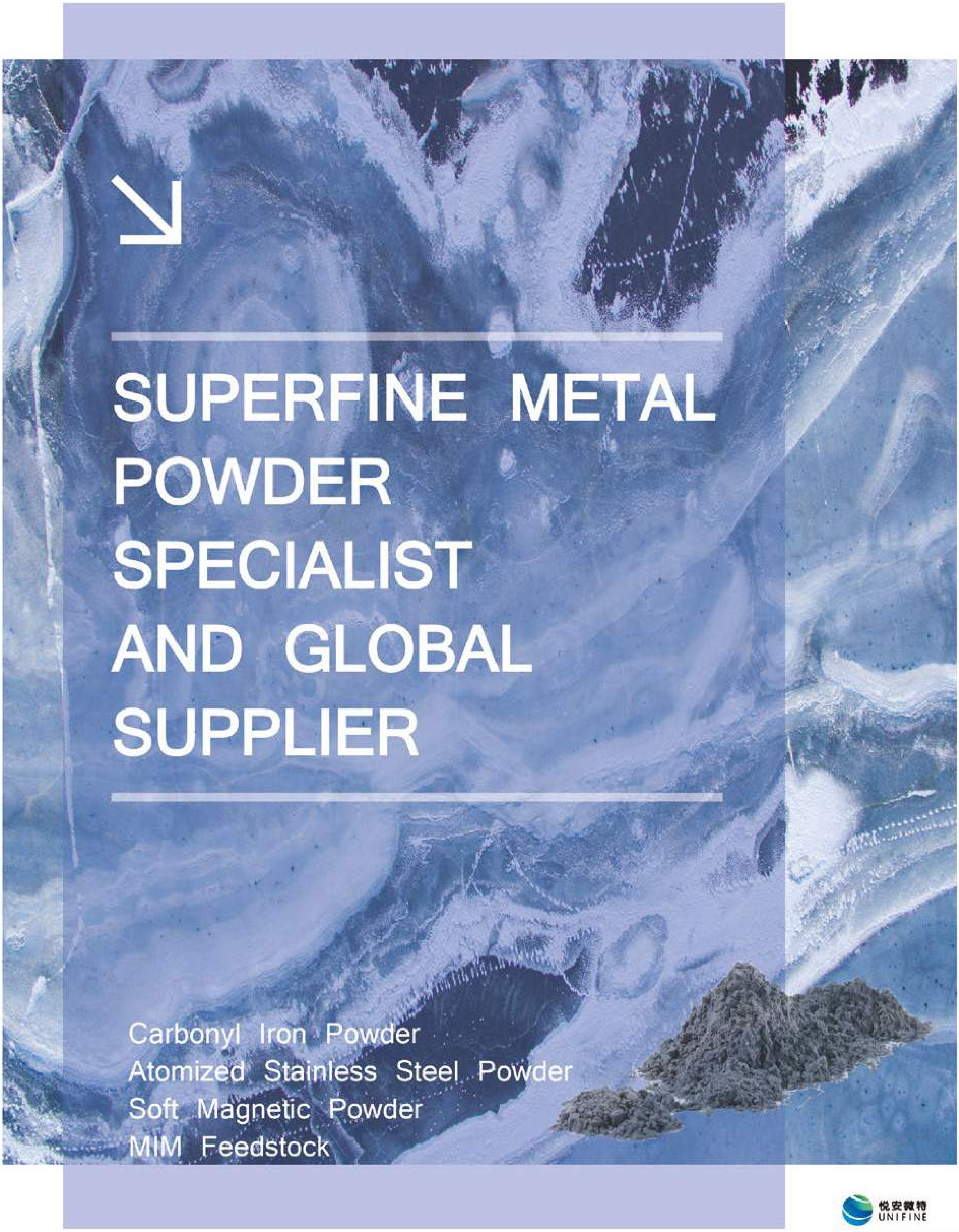
In 2017, a new 316L stainless steel powder was introduced specifically for the production of smart wearable devices and jewellery. The tap density of the powder is stated to be above 4.8 g/cm^3, with the sintered density achieved reported as 7.95 g/cm^3 after sintering at 1390°C. Other types of powders available are 17-4PH Super, which is used for medical devices. With a sintered density of 7.7 g/cm^3, and possessing the characteristics of high corrosion resistance and strength, LDNMT reports that it has seen sales of this product grow by 40% over the last year. Sales of the soft magnetic powders FeSi/FeSiCr and Kovar-F15, used in electronic devices, saw growth of 25% and 28% respectively.

The company told *PIM International*, "LDNMT supplies around 50% of China's domestic market for MIM stainless steel powder. We are well positioned and growing to become one of the largest suppliers of MIM stainless steel materials in China."

www.ldpowder.com

Submitting news..

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


SUPERFINE METAL POWDER SPECIALIST AND GLOBAL SUPPLIER
Carbonyl Iron Powder
Atomized Stainless Steel Powder
Soft Magnetic Powder
MIM Feedstock

Nondestructive testing of MIM418 superalloy turbine wheels using X-ray inspection

Turbine wheels are key components within automotive turbochargers and help to improve the fuel efficiency of internal combustion engines and reduce emissions. Because these turbine wheels operate at high temperatures and cyclic speeds, they are often produced using superalloys such as the cast nickel-base grade K418, which shows good high temperature strength, good fatigue properties and good corrosion resistance. Additionally, this grade of superalloy has been found to be more cost competitive when using the Metal Injection Moulding approach for producing the complex geometrical shapes required for such applications. The MIM 418 superalloy material also has a finer microstructure resulting in significantly improved mechanical properties compared with cast K418 superalloy. However, cast K418 superalloy turbine wheels can result in production defects.

Nondestructive testing (NDT) and metallographic observation have shown that macro-cracks are prone to form in the thicker, centre part of the MIM superalloy turbine wheel due to uneven shrinkage of the thicker section during the debinding and sintering stages of the MIM process. Micro defects such as porosity have also been found, but more often in the thinner blade section of the turbine wheel than in the centre of the wheel. Whilst Hot Isostatic Pressing (HIP) can significantly decrease or eliminate this micro porosity in the blades, any macro cracks in the thicker section will not be healed by HIP.

Jing Yanhong and research colleagues from the General Research Institute for Nonferrous Metals in Beijing, the Beijing Special Equipment Inspection & Testing Center, and the Beijing University of Science and Technology, China, have undertaken work to nondestructively test sintered and HIPed MIM 418 superalloy turbine wheels for the existence of defects using three different X-ray instruments and analysis software. The results of their work were published recently in *Rare Earth Materials and Engineering* (2017, Vol. 46 (2), pp 317-321). The authors reported that eight MIM 418 turbine wheels were produced for testing by mixing the nickel-base superalloy powders with a binder consisting of 60% paraffin wax (PW) + 15% high density polyethylene (HDPE) + 15% polypropylene (PP) + 10% stearic acid (SA) to produce the feedstock with excellent rheological

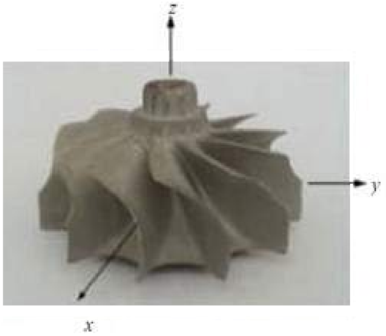


Fig. 1 Transillumination direction of X-ray inspection (From paper: 'X-ray inspection of MIM 418 superalloy turbine wheels and defect analysis' by Y Jing et al Rare Earth Materials and Engineering, Vol. 46, No 2, 2017, pp 317-321)

Fig. 2 Sectional image of the sintered turbine wheel (From paper: 'X-ray inspection of MIM 418 superalloy turbine wheels and defect analysis' by Y Jing et al Rare Earth Materials and Engineering, Vol. 46, No 2, 2017, pp 317-321)

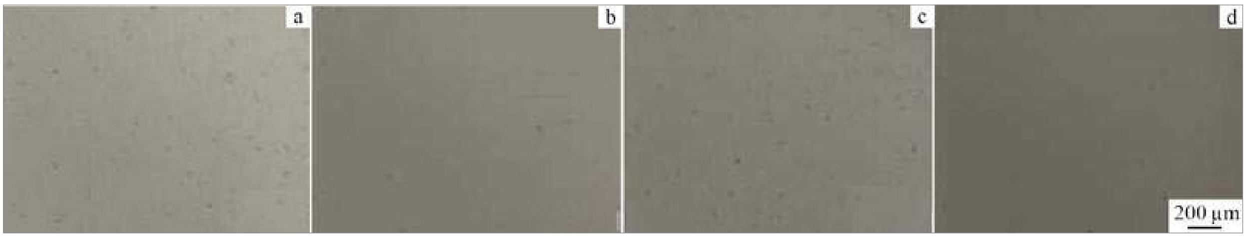


Fig. 3 OM images of the samples taken from MIM turbine wheels: (a) centre section of the sintered sample, (b) centre of the sintered & HIPed sample, (c) blade section of the sintered sample, and (d) blade section of the sintered & HIPed sample (From Paper: 'X-ray inspection of MIM 418 superalloy turbine wheels and defect analysis' by Y Jing et al, Rare Earth Materials and Engineering, Vol. 46, No 2, 2017, pp 317-321)

properties at 160°C. Critical powder loading in the feedstock was put at 65 to 67 vol.%. Following debinding the turbine wheels were sintered at 1240°C in vacuum and HIPed at 1210°C under argon atmosphere. The volume shrinkage of the turbine wheels was shown to be 12.7%. The MIM turbine wheels have a large sectional thickness difference with the thinnest part of the blades being just 2 mm thick and the thickest part in the centre of the wheel being around 30 mm.

The authors stated that the MIM 418 turbine wheels were subjected to X-ray inspection using three different X-ray instruments, including film X-ray, real-time imaging X-ray and microfocus X-ray in order to find serious defects such as cracks which can negatively impact the fatigue properties. Fig. 1 shows the transillumination direction of the X-ray inspection of the turbine wheels. One of the turbine wheels was cut in half and polished (Fig. 2) for observation of any defects using an optical microscope (OM) and scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS).

The authors reported that the cut turbine wheel showed two cracks in the thicker, centre part, having lengths of around 10 mm and 0.3 mm in width, which they attributed to tensile stresses in the centre part caused by shrinkage during sintering, and also on the existence of oxides on the crack surface which were difficult to remove during debinding and vacuum sintering of the wheel. The cracks were found to exist in all eight MIM samples when using XXQ-2505 film X-ray equipment with 2 mm focus. Because of the large difference in thickness between the blades and the centre of the parts these sections were tested separately with the XXQ-2505 film X-ray equipment using different process parameters. Testing with the real time imaging X-ray equipment CDZ-302P-A found cracks only in No. 2 and No. 4 turbine wheels, indicating that film X-ray testing is more sensitive than the real-time imaging under the same conditions. The testing results obtained using microfocus X-ray equipment showed that even though this equipment has very high resolution, it could not detect defects such as cracks in the centre of the part due to underexposure.

The researchers state that because the specific surface area in the thinner blade sections of the turbine wheel is larger and the shrinkage during debinding and sintering more homogeneous, there were no cracks found in the blades. However, X-ray inspection showed a relatively high level of porosity with almost spherical pores and with a higher level of porosity and even larger pores in the blade sections than in the centre. This the authors attributed to the slight separation of binder and powder that can occur during moulding due to frictional force between powder particles, the binder and the mould. This type of separation is more important in the blade section which can lead to more binder and less powder being injected into this section compared with the thicker centre of the part as verified by X-ray inspection.

Fig. 3 shows the level of porosity in the centre and blade sections after sintering and after sintering and HIPing of MIM418 superalloy turbine wheel parts. After HIP the porosity of the samples decreases significantly and even almost disappears. However, as already mentioned, HIP does not heal the cracks in the thicker section of the turbine wheel. The authors concluded that the results show that, under the same testing condition, the film X-ray equipment has higher sensitivity than the real-time imaging X-ray for NDT. However, the microfocus X-ray testing is not suitable for the NDT testing of the MIM418 turbine wheels due to its low power in penetrating thicker sections.

www.sciencedirect.com/journal/rare-metal-materials-and-engineering

Vacuum sintering of 17-4 PH stainless steel for orthodontic brackets

Orthodontic brackets are extensively used worldwide in the dental sector to change the position of teeth or align teeth of the lower jaw to the upper jaw. The production of the complex-shaped bracket components that are required was one of the early applications for Metal Injection Moulding, and remains a major product for the industry. In the case of Indonesia, where malocclusion is still a common problem encountered in the teeth of the population, imported orthodontic brackets are said to be (a) relatively expensive and (b) unsuitable for the dental structure of most Indonesian people.

To overcome these issues, researchers at the University of Indonesia and Yeungnam University, both located in Depok, have been undertaking research to create a suitable material and manufacturing technology for the lower cost production of orthodontic brackets locally. In a paper published in *Solid State Phenomena* (Vol. 266, 2017, pp 231-237) Bambang Suharno and colleagues have reported on the use of MIM to produce orthodontic brackets from a 17-4 PH stainless steel (AISI 630) – a material commonly used for this application. The research was particularly focused on whether vacuum sintering could provide more optimum properties than using hydrogen atmosphere for sintering.

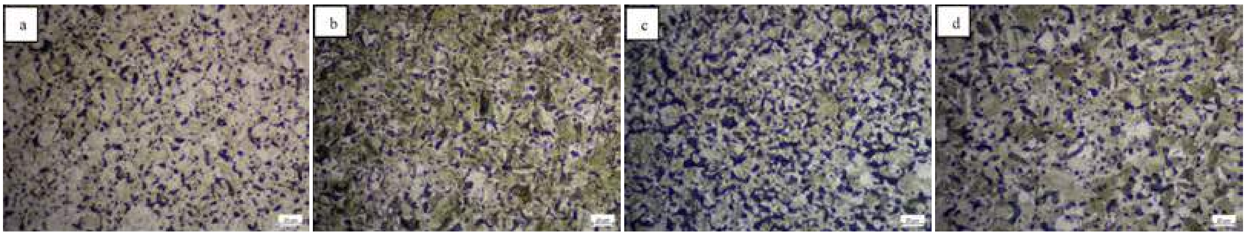


Fig. 1 Microstructures of 17-4 PH stainless steel sintered at (a) 1320°C (b) 1340°C, (c) 1360°C and (d) 1380°C (From paper: 'Vacuum Sintering process in MIM for 17-4 PH Stainless Steel as Material for Orthodontic bracket', by B Suharno et al, Solid State Phenomena, Vol. 266, 2017, pp 231-237)

The authors used a commercial grade 17-4 PH stainless steel feedstock to injection mould 5 mm x 5 mm x 5 mm cube test pieces at pressure of 2700 psi. During injection moulding the temperature of the outer barrel was maintained at 100°C, nozzle temperature was 200°C, and mould temperature was 50°C. Debinding was performed in two stages, solvent debinding and thermal debinding. Solvent debinding was carried out in hexane at 50°C for 90 min with agitation. The debound parts were then vacuum dried for 1 h at 50°C, followed by thermal debinding using a heating rate of 1°C/min up to 510°C, holding for 60 min and then cooled. Sintering was done under vacuum with a heating rate of 5°C/min in vacuum at four different temperatures - 1320°C, 1340°C, 1360°C and 1380°C. Fig. 1 shows that similar microstructures are obtained in the 17-4PH stainless steel sintered at the different temperatures. The light green colour is α-ferrite and a darker green colour is martensite. The existence of martensite leads to optimum hardness in the sintered material when sintered at 1360°C.

Fig. 2 shows the effect of sintering temperature on density achieved in the MIM 17-4 PH samples. As can be seen sintered density is lower than that required for this material according to MPIF Standard 35 which stipulates density to be in the range 98-99% of theoretical. The lower density achieved in vacuum sintering is attributed by the authors to the

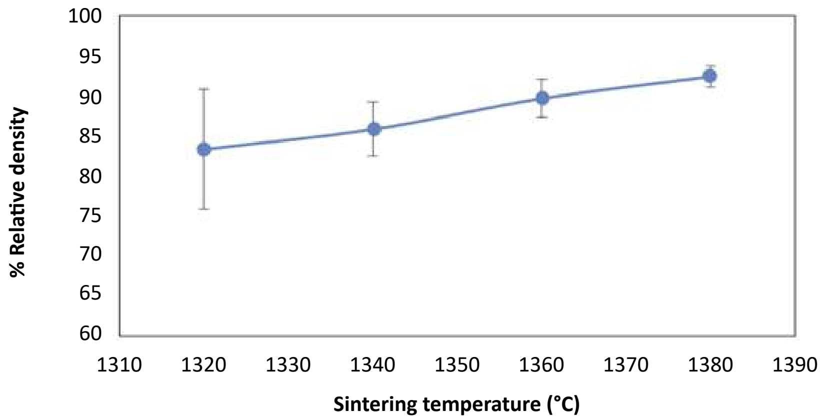


Fig. 2 Effect of sintering temperatures on relative density of 17-4 PH stainless steel (From paper: 'Vacuum Sintering process in MIM for 17-4 PH Stainless Steel as Material for Orthodontic bracket', by B Suharno et al, Solid State Phenomena, Vol. 266, 2017, pp 231-237)

presence of silicon oxide inclusions, which are believed to form during the thermal debinding stage and which prevent necking at the initial stage of sintering. Silicon oxide content can be reduced by the addition of graphite or by using hydrogen atmosphere in sintering to react with the oxide layer. Another cause for lower density could be the injection moulding pressure used which resulted in lower density and a nonhomogeneous distribution of density in the green compacts. Lower green density will affect shrinkage behaviour in sintering leading to increased internal stresses and will ultimately influence final sintered density.

The authors also studied the influence of sintering temperature needed to achieve the hardness properties necessary to meet the required levels in commercial 17-4 PH stainless steel orthodontic brackets (Fig. 3). Optimum hardness of 395 HV was achieved at a sintering temperature of 1360°C, which is far higher than that achieved in commercial orthodontic brackets, and the authors consider that vacuum sintering of MIM 17-4 PH stainless steel materials is, therefore, commercially feasible.

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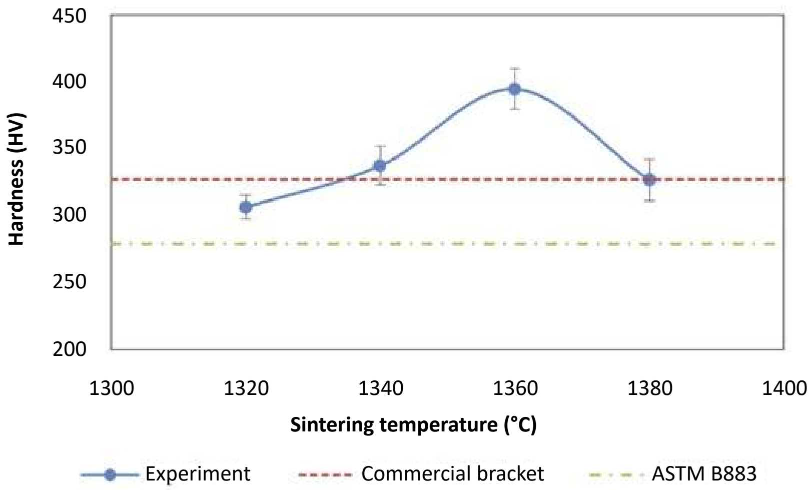

Fig. 3 Effect of sintering temperature on the hardness of 17-4 PH stainless steel (From paper: 'Vacuum Sintering process in MIM for 17-4 PH Stainless Steel as Material for Orthodontic bracket', by B Suharno et al, Solid State Phenomena, Vol. 266, 2017, pp 231-237)

Innovnano to present data on the fatigue resistance of its 2YSZ powder at ceramitec 2018

Innovnano, Coimbra, Portugal, has developed a 2%mol Yttria Stabilised Zirconia (2YSZ) powder that offers beneficial properties including high flexural strength, ageing resistance and outstanding fracture toughness, making the material well-suited to cyclic fatigue conditions. The company is set to present its findings in a presentation titled 'YSZ materials for harsh working environment obtained by emulsion detonation synthesis' during the Technical Ceramics Day, April 12, at Ceramitec 2018, Munich, Germany.

Innovnano, a subsidiary of CUF, is a specialist manufacturer of industrial-scale quantities of nanostructured zirconia-based ceramic powders. This new powder is produced using the company's Emulsion Detonation Synthesis (EDS) process and has been tested for cyclic fatigue under standard conditions (one million cycles at 20 Hz; load range: 110-320 MPa) and extreme conditions (maximum load 1100 MPa).

During the tests, Innovnano 2YSZ is said to have lost just 13% of its flexural strength after cycling under standard conditions and to have resisted one million cycles without failure under extreme conditions. The maximum stress loading is said to correspond to almost double the values obtained with benchmark 3YSZ. Innovnano stated that this performance demonstrates that Innovnano 2YSZ is ideal for structural ceramic applications and has great potential as a solution for areas in which exceptional cyclic fatigue resistance is required.

www.innovnano-materials.com

EPMA's Functional Materials seminar to identify applications in EVs

The European Powder Metallurgy Association is to hold a Functional Materials Seminar in Jülich, Germany, April 16-17, 2018. The seminar will be on the topic of 'Functional PM Components in Next Generation Electric Cars' and will discuss the range of functional materials that can be produced by PM processes such as SMCs, thermoelectric materials, porous heat pipes, permanent magnets, hydrogen carriers and fuel cell plates, among many others.

During the seminar a number of industry experts will present the potential uses of these materials in cars, followed up by a workshop preparing a consortium for a Horizon2020 application.

www.seminars.epma.com

NiO-YSZ porous anode support for SOFCs produced by Ceramic Injection Moulding

The Solid Oxide Fuel Cell (SOFC) is a promising energy conversion device that directly produces electrical power from a wide variety of fuels at high temperatures. The fuel cells consist of three main parts: the anode, cathode and electrolyte – the latter being made from a solid oxide such as yttria stabilised zirconia (YSZ) which conducts the negative oxygen ions from the cathode to the anode. The ceramic anode layer made of Ni-YSZ must be sufficiently porous (30-40% porosity) to allow the fuel to flow towards the electrolyte.

Researchers at Chulalongkorn University, Bangkok, Thailand, have been studying the use of CIM to produce the porous anode support for SOFC applications, and the results of their recent work have been published by Nutthita Chuankrerkkul *et al* in *Key Engineering Materials* (Vol. 75, 2017, pp 467-470). The authors state that one route to achieving the 30 - 40 vol.% porosity level required in the nickel oxide-yttria stabilised zirconia (NiO-YSZ) ceramic anode layer is the use of pore formers and a wax based binder system for the CIM feedstock. However, this type of feedstock requires the use of organic solvents, which are environmentally unfriendly, for the removal of the wax binder during the debinding step, and the researchers have instead used an alternative binder system based on polyethylene glycol (PEG) which can be removed in debinding using water.

CIM feedstock for the porous anode support was produced using a powder mixture of 40 wt.% nano sized NiO powder and 60 wt.% nano sized YSZ powder. Optimum binder composition of the feedstock was stated to be important for successful injection moulding, and because of the high surface area of the nanopowder mixture and the need to high level of porosity, a 36-38 vol.% powder loading was used. The nanopowder mixture was blended with a binder based on polyethylene glycol (PEG) and a small amount of polyvinyl butyral (PVB). CIM specimens having a dimension of 5 x 5 x 55 mm were produced at an injection temperature of 190°C and pressure of 44 MPa. PEG removal was performed in a water bath equipped with stirring system. Rate of binder removal was studied by comparing the weight loss of samples after soaking in water at temperature of 40°C for various times. The sintering tests were done at temperatures of 1300°C, 1350°C and 1400°C. Table 1 shows the density of the sintered specimens, flexural strength in three-point bending test, and Vickers hardness at the different sintering temperatures. Fig. 1 shows the microstructure of NiO-YSZ specimens sintered at 1350°C.

It was also found that the porosity, mechanical properties and microstructure of the NiO-YSZ CIM specimens can be controlled by adjusting the sintering temperatures and holding times. As shown in Table 1 sintering at high temperature resulted in components with high density and less porosity, and the authors have proposed using the temperature of 1350°C which gives specimens having porosity of around 25%. It is expected that after performing a reduction of NiO, approximately 40% porosity would be achieved, as required by the SOFCs system.

www.scientific.net/KEM

Sintering Temperature (°C)	Flexural Strength (MPa)	Hardness (Hv)	Density (g/cm^3)	% Theoretical density
1300	12.4	162	3.99	65.5%
1350	28.5	387	4.41	75.0%
1400	27.2	n/a	5.24	86.1%

Table 1 Sintered properties of specimens sintered at 1300°C, 1350°C and 1400°C (from paper: 'Characterisation of NiO-YSZ Porous Anode-Support for Solid Oxide fuel Cells Fabricated by Ceramic Injection Moulding', by N Chuankrerkkul, et al, Key Engineering Materials ,Vol. 75, 2017, pp 467-470)

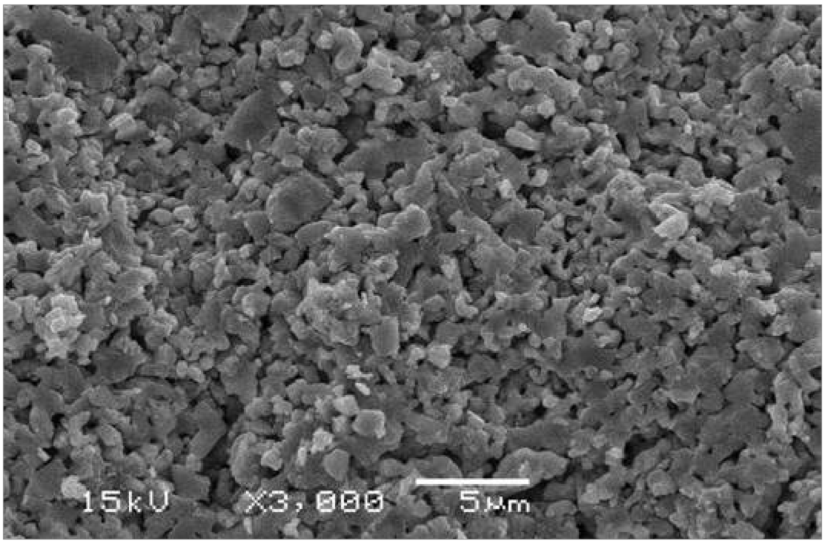

Fig. 1 Microstructure of NiO-YSZ sintered at 1350°C (from paper: 'Characterisation of NiO-YSZ Porous Anode-Support for Solid Oxide fuel Cells Fabricated by Ceramic Injection Moulding', by N Chuankrerkkul, et al, Key Engineering Materials ,Vol. 75, 2017, pp 467-470)

Influence of backbone binders on the rheological performance of CIM feedstocks

Typical binders used in Powder Injection Moulding feedstock comprise three key components; (1) main body (2) backbone, which should be non-reacting during debinding and which maintains part shape prior to sintering and (3) any other additives such as surfactants. Each ceramic powder particle in the feedstock must be coated with the binder, but only the smallest effective amount of binder should be used in order to achieve the required viscosity for injection moulding.

This can be challenging in the case of ceramic powders such as Al_2O_3 which are often a magnitude smaller (typically from 0.1 µm to 5 µm) than metallic powders, and the tendency for finer ceramic powders to form agglomerates despite the high shear rates during mixing of the binder and powder and subsequent processing. This brings increasing demands on the selection of polymer binder which should have low viscosity but at the same time have good adhesion to the powder. Polyethylene glycol (PEG) or ethylene-based block copolymers used as the backbone binder in CIM feedstock have provided good adhesion of the binder to the ceramic powder particles. Most of the effects of binder/powder interactions in CIM have been investigated from a standpoint of rheological performance.

Recently, Berenika Hausnerova and colleagues at the Tomas Bata University in Zlin, Czech Republic, have been investigating the role of particular binder components and their interactions with ceramic powders in CIM feedstock as well as with mould channel walls, as a basis for the development of a novel, environmentally friendly CIM feedstock. Their most recent results, reported in a paper published in *Polymer Engineering and Science*, Vol. 57, 2017, pp 739-745, have shown that binders containing carnauba wax (CW) or acrawax (AW) could be used as a substitute for low density polyethylene (LDPE) and have exhibited superior debinding properties with gradual binder extraction up to the late stage of thermal debinding.

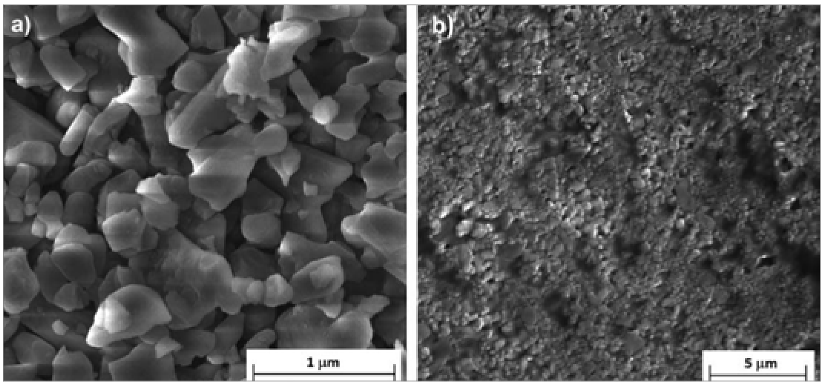

Fig. 1 (a) SEM of Al_2O_3 powders used in the investigation; (b) SEM of Al_2O_3 feedstock based on CW binder (from paper: 'Effect of Backbone Binder pm Rheological Performance of Ceramic Injection Moulding Feedstock' by B Hausnerova et al, Polymer Engineering and Science, Vol. 57, 2017, pp 739-745)

LDPE1	LDPE	PW	PEG 6000			SA
	40	10	49			1
LDPE2	LDPE	PW	PEG 6000	PEG 4000	PEG 1000	SA
	40	10	25	14	10	1
LDPE3	LDPE	PW	PEG 6000	PEG 4000	PEG 1000	OA
	40	10	25	14	10	1
Carnauba1	CW	PW	PEG 6000			SA
	40	10	49			1
Carnauba2	CW	PW	PEG 6000	PEG 4000	PEG 1000	SA
	40	10	25	14	10	1
Carnauba3	CW	PW	PEG 6000	PEG 4000	PEG 1000	OA
	40	10	25	14	10	1
Acra1	AW	PW	PEG 6000			SA
	40	10	49			1
Acra2	AW	PW	PEG 6000	PEG 4000	PEG 1000	SA
	40	10	25	14	10	1
Acra3	AW	PW	PEG 6000	PEG 4000	PEG 1000	OA
	40	10	25	14	10	1
Commercial	Licomont					SA
	99					1

Table 1 Compositions of binders used in CIM feedstock – content given of individual components in wt.% (from paper: 'Effect of Backbone Binder pm Rheological Performance of Ceramic Injection Moulding Feedstock' by B Hausnerova et al, Polymer Engineering and Science, Vol. 57, 2017, pp 739-745)

The proposed binders have been evaluated for their suitability for Ceramic Injection Moulding feedstock using Al_2O_3 powders having a particle size distribution D50 of 0.8 µm. An SEM image of the Al_2O_3 powders used is shown in Fig. 1(a). Powder loading in the feedstock was 50 vol.% and the binder and powder were mixed in a Z-blade mixer at a temperature dependent on the melting temperature of the particular binder components. Compounding for 30 min was sufficient to obtain a steady mixing torque and well dispersed feedstock as illustrated in Fig. 1(b).

Polyolefine-based binders contain LDPE, PW and PEG of different molecular weights. In the newly developed feedstocks, LDPE was substituted with CW or AW. In addition, all feedstock compositions used contain 1 wt.% stearic acid (SA) as surfactant. Detailed composition of the feedstocks studied is summarised in Table 1. The table also includes a commercially available, partially-water soluble binder with a density 1.07 g/cm^3 (Licomont EK 583, eMBe) which was evaluated for comparison. The rheological properties of the LDPE backbone binder, the newly developed binders with waxes AW and CW, and the commercial binder are shown in Fig. 2, along with processing temperatures. It is noteworthy that the use of CW as a backbone binder allowed processing at 100°C. LDPE feedstock was found to show more than one order of magnitude higher viscosity than CW or AW binder systems.

Overall, the rheological performance of the feedstocks investigated is in accordance with the authors' quantitative analysis of the specific interactions carried out on low molecular weight substitutes of the binder components in previously published work, as well as with the findings also obtained earlier from contact angle measurement of the wettability of the respective binders. In the present study, the authors also reported on the effect of Al_2O_3 on the thermal properties of binders and evaluated the melting properties of Al_2O_3 /binder mixtures in comparison with those of neat binders. The lowering of melting point was the most pronounced in case of PEG based binder, while no change was observed for LDPE/Al_2O_3. The addition of hydrophilic PEG to feedstock containing Al_2O_3 initiated an additional shift of melting temperature, which was different for carnauba and acrawax.

The authors stated that in general, a decrease in melting temperature in a feedstock blend can be caused both by morphological effects, such as for example a decrease in lamellar thickness, and to thermodynamic factors (interchain interactions, interactions with particular material, and change in crystal size).

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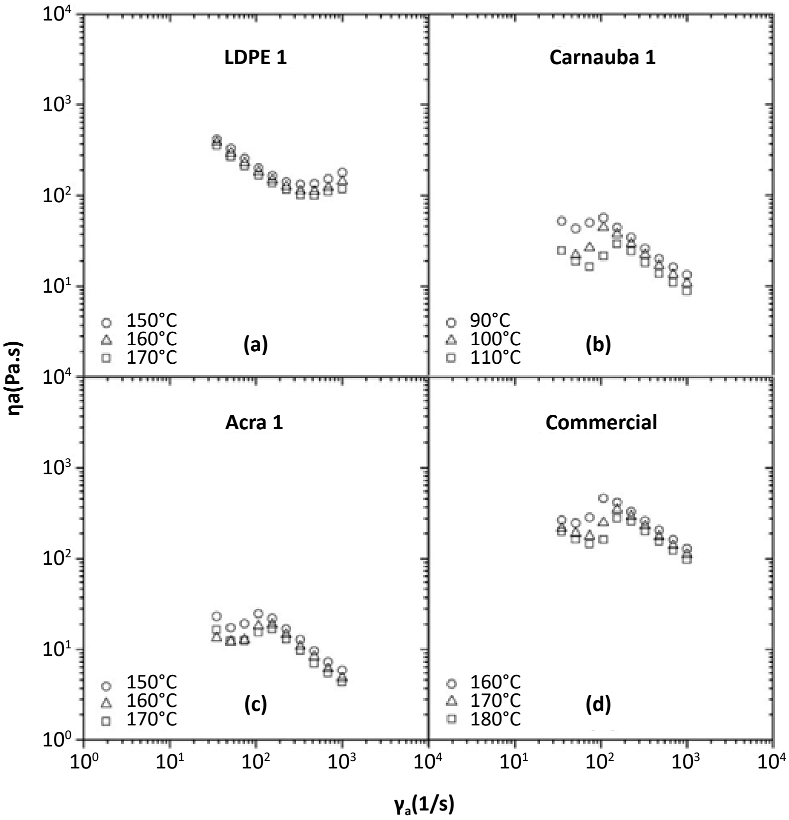
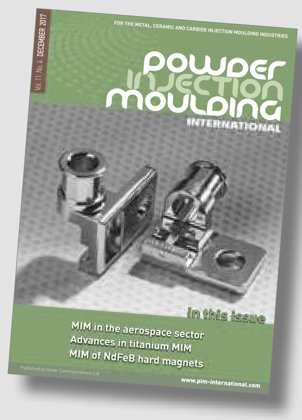


Fig. 2 Flow properties of Al_2O_3 feedstocks (50 vol.%) based on binders with PW, PEG 6000, and LDPE (a), or CW (b), or AW (c) and commercial composition (d). (from paper: 'Effect of Backbone Binder pm Rheological Performance of Ceramic Injection Moulding Feedstock' by B Hausnerova et al, Polymer Engineering and Science, Vol. 57, 2017, pp 739-745)

Erratum



The picture of the Swatch watch on page 91 of the print issue of *PIM International* Vol. 11 No. 4, December 2017 was published in error.

Nippon Piston Ring: Leading Japanese MIM producer builds on automotive success

It is thirty years since Japan's Nippon Piston Ring Co. Ltd. (NPR) first began developing its MIM production capability. The company has since pioneered the use of MIM in key automotive engine systems, from fuel injector components to rocker arm applications. The following article reviews the company's growth and presents insight into its most significant component developments, while considering some of the opportunities offered by new MIM applications.

Nippon Piston Ring Co., Ltd. was established in Saitama, Japan, in 1934 and is a specialist producer of precision sliding parts with high wear and friction resistance, ranging from piston rings to key components for internal combustion engines such as valve seat inserts, camshafts and cylinder liners. The company reported FY 2016 sales of ¥52.1 billion (approx. $470 million), of which more than 50% was exported.

NPR first successfully commercialised sintered valve seat inserts, now commonplace in the automotive industry, in 1971 and by 1976 it had developed composite valve seat inserts. "We were one of the first companies to start the mass production of sintered valve seat inserts. Taking advantage of the high degree of freedom in material design provided by Powder Metallurgy, along with the possibilities offered by diffusion bonding, we also succeeded in the mass production of Powder Metallurgy camshafts (Fig. 1). This lightweight alternative to conventional cast products also brought the benefit of higher contact pressure resistance," stated Shunsuke Takeguchi, General Manager of NPR's New Product Business Promotion Department.

The company first began the development of its Metal Injection Moulding capability in 1988, with mass production starting in 1990. "In the beginning, we used our original binder for thermal debinding. However, we adopted the AMAX process in 1992 and began mass production using this technology in 1993. In 1998, our MIM process

Fig. 1 Diffusion bonded camshafts and other MIM and PM parts manufactured by Nippon Piston Ring

Fig. 2 Top: Aerial view of Nippon Piston Ring's Tochigi plant; lower left: view of the injection moulding area; lower right: view of a batch debinding and sintering area

was used for the production of valve parts for fuel injection systems for cars, where magnetic properties are important," explained Takeguchi.

In 1999 the company started production of its revolutionary MIM roller rocker arm components. These complex automotive engine parts, with high fatigue strength and the ability to withstand a harsh operating environment, were used in the Honda Motor Co. Ltd's F20C engine for its S2000 sports car, released in celebration of the company's 50th anniversary [1]. The roller rocker arm parts were the world's first stress loaded engine parts to be mass produced by the MIM process and have won numerous awards and recognition, including being presented as a successful example of supplier excellence by Honda.

In 2014, NPR acquired the MIM operation of Sumitomo Metal Mining Co. Ltd., which manufactured parts under the Metamold® brand, further consolidating its position as a leading MIM parts manufacturer for the international market. The company's MIM production facility is located at the Nogi branch of NPR's Tochigi plant, which has a site area of 3,805 m^2 (Fig. 2). "Our batch type sintering furnaces are some of the largest in Japan and enable us to produce approximately four hundred different types of products. We are also one of the few MIM manufacturers to be IATF16949 certified and have a high standard quality control system," stated Takeguchi.

Delivering highly-complex MIM parts

MIM's ability to deliver complex components in high volumes has enabled the technology to grow rapidly worldwide and NPR continues to push the capability of the process with parts that feature very high levels of complexity combined with exceptional fatigue resistance.

Kyosuke Mori, from NPR's Product Engineering Department II explained, "We manufacture numerous geometries that cannot be processed by conventional technologies. In our ball screw components, we split a single component design into two parts by carefully selecting the positions of the split surface and pin/hole. The dimensional tolerances are set according to the requirements of the component. We then sinter each split piece separately before assembly into the final product. Managing this type of process is extremely difficult; however, we offer high-quality products by performing dimensional control measurements of the split pieces, along with visual checks and other necessary processes. We have supplied numerous automotive parts using this technology to-date." Deflector and tube-type ball screw components are shown in Figs. 3 and 4.

NPR also has specific expertise in incorporating complex threads into metal injection moulded components. "Metal cutting tools are generally used to form external threads. However, we can also mould internal threads. To ensure defect-free moulding, it is important to maintain moulding precision and an optimal balance in the binder to provide the necessary material flow and shape retention. With the application of our MIM technology, we offer various types of special threads that cannot be made by machining, such as irregular-shaped and multi-start threads. This provides a significant cost advantage to our clients," stated Mori. A component with a trapezoidal octuple-thread is shown in Fig. 5.

Fig. 3 MIM ball screw parts (deflector type)

Fig. 4 MIM ball screw parts (tube type)

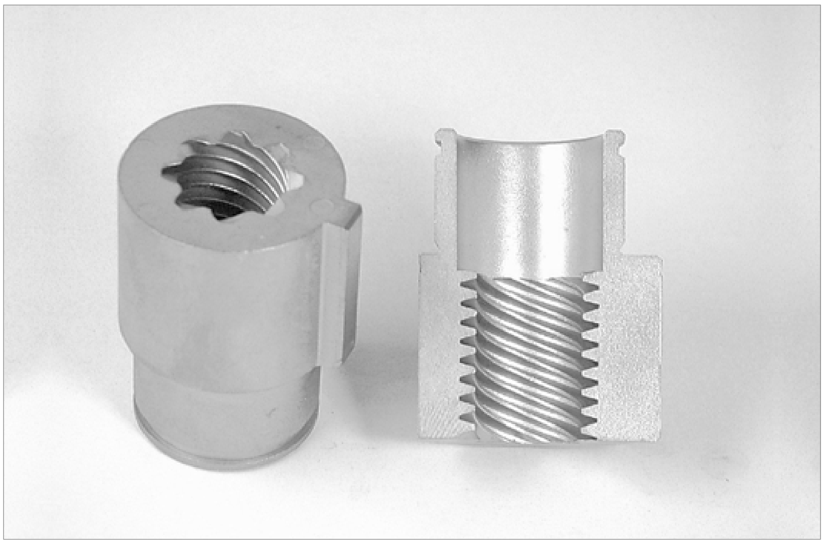


Fig. 5 MIM component with trapezoidal octuple-thread

	Composition	Treatment	U.T.S. (MPa)	Y.S. (MPa)	Elongation %	Hardness	C.T.E. 20 ~ 100°C (x10⁻⁶)
Fe-2Ni-C	C-2Ni	○	1800	1430	2.0	50 HRC	
			1450	1250	6.0	40 HRC	
			1000	860	10.0	30 HRC	
SNCM630	0.3C-3Ni-3Cr-0.4Mo	○	1120		3.0	650 HV	
SCM415	0.15C-1.0Cr-0.25Mo		450	240	25.0	120 HV	
SUS316L	18Cr-12Ni-2Mo		520	170	40.0	130 HV	
SUS420J2	0.3C-13Cr	○				50 HRC	
SUS440C	1.0C-18Cr	○				57 HRC	
SUS630	17Cr-4Ni-3Cu	○	1330	1230	13.0	44 HRC	
XM27	27Cr-1.5Mo		570	420	40.0	220 HV	
SKD11 (D2)	1.5C-12Cr-1.0Mo-0.3V	○				61 HRC	
SKH51 (M2)	0.85C-4.2Cr-5Mo-6W-1.9V	○				64 HRC	
Kovar	29Ni-17Co		540	330	30.0	140 HV	4.9
Invar	36Ni		490	290	26.0	140 HV	1.6
Super Invar	32Ni-5Co		450	250	20.0	150 HV	0.3
Pure Ti	Ti		520	430	12.0	170 HV	
Ti-6Al-4V	Ti-6Al-4V		900	800	6.0	300 HV	

Table 1 Mechanical properties of NPR's MIM alloys

Method	Processing method	Liquidity	Shape retainability	Processing time	Characteristics
Solvent debinding	Extraction by immersion in liquid solvent	0	+	4 – 12 hours	Short processing time Use of solvent
Thermal debinding	Sublimation by heating	+	0	20 – 48 hours	Less deformation than general thermal sintering due to NPR's in-house technology
Free debinding (continuous debinding and sintering)	Sublimation by heating (performing debinding and sintering continuously in a furnace)	-	+	Continuous processing with sintering	Able to shorten manufacturing time due to continuous debinding with sintering

Table 2 Characteristics of the debinding processes used at NPR

MIM technology and materials

The company currently processes twelve alloy types for mass production. However, in the past it processed nearly fifty different types of material (Table 1). "We aim to offer the best solution that meets the client's requirements. We are also capable of evaluating the durability and reliability of MIM materials and make relative comparisons with existing products manufactured by other processes, thanks to the expertise that we have cultivated through our automotive part production," stated Mori.

Commenting on the approach to debinding at NPR, Mori explained, "Our debinding methods are largely divided into two types; thermal debinding and solvent debinding. For the more conventional thermal debinding processes, debinding and sintering are either performed in two separate furnaces or by a 'free debinding' process, in which debinding and sintering take place concurrently in a continuous process. We use these three methods in accordance with the specific requirements of each application and its materials." Table 2 gives a breakdown of the characteristics of the debinding processes used at NPR.

Production volumes and markets

To-date, NPR has mass produced around a thousand different types of MIM part, with four hundred currently in mass production. Whilst, as a key supplier to the automotive industry, the company is used to working with extremely high volumes, it also has the ability to cater for smaller production runs. "Our minimum lot size is about five hundred parts and the manufacturing experience that we have accumulated over a long period of time covers a variety of products ranging in size and type," stated Mori. "In terms of our track record for mass production, we have been producing approximately one million automotive parts per month for over ten years and can respond flexibly to the needs of our clients."

Almost half (47%) of NPR's MIM products, by volume, are for the automotive market (Fig. 6). The remaining products are for various types of non-automotive applications such as industrial machines, security systems and locks, taps and related products, consumer applications and others (Fig. 7-9, 12). In terms of the materials processed, the Fe-Ni-based materials widely adopted in the automotive field are by far the most frequently used.

Case study: Rocker components

As previously mentioned, one of NPR's best-known products are the roller rocker components for the Honda S2000's variable valve lift system. A small, light engine was required that could achieve the high-RPMs and high power output desired for the vehicle concept. It was therefore decided that a roller follower instead of the conventional

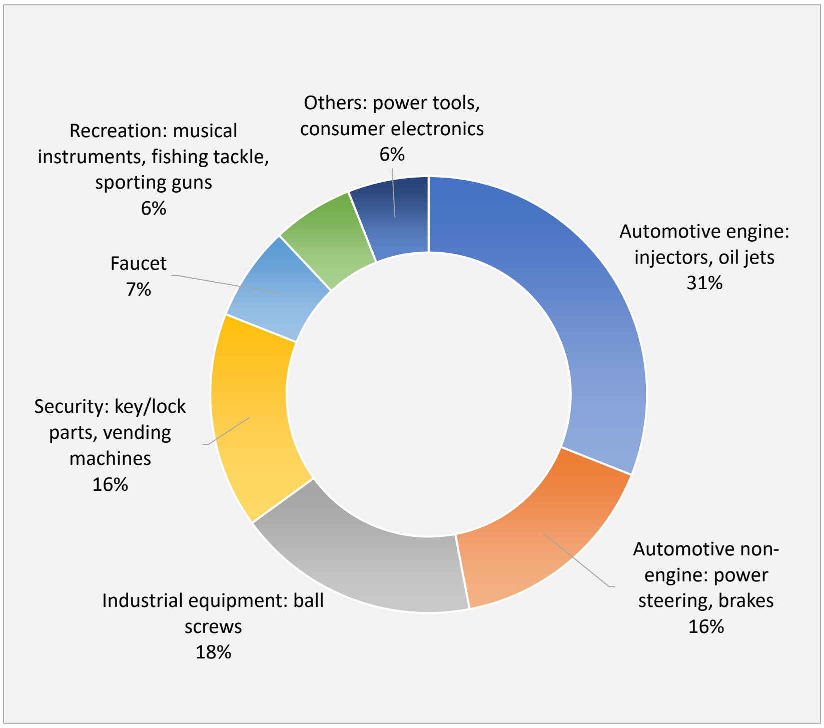


Fig. 6 Breakdown of NPR's MIM markets, with application examples for each sector

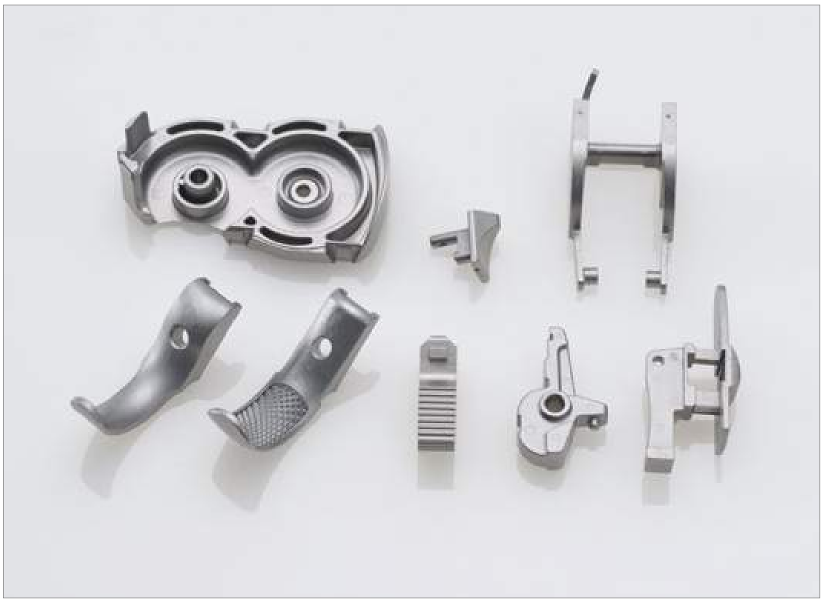


Fig. 7 MIM parts for sporting guns

Fig. 8 MIM parts for security applications

Fig. 9 MIM parts for consumer/recreation applications

Fig. 10 Roller rocker parts for the F20C engine used in the Honda S2000 car

Fig. 11 MIM automotive fuel injector parts

cam slipper-type followers would be adopted in the new roller rocker.

In terms of the system's operation, a built-in VTEC mechanism was located coaxially within the roller elements to reduce size and weight to the minimum levels. However, to make the roller rocker with conventional forging and machining technology, heavy cutting was required because the roller mounting groove had to be hollowed out. This resulted in poor productivity and increased costs, hindering mass production. To overcome this issue, NPR developed the coaxial VTEC roller rocker using MIM technology (Fig. 10).

Material selection

Masahiro Kimura, Assistant Senior Engineer in NPR's Technical Development Department Group II explained, "For the roller rocker's material we chose the MIM equivalent to chromium molybdenum steel, with which we had previous experience in mass production. This enabled us to achieve both the required strength and workability. Properties of the component were further improved through carburising and quenching operations."

"For the metal powder, we decided to use water atomised powders to reduce the costs in such a high-volume application. By overcoming some of the drawbacks of the conventional water atomisation process, a new water atomisation method was developed using supersonic water jets which can deliver the desired spherical powder shape and low oxygen content. This powder was specifically developed for the roller rocker application."

Moulding

For the binder system, NPR used polyethylene as the main component, which provides high and stable fluidity in a relatively wide temperature range. The optimum feedstock blend for the application was obtained by compounding the new water atomised powder with a reduced amount of binder. "The filling balance was initially unstable in the area of the control screw. However, we resolved

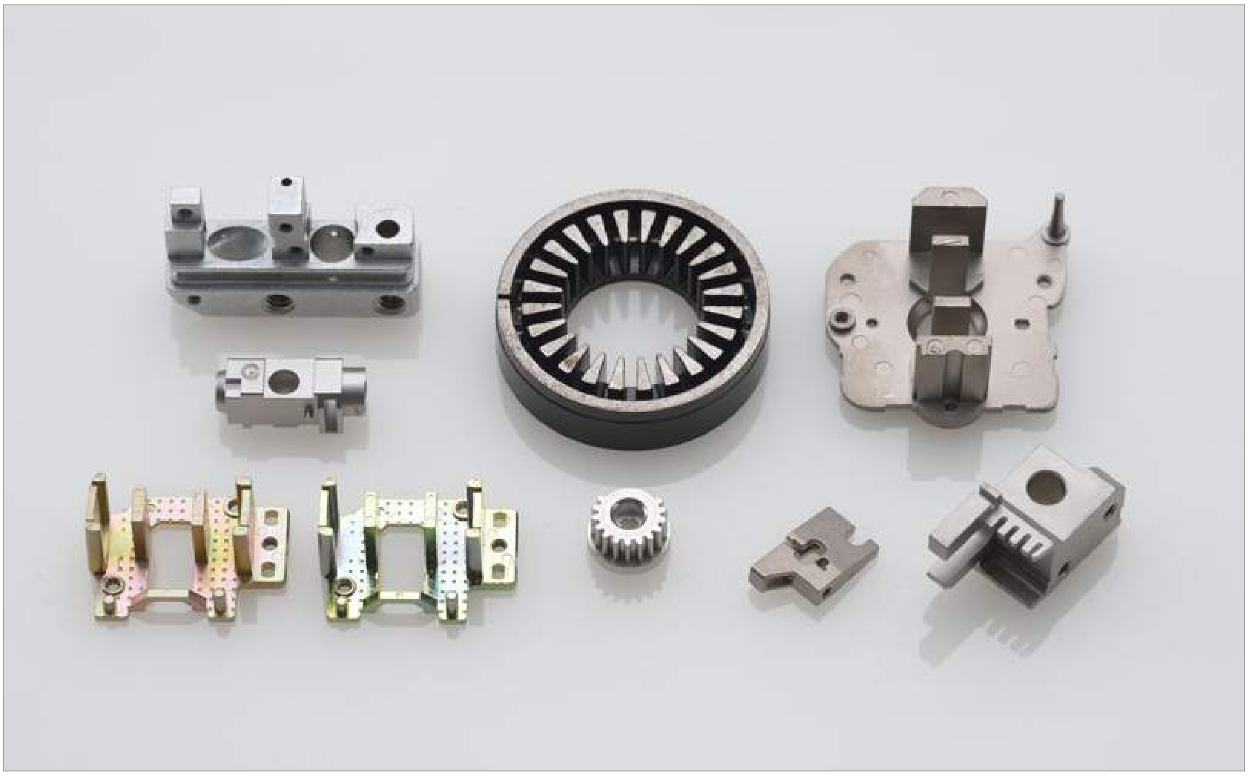


Fig. 12 A selection of MIM components for various industrial sectors

the issue by changing the pressure control of the injection cylinder. Moreover, we were able to significantly improve the quality by gradually reducing the holding pressure after filling, from what was a constant pressure."

Debinding

For the debinding stage of the process, solvent debinding was adopted as it enabled a shorter debinding time, consequently shortening the entire manufacturing time. Since the process does not require heating during debinding, NPR stated that it enabled higher dimensional precision and greater design freedom for these complex-shaped and relatively large parts.

Sintering

Sintering was performed in a vacuum furnace, where dimensional precision was improved by modifying the sintering trays and setter plates to counter deformations such as warping, which have a negative impact on final component dimensional tolerances. Spacers in the shape of the hollow gaps were also used to maintain critical tolerances.

"As a result, we were able to use the MIM process to enable the near-net shape fabrication of the roller rockers. These components could not have been viably manufactured using conventional processes such as forging and machining. The use of MIM consequently led to a reduction of machining steps by half."

Case study: High precision fuel injectors

Fuel injector components for automotive engines are today an important application for MIM technology. These injector components were first developed by NPR in the late 1990s in order to deliver improved responsiveness, reduced weight and lower costs. To achieve both the performance and cost reductions, the conventional needle-type armature inside the injectors was replaced with a ball-type armature. The armature body was also designed to be hollow so as to reduce the armature's movement and improve fuel flow control. Example components are shown in Fig. 11.

Using the conventional processes of forging and machining for such a design raised a number of production issues that could lead to increased machining costs and decreased productivity. Mass production was also considered to be unfeasible if machining was required to create the lateral fuel passage hole. To address these issues, the MIM process was investigated during the development of the armature body.

Material selection

Replacing the conventional electromagnetic stainless steel with the Permalloy PB greatly improved magnetic flux density and reduced coercivity. Thanks to MIM, processing requirements were also minimised and a magnetic annealing step was eliminated. The magnetic properties of NPR's MIM alloys are shown in Table 3.

	Composition	Permability	Coercivity (A/m)	Magnetic flux density (T)
Permalloy	Fe-47Ni	15000	16	1.46
Permendur	Fe-49Co-2V	3100	200	2.30
Fe-3Si	Fe-3Si	12600	32	1.52
SUS410L	Fe-13Cr	2860	127	1.31
Pure Fe	Fe	5000	1194	1.50

Table 3 Properties of NPR's MIM magnetic alloys

Moulding

"Since the production volumes for these parts are extremely high, achieving stable production was essential and we focused on optimising all aspects of moulding as we continued the development. We used a plastic moulding simulation tool that was adapted for the MIM process by measuring and employing the physical property value of the feedstock to be used. For moulding, multi-cavity moulds were used to support the high production volumes and the simulation data was helpful in minimising the moulding variations of each cavity."

Based on an analysis of the simulation results, NPR determined the shape, quantity and size of the runners and the position and quantity of the gates to design the optimal mould. "We also took various measures from the viewpoint of preventing defects such as scratches and to minimise handling as much as possible. This included eliminating product sorting, with automatic gate cutting after moulding, and automated placement of parts for the debinding and sintering processes," commented Kimura.

These MIM fuel injector components have served as a mainstay of NPR's MIM business. MIM injector parts were further developed in 2003 and 2005 and these three models have been produced in volumes of up to a million parts per month.

Future prospects

NPR believes that there are a growing number of opportunities for MIM products in the automotive industry. For example, small and complex parts that make up assemblies produced by tier 1 suppliers, or 'mega suppliers', often feature multiple parts that could be consolidated into one component using the MIM process. "Considering these opportunities, it is important that we continue to raise awareness of the MIM process among designers within the mega suppliers," commented Hiroshi Sato, General Manager of NPR's Product Engineering Department II.

NPR believes that further opportunities in the automotive industry are also available thanks to the rapid growth of electric and hybrid vehicles, with technologies such as electric power steering and brake systems seeing increased use.

Business expansion into non-automotive markets

"Because of the nature of our business, the majority of our products are for automotive engine components. However, we are striving to increase our sales in other non-automotive areas. Recent developments in the MIM market show that the large applications that are adopting a large number of MIM products, for example consumer electronics and industrial machines, are steadily growing," stated Sato.

One of the strategies of NPR is also to enter into new markets such as medical and aerospace. "To enter into these markets, it is necessary to offer materials such as pure titanium and titanium alloys. Although we have previous experience of producing titanium and titanium alloy MIM products in small volumes, we do not yet have any mass production experience. In preparation to mass produce these materials, we are currently evaluating our manufacturing processes and facilities".

"With regard to expanding our business to the medical field, we are continuing our sales activities focused on North America as the main market. Currently, our production is based only in Japan. However, expanding production overseas may also be an option. We have identified MIM as a key technology to enable us to expand our production volumes and support our company's growth, and will continue our efforts to contribute to the global expansion of the MIM market in the future," concluded Sato.

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Ceramic Injection Moulding: Binder innovations and Additive Manufacturing open up new opportunities

Ceramic Injection Moulding (CIM) is receiving ever more attention as a process for the production of complex shaped components in high volumes and to tight tolerances. As Dr Karin Hajek from Germany's Inmatec Technologies GmbH explains, there have to-date been a number of obstacles to the increased adoption of the technology. The availability of a wider range of binder systems, combined with the opportunities presented by ceramic Additive Manufacturing, may now open the door to a new generation of applications.

Ceramic Injection Moulding is a shaping technology for ceramic parts which offers significant advantages where complex geometries and high part numbers are required. The technology offers the ability to produce complex, three-dimensional designs to a high dimensional accuracy and with a high quality surface finish. Using ceramic powders, parts can be manufactured to the highest technical and aesthetic specifications. Narrow tolerances enable the production of complex parts in a single moulding step, so expensive post-processing is rarely needed and the mass production of highly complex ceramic parts is supported thanks to a high degree of process automation.

When looking to the worldwide Powder Injection Moulding (PIM) market, the Metal Injection Moulding (MIM) industry holds the major share, with global growth estimated to be in excess of 12% a year. Broadly speaking, CIM is thought to be around ten years behind MIM in terms of market penetration, but a faster development of the CIM market has been observed. This is supported by positive sales growth for CIM feedstock at Inmatec Technologies GmbH. In 2017, the company experienced its best year ever and the outlook for 2018 is even more promising.

One of the main drivers for the MIM business comes from Asia and is fuelled by the dynamic computing, communication and consumer electronics sector (3C), for which MIM parts are made by the million.

CIM parts are used for structural as well as for functional applications. However, it is the market for coloured zirconia for aesthetical applications that has grown very strongly over

Fig. 1 Ceramic Injection Moulding feedstock manufactured by Inmatec Technologies GmbH

Fig. 2 A CIM thermal insulation tube in the green, sintered and finished states. The tube was produced with the INMAFEED alumina based feedstock INMAFEED K1008. The dimensions are remarkable: the overall length is 89 mm and the green weight is 335 g (Courtesy Kläger Spritzguss GmbH & Co. KG, Germany)

the last ten or so years. Swiss producers of luxury watches have boosted the market with new shapes and designs, and the automotive industry also is looking for new aesthetic applications for luxury interiors.

"Swiss producers of luxury watches have boosted the market with new shapes and designs, and the automotive industry also is looking for new aesthetic applications for luxury interiors"

The production of translucent CIM parts is also a strong growth area for the industry. The production and processing of feedstock based on ultrapure Al_2O_3 powder for translucent CIM parts is, however, challenging. Parts without any grey tint can be achieved, along with high mechanical strength in the final part, but the entire processing chain must be strictly controlled so that any contamination with metallic particles is prevented. The sintered products must also be free of porosity and the grain size should be smaller than the wavelength of visible light, i.e. 400 nanometres. Substantial efforts have been made at Inmatec to develop this type of feedstock.

In the world of functional ceramics, the demand for more complex multifunctional components has led to the application of CIM parts in a wide range of industries. One example is an innovative glow plug for diesel engines which helps to reduce NOx emissions. These are made from four different ceramic materials, which are co-sintered in one step.

Titanates, with a positive thermal coefficient (PTC), enable the reduction of electrical consumption by 90% in some applications. This enables a longer period of operation for battery powered devices. Here, CIM parts have the added benefit of enabling the production of complex 3D geometries and uniquely thin walls.

These unique advantages of CIM will become even more important in the future, particularly for the automotive industry as it moves towards electrification. The continuously increasing need for electronic parts, coupled with miniaturisation and reduction of powder consumption, is expected to lead to a further acceleration of growth in the CIM industry beyond 2020.

What will accelerate CIM's growth?

In order to answer this question, we need to look at the obstacles currently restraining the rapid development of CIM parts. At least three related factors can be identified.

Firstly, CIM parts to be produced now and in the future might have or need:

- Very thin walls combined with long flow distances
- Smaller or larger dimensions than usually considered for CIM parts
- The possibility to allow for green machining
- Green part stability for mass production
- A robust process.

Secondly, the production of all ceramic parts takes time. The production of CIM parts, however, takes even more time because of long debinding and sintering steps.

Thirdly, for the efficient production of high volumes of CIM parts, multi-cavity or multiple moulds are required and the design and construction of these also takes time. Moreover, a mould is needed at an early stage of the development process in order to evaluate prototype parts. This means that a relatively high investment must be made with no guarantee of a successful outcome.

In order to address these factors, Inmatec has worked to meet the requirements of the CIM market. The demand for extending the dimensional range of CIM parts, as well as the other requests, have fuelled the R&D activities within our company. Alternative thermoplastic binder systems have been developed and evaluated and there are now three binder-systems available based on differing thermoplastic base materials. These are available either in combination with the ceramic materials that we offer as 'standard feedstocks' or as custom ceramic feedstocks.

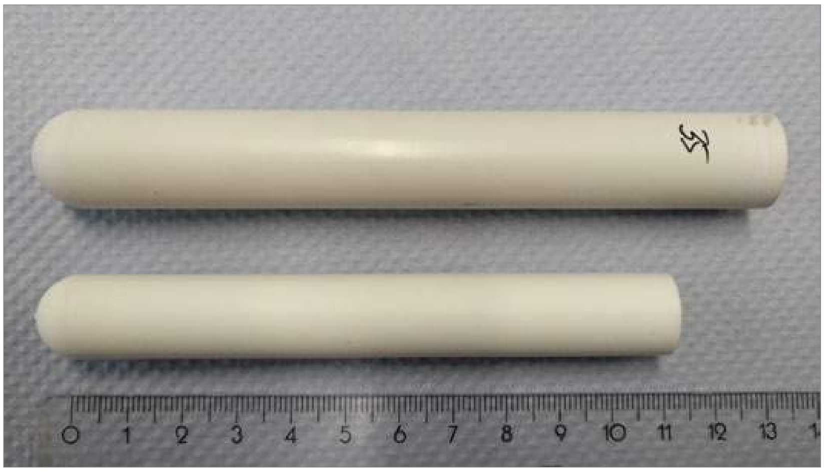

Fig. 3 A CIM tube in green and sintered state made from the feedstock INMAFLOW K2012. The flow distance is more than 130 mm combined with a wall thickness of 2.3 mm (Courtesy Fraunhofer IKTS, Germany)

The three-binder strategy for CIM feedstocks at Inmatec

Depending on the properties of a CIM part, its production volumes and any regional regulations and market needs, a customer can choose one or more ceramic feedstocks based on Inmatec's three different binder systems.

Polyolefin/wax based systems
Inmatec's INMAFEED feedstocks work on the basis of a polyolefin/wax-based binder system. The debinding stage is based on a two-step process, with the first step in water and the second step in a thermal debinding furnace with precise temperature control. This wax-based system has been approved for many years and works without any chemicals or acids. An example part is shown in Fig. 2. The total time needed for debinding parts ranges from 32 to 120 hours, depending on part geometry, wall thickness and the physical properties of the ceramic powder used in the feedstock. Very fine powders, such as zirconia, need the longer debinding times.

Polyamide based systems
INMAFLOW feedstocks work on the basis of a polyamide-based binder system. These show low viscosity and are perfect for thin walled parts and long flow distances (Fig. 3). Debinding is again a two-step process, with the first step in acetone and the second step via thermal debinding, as with the wax-based feedstocks. Here, the debinding time ranges from 32 to 70 hours.

"for the fast production of high volumes of CIM parts, multi-cavity or multiple moulds are required and the design and the construction of these also takes time"

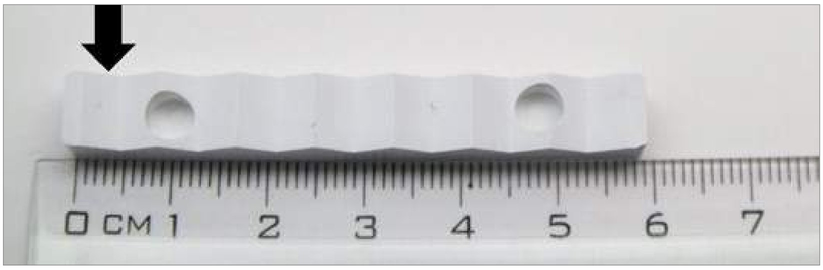

Fig. 4 The test part is a bar with rectangular geometry, a toothed surface and two out-of-line cores. The arrow marks the gate position

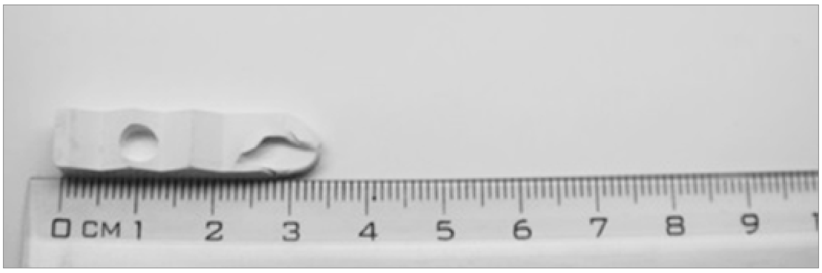

Fig. 5 Typical flow behaviour is observed with the INMAFEED zirconia feedstock. The flow fronts are not completely joined, however this would happen by completely filling the mould

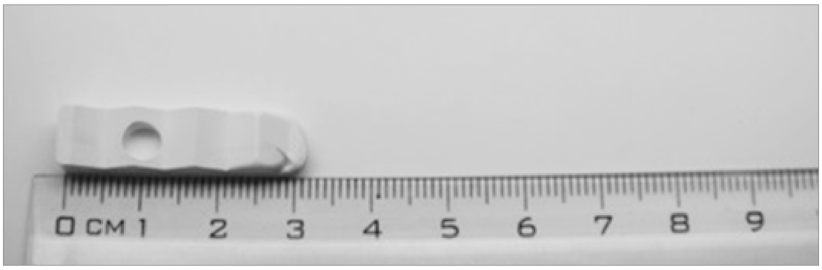

Fig. 6 The flow behaviour which can be observed with the INMAFLOW zirconia feedstock looks similar to that of a plastic material. The flow fronts are almost completely joined

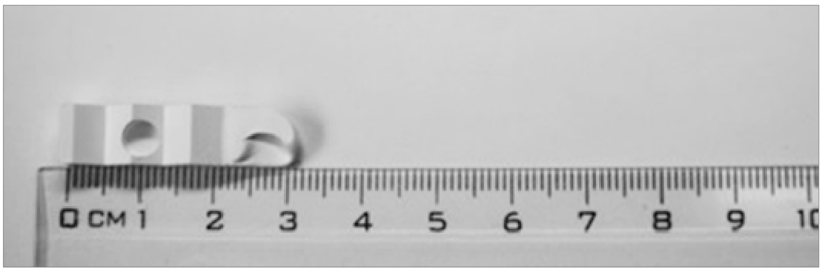

Fig. 7 The typical flow behaviour is observed with the INMAPOM zirconia feedstock. The flow fronts are again not completely joined, this will happen by completely filling the mould

Polyacetal (POM) based systems
Our INMAPOM feedstocks are based on a polyacetal binder (POM), a semi-crystalline thermoplastic material with good processing characteristics, high dimensional stability, high rigidity and good warm strength. Debinding takes place during a single debinding stage in a catalytic debinding oven.

The catalytic debinding process, which is widely used in the PIM industry, can be used as part of a fully automated production process with continuous 24/7 operation. The time needed for debinding ranges from 2 to 24 hours, increasing significantly with greater wall thicknesses.

Process support and application development

All three binder systems can be operated in a semi-automated or fully automated production process and the necessary equipment for efficient debinding of PIM parts is widely available. However, support for optimising the processes can be provided by Inmatec. We are able to perform injection moulding trials and all debinding and sintering processing in our own CIM laboratory.

When it comes to selection of the appropriate binder system, this is ideally based on a part's properties. To demonstrate the differing flow behaviour that the feedstocks exhibit, the flowabilities of the feedstocks have been compared using a unique test mould (Fig. 4) developed by Germany's Expertenkreis Keramikspritzguss (CIM Expert Group), a network CIM industry suppliers, researchers and component manufacturers.

It is known that the flow front of a ceramic feedstock behaves differently to that of a thermoplastic material. Plastic materials show a swelling behaviour during injection as well as a willingness to join flow fronts again after splitting. Ceramic feedstocks do not show any swelling behaviour and, once split, the separate flow fronts cool down and are usually difficult to reunite. In this test mould, the flow front of the injected feedstock is split immediately by the first core. The behaviour of the feedstock flow behind the core is interesting.

The flow behaviour has been observed by means of a short-shot study conducted with all three feedstocks, processed with the same zirconia powder. What can be seen is the differing flow behaviour of each of the three feedstock types (Figs. 5-7). This behaviour should be taken into consideration when deciding on a feedstock for a new application, although other factors such as part size, geometry and number should also be considered.

We are able to support customers along the whole process chain, from part design and feedstock selection to series production. Our injection moulding experts are, in particular, able to support customers during development and optimisation of their injection moulding operations.

Embracing ceramic Additive Manufacturing

Coming back to the original question of what will accelerate the growth of the CIM industry in the immediate future, another processing innovation of great importance is Additive Manufacturing (AM). In general, Additive Manufacturing technologies offer the possibility for tool-free production of components with complex geometries in a short period of time.

In relation to the argument regarding the long time period needed for a CIM component to reach series production, Additive Manufacturing now offers a number of solutions for the ceramics industry, one being Fused Filament Fabrication (FFF), also known as Fused Deposition Modelling (FDM™). This is a manufacturing method in which an endless filament is used as a semi-finished product which is melted and deposited under a heated nozzle.

Ceramic filaments for FFF have been developed by Fraunhofer IKTS, Dresden, and Inmatec (Fig. 8) [1]. With a ceramic filament made from INMAFLOW K2010, ceramic parts have been produced using FFF which show a dense microstructure after sintering thanks to the highly filled nature of the feedstock (Fig. 9).

The advantage of this shaping process is not only its speed, but also the fact that no mould is needed. The part is directly formed during deposition of the filament made from the ceramic feedstock [2]. INMAFLOW K2010 filament satisfies the elasticity and printability requirements of standard FFF devices and after printing, the parts are debound in a solvent and processed further in a typical CIM-like way.

Fig. 8 Filament coil made from INMAFLOW 2010 (Courtesy Fraunhofer IKTS, Germany). This was developed as part of a project supported by Germany's Federal Ministry for Economic Affairs and Energy (BMWI) with the "Zentrales Innovationsprogramm Mittelstand" [1]

Fig. 9 A sintered Al_2O_3 laval nozzle made by FFF (Courtesy Fraunhofer IKTS, Germany)

The synergy of CIM and AM

What CIM and ceramic AM have in common is that ceramic parts with complex three-dimensional designs can be produced. By combining the shaping methods, a customer has the possibility to evaluate and test real parts in operation. It is simple and quick to modify parts as often as needed – they can easily be manufactured with the same or altered geometry again by FFF.

Having established the optimal part design using FFF, the next step can be undertaken; the construction of a mould to transfer the production of the part to volume production by CIM. The parts that are produced in high volume by Ceramic Injection Moulding use the same raw material - the ceramic feedstock.

Currently, the quality of the FFF part in terms of surface finish and tolerances is still lower than the high dimensional accuracy and high surface quality of CIM parts. In principle, however, the necessary machinery for FFF is already available. Going from three to five axis movement and adding machine heads for subtractive processes, for example machining or drilling, will extend the possibilities and the quality of FFF parts substantially. FFF parts can then be created that meet the highest technical demands and aesthetic standards. Cost reduction and faster component development times are therefore important advantages that AM offers to CIM producers.

Combining AM and CIM for finished products

In the near future, there will be a further connection between AM and CIM. For example, parts could be injection moulded by CIM, followed by an individualisation process by AM. The combination of these technologies would allow the individualisation of production components produced in high volumes.

A piece of ceramic jewellery, for example, could be created with unique details. This flexibility could be taken to the point where the end customer chooses or designs individual features which are then applied to a piece of jewellery. This combination of ceramic shaping technologies could also lead to customised medical products covering a very wide range of applications, from dental parts to surgical tools and other medical devices.

Outlook

Through the ongoing development of binders and feedstocks for advanced ceramic forming applications, Inmatec is well positioned to support its customers in the creation of new applications using CIM and AM technologies. This development is also taking place on a European level through participation in European Research Projects such as CerAMfacturing [3] where, amongst others, the combination of AM and CIM is a main topic.

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[3] European Research Project, CerAMfacturing, Grant Agreement No 678503

Nishimura Advanced Ceramics targets the international PIM feedstock market

This year, Japan's Nishimura Advanced Ceramics Ltd. celebrates a hundred years of technical ceramics production, having begun with electromagnetic devices and later ceramic insulators, as well as a range of further ceramic products over the intervening period. The family-run company, now in its fourth generation, was also an early developer of PIM technology in Japan, having begun its development and production of CIM feedstock and parts over thirty years ago. *PIM International* reports on the company's ambitions for growth in international markets, in particular with respect to PIM feedstock.

Nishimura Advanced Ceramics was founded by Masajiro Nishimura in 1918 and has remained a family-run company for a century, with Yoshihiro Nishimura taking over the reins from his father Yoshio in 2011. Yoshihiro's son, Ko, is also in the family business with the role of Overseas Sales Manager. The company, located in the Yamashina district of Kyoto, was until January 2017 known as Nishimura Porcelain Ltd. Its current name was chosen to reflect the growing range of structural ceramic components that have been added to the company's production in recent decades, and to strengthen the company's image as it seeks to expand its exports business globally for its range of advanced ceramic materials, and particularly for Ceramic and Metal Injection Moulding feedstocks.

The company's product range includes functional and structural ceramic parts made from conventional 96% purity (N-96 grade) and 99.9% purity (N-999) alumina, translucent and light reflecting alumina, heat radiating alumina, porous alumina, zirconia (including coloured grades and high fracture toughness grade), aluminium nitride and many more ceramics. End user industries include automotive, electrical and thermal insulation, electronics and sensors, energy and power generation, wear and corrosion protection, medical technology and mechanical and chemical process engineering. Nishimura Advanced Ceramics Ltd uses dry and wet pressing, casting, and Cold Isostatic Pressing as well as extrusion and injection moulding equipment to manufacture its range of ceramic structural parts.

Fig. 1 These zirconia connectors are examples of typical CIM parts manufactured by Nishimura Advanced Ceramics

Fig. 2 An injection moulding machine at Nishimura Advanced Ceramics

Fig. 3 Nishimura Advanced Ceramics uses a range of furnaces for sintering from 1300 to 1700°C

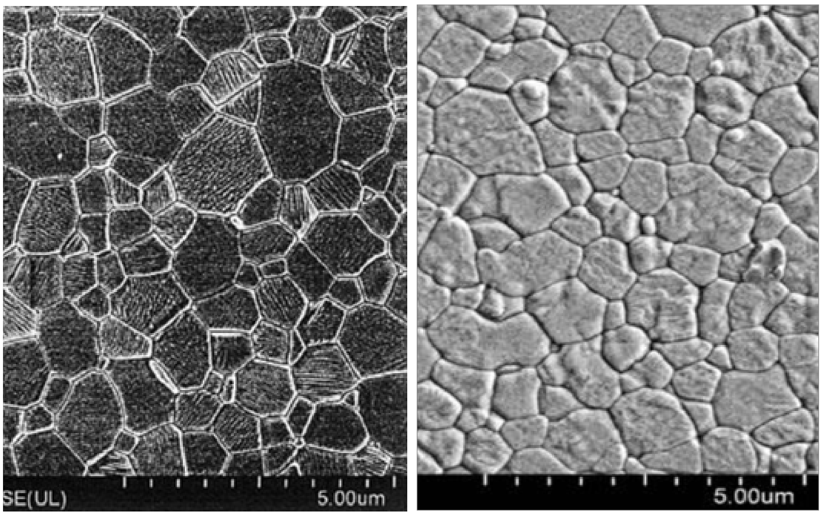

Fig. 4 Left: typical microstructure of a sintered N-9000NS alumina ceramic; Right: microstructure of sintered N-650 zirconia grade

Over a hundred proprietary ceramic formulations are now offered by the company for the various components being produced, including in-house developed alumina and zirconia CIM feedstock using a proprietary binder first developed in 1986 and used in the production of CIM components for the past thirty years. New ceramic material formulations and applications have been developed through in-house R&D projects and in projects in partnership with customers. The company has also greatly benefited from its long term technical cooperation with the Kyoto Municipal Institute of Industrial Technology and Culture.

Ceramic Injection Moulding feedstock

According to Nishimura Advanced Ceramics, its CIM feedstock offers a number of advantages over other commercially available systems. Nishimura uses its own proprietary binder formula that is mixed and kneaded with ceramic powders in batches that range from two to fifty kilogrammes.

The feedstock is said to have low thermal degradation properties, allowing it to be recycled up to ten times compared with what the company suggests is three times for many other commercial CIM feedstocks. It is also said to be less abrasive on the moulds used during injection moulding.

Alumina powder (96% and 99.9% purity and in black or white colour) and zirconia powder (also in different colours and including high fracture toughness ZrO_2) are often used for CIM feedstock, but other types of ceramic feedstock can also be produced. These include aluminium nitride ceramic, introduced in the 1990s, and the more recently available high heat radiating alumina (N-9H grade) for LED parts and heat sinks, translucent alumina (N-9000NS grade), light reflecting ceramic (N-9000T grade), ytrria and others. Research is currently ongoing

Fig. 5 Nishimura Advanced Ceramics' management team. From left to right: Ko, Yoshihiro and Yoshio Nishimura

into apatite and nano apatite powders, which show excellent biocompatibility in medical applications.

Fine-grained 99.99% purity translucent alumina (1-3 µm - N-9000NS grade) has been used to produce ceramic injection moulded parts which can have > 83.7% light transmittance and extremely smooth surfaces. Feedstock is also produced using submicron size N-9000NS alumina powders. In conventional applications, N-9000NS alumina is used in semiconductor devices, substrates for electronic parts, reaction tubes, crucibles, medical equipment and analysers. A potential future application area is in orthodontic brackets.

CIM component production

To produce CIM components, Nishimura Advanced Ceramics Ltd uses four domestically produced injection moulding machines with a clamping force of up to 32 tons (Fig. 2). According to the company, all of its machines have been modified in-house for Ceramic Injection Moulding using either single shot moulds or moulds with up to eight cavities for the smallest CIM parts. The largest CIM part in production has dimensions of 20 mm x 16 mm x 100 mm. The moulds are produced externally by a partner company.

Debinding of the moulded CIM parts is done in air or using protective atmosphere furnaces at temperatures up to 450°C, depending on the material being processed. Debinding using the company's proprietary binder is said to be faster than that needed for conventional binders. Where an additional shaping step is required, a limited number of CIM parts are also machined in the green state.

Parts are then sintered to high or full density in Nishimura's thirteen batch furnaces (Fig. 3). While the furnaces offer a temperature range of 1300 - 1700°C, most of the high purity alumina and zirconia are sintered at the lower temperature. A typical microstructure of a sintered alumina part made from grade N-9000NS is shown in Fig. 4 (left) and shows the

"Fine-grained 99.99% purity translucent alumina has been used to produce ceramic injection moulded parts which can have > 83.7% light transmittance and extremely smooth surfaces"

Fig. 6 Preparation of a mould for Ceramic Injection Moulding at Nishimura Advanced Ceramics

optimised regular grain size which can be obtained with Nishimura Advanced Ceramic's alumina feedstocks. Fig. 4 (right) shows a typical sintered microstructure of grade N-650 zirconia produced by Nishimura Advanced Ceramics Ltd. Zirconia has excellent hardness, toughness and wear resistant properties; it also offers good electric insulation and chemical resistance.

CIM applications

The company produces a range of CIM parts for applications including the medical industry, smart phones, nozzles, watch parts and parts for electronic devices. Temperature sensor parts are also an important application area and these are made from a high thermal emittance alumina ceramic designated N-9H, which has recently been introduced by the company and uses radiation, rather than conduction or convection, to transfer heat.

The patented N-9H alumina ceramic, whose properties are shown in Table 1, has found successful applications in heat sinks and LED devices. The company currently produces heat sinks and LED parts using non-CIM manufacturing processes, but states that these parts can be produced by CIM to meet design requirements. A further selection of CIM ceramic components currently in production at Nishimura Advanced Ceramics Ltd is visible in Fig. 7.

MIM feedstock production

Although the company does not manufacture any MIM parts, commercial MIM feedstock production at Nishimura Advanced Ceramics dates back to 1989. The blending of the metal powders and binder material for MIM feedstock is done in a separate section of the plant to avoid any cross contamination with ceramic

	N-9H	AlN	Cu	Al
Emissivity (100°C)	0.97	0.93	<0.1	<0.05
Thermal conductivity [W/m · K] (25°C)	39	180	394	238

Table 1 Emissivity and thermal conductivity properties of the N-9H alumina developed by Nishimura Technical Ceramics

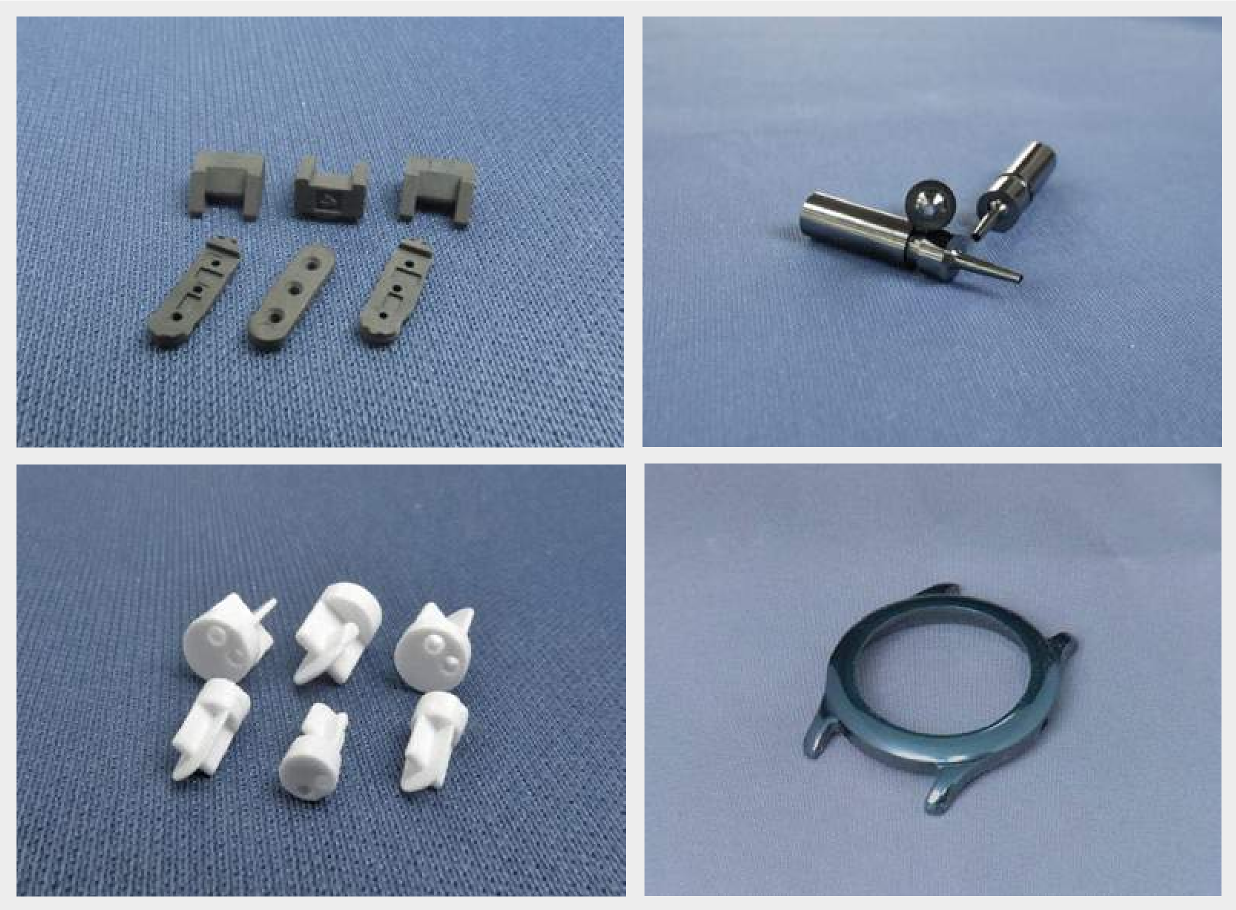

Fig. 7 CIM components manufactured by Nishimura Advanced Ceramics. Top left: black alumina (92%) electronic components; top right: black zirconia yarn guides; lower left: forsterite camera parts; lower right: zirconia watch cover

materials. The binder used for MIM is different from that used in CIM feedstock but is also stated to have unique properties allowing for faster debinding and less deformation in the debinding step, and with little carbon residue in the sintered parts.

Nishimura Advanced Ceramics currently processes the customer's own metal powders to produce the feedstock, but plans to introduce its own metal powder supply for MIM feedstock in 2018. The range of metal powders currently processed includes 316L stainless steel, Fe-2Ni, Fe-Co, tungsten carbide, and other special steels.

International marketing of CIM and MIM feedstock

The company is confident that it will continue to develop new ceramic components using its CIM technology in the years ahead, despite the challenges of the relatively high manufacturing cost of CIM for the domestic market in Japan. It believes there is considerable potential for CIM components in a number of market sectors particularly in the medical sector and in smart phone applications. In addition to producing its CIM feedstock for in-house use, the company has also achieved success in marketing its CIM feedstock, with its proprietary binder, to the international market.

The company has been involved in the development of metal powder feedstock since 1989, at which time it began a development project with one of Japan's large steel producers to develop a stainless steel MIM feedstock for the production of injection moulded and sintered watch cases. The company did not enter MIM production, but has instead focused over the past twenty years on producing MIM feedstock on a relatively small scale for external MIM producers, using its experience in developing binders and feedstock for CIM.

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Advanced PIM processes demonstrate the potential for MIM and CIM to reach new markets

A technical session at the Euro PM2017 Congress and Exhibition, held in Milan, October 1-5, 2017, focused on the development of advanced processes for PIM. This report reviews three papers from this session that present the advanced processes required to produce MIM parts with high aspect ratios, to injection mould short-fibre Ceramic Matrix Composites and to manufacture alumina-rich porcelain parts.

Production of MIM parts with a high aspect ratio

As previously reported in *PIM International* [1,2], there has in recent years been growing interest in the production of larger parts with higher aspect ratios, particularly for the 3C market (Fig. 1). A paper presented by Marko Maetzig and Hartmut Walcher (Arburg GmbH + Co KG, Germany) and Martin Bloemacher and Sven Fleischmann (BASF SE, Germany) discussed this trend, addressing the production of thin-walled parts with a high aspect ratio for smartphone applications [3]. The main focus of this development was to achieve a faultless surface finish and narrow part tolerances.

The authors stated that, to-date, most MIM parts have been produced using an isothermal mould temperature concept, where the mould temperature is kept at the de-moulding temperature. This temperature is significantly lower than the temperature of the molten feedstock. Because the feedstock has a high thermal conductivity, it has to be injected at high speed to allow for good filling of the cavities without premature freezing of the feedstock. Typically, this results in higher pressures at the near gate area and lower pressure at the end of the flow path and produces green parts with inhomogeneous density distributions. It was said that these inhomogenei-ties can lead to larger dimensional tolerances and internal stresses in the part. Depending on the feedstock used, black demixing lines, close to the gate, can also be observed. The black lines indicate a lack of powder

Fig. 1 Arburg's MIM smartphone housing (Courtesy Arburg GmbH + Co KG) [2]

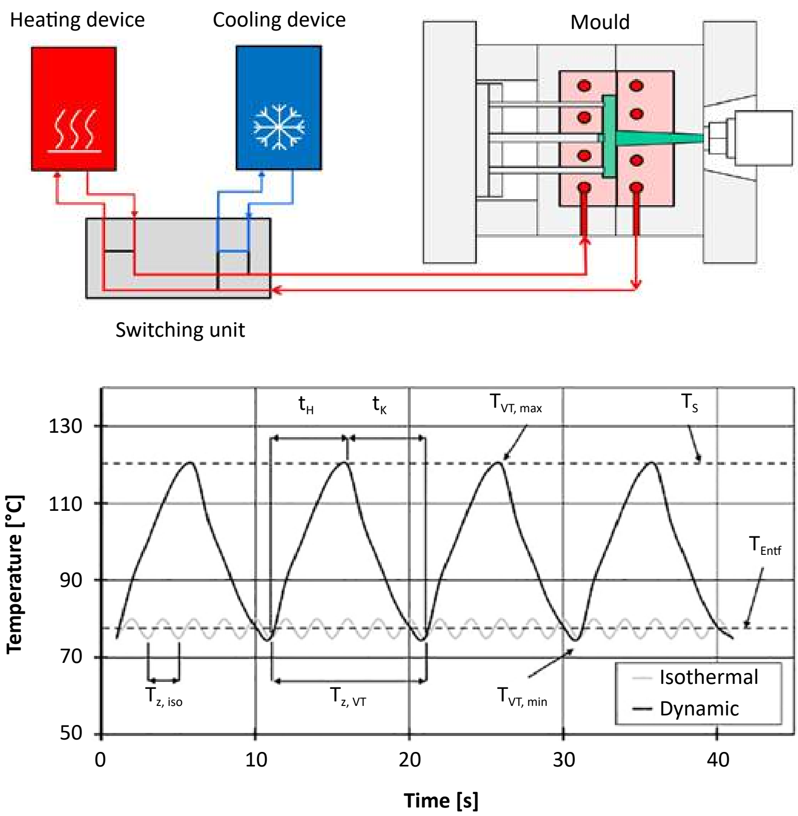

Fig. 2 Principal setup and temperature profile of dynamic mould temperature control [3]

and an excess of binder, which can result in surface defects. To solve this problem and to produce green parts with almost constant green density, the authors proposed the use of dynamic mould temperature control. A principal setup and temperature profile for such control is shown in Fig. 2.

A test mould, with interchangeable inserts, was developed for the production of a smartphone back cover and a smartphone frame. Green parts of both geometries are shown in Fig. 3. The overall dimensions of the green parts are reported to be 160 mm x 80 mm and wall thickness of the back cover ranges from 1.2 mm at the gates to 1 mm towards the outer edges. The frame has a wall thickness of 1 mm.

The test mould has been designed for dynamic mould temperature control and utilises interchangeable liquid medium controlled hot runner systems for the different parts to be produced. The use of a liquid heat transfer medium controlled hot runner and a specially designed oil temperature control unit allows the generation of a constant feedstock temperature, with deviations of less than +/- 1 K. Further more, the test mould was designed in such a way that a cycle time of less than 60 s can be achieved with a temperature difference of 70 K within each injection moulding cycle. The control of the cavity temperature is achieved using two temperature control units for heating and cooling, a valve switching unit and an energy battery to avoid high energy consumption.

All of the tests used Catamold 17-4PH Plus feedstock from BASF. This feedstock has been specially developed for the production of parts with long flow paths and thin walls and is said to exhibit high pressure transducability and good flowability. An Arburg Allrounder 470S 1100 – 170 moulding machine, dedicated to MIM processing, was used for all experiments. During injection moulding, the filling pressure was limited to values below 600 bar, resulting in a filling time of approximately 9 s. The cycle time for the production of one part was 56 s. A process chart is shown in Fig. 4.

The green parts produced were removed using a robot system and placed on a conveyer. Prior to debinding, the sprues were removed by cutting and grinding to provide a flat surface for debinding and

Fig. 3 Green parts of smartphone back cover (left) and smartphone frame (right) [3]

sintering. The parts were then debound and sintered at DSH Technologies LLC, using an Elnik catalytic debinding furnace and an Elnik sintering furnace. Catalytic debinding was conducted over 3 h and sintering was carried at 1370°C for 75 min under a hydrogen atmosphere. A total of 132 parts were sintered in one sintering cycle. After sintering, the dimensions of the parts were checked using a Zeiss Accura coordinate measuring machine, the length of the part being measured at two positions and the width at four positions.

The authors stated that the advantage of using dynamic mould temperature control was clearly demonstrated during the injection moulding tests. The two moulded parts can be seen in Fig. 5, with the part on the left being produced using isothermal mould temperature control. Because of the small wall thickness, the available injection pressure of the moulding machine was not sufficient to fill the part completely. There were also demixing lines around the gates. The part on the right was produced using dynamic mould temperature control, was filled completely at pressures below 600 bar and showed no demixing lines.

After sintering, the surface of the part was very uniform with no visible defects. For external surfaces this is an important result as it reduces the effort required for polishing. The authors stated that the first polishing trials of the sintered parts have already shown promising results.

Prior to measuring the dimensions of the parts, the repeatability of the measurements was checked on one part. Each measurement was repeated 20 times and the results, summarised in Table 1, were said to show a high level of repeatability.

The results of measurements on 66 sintered parts are summarised in Table 2, where xq is the mean value, s is the standard deviation, x_{max} and x_{min} are the maximum and minimum values, respectively, and tol is the tolerance. The results show that

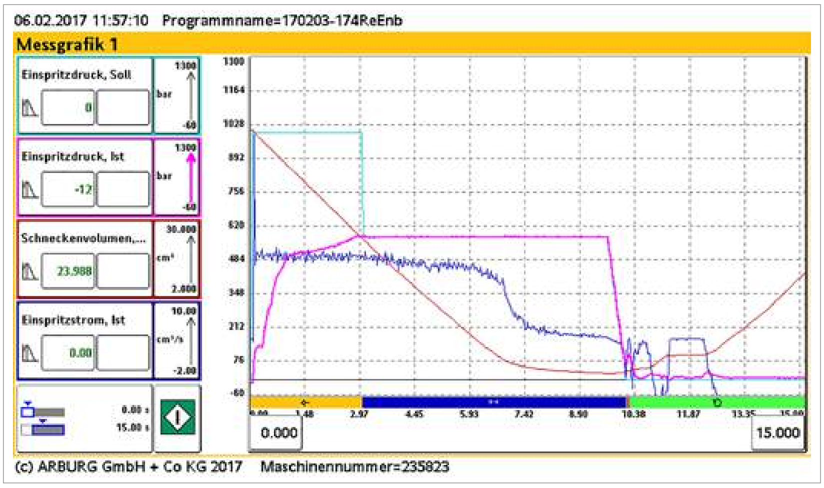

Fig. 4 Process chart for the injection moulding process [3]

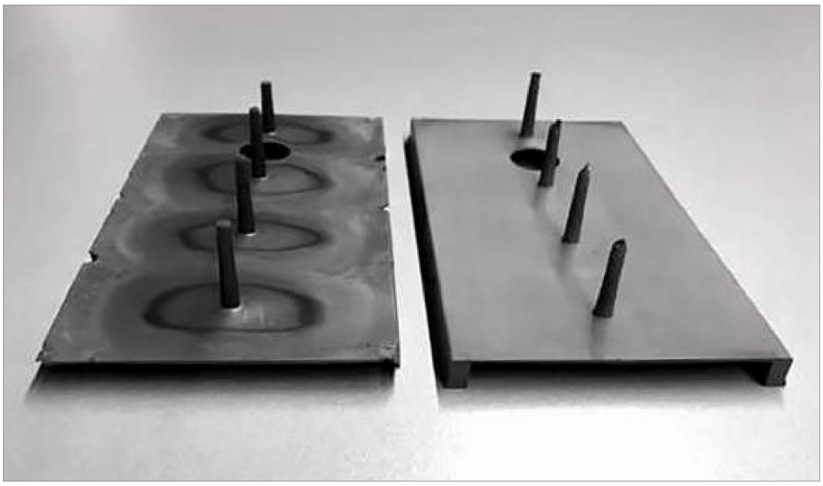

Fig. 5 Green parts produced without and with dynamic mould temperature control [3]

Value	L1	L2	B1	B2	B3	B4
s (x)	0.004	0.003	0.000	0.004	0.003	0.001
x_{max} - x_{min} [mm]	0.011	0.010	0.002	0.017	0.013	0.004

Table 1 Repeatability of measurements [3]

Value	L1	L2	B1	B2	B3	B4
xq [mm]	134.974	135.002	65.998	65.736	65.131	66.096
s (n)	0.067	0.065	0.032	0.057	0.049	0.047
x_{max} [mm]	135.105	135.122	66.075	65.890	65.251	66.198
x_{min} [mm]	134.859	134.873	65.922	65.624	65.017	65.991
x_{max} - x_{min} [mm]	0.246	0.249	0.153	0.266	0.234	0.207
tol +/- [mm]	0.123	0.124	0.076	0.133	0.117	0.104

Table 2 Results of dimensional measurements [3]

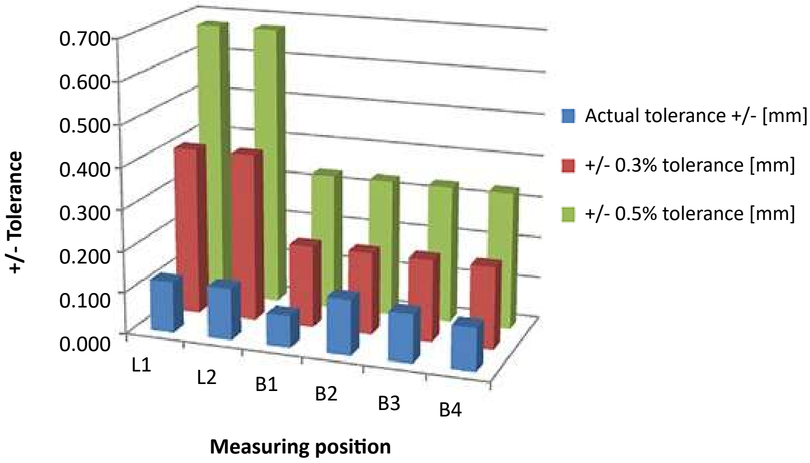

Fig. 6 Comparison of achieved tolerances with typical tolerances [3]

there are some minor differences in the mean values of the different measuring positions, but very high consistency within the sample. Although this consistency still has to be proved over several sintering runs, it can be assumed that very narrow tolerances can be achieved for each measuring position if comparing the achieved tolerances with commonly observed MIM tolerances of +/- 0.3% to +/- 0.5% (Fig. 6).

The authors added that proof of the repeatability of the achieved tolerances is still in progress, as are trials on sintering in continuous debinding and sintering furnaces.

Processing of short-fibre Ceramic Matrix Composites by CIM

In this presentation, attention was turned to Ceramic Injection Moulding. A paper from Volker Piotter, Metin Tueluemen, Alexander Klein, Rainer Oberacker and Benjamin Ehreiser (Karlsruhe Institute of Technology, Germany) reported on a study of the processing of short-fibre Ceramic Matrix Composites (CMC) by Ceramic Injection Moulding [4].

The authors stated that in recent years, the concept of improving material properties by embedding ceramic fibres into a ceramic matrix has transitioned from laboratory research to industrial utilisation. Typical applications for Ceramic Matrix Composites are, for example, aero engine parts subjected to high temperatures and stresses. It was added that the intention of the fibre addition has not primarily been to enhance the intrinsic properties, but rather to change brittle failure behaviour to a stepwise failure enabled by restricted crack propagation.

Currently, infiltration based methods are mainly applied for the processing of CMCs. However, these techniques are rather expensive and exploring new mass markets for CMCs would require the availability of cost-effective, large scale processes. In this context, Ceramic Injection Moulding would be a very attractive option, but a number of technical challenges need to be solved.

The reported study, therefore, was aimed at the development of feedstocks containing ≥ 50 vol.% solid content, with this solid content containing a maximum of 50 vol.% of fibres, assessing the required injection moulding conditions and investigation of injection moulded CMC samples in the green and sintered states.

A commercial fibre type (Nextel 610, 3M) was used in the study, with the fibres being chopped to a median length of 3.2 mm at a diameter of 11 µm. The matrix powder used was an Al_2O_3 type with a median particle size of $D_{50} \leq 200$ nm. The binder used (at 50 vol.% in the feedstock) was KIT's proprietary GoMikro-system, consisting of polyethylene, paraffin wax, stearic acid and other additives.

The authors reported that the fibre content of the solid filler was varied between 10 and 50 vol.%, whereas the solid content was kept constant at a 50:50 volume ratio of solid filler to organic binder. The additive (dispersant) concentration varied between 1.1 and 5.5 mg/m². As demonstrated in Fig. 7, compounding took place

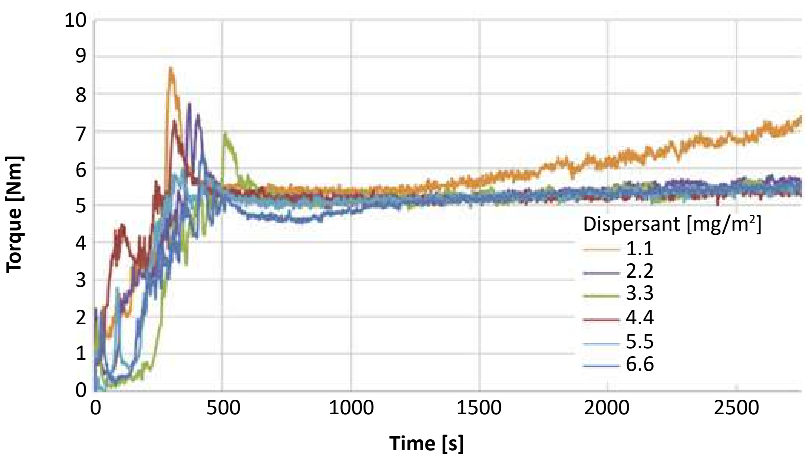

Fig. 7 Mixing torque over compounding time for different amounts of dispersant at a constant fibre volume content (10 vol.%) in the feedstock. After the initial mashing up hysteresis, the torque inclined to a nearly constant value except for the 1.1 mg/m² sample [4]

until a nearly constant mixing torque was reached. Only in the case of the 1.1 mg/m² dispersant level did the torque increase significantly, as this amount of dispersant was insufficient to cover the solid content in the feedstock. This composition, therefore, showed slight dosing problems in injection moulding and a limited suitability for further processing.

The fibre content had a clear influence on mixing torque (Fig. 8), with the torque increasing steadily with the degree of fibre filling.

After compounding, viscosities were measured using a high pressure capillary rheometer. The results of the rheological characterisation are shown in Fig. 9. All feedstocks showed the typical shear-thinning behaviour. However, the influence of the fibre content was less significant than had been expected. For example, even for a 25 vol.% fibre content in the original feedstock (i.e 50 vol.% in the debound part), the viscosity was virtually identical to that for pure powder filling.

Injection moulding trials were performed on a Battenfeld Microsystem 50 machine, which was equipped with a special PIM screw featuring a reduced compression ratio and volume. The maximum feedstock temperature was chosen as 160°C.

As a demonstrator part, a miniaturised tensile specimen was chosen (Fig. 10). Subsequently, the samples were pre-debound by dissolution in hexane for 24 to 48 h. The debound samples were then sintered at four different temperatures for 2 h in an air atmosphere.

Fig. 11 shows the relative densities of the samples sintered at different temperatures. The highest densities were obtained using pure powder filling; for example, density exceeded 90% once the temperature had been increased from 1150°C to 1350°C. It was stated that the fibre filling, however, obviously restricted the sintering process considerably, as demonstrated by the decrease of the achieved densities with rising fibre content for all sintering temperatures.

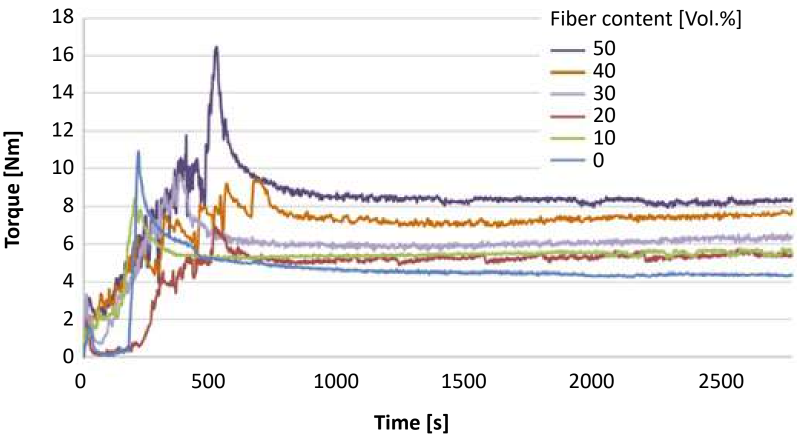

Fig. 8 Torque profile over compounding time depending on fibre content (in terms of the green part) at a constant dispersant content of 3.3 mg/m² [4]

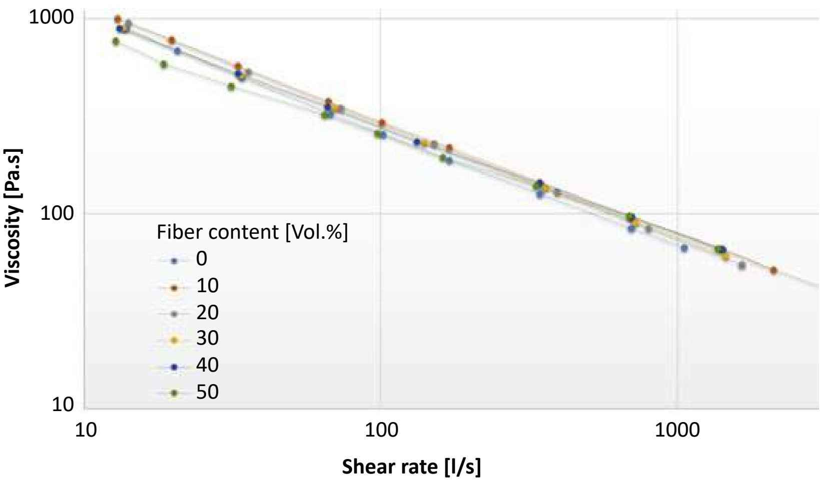

Fig. 9 Feedstock viscosity vs shear rate depending on the fibre content, given as a percentage of the solid content of the feedstock [4]

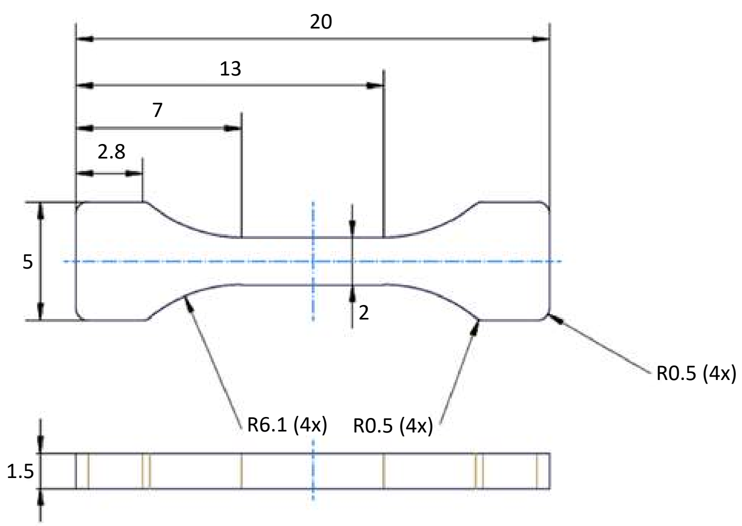

Fig. 10 A drawing of the miniaturised tensile specimen used as a demonstrator part [4]

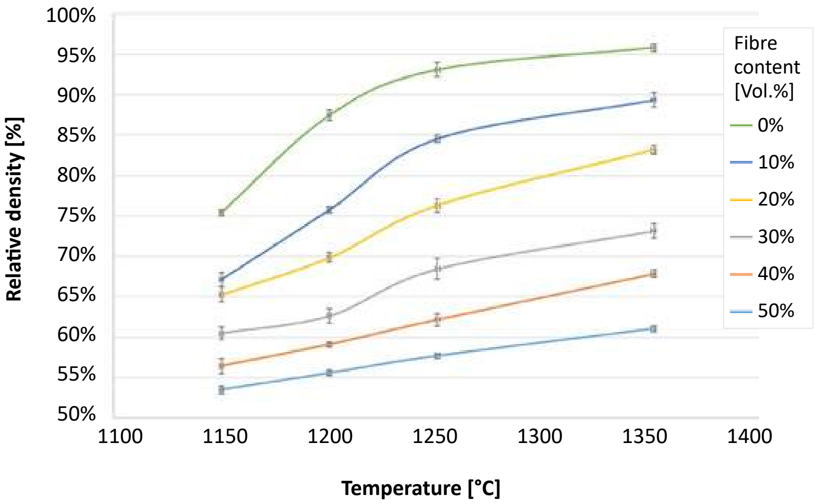

Fig. 11 Relative density of the sintered samples as a function of the sintering temperature and the fibre loading [4]

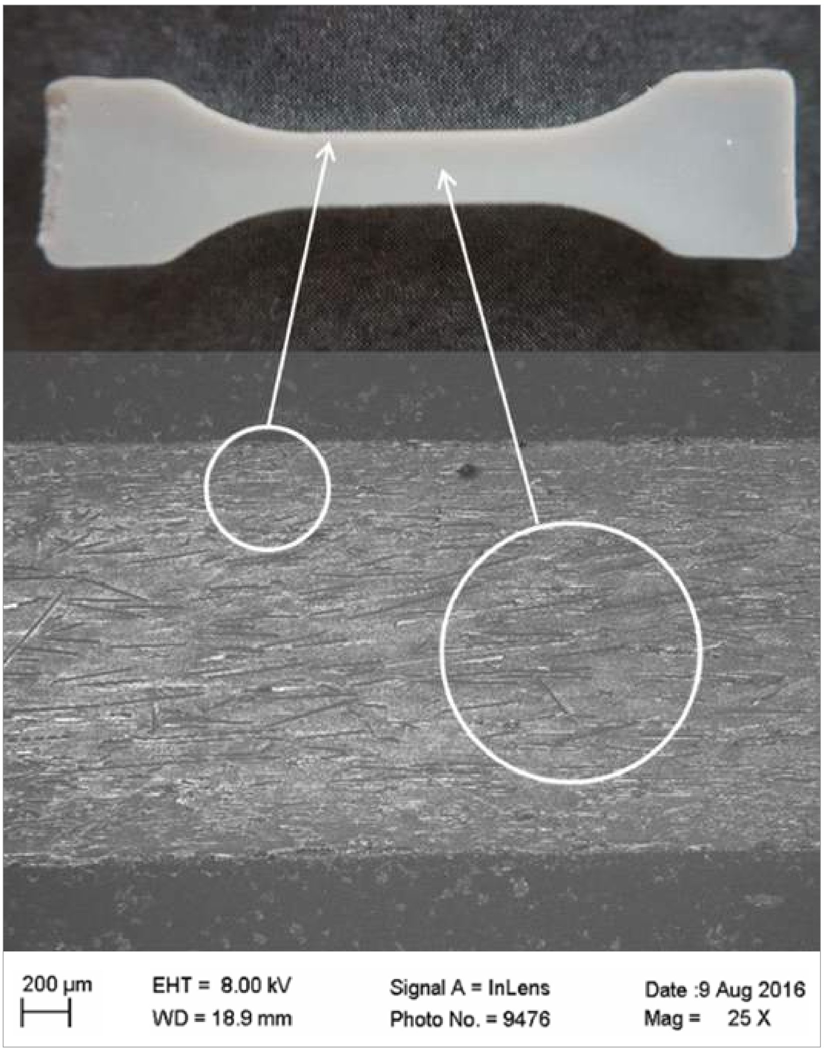

Fig. 12 Tensile specimen made of CMC feedstock (green body, above). SEM picture of the same sample, showing the high degree of fibre orientation near to the surface (high shear area) and a lower degree of orientation in the bulk, i.e. in the low shear rate area (below) [4]

It is known from the processing of fibre-filled plastics that the shear rate profile plays a dominant role in the orientation of the fibres. The same effect should also occur in the case of CMC-PIM feedstocks. In an initial investigation, green parts were ground and examined by SEM (Fig. 12). It could be proven qualitatively that the highest fibre orientation occurs in the central zone of the specimen. Additionally, with reference to the cross-sectional area of the specimen, it was found that the apparent degree of orientation is higher in the near surface region than in the bulk.

The authors concluded that, at this early research stage, some basic correlations had already been identified (as discussed earlier in this report), but that there were further crucial parameters that need to be investigated, particularly the average fibre length, which decreases during compounding and injection through fibre cracking.

Development and characterisation of alumina-rich porcelain parts processed by CIM

A poster presentation, attached to the same session, came from Guillermo Larraz Nogues, Alberto Gallego Bravo, Cristina Berges Serrano and Gemma Herranz Sanchez-Cosgalla (Universidad de Castilla-La Mancha, Spain) and addressed the development and characterisation of alumina-rich porcelain parts processed by Ceramic Injection Moulding [5].

Currently, the authors claimed, only a few companies produce commercial feedstocks for CIM and these feedstocks are mainly based on zirconia and alumina. Due to their properties and high costs, the final parts obtained from these base powders are only suitable for specific high technology and/or high cost sectors, such as aerospace, medical or jewellery. However, new potential markets, in which aesthetics are more relevant

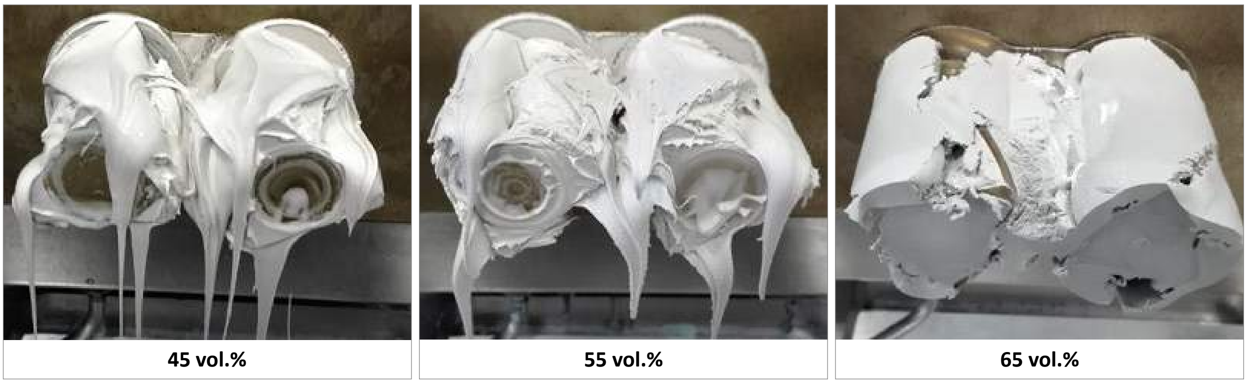

Fig. 13 The binder was mixed with different quantities of powder. The fluidity of the feedstock decreases as the porcelain load increases [5]

than mechanical properties, are said to be awaiting cost-effective feedstocks.

In the reported study, the authors addressed the goal of producing finished porcelain pieces by CIM showing good mechanical properties as well as meeting customer requirements in terms of price. For this purpose, a new low-cost porcelain with a high content of alumina (up to 70%) has been specifically adapted for the CIM process. This has been mixed with a binder system in different proportions and the optimum mixture, selected from rheological studies, has been injected using a low pressure injection machine. After optimising the debinding and sintering process, final parts were characterised by means of micro-hardness and three-point bending experiments.

The aluminous porcelain powder used had the composition presented in Table 3 and was produced by milling, atomisation and calcination with the aim of adapting it for the CIM process. The particle size parameters D_{10}, D_{50} and D_{90} were 1.8 µm, 4.9 µm and 13.3 µm respectively. This particle size distribution results in a distribution slope parameter (S_W) of 2.96, which is considered a suitable value for a powder for moulding. Powder density was measured with a pycnometer, while tap density was measured using the UNE-EN ISO 3953 standard procedure. Values of 3.21 g/cm^3 and 1.21 g/cm^3 were obtained, respectively.

% SiO_2	% Al_2O_3	% Fe_2O_3 + TiO_2	%CaO	%MgO	%Na_2O	%K_2O
26-27	69-70	0.2	0.1	0.1	0.8	2.9

Table 3 Chemical analysis of the fired powder [5]

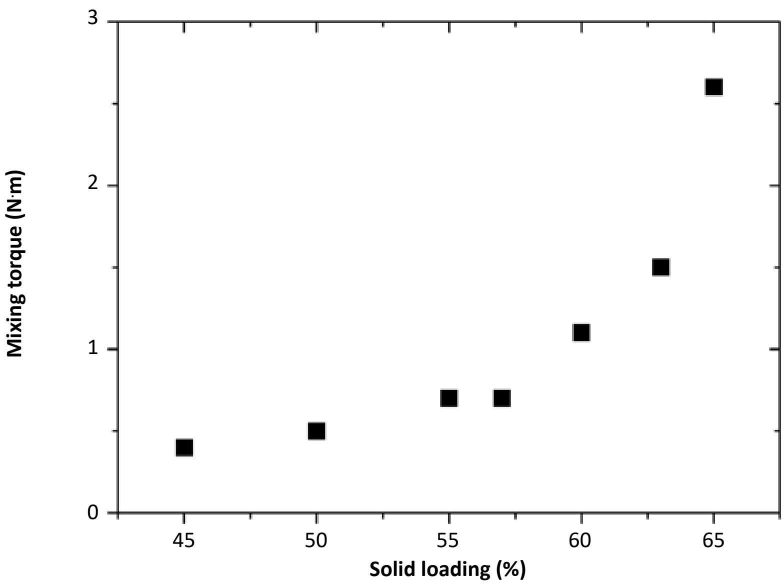

Fig. 14 Torque value as a function of the porcelain powder content (vol.%) [5]

The porcelain powders were mixed with a multicomponent binder system based on high density polyethylene (HDPE), paraffin wax (PW) and stearic acid (SA). For confidentiality reasons, the exact proportions of binder components used in the study were not revealed. The binder was mixed with different quantities of powder (between 45 and 65 vol.%) in a rotor mixer (Fig. 13). The mixing conditions for all feedstocks were 30 rpm and 160°C. The feedstocks were granulated in a mill.

Fig. 14 shows the evolution of a stabilised torque value as a function of the solid loading. Mixing torque increased slowly from 0.4 N·m at soft mixture to 0.7 N·m at 57 vol.%, as expected, until a drastic increase occurred above that solid loading. In order to verify the reproducibility of the process, several mixtures of each ceramic loading were performed, stabilising all of them at the same torque value. This signified that the mixing procedure was correct and reproducible. With reference to the dramatic torque increase above 57 vol.% ceramic loading, this could be an indication that the critical powder content is close, with all particles closely packed and all spaces between filled with binder. This situation leads to a high viscosity, which brings difficulties during the moulding process.

The rheological behaviour was evaluated using a capillary viscometer. The rheological tests were carried out in a temperature range of 150°C – 175°C. Feedstock was left in the barrel for about 6 min to ensure thermal equilibrium before starting the test. The range of selected shear rates was between 10 and 7000 s^{-1}.

An example of the rheology measurements is given in Fig. 15, which shows the results of the viscosity dependence with shear rate for 57 vol.% ceramic loaded feedstock at 160°C and 170°C. At low shear rates (<100 s^{-1}), a plateau could be observed in which viscosity remains almost constant, independent of the shear rate. This is observed especially at 170°C and can be assessed as signifying Newtonian flow. However, as the shear rate increases, the viscosity subsequently begins to decrease, according to a pseudo-plastic behaviour that is desirable for the PIM process. For feedstocks with a porcelain loading lower than 60 vol.%, the viscosity was less than 1000 Pa·s when shear rates varied between 100 and 1000 s^{-1}, the generally accepted restriction during injection moulding processing.

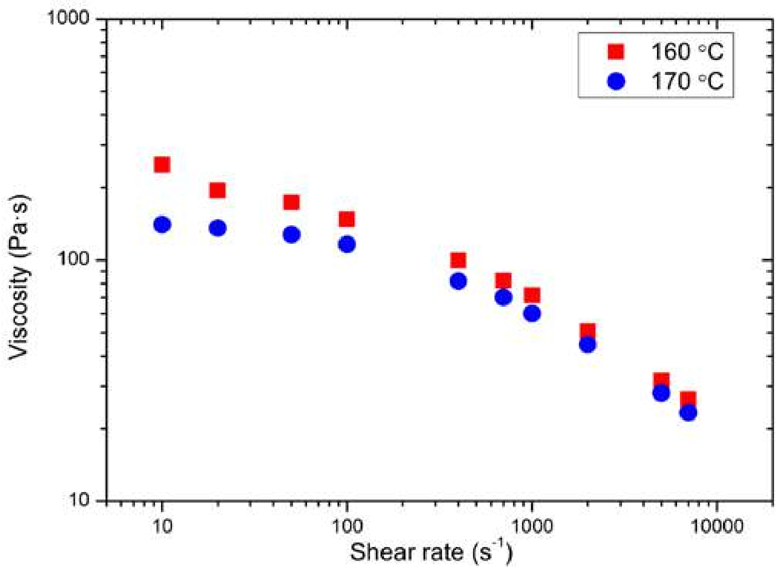

Fig. 15 Viscosity versus shear rate of 57 vol.% porcelain feedstock at 160 and 170°C [5]

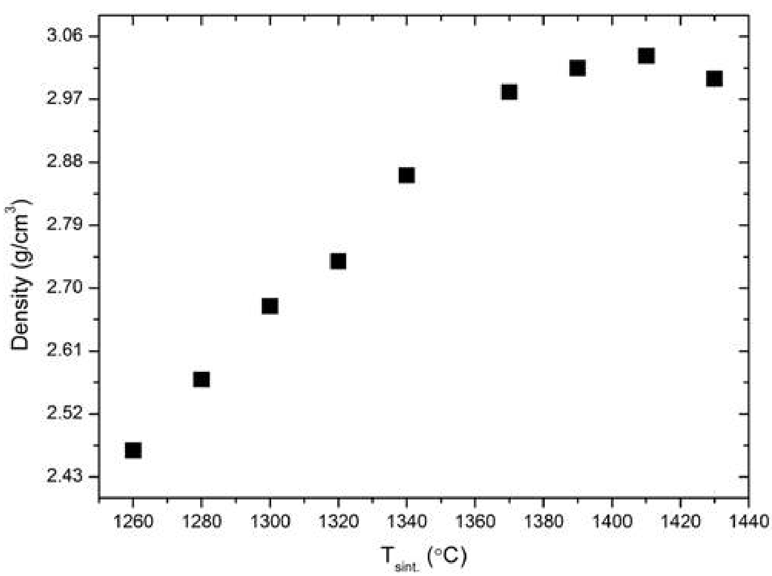

Fig. 16 Densities of final parts as a function of sintering temperature [5]

A low-pressure injection moulding machine was used to obtain bars with a rectangular cross section of 11.90 mm x 3.68 mm x 65.8 mm (green body dimensions). Taking into account both the machine limitation and the rheological properties (torque values and viscosity dependence), for the purpose of avoiding viscosity variations during the low-pressure injection process, which would lead to defective parts, the authors decided to inject a 55 vol.% ceramic loaded feedstock.

The authors stated that the injection process was performed using a piston pressure between 110 and 240 bar at temperatures in the range 150°C - 175°C. Green parts, free from defects, were successfully obtained at approximately 140 bar and 160°C. Thermal debinding of the parts was optimised and consisted of three temperature stages (120°C, 380°C and 440°C) in an oven under flowing air. Sintering was performed in a furnace at temperatures between 1250°C and 1430°C in air. The sintering process was optimised, with the highest density of parts being obtained at 1410°C (Fig. 16). For a 1 hour treatment at this temperature, a density of 3.04 g/cm^3 was achieved and the water absorption coefficient was 0%, signifying zero open porosity. The lower density reached in sintered parts, compared with the powder density, is due to the presence of vitreous phases typical of porcelain bodies. An isotropic linear shrinkage of approximately 15% was observed during sintering.

Mechanical properties of the final pieces were assessed by micro-hardness and three-point bending tests. Micro-hardness was measured on polished surfaces using a 1000 g load. Bending tests were performed using a tensile testing machine.

It was observed that both micro-hardness and bending strength increased with densification of sintered pieces, reaching values of 800 HV and 200 MPa respectively. The results obtained in the reported study for the highest density porcelain pieces are summarised in Table 4.

On the basis of these preliminary results, the authors claim to be optimistic that higher solid loading feedstocks could be moulded with a high pressure injection machine. This would, it was said, produce an increase in densification and, therefore, an improvement in all mechanical properties. Further experiments are reported to be in progress on this basis.

Sintering temperature (°C)	1410
Sintering time (h)	1
Sintering atmosphere	Air
Density (g·cm³)	3.04
Linear shrinkage (%)	15
Water absorption coeficient (%)	0
Vickers hardness (HV)	800
Flexural strength (MPa)	200

Table 4 Sintering conditions, density and mechanical properties of optimised final parts [5]

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[1] *PIM International* Vol. 10 No. 4, pp 7

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[4] Processing of short-fibre CMC by Ceramic Injection Moulding, V Piotter, M Tueluemen, A Klein, R Oberacker and B Ehreiser, as presented at Euro PM2017 Congress and Exhibition, Milan, Italy, October 1-5, 2017, and published in the Conference Proceedings by the European Powder Metallurgy Association (EPMA).

[5] Development and characterisation of rich-alumina porcelain parts processed by CIM, G Larraz Nogués, A Gallego Bravo, C Berges Serrano and G Herranz Sánchez-Cosgalla, as presented at Euro PM2017 Congress and Exhibition, Milan, Italy, October 1-5, 2017, and published in the Conference Proceedings by the European Powder Metallurgy Association (EPMA).

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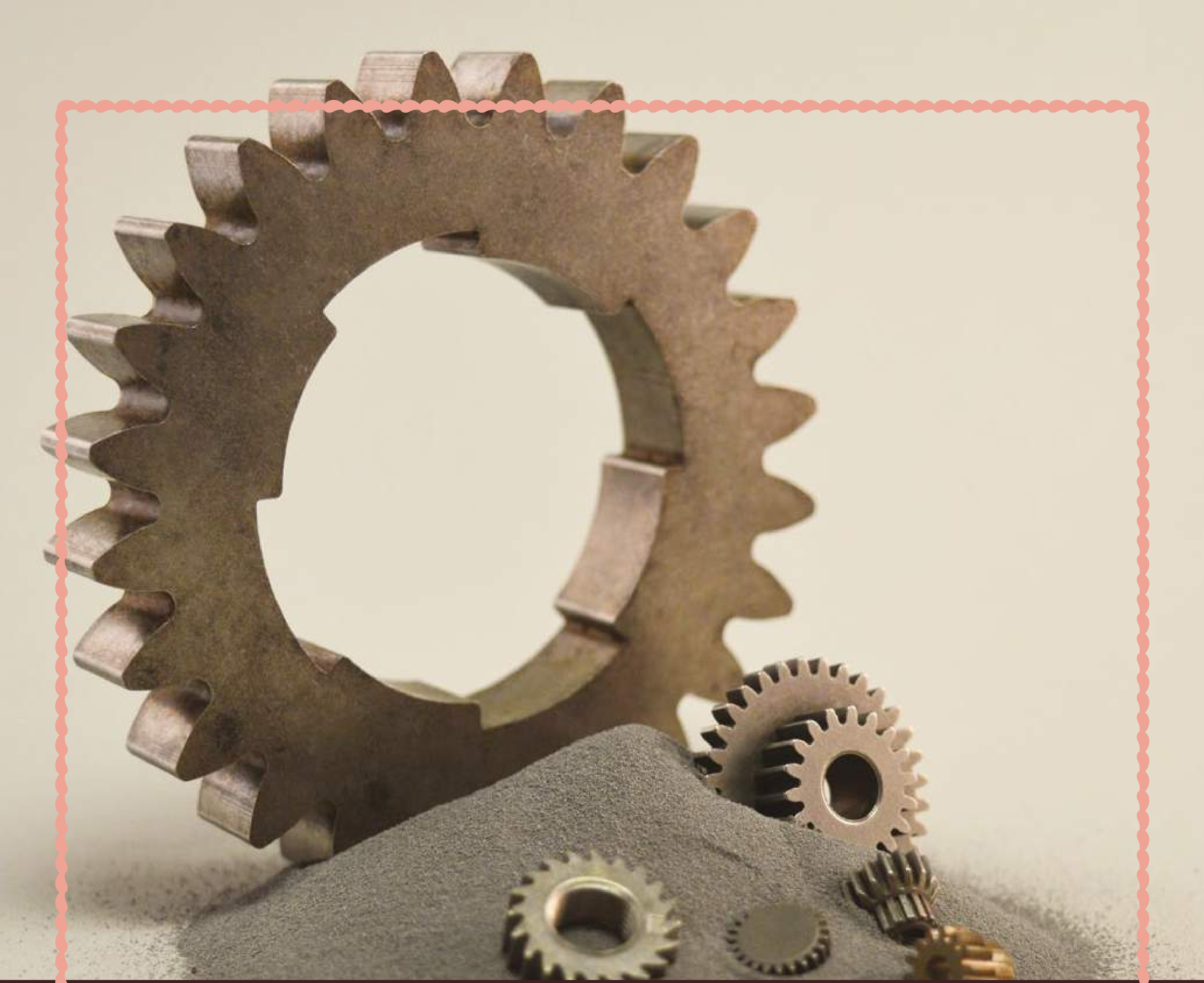


Advances in the PIM of Ti-6Al-7Nb and Ti-6Al-4V for biomedical and load bearing applications

A technical session at the Euro PM2017 congress and exhibition, held in Milan, October 1-5 2017, addressed the processing of biocompatible materials by Powder Injection Moulding. This article reviews three papers from this session that focus on the processing and fatigue behaviour of Ti-6Al-7Nb and Ti-6Al-4V alloys, as well as an investigation into the production of a gas tight platinum-alumina-Ti-6Al-4V feedthrough for implantable medical devices by Powder Injection Moulding

The effects of oxygen on the fatigue behaviour of MIM Ti-6Al-7Nb

Ti-6Al-7Nb is a suitable alloy for load bearing medical applications thanks to its biocompatibility, mechanical properties and corrosion resistance. It has been developed as a preferred alternative to Ti-6Al-4V in biomedical applications because of concerns over the toxic properties of vanadium. A paper by Alexandra Amherd Hidalgo *et al* assessed the effects of oxygen content on the fatigue behaviour of the MIM Ti-6Al-7Nb alloy [1].

It is well known that oxygen plays a critical role in the mechanical properties of titanium alloys. While oxygen acts as a potent strengthener, it has been reported that the ductility in α/β titanium alloys processed by MIM is maintained up to a certain level of oxygen (~0.38 wt.%), where an extreme drop occurs. However, much uncertainty still exists regarding the influence of oxygen on dynamic properties. A full study was deemed necessary to understand the influence of oxygen on the static and dynamic properties of α/β Ti alloys.

The gas atomised Ti-6Al-7Nb powder used in the reported study was produced from a commercially rolled rod material by the Electrode Induction Melting Gas Atomisation (EIGA) process at Helmholtz-Zentrum Geesthacht, Germany. A size fraction sieved below 45 µm was used. The interstitial contents, measured by melt extraction, were 0.21 wt.% oxygen, 0.004 wt.% nitrogen and 0.007 wt.% carbon.

Fig. 1 The Euro PM2017 congress and exhibition was held in Milan, Italy, and attracted over a thousand participants (Courtesy EPMA)

Process	Treatment time [min]	Oxygen content [wt.%]	Colony size [µm]
MIM	No treatment	0.26	137 ± 8
	1	0.43	137 ± 1
	180	0.45	136 ± 3
MIM + HIP	No treatment	0.26	136 ± 4
	180	0.47	134 ± 9

Table 1 Colony size of MIM and MIM + HIP specimens with different oxygen contents. Each value is the average value of three measurements. The oxygen content error is less than 0.01 wt.% [1]

Feedstock was produced in a Z-blade mixer at 120°C for 2 h. Powder was mixed with 35.5 vol.% of a multicomponent binder consisting of ethylene vinyl acetate (EVA), paraffin wax and stearic acid. Extrusion was carried out to achieve feedstock homogenisation. Injection moulding was performed on an Arburg 320S injection moulding machine, with four-point bending fatigue specimens being produced. Debinding was undertaken in two stages, chemical debinding in a hexane bath at 40°C for 15 h, followed by thermal debinding in the sintering furnace at 450°C under 5 mbar argon flow at 150 l/h.

Specimens were treated in order to have different oxygen contents. Initially, specimens were pre-sintered, then thermally treated under an oxygen-argon atmosphere and finally sintered. The pre-sintering step was performed after the thermal debinding process, in a single thermal cycle, in a cold-wall furnace with Mo-shielding and tungsten heater, at 800°C under high vacuum for 1 hour using molybdenum supports with yttria coating.

During the thermal treatment, two groups of specimens were exposed to an oxygen (20%)-argon atmosphere of 1 bar with flowing gas continuously renewed at a rate of 35 l/h for 1 h and 180 min respectively, at a temperature of 400°C inside a hot-wall furnace. Finally, specimens were sintered in a cold-wall furnace at a temperature of 1350°C for 4 h under high vacuum, again using molybdenum supports with yttria coating. Some specimens were subjected to sintering only, as reference specimens to obtain low oxygen specimens. Several low oxygen and 180 minutes treated specimens were subjected to Hot Isostatic Pressing (HIP) at 920°C at 1,000 bar for 120 min.

The MIM specimens contained 3% residual porosity, while the HIP specimens were fully dense. The oxygen contents of the specimens are reported in Table 1. Higher

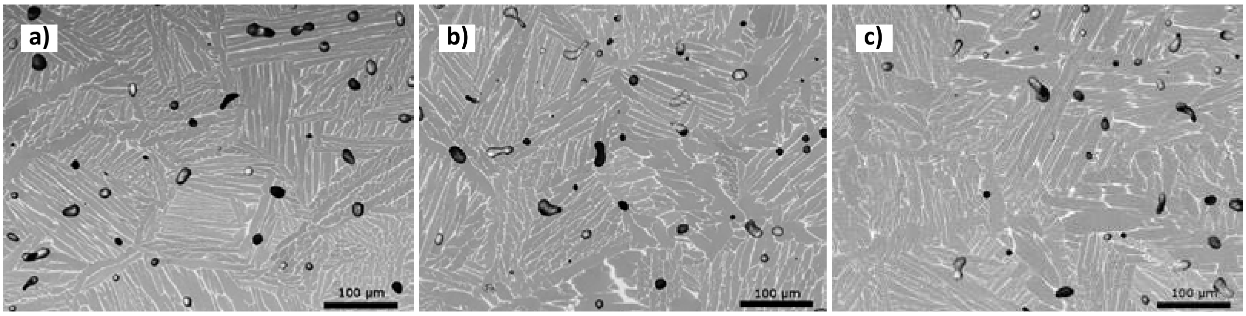


Fig. 2 MIM specimens containing (a) 0.26 wt.%, (b) 0.43 wt.% and (c) 0.45 wt.% of oxygen [1]

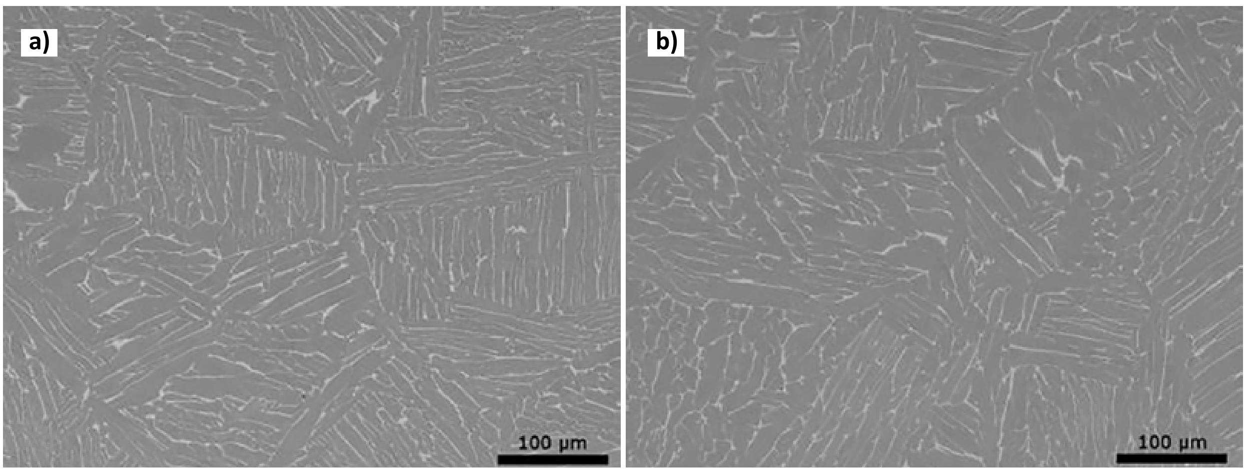


Fig. 3 MIM + HIP specimens containing (a) 0.26 wt.% and (b) 0.47 wt.% of oxygen [1]

oxygen content is acquired when the specimens are exposed longer under an oxygen-argon atmosphere. However, the oxygen increase is not linear with increasing exposure time.

The microstructures of the specimens are shown in Figs. 2 and 3. MIM specimens showed a maximum pore size of around 50 µm. A lamellar colony structure was formed inside the prior β grains and the average diameters of these lamellar colonies are shown in Table 1. No significant variation of colony size was observed due to the increase of oxygen. However, α lamella width was slightly enlarged. It was stated that oxygen, as α stabilising element, increases the β transus temperature. Then, the precipitation of α phase takes place at higher temperatures, leading to coarser α lamellae and a higher amount of α phase.

After shot peening of the specimens, high cycle fatigue tests were undertaken in a four-point bending configuration under air at room temperature using a resonance machine. The experiments were performed under load control at R=σmin/σmax of 0.2 with a cyclic frequency of ~95 Hz (sine wave). The maximum number of cycles was set at 10^7 cycles in presenting the fatigue life data.

Fig. 4 shows the S-N curves for MIM specimens containing different oxygen contents. In general, the specimens do not present a well-defined fatigue limit. As-sintered specimens with low oxygen content show 425 MPa fatigue strength at 10^7 cycles. The variation in oxygen content from 0.26 wt.% to 0.43 wt.% caused a slight decrease in fatigue resistance. Interestingly, the variation from 0.43 wt.% to 0.45 wt.% oxygen showed a more significant reduction. This fact could probably be related to the alloy's ductility drop, reported at oxygen contents over 0.38 wt.%. However, the alloy with 0.45 wt.% oxygen content still delivered 350 MPa fatigue strength at 10^7 cycles. The observed reduction of fatigue properties on increasing oxygen content could be attributed to wider alpha lamellae.

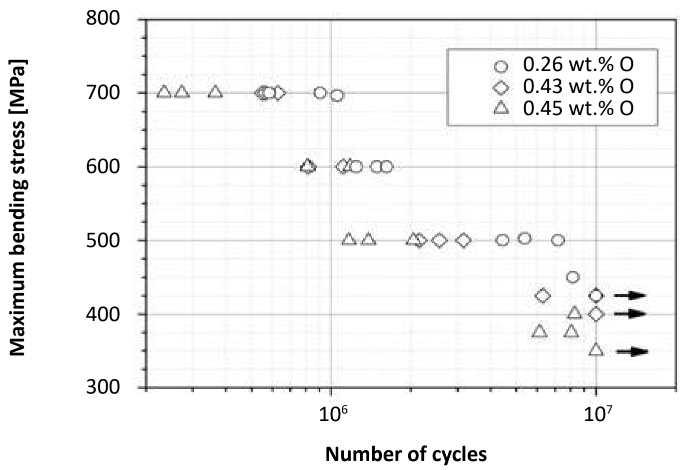


Fig. 4 Influence of oxygen on the S-N curve of MIM Ti-6Al-7Nb [1]

SEM assessment of the fracture surfaces of specimens subjected to 700 MPa bending stress showed a crack initiation point located in a sub-surface level where the surface was exposed to tensile stress during the four-point bending fatigue test. It was observed that crack propagation of specimens containing different oxygen contents had different surface topology. At low oxygen contents, crack propagation was smooth, while, at high oxygen contents, faceted fracture was observed.

In order to evaluate the impact of residual porosity on the fatigue behaviour of Ti-6Al-7Nb, the graphs in Fig. 5 compare the S-N curves of specimens processed by MIM

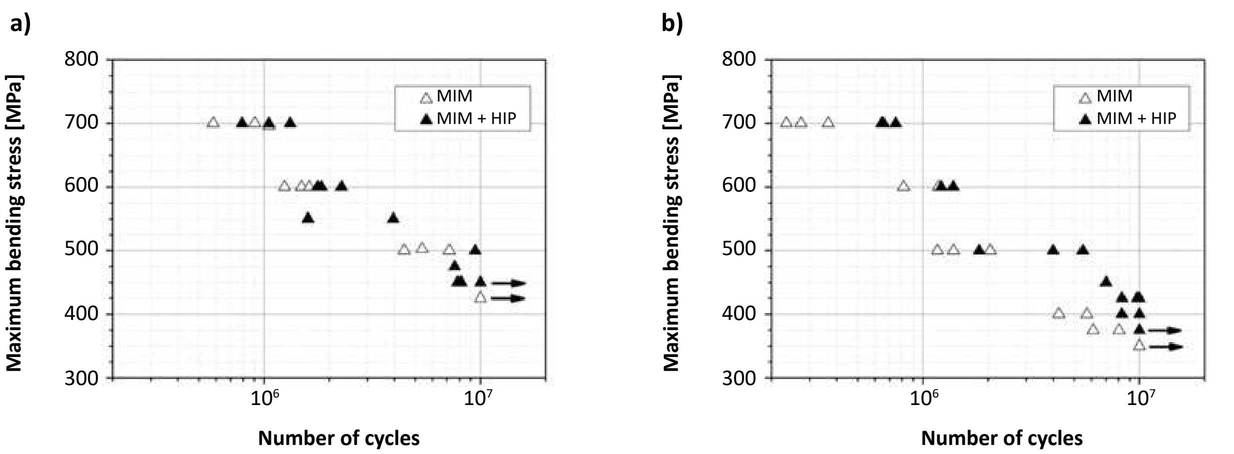
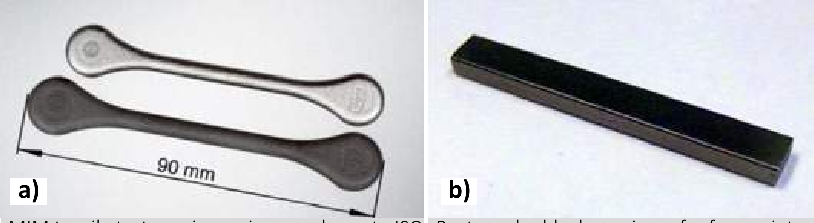


Fig. 5 Influence of the typical residual porosity of MIM specimens on the S-N curve of Ti-6Al-7Nb at (a) 0.26 wt.% and (b) around 0.45 wt.% of oxygen content [1]



MIM tensile test specimens in accordance to ISO 2740 (sintered and green part)

Rectangular block specimen for four point bending fatigue test

Fig. 6 MIM produced specimens [2]

and by MIM + HIP. At low oxygen contents (Fig. 5a), the 3% of residual porosity after the MIM process had no significant impact on the fatigue behaviour. Moreover, if the oxygen level was increased to values around to 0.45 wt.% (Fig. 5b), the residual porosity reduced the fatigue strength at 10^7 cycles by just 25 MPa. Similar scattering of fatigue strength values was found in both un-HIPed and HIPed specimens.

SEM assessment of the fracture surfaces of the MIM + HIP specimens, containing low and high oxygen levels and subjected to 700 MPa bending stress, showed, in the absence of porosity, that there is a clear vision of the crack propagation zone, where the lamellar colonies could be distinguished. It appeared that crack propagation was along the lamellae. Similar to the fracture of the MIM specimens at high oxygen contents, the fractured surface of the lamellar colonies had a flat morphology, probably caused by the embrittlement of the α phase.

The overall conclusion drawn by the authors was that, in general, oxygen reduced the fatigue properties of the MIM specimens. The increase of oxygen from 0.26 wt.% to 0.43 wt.% reduced the fatigue strength at 10^7 cycles by just 25 MPa. However, the small variation of oxygen from 0.43 wt.% to 0.45 wt.% led to a reduction of 50 MPa. Although ductility at 0.45 wt.% oxygen content drops, the fatigue strength at 10^7 cycles was found to be at 350 MPa.

It was therefore proposed that further studies need to be carried out in order to evaluate the relationship between tensile and fatigue properties. Moreover, at oxygen content around 0.26 wt.%, the fatigue properties of MIM specimens are not improved by a subsequent HIP treatment, showing that porosity is not the most important factor in determining fatigue behaviour. The fatigue strength at 10^7 cycles of specimens containing around 0.45 wt.% of oxygen was improved by just 25 MPa after HIP.

Improving the fatigue properties of MIM Ti-6Al-4V by the addition of yttrium

Members of the Helmholtz-Zentrum group also presented a poster contribution that won the EPMA's Peter Brewin Best Poster Award at Euro PM2017. This paper, authored by Wolfgang Limberg *et al* focused on the enhancement of the fatigue properties of MIM-processed Ti-6Al-4V by adding yttrium [2].

A fine microstructure is a very important contributor to good mechanical properties at room temperature, both for statically loaded and dynamically loaded parts. MIM is a near net shape production process with sintering, as a heat treatment at high temperature, as the last integrated step. Therefore, the possibilities for microstructural refinement of MIM-processed α-β-titanium alloys such as Ti-6Al-4V are limited.

However, yttrium oxide is a strong colony refining agent. The colony size of MIM-processed Ti-6Al-4V can be reduced from 130 μm down to 50 μm by the addition of only 0.1 wt.% Y_2O_3. Also, an important parameter which influences the mechanical properties of titanium materials is the oxygen content.

When pure elementary yttrium is added to Ti-6Al-4V instead of Y_2O_3, the Y_2O_3 is formed in-situ during the sintering process. Oxygen is scavenged from the titanium matrix, increasing ductility, and the Y_2O_3 particles formed lead to colony refinement. The aim of the reported study, therefore, was to observe the kinetics of the solution of yttrium and

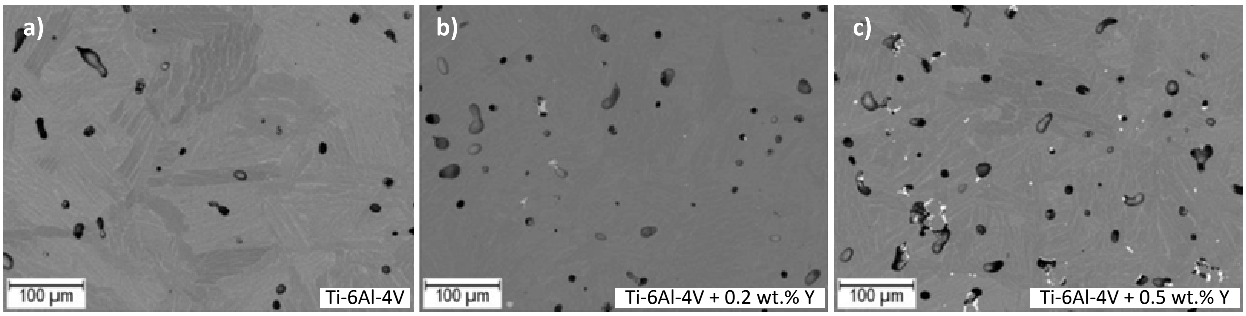


Fig. 7 SEM (BSE-mode) micrographs of MIM processed Ti-6Al-4V with different yttrium powder additions [2]

Specimen composition		YS [MPa]	UTS [MPa]	ε_f [%]	Porosity [%]	Oxygen content [µg/g]	Nitrogen content [µg/g]
Initial Powder						1600	200
Ti-6Al-4V		771	884	15.3	3.5	2356 ± 78	501 ± 104
Ti-6Al-4V + Y	0.2 wt.%	739	849	13.2	4.3	2380 ± 76	536 ± 24
	0.5 wt.%	692	794	12.5	5.0	2501 ± 94	590 ± 72

Table 2 Mechanical properties, porosity and impurity contents of MIM-processed Ti-6Al-4V [2]

formation of Y_2O_3 during sintering of Ti-6Al-4V and to investigate the influence of the yttrium addition on fatigue properties.

The Ti-6Al-4V alloy powder used for the reported experiments was a plasma atomised Grade 5 powder produced by AP&C. The powder fraction used had a particle diameter < 45 µm. The yttrium powder was a commercially available powder with a purity of > 99.9%. The particle shape was square-edged with a rough surface. A fine powder fraction < 32 µm was extracted from the powder by sieving. The initial oxygen content of the yttrium powder was determined as 0.9 wt.%. The binder system used for feedstock preparation was a mixture of ethylene vinyl acetate (EVA), paraffin wax (PW) and stearic acid (SA).

The entire powder handling was performed in a glove box under an argon atmosphere to protect the fine powder from further oxidation. The residual oxygen in the argon atmosphere was eliminated by a gas purifier, so that the oxygen content in the glove box could be kept below 1 ppm.

Three different feedstocks were produced, all containing 10 wt.% of the binder: pure Ti-6Al-4V, Ti-6Al-4V + 0.2 wt.% Y powder and Ti-6Al-4V + 0.5 wt.% Y powder.

For tensile tests, standard MIM specimens, according to ISO 2740 (Fig. 6a), were injection moulded. For fatigue tests in four-point bending mode, rectangular block specimens (Fig. 6b) with green part dimensions of 50 mm x 6.3 mm x 3.4 mm were injection moulded.

Fig. 7 shows SEM images (BSE-mode) of cross-sections of the sintered tensile test specimens of Ti-6Al-4V with and without the addition of yttrium powder. The micro-structure consists of lamellar α/β colonies. The α-lamellae appear dark grey, while the β-phase between the α-lamellae is visible as a light grey colour and the pores as black dots. EDX and X-ray diffraction experiments have shown that the white structures are Y_2O_3 particles, which were formed during the sintering process.

The pores in the pure Ti-6Al-4V and the Ti-6Al-4V with the addition of 0.2 wt.% yttrium were homogeneously distributed; only the specimens with the addition of 0.5 wt.% Y showed a few pore clusters with sizes up to 100 µm, partially filled with Y_2O_3. The residual porosities of the different specimens are listed in Table 2. The porosity increased with the addition of yttrium, in accordance with earlier investigations by this research group, where coarser yttrium powders were used.

The colony sizes of such alloys were assessed using EBSD-patterns, determining the average colony area $\bar{A}$ by image analysis and calculating the average mean colony diameter by taking the square root of $\bar{A}$. Fig. 8 shows EBSD-patterns of the cross-sections of MIM-processed Ti-6Al-4V and Ti-6Al-4V with 0.5 wt.% yttrium powder addition, respectively. The differently coloured areas are α/β colonies with different orientations. The two images at the outside (a1 and b2) showed the differently oriented α-colonies of the Ti-6Al-4V specimen. The initial colony size was measured as 125 µm on average. A decrease in average colony size from 125 to 90 µm by adding 0.5 wt.% yttrium was found.

The two images in the centre of Fig. 8 (a2 and b1) show regions of differently oriented β-phase between the α-lamellae. The β-phase images show the same regions

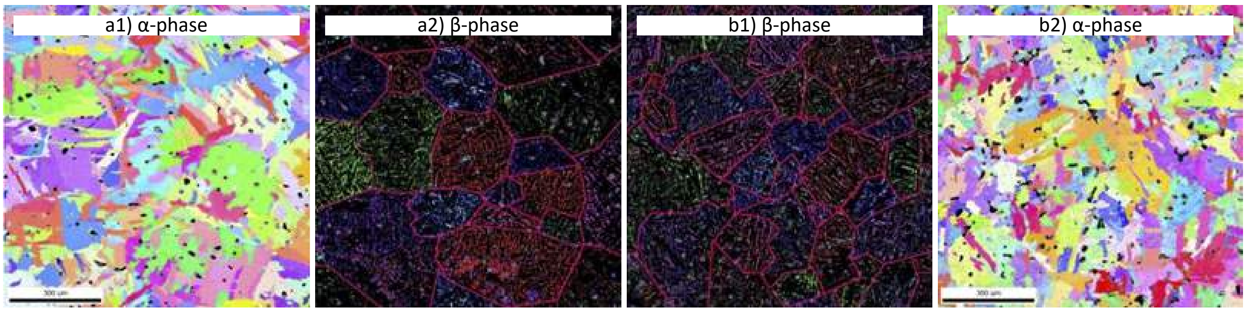


Fig. 8 EBSD-patterns of MIM processed (a) Ti-6Al-4V and (b) Ti-6Al-4V with 0.5 wt.% yttrium [2]

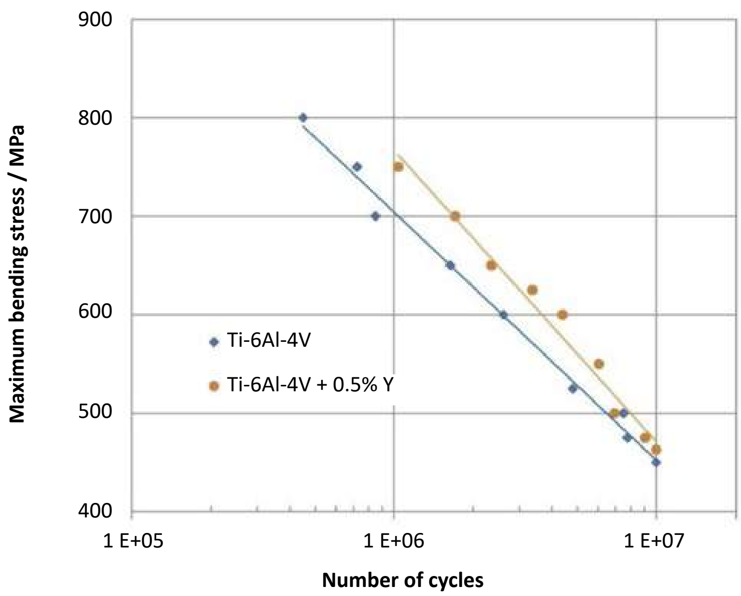


Fig. 9 Fatigue properties of MIM-Ti-6Al-4V and MIM-Ti-6Al-4V with an addition of 0.5 wt.% yttrium (R = 0.2) [2]

as the α-phase images. Since the α-lamellae grow into the β-grains during cooling after sintering, the β-phase images in Fig. 8 represent the size of the prior β-grains. The addition of yttrium powder hinders the growth of β-grains during the sintering of Ti-6Al-4V. The prior β-grain size of the Ti-6Al-4V samples was determined as 235 µm. Through the addition of 0.5 wt.% yttrium, the prior β-grain size decreases to 175 µm.

The decrease in colony size of Ti-6Al-4V through the addition of 0.5 wt.% yttrium is 28%, the same difference as between the prior β-grain sizes. This leads to the conclusion that colony refinement is mainly caused by the hindering of β-grain growth and not by the heterogeneous nucleation of α-lamellae.

The concentrations of oxygen and nitrogen in the alloy powder and in the MIM-manufactured specimens after sintering were analysed. The impurity levels of the various MIM specimens are listed in Table 2. While the alloy powder had an initial oxygen content of 1600 µg/g, this increased to 2356 µg/g during sintering at 1400°C. Earlier work by this group demonstrated that, under optimised conditions, the oxygen increase during MIM-processing can be limited to 350 µg/g. This reported higher oxygen pick-up could be one reason for the small colony sizes and high porosities.

Compared to the sintered yttrium-free specimen, a further increase of 24 µg/g of oxygen for 0.2% and 145 µg/g of oxygen for 0.5% yttrium is found in the yttrium-containing sintered specimens. Based on these results, it is possible to estimate the effectiveness of the oxygen scavenging effect. Based on the stoichiometry of Y_2O_3, the 0.5 wt.% yttrium added to the alloy needs 0.135 wt.% oxygen to fully transform to Y_2O_3. This results in 1350 µg/g bound to yttrium in the form of oxide. Only a minor part of this oxygen was introduced into the alloy mixture with the yttrium powder (145 µg/g for 0.5% yttrium) and, therefore, nearly 90% of the oxygen now bound as oxide was scavenged from the titanium matrix by the yttrium.

Tensile tests, according to ISO 6892-1, were conducted in air at room temperature. The gauge length was 30 mm and the gauge diameter 4.3 mm. The strain was measured by a laser extensometer. All tensile test specimens were tested in the as-sintered condition.

The results of the tensile tests are listed in Table 2. The MIM-processed specimens of pure Ti-6Al-4V, sintered at 1400°C, showed a yield strength (YS) of 771 MPa, an ultimate tensile strength (UTS) of 884 MPa and a plastic elongation (ε_f) of 15.3%. The addition of yttrium leads to a decrease of UTS, YS and ε_f. This decrease in strength could be caused by a reduced oxygen content in the titanium matrix due to the scavenging effect of yttrium, but also, at least partially, by the increase of porosity. For an increase in porosity of 1.5%, a decrease in ultimate tensile strength of approximately 35 MPa has been observed in previously reported work. This means that, of the decrease of 90 MPa by the addition of 0.5 wt.% yttrium to Ti-6Al-4V, 35 MPa was caused by the increased porosity and 55 MPa was caused by the oxygen reduction due to the scavenging effect.

Prior to fatigue testing, the rectangular block specimens were surface treated by shot peening to minimise the influence of surface defects. The fatigue tests were conducted in air at room temperature on a resonance testing machine, with a cyclic frequency of 95 Hz at a load ratio R = σmin/σmax of 0.2.

The results of the fatigue tests are shown in Fig. 9. For pure Ti-6Al-4V, the fatigue strength at 10^7 cycles is 450 MPa. In contrast to the tensile test results, the addition of 0.5 wt.% yttrium increases the fatigue strength at 10^7 cycles from 450 to 470 MPa. At lower numbers of load cycles (10^6), the fatigue strength increases by a greater degree, from 705 to 765 MPa. So, the colony size seems to have more influence on the fatigue behaviour than the porosity or the oxygen content, at least at oxygen values which do not lead to embrittlement.

For in situ diffraction studies, cylindrical specimens were prepared out of the gauge regions of tensile test samples. Specimens were pre-sintered at a temperature of 850°C for 30 min, to achieve a sufficient mechanical stability for further handling. The porosities of the pre-sintered specimens were between 25 and 30%. Only the pure Ti-6Al-4V and the Ti-6Al-4V with the addition of 0.5 wt.% Y powder were investigated by synchrotron X-ray diffraction.

The X-ray diffraction experiments were carried out at the German Electron Synchrotron facility. Sintering was performed in a vacuum of 2 x 10^{-5} mbar. During heating to sintering temperature, diffraction patterns were recorded every 10 sec on a flat panel detector with an exposure time of 0.1 sec. Diffraction diagrams were generated from the detector images.

Fig. 10 shows the diffraction diagrams of Ti-6Al-4V with the addition of 0.5 wt.% yttrium, taken at different temperatures during heating up. With increasing temperature, all peaks shift to lower values of 2Θ, this effect being caused by the thermal expansion of the lattice. The intensity of the main Y_2O_3 peak at 2Θ = 2.3° shows a strong increase between 800 and 900°C. Thus, in this temperature range, the formation of Y_2O_3 accelerates.

Fig. 11 shows the 2Θ-region between 2.2 and 2.7°, where the peaks for yttrium and Y_2O_3 with the highest intensity (labelled in red) are located. A small amount of yttrium is already oxidised during or before pre-sintering. The main yttrium-peak at 2Θ = 2.5° disappears between 1000 and 1025°C, indicating that all yttrium is dissolved in the titanium matrix or oxidised and elemental yttrium is no longer present at 1025°C.

In the same temperature range, the main Y_2O_3 peak at 2Θ = 2.3° reaches its highest intensity and then remains constant up to 1300°C. This means that there is no further oxidation of yttrium above 1025°C and only a small amount of yttrium was dissolved before oxidation.

No formation of yttrium titanates, such as $Y_2Ti_2O_7$ or Y_2TiO_5, was observed. The authors proposed that this should be part of further investigations, using transmission electron microscopy of the interface between Y_2O_3 particles and the Ti-6Al-4V matrix.

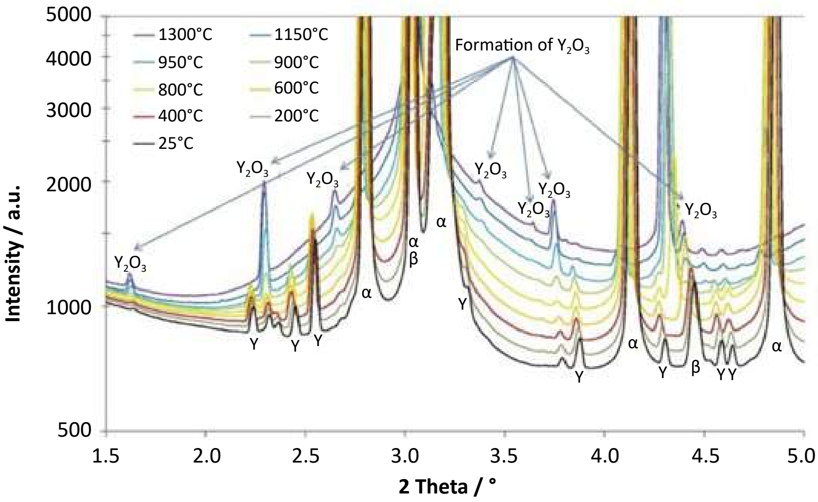


Fig. 10 X-Ray diffraction diagrams of Ti-6Al-4V with an addition of 0.5 wt.% yttrium at different temperatures [2]

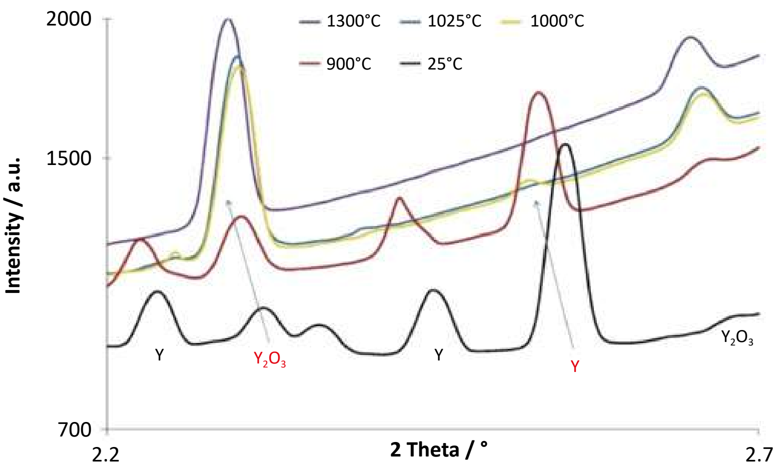


Fig. 11 2Θ-region between 2.2 and 2.7° of X-Ray diffraction diagrams of MIM-Ti-6Al-4V with an addition of 0.5 wt.% yttrium [2]

A gas tight platinum-alumina-Ti-6Al-4V feedthrough for implantable medical devices without brazing

Finally, D Vincent *et al* (Universite Grenoble Alpes, France) reported on the development of a PIM based manufacturing process that avoids brazing in the production of gas tight, platinum-alumina-Ti-6Al-4V feedthroughs for implantable medical devices [3].

Implantable medical devices such as cardiac defibrillators, cochlear implants and neuromuscular micro stimulators are widely used to restore body functions, improve quality of life and save lives. For safe and sustainable implantation in the human body without the release of toxic products and device degradation by body fluids, a hermetic housing is required. A hermetic electrical feedthrough is essential for a safe and functionally active implantable biomedical device and needs to be as small as possible for patient comfort and safety reasons.

a) Mono-pin circular feedthrough **b) Racetrack shape multi-pin feedthrough**

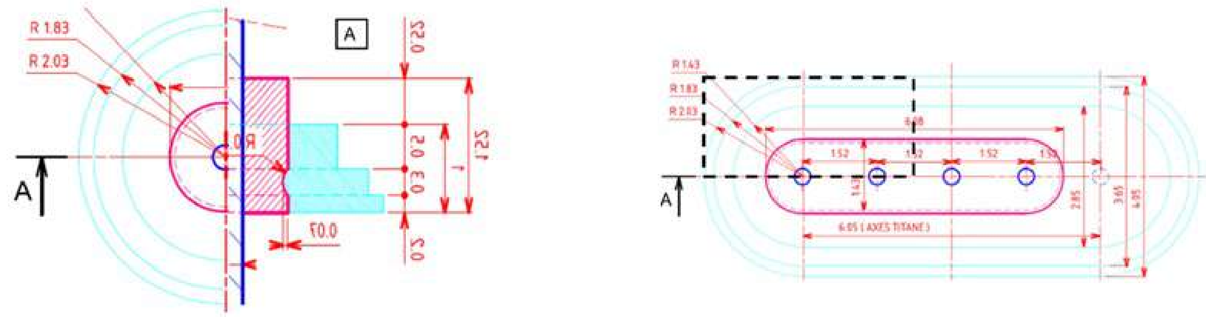


Fig. 12 Feedthroughs investigated by the PIM process [3]

Metals for the packaging and ceramics for the insulating material are the most common materials used to make a hermetic feedthrough. To bond metal and ceramic materials, brazing is to date the most widely used technique when a mechanically reliable gas-tight bonding is required. However, for implantable devices, a high content of expensive noble metals, for example gold, is often added to the brazing paste. The most commonly used ceramic/metal feedthrough is made of pure platinum pins surrounded by alumina. This kind of complex multi-material component fabrication by conventional processes needs complex and expensive steps due to the dimensional accuracy required in each part and the brazing process (insulator and ring machining, metallisation for brazing, paste brazing elaboration, brazing, etc.).

Powder Injection Moulding can open new opportunities to manufacture hermetic feedthroughs between metal electrodes, ceramic insulator and metal ring for packaging welding. The process allows the manufacture of complex multi-material and gas tight components with a reduced number of production steps. Also, it allows an appropriate choice of materials and treatment conditions to bond ceramic and metal without brazing. The reported study was aimed at exploring the potential of PIM to create a hermetic feedthrough without the use of brazing.

Pure platinum wires, 0.33 mm in diameter, were cut to obtain 25 mm length pins. The pins were then ultrasonically cleaned in an ethanol/acetone bath. An alumina feedstock, a mixture of ceramic powder and binder, was supplied with 99.8% purity. The Ti-6Al-4V feedstock used was prepared with 32 vol.%. of binder. The binder was a mixture of polyethylene, paraffin and stearic acid. The Ti-6Al-4V powder was produced by argon gas atomisation with 0.012% carbon, 0.15% oxygen and 0.09% nitrogen as respective interstitial impurity levels.

Two feedthrough designs were investigated; a circular feedthrough (Fig. 12a) and a multi-pin feedthrough with a racetrack shape (MPFR), (Fig. 12b). The PIM feedthrough process steps consisted of:

1. Over-moulding of pins by alumina feedstock
2. Debinding and sintering of alumina at 1600°C under air
3. Sintered alumina over-moulded by Ti-6Al-4V feedstock
4. Debinding and sintering of Ti-6Al-4V. Two sintering treatments were carried out at 1250°C for 2 h under argon and vacuum.

The various process steps are illustrated in Fig. 13.

At the end of the process, the feedthroughs were polished to obtain a low roughness surface in order to characterise both interfaces: insulator-pin and insulator-Ti-6Al-4V ring and sintered Ti-6Al-4V ring. Each interface was observed by scanning electron microscopy (SEM) with an energy dispersive X-ray detector. Interface defects, diffusion profiles and sintered Ti-6Al-4V microstruc-

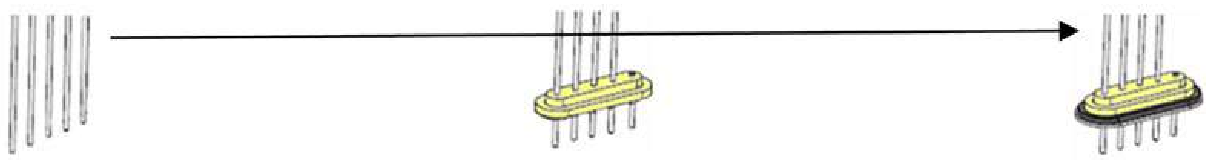


Fig. 13 PIM process steps for feedthrough manufacturing [3]

ture were analysed to define the impact of treatment conditions on the interface quality and the gas tightness level.

The mechanical properties of sintered Ti-6Al-4V were measured by hardness testing. For each sample, Vickers hardness near and far from the alumina interface was measured with a 20 kg load. The mechanical interface resistance was tested in a tensile test, for the pin/alumina interface, and, by a compressive test, for the alumina/Ti-6Al-4V interface. Tests were carried out only on the circular feedthrough.

Sealing measurement was performed with a helium detector. The feedthrough was positioned on a metallic pad connected to a vacuum system and a helium detector. A Viton seal ensured the tightness between the metallic part and the feedthrough. When the vacuum was reached, helium was sprayed on the feedthrough and the leakage rate was measured by the helium detector.

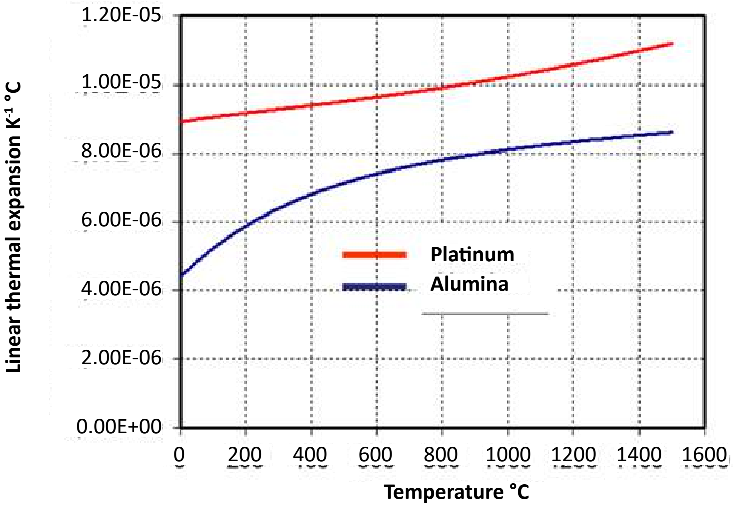


Fig. 14 Linear thermal expansion of platinum and alumina versus temperature [3]

The platinum pin-alumina interface

Considering firstly the platinum pin-alumina interface, dilatometer measurements (reported in Fig. 14) indicate that platinum thermal expansion is higher (11×10^{-6} °C^{-1}) than that of alumina (9×10^{-6} °C^{-1}) at 1400°C. Thus, after alumina sintering without interaction between both materials, a delamination will appear during cooling.

The SEM observations, illustrated in Fig. 15a and Fig. 15b, showed the interface between platinum pin and alumina. Near the platinum pin, no cracks in the alumina or delaminations are observed. These results show that the alumina shrinkage around the pin during sintering does not create any defect by a physico-chemical interaction between alumina and platinum.

Alumina/platinum direct bonding has already been reported in the literature. The bonding quality depends on surface interactions, such as Van Der Waals adhesion and the chemical bonding between atom surfaces. Several factors can impact the bonding strength, namely bonding atmosphere, surface impurity with the formation of glass phase in the alumina surface and grain orientation.

To assess mechanical interface resistance, tensile tests were performed on the mono-pin circular feedthrough. For each test, the rupture occurred consistently at 100 N in the platinum pin and never at the platinum/alumina interface. These results highlight the good mechanical resistance of the Pt/alumina interface. According to the sample geometry and rupture load, shear stress can be calculated and is higher than 40 MPa.

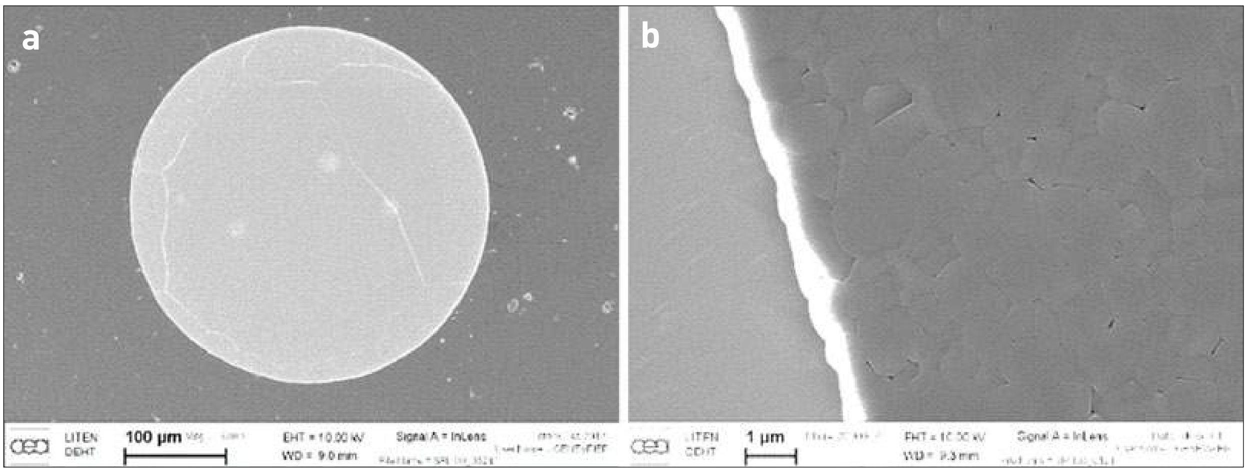


Fig. 15 Pin - alumina interface observed by SEM [3]

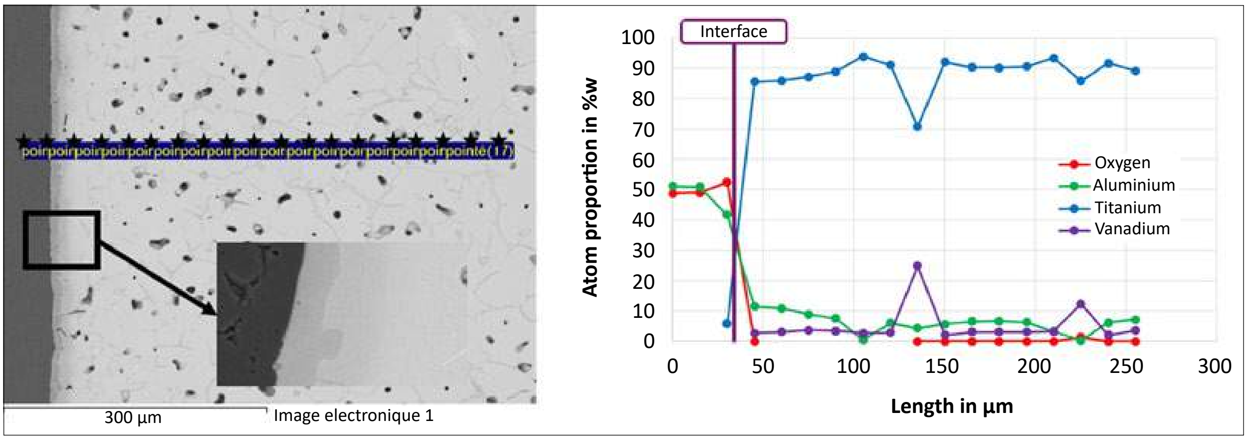


Fig. 16 Micrograph of a sintered Ti-6Al-4V ring with EDX profile of elemental diffusion at the alumina-Ti-6Al-4V interface [3]

After alumina sintering, the leakage rate measurement on the Pt pin - alumina interface was lower than 1 x 10^{-9} mbar. To simulate the second thermal treatment, pin - alumina parts were heated at 1250°C under vacuum or argon. The leakage rate was also found to be below 1 x 10^{-9} mbar. The same gas tightness measurement was also performed on parts used for tensile tests with the same results. All results show that the bonding between platinum pin and alumina is strong enough to obtain a hermetic interface fulfilling the feedthrough requirements.

The alumina/Ti-6Al-4V interface

Turning next to the alumina/ Ti-6Al-4V interface, SEM observations in back scattered electron (BSE) mode of sintered Ti-6Al-4V rings were carried out to check the densification of the Ti-6Al-4V at several positions. The porosity was measured by BSE image analysis. 2.8% of porosity is estimated on parts sintered at 1250°C, which is high enough to ensure the tightness in Ti-6Al-4V.

At the interface between alumina and Ti-6Al-4V shown in the Fig. 16, two different phases were identified, a first 5 µm thick phase in dark grey in direct contact with alumina and a second 5-10 µm thick phase in clear grey.

EDX analysis profiles were derived at the alumina/Ti-6Al-4V interface and are reported in Fig. 16. They show an aluminium content in the Ti-6Al-4V higher in the region close to the interface (0-50 µm) than in the Ti-6Al-4V raw material (10% wt.% versus 6% wt.%). Beyond 50 µm, the Al content decreases slowly to 6%.

These observations and analysis are in agreement with information in the published literature. It has been highlighted that Ti-6Al-4V can reduce alumina at high temperature and enhance aluminium and oxygen diffusion in Ti-6Al-4V. The aluminium diffusion leads to the formation of intermetallic phases such as TiAl and Ti_3Al. For the feedthrough application, it is preferable to avoid the formation of these phases because they are hard and brittle and can lead to an abrupt fracture under stress.

By EDX analysis, it is difficult to measure the oxygen diffusion, but several authors have described the impact of oxygen on Ti-6Al-4V mechanical properties. An increase in oxygen content in Ti-6Al-4V leads to hardening with a strong diminution of elongation to fracture.

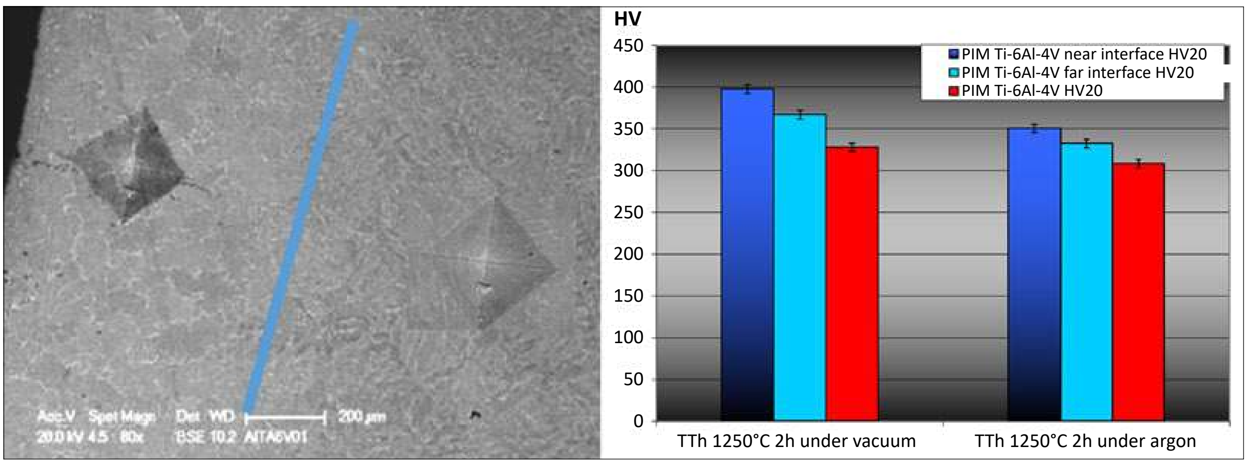


Fig. 17 Ti-6Al-4V microstructure close to and far from the alumina insert; hardness measured in Ti-6Al-4V [3]

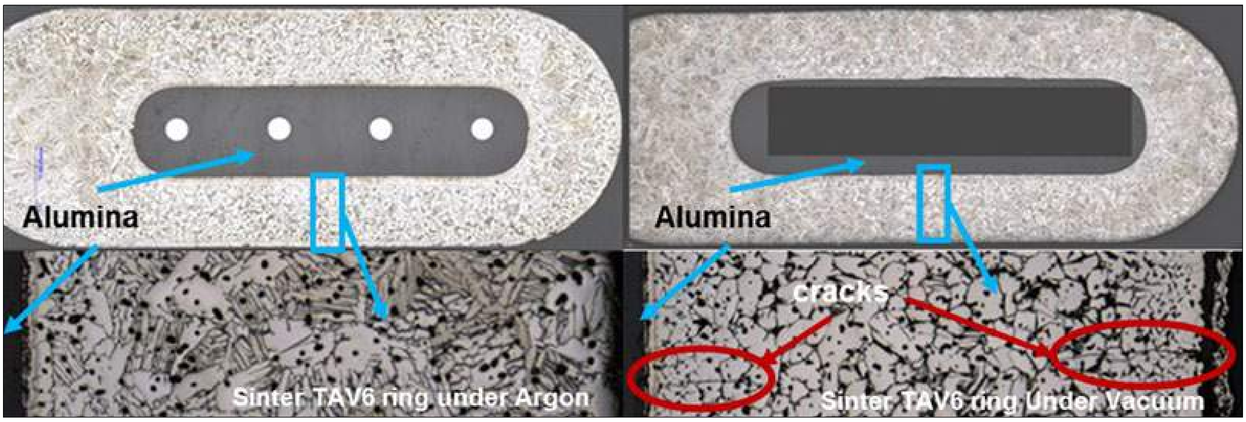


Fig. 18 Microstructure of MPFR sintered under argon and under vacuum [3]

The oxygen diffusion must therefore be limited to avoid a high degree of Ti-6Al-4V embrittlement.

The microstructure of sintered Ti-6Al-4V under vacuum is shown in Fig. 17 (left). The microstructure was observed close to the alumina/Ti-6Al-4V interface, where the grain morphology is equiaxed, and at 400 µm from the interface, where the grain shape is lamellar as is normal for sintered Ti-6Al-4V. The histogram, presented in Fig. 17 (right), shows the hardness measured in both microstructures for a Ti-6Al-4V ring sintered under vacuum and argon. The results show that, whatever the treatment carried out, the Ti-6Al-4V hardness is higher near the alumina/Ti-6Al-4V interface. The sintering atmosphere makes the Ti-6Al-4V harder under vacuum than under argon. In the ring sintered under vacuum, failures are observed from the Vickers indentation near the alumina/Ti-6Al-4V interface, but not from the indentation further into the alumina.

These results show that, during the Ti-6Al-4V ring sintering, titanium reduces alumina, leading to aluminium and oxygen diffusion. Aluminium and oxygen diffusion modifies the Ti-6Al-4V's mechanical properties, through hardening and embrittlement.

The leakage measurements, performed on circular feedthroughs, show that, for parts sintered under argon or vacuum, the rate of leakage is lower than 1 x 10^{-9} mbar.

Multi-pin feedthroughs

Finally, considering multi-pin feedthroughs, these were produced with the same process as the mono-pin circular feedthroughs. Four platinum pins were over-moulded by an alumina feedstock. After alumina sintering, alumina was over-moulded by Ti-6Al-4V feedstock and sintered (under vacuum or argon).

For each feedthrough, the Ti-6Al-4V ring sintered under argon, the Ti-6Al-4V ring sintered under vacuum and the microstructures were observed by SEM (Fig. 18). Under argon, the Ti-6Al-4V microstructure presents a mix of lattice grains and an equiaxed grain structure. On sintering under vacuum, the lattice structure is almost totally absent. Under vacuum, cracks are visible in the middle of Ti-6Al-4V ring; under argon, no cracks are observed.

As the thermal expansion of Ti-6Al-4V is higher than that of alumina, the racetrack design induces more tensile stress in the Ti-6Al-4V ring during cooling than in the circular design. Under vacuum, Ti-6Al-4V embrittlement is observed and the tensile stress leads to the crack formation. Under argon, the Ti-6Al-4V embrittlement is less and the mechanical resistance is sufficient to avoid failure.

The last step in producing a complex feedthrough by PIM is the welding of the Ti-6Al-4V ring onto a Ti-6Al-4V plate. This welding was carried out by µTIG (µ Tungsten Inert Gas) under argon. Fig. 19 shows an example of a MPFR welded onto a titanium T40 plate. The SEM observations do not show any failure and the gas tightness was found to be below 1 x 10^{-9} mbar.

The authors concluded by stating that characterisation carried out on the circular feedthrough has shown that despite any atomic interdiffusion, the bonding after alumina sintering at high temperature between platinum and alumina is mechanically strong and allows interfaces with good gas

"...the bonding after alumina sintering at high temperature between platinum and alumina is mechanically strong and allows interfaces with good gas tightness to be obtained."

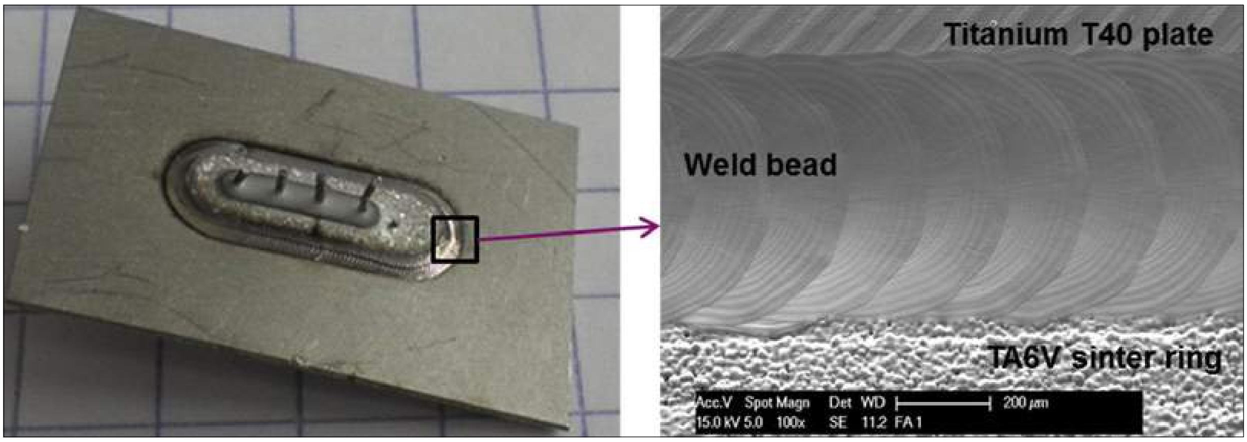


Fig. 19 MPFR with a racetrack shape weld on Ti-6Al-4V T40 plate [3]

tightness to be obtained. A very low leakage rate was measured, lower than 1 x 10^{-9} mbar; and, during Ti-6Al-4V sintering, titanium reduces alumina. The aluminium and oxygen diffusion into Ti-6Al-4V induces microstructural and mechanical modifications. The sintered Ti-6Al-4V near to the alumina interface is harder and has an equiaxed grain structure. The interface tightness is measured with a leakage rate less than 1 x 10^{-9} mbar.

Characterisation carried out on a racetrack feedthrough has confirmed the circular feedthrough results, but shown that, if the design induces mechanical stress, cracks can occur. To avoid critical cracks, it is necessary to control heat treatment conditions to limit aluminium and oxygen diffusion into Ti-6Al-4V. In this reported study, the sintering atmosphere is a critical parameter.

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