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## INTERNATIONAL



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Additive Manufacturing of ceramics**

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## Publisher & editorial offices

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

## Managing Director and Editor

Nick Williams  
Tel: +44 (0)1743 454991  
Email: nick@inovar-communications.com

## Publishing Director

Paul Whittaker  
Tel: +44 (0)1743 454992  
Email: paul@inovar-communications.com

## Consulting Editors

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

Dr Yoshiyuki Kato  
*Kato Professional Engineer Office, Yokohama, Japan*  
Professor Dr Frank Petzoldt  
*Deputy Director, Fraunhofer IFAM, Bremen, Germany*

Dr David Whittaker  
*DWA Consulting, Wolverhampton, UK*

Bernard Williams  
*Consultant, Shrewsbury, UK*

## Production

Hugo Ribeiro, Production Manager  
Tel: +44 (0)1743 454990  
Email: hugo@inovar-communications.com

## Advertising

Jon Craxford, Advertising Director  
Tel: +44 (0) 207 1939 749, Fax: +44 (0) 1743 469 909  
E-mail: jon@inovar-communications.com

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For the metal, ceramic and carbide injection moulding industries

## A bright future for Ceramic Injection Moulding

In the world of product design and engineering, the Metal Injection Moulding (MIM) industry still has a long way to go in order to achieve high levels of awareness amongst designers and engineers. The situation is in some respects even more challenging for Ceramic Injection Moulding (CIM), where there is not only a limited awareness of the process itself, but also a more general misunderstanding of the properties and capabilities of technical ceramics.

With this issue of *PIM International*, which will be distributed widely at Ceramitec, the leading international ceramics exhibition held in Munich, Germany, October 20-23, we hope to make a small contribution towards addressing this issue. For those who are new to Ceramic Injection Moulding, the technology is by no means new. Early versions of the process date back to the 1930s and 1940s, however since the late 1970s technical advances have enabled a much wider use of the technology.

Today CIM applications can be found in the automotive, aerospace, 3C, medical and dental sectors, to name just a few. Wherever high precision, complex shaped components are required in high volumes and with unique properties such as high wear, corrosion or heat resistance, CIM products can be used.

As we discover in this issue, the appeal of CIM has rapidly spread beyond industrial applications. CIM watch cases and components have become hugely popular with many of the world's finest watchmakers thanks to the material's high strength, toughness and hardness combined with the precision and net-shape abilities of the CIM process. Additionally, the material's low thermal conductivity makes it very comfortable against the skin, and a high refractive index allows for beautiful surface finishes to be achieved.

Nick Williams  
Managing Editor

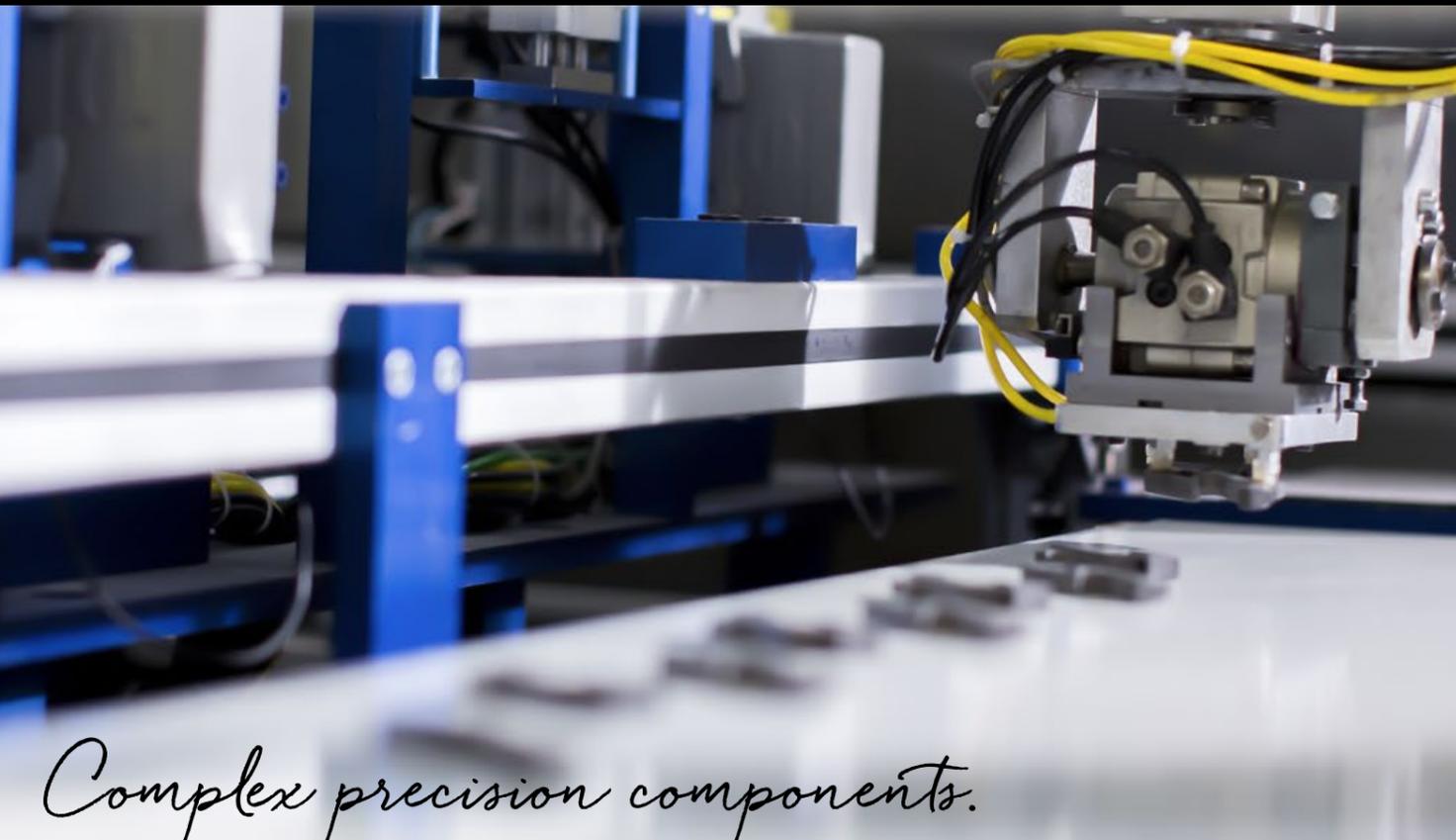


## Cover image

Compounded CIM feedstock  
(Photo courtesy Inmatec Technologies, Germany)

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Ceramic Injection Moulding has come a long way in recent years, with the industry blossoming from the manufacture of simple engineering components to the production of advanced dental implants and beautiful watch components for the world's most prestigious luxury brands. As Dr Georg Schlieper reveals, behind this success are specialist feedstock producers such as Germany's Inmatec Technologies.

### 53 Ceramic Injection Moulding adds to the allure of luxury watches

Recent years have seen an increasing number of leading luxury watchmakers embracing Ceramic Injection Moulding for watchcases, bezels, bracelet segments and even movement parts. We report on recent Ceramic Injection Moulded applications from the luxury market.

### 63 Micro Powder Injection Moulding: Processes, materials and applications

Micro Powder Injection Moulding (MicroPIM) continues to attract attention as a unique process by which to manufacture metal and ceramic micro components in medium to high volumes. In the exclusive review for *PIM International* magazine Dr Volker Piötter and Dr Tassilo Moritz present a status report on the technology, from binder developments to best practice in process simulation, injection moulding and sintering. A number of innovative application areas are also reviewed.

### 73 Additive Manufacturing of Ceramics: Opportunities and solutions for the CIM industry

The Additive Manufacturing of ceramics has the potential to not only transform the way the ceramic industry addresses prototype development, but also presents an opportunity to open up new applications that until now were impossible to produce with conventional ceramic technology. Dr Johannes Homa and Dr Martin Schwentenwein present the state of the art in ceramic AM and review the technical and commercial considerations with specific relevance to the CIM community.

### 81 POWDERMET 2015: Advances in MIM materials and processing highlighted in San Diego

POWDERMET2015, which took place in San Deigo, California, from May 17-20, continues to encourage MIM producers, industry suppliers and researchers from around the world to share their latest research and developments. Dr David Whittaker reviews a selection of papers that reflect a number of key areas for MIM process enhancement and industry growth.

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# Industry News

To submit news for inclusion in *Powder Injection Moulding International* please contact Nick Williams, [nick@inovar-communications.com](mailto:nick@inovar-communications.com)

## Hoeganaes Corporation introduces metal powders for MIM and Additive Manufacturing

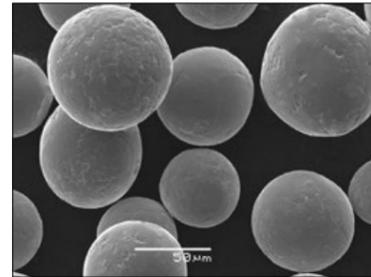
Hoeganaes Corporation, based in Cinnaminson, New Jersey, USA, has launched AncorAM™, a new product line of metal powders engineered for advanced forming processes including Additive Manufacturing, Metal Injection Moulding, and Hot Isostatic Pressing. The first offering in this series includes AncorTi™ titanium powder, a spherical powder available in Ti6Al4V alloy and commercially pure grades. Ti6Al4V alloys exhibit a high strength to weight ratio with excellent corrosion resistance and biocompatibility. This range of properties makes the alloy a perfect candidate to manufacture parts for aerospace, medical, chemical and marine applications.

Hoeganaes has taken this step into the specialist powders market

in conjunction with a multi-million dollar expansion of its Innovation Center in Cinnaminson. Additions to the research and development facility include a new Advanced PM Machining Lab and 3D printer. Chief among the technological upgrades is a pilot atomising facility dedicated to the development of advanced metal powders.

A leader in the development and production of ferrous powders primarily for automotive and industrial applications, Hoeganaes stated that its investment provides the basis for long-term growth in new arenas, including the aerospace and medical industries.

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Metallurgy. The company is a leading contributor to the approximately one million metric tons of iron powder used to produce press-and-sinter parts worldwide.

Hoeganaes is already collaborating with new customers to supply AncorTi for aerospace applications using Additive Manufacturing. For more information on AncorAM solutions, contact Paul Taylor, [paul.taylor@hoeganaes.com](mailto:paul.taylor@hoeganaes.com), [www.hoeganaes.com](http://www.hoeganaes.com)

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## MIM specialist ARC Group further expands its Additive Manufacturing capacity

ARC Group Worldwide, Inc., a leading international MIM parts producer with operations in the USA and Europe, has announced that its 3D printing/Additive Manufacturing division, 3D Material Technologies, LLC, (3DMT) has further expanded its metal Additive Manufacturing capacity with the purchase of new machines from Concept Laser and EOS.

The new machines, a Concept Laser M2 Cusing 400W Dual Laser and an EOSINT M280, will add to 3DMT's already significant metal 3D printing experience and capacity, as well as its leading plastic 3D printing capabilities.

"We have recently experienced increased demand for our Additive Metal/3D printing services from the aerospace industry, Department of Energy, Department of Defence, and medical device markets, which led us to purchase these new machines. Further, with production jobs from our existing customers tying up significant existing capacity, the additional equipment allows us to continue to use our expertise in

powder metals to increase capacity and develop new materials not currently being offered," stated Ashley Nichols, General Manager of 3DMT.

"Notably, we will be working on new aluminium alloys, as well as high strength, high temperature ferrous alloys, which are of great interest to our customers. We have been quite encouraged by this recent increase in adoption of metal 3D printing by clients who traditionally used legacy manufacturing processes, and believe this is potentially indicative of the future market opportunity that additive metals may have," added Nichols.

Overall, these additional machines will significantly increase 3DMT's capacity and reduce lead time to support customers in the aerospace, defence, dental, and medical industries, as well as in ARC's Metal Injection Moulding, injection moulding, tooling, and various capabilities. Further, these new machines have the capability of making a wide variety of parts out of Inconel 625, Inconel 718, titanium, aluminium, stainless steel, cobalt chrome, and maraging steel.

www.3dmaterialtech.com ■

## World PM2016 Congress & Exhibition: Call for Papers issued

The World PM2016 Congress & Exhibition, organised and sponsored by the European Powder Metallurgy Association (EPMA), will take place in Hamburg, Germany, from October 9-13, 2016.

The Powder Metallurgy World Congress is held in Europe once every six years and is therefore an essential destination for those in the international PM community to meet suppliers, producers and end-users and to discover the latest innovations in the state-of-the-art PM technology.

A Call for Papers has now been issued and abstracts can be submitted online until November 12<sup>th</sup> 2015. World PM2016 is an all topic event and includes sessions on:

- Additive Manufacturing
- Hard Materials and Diamond Tools
- Hot Isostatic Pressing
- New Materials and Applications
- Powder Injection Moulding
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## Advanced Materials Technologies diversifies with new 3D metal printing centre

Advanced Materials Technologies Pte Ltd (AMT), Singapore, has announced that it is now offering metal Additive Manufacturing services to complement its existing range of mass manufacturing processes. The company specialises in the high volume manufacture of complex precision components via Metal Injection Moulding and Ceramic Injection Moulding.

In addition to prototyping parts, AMT stated that it is also leveraging Additive Manufacturing technology to meet demands for small volume batch production. One of the main advantages of 3D metal printing for small volume production is the potential elimination of tooling. This leads to direct production being possible without costly and time-consuming tooling.



Having established a 3D Metal Printing Centre, AMT is able to offer a complete solution to customers seeking to accelerate their development of ideas and translation into final product.

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## European PM and MIM sectors see continued growth

The European PM industry is benefiting from the gradual economic recovery in Europe, and particularly from a rejuvenated automotive industry over the past couple of years. Both ferrous and copper-based powder shipments to the PM industry were reported by the European Powder Metallurgy Association (EPMA) to have increased by 7.1% in 2014 compared with the previous year to over 197,612 tonnes.

The EPMA expects this growth trend to continue for 2015 with an anticipated 5% increase for PM powder shipments over the whole year. The MIM sector is also reported by the EPMA to have put in another steady performance in 2014 gaining by around 10% by sales value to an estimated €280 million (\$310 million).

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## Miniature metal check valve made by Micro Metal Injection Moulding

A check valve is a valve that normally allows fluid, be it liquid or gas, to flow through it in only one direction. In some check valves a spherical ball is used to block or release the flow. Japanese Metal Injection Moulding (MIM) producer Taisei Kogyo Co. Ltd., based in Neyagawa City, Osaka Prefecture, recently introduced

what the company claims is the world's smallest MIM-made metal check valve at the 6th Medical Equipment Exhibition held in Tokyo, June 24-26, 2015, using its MicroMIM technology. The check valve has a diameter of just 2.4 mm and wall thickness of 0.2 mm, which is considered to be the limit of conventional MIM technology.

The function of this MicroMIM check valve component is to control the flow of blood to the heart, however the design is not limited to just medical applications but also to other industries. Fig. 1 shows the internal structure of the check valve and its operation in the 'stopped' and 'flowing' states. Fig. 2 shows the MicroMIM check valve and its inner structure.

Taisei Kogyo reports that it has been successful in the series production of the small part having such a highly complex internal shape thanks to its expertise in MicroMIM. The company states that such a complex shape would be impossible to machine, and some of the internal features would also be impossible without Metal Injection Moulding.

[www.metal-injection-tech.com](http://www.metal-injection-tech.com)

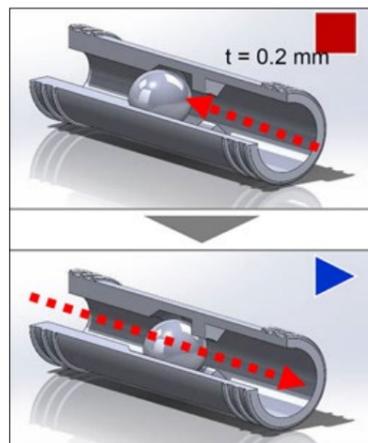


Fig. 1 Image of the inside of the check valve in operation, above is when it is 'stopped' and below is when it is 'flowing'

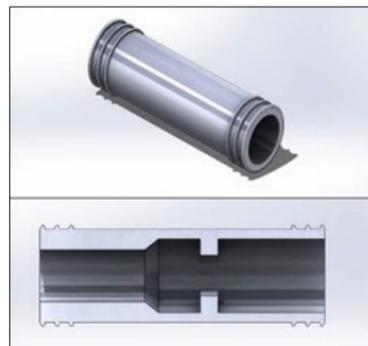


Fig. 2 Appearance and inner structure: the thickness of 0.2 mm is only possible only with MicroMIM



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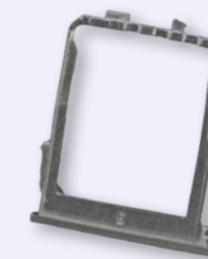
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## Novel PIM method developed for manufacture of integrative membrane carriers

The mass production of carrier devices incorporating membranes as quasi-monolithic devices is often problematic, and a way to produce these devices as an integrated product would offer distinct economic advantages. As these membrane devices are made from both plastic and also metals and/or ceramics, a suitable manufacturing method

capable of volume production is required.

Researchers at the Institute of Applied Materials at the Karlsruhe Institute of Technology (KIT) in Eggenstein-Leopoldshafen, Germany, have adapted micro powder injection moulding technology developed previously at KIT to overcome problems in producing  $\mu$ PIM parts with significant

differences in wall thicknesses required in the various membrane carrier combinations.

Dr Volker Piottter and his colleagues at KIT reported in the May 2015 issue of *Microsystems Technologies* (Vol. 21), that in order to produce the integrated membrane carriers containing different wall thicknesses, a demonstrator tool design was developed. This tool design enabled the generation of thin membranes by a controlled piston movement in the injection moulding step. Simulation calculations were used to determine the layout of the runner system, and the resulting filling behaviour and dimensional accuracies of the moulded parts.

The twin-piston moulding tool also enabled the subsequent embossing of the moulded feedstock in the membrane cavity which allows further consolidation and results in a defined adjustment of the membrane thickness in the range from 600  $\mu$ m to less than 200 $\mu$ m. The combined injection + embossing process could further reduce minimum membrane thickness by a half.

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## PIM International magazine's digital archive expanded

The free-to-access digital archive of past issues of *PIM International* magazine has recently been expanded to include every issue dating back to March 2013.



The recent additions include all issues from 2013 (Volume 7) and feature a number of exclusive company profiles including Element 22, CMG Technologies, Maxon Motor, Elnik Systems and Polymer Technologies Inc. Past issues can be downloaded in a convenient PDF format.

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## Positive outlook for North American MIM and HIP industries

In his state of the PM industry keynote presentation at POWDERMET2015, San Diego, USA, May 18 2015, Richard Pfingstler, President of the Metal Powder Industries Federation, stated that despite some "bumps in the MIM business road," namely the saturated firearms market, 2014 was a good year for the North American MIM industry. He added that the forecast for the next three to five years also looks positive.

According to a 2014 survey by the Metal Injection Molding Association (MIMA), the MIM industry is still ascending its growth-cycle curve at a growth rate well above that of the GDP. Some, it was stated, have even suggested that the industry is still in the steepest segment of its growth curve. The MIMA survey reported the following primary end-user markets in North America by weight of parts shipped: firearms, 28%; general

industrial, 24%; medical/dental, 19%; automotive, 15 %; electronics, 9%; and miscellaneous, 5%.

Pfingstler stated, "While the firearms market currently remains somewhat sluggish, it will most likely stabilize into a more normal growth pattern. However, overall the MIM industry is set to enjoy a 10% growth rate in 2015, certainly an enviable position."

The Metal Injection Molding industry has begun selling into the automotive market in North America, following the trend in Europe towards the adoption of MIM automotive components. "Automotive engineers are designing more MIM parts, which points to significant potential growth as MIM becomes more accepted. MIM parts are being designed for engines, electrical systems, and chassis hardware," concluded Pfingstler.

The Hot Isostatic Pressing (HIP) market registered gains last year that should continue into 2015. "The use of PM HIPed products for the oil-and-gas market will increase, despite declines in oil drilling and fracking. This is mainly due to long lead times for necessary replacement parts. HIPed PM aerospace parts are another growing market," stated Pfingstler.

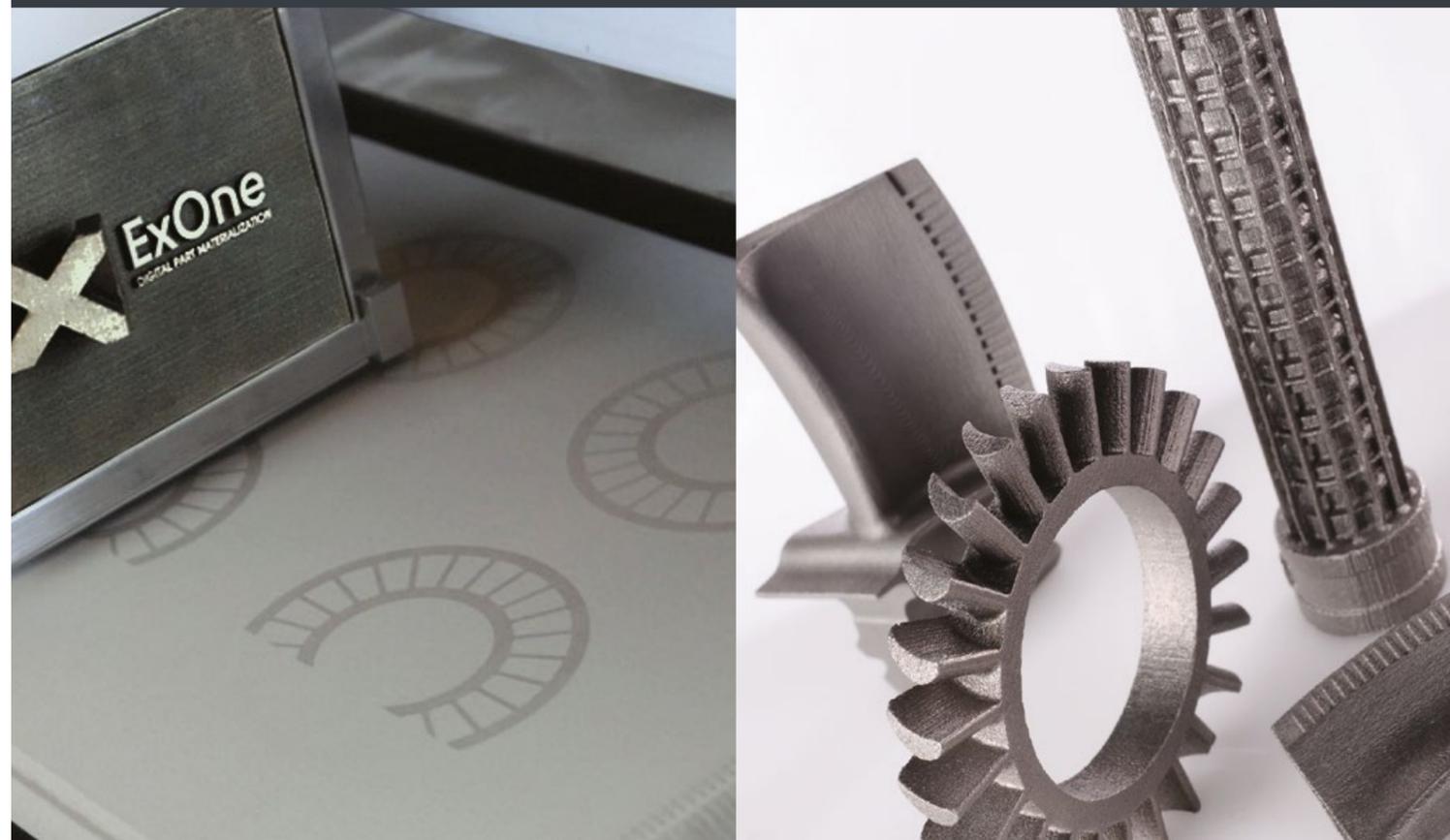
HIP densification of MIM parts remains a robust growth business, and there is new interest in HIP from the Additive Manufacturing sector. "Additive Manufacturing offers an exciting niche business for PM and metal powder producers. Without a doubt AM presents some very interesting opportunities as a new PM technology."

It was stated that a number of powder makers are working on qualifying gas and water atomised powders for AM applications made by laser-based, electron-beam, and ink-jet processes.

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## Slovakian MIM producer Gevorkyan to double the size of its operation

Gevorkyan, s.r.o., is a family owned Powder Metallurgy producer established 20 years ago in Slovakia by Armenian military aircraft engineer Artur Gevorkyan. Today, the company supplies PM and MIM parts to a wide range of end-user industries including the automotive, oil and gas, hand tools, locks and garden equipment sectors.

Over the last three years the company has not only invested in the modernisation of its PM operations, but has also established a new MIM division. Artur Gevorkyan told *PIM International* that the development of MIM technology was an important strategic target for next decade. Around 70% of new company projects are already coming from MIM customers, with conventional PM accounting for the

remaining 30%. These figures, stated the company, are steering investment plans.

This year, the company has invested in a new MIM continuous debinding and sintering furnace from Cremer Thermoprozessanlagen

GmbH, a new furnace for special materials from Elnik Systems, as well as injection moulding machines from Arburg GmbH + Co KG. The company is also installing a HIP plant which will primarily be used for parts destined for the firearms and aerospace industries.

Over the last year, the company has taken on 70 new employees and built a new fully-automated



Engineering discussions at Slovakian MIM specialist Gevorkyan, s.r.o

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warehouse. In relation to the enlargement of the MIM operation, new investments have been made in the company's quality department, where 3D inspection technology and equipment for the chemical analysis of materials have been installed. The company has its own tool room, where two CNC Hermle mills and Fanuc electro-erosion machines are installed.

In Gevorkyan's R&D department, 50% of the company's engineers work exclusively on MIM technology. By the end of 2015, the company plans to install a 3D printer as well as software for the simulation of the injection moulding process. The company currently offers rapid prototyping of MIM parts by CNC machining samples from feedstock blocks. "This is desired mainly by customers, who do not have any experience with MIM materials," stated Artur Gevorkyan.

In last twelve months, Gevorkyan has received 73 new tooling orders for MIM projects. The company plans to add an additional 3,500 m<sup>2</sup> of floorspace to its MIM production facility, bringing the total area for PM

and MIM to 7,000 m<sup>2</sup>. Artur Gevorkyan commented that the company intends to remain reasonably independent of the automotive industry, maintaining automotive production at 30% of its total portfolio. The company plans to change from present sales ratio of 85% PM and 15% MIM technology to 50% PM and 50% MIM, as well as to increase sales from the present €20 million to €40 million by the end of 2018. The majority of the company's customers come from Europe and North and South America.

The company actively promotes MIM technology to its customers, with technical days organised at Gevorkyan as well as at customers' plants. Together with increasing sales and a growing workforce, Artur Gevorkyan's told *PIM International* that his goal is to keep the special atmosphere of a family company with close relation between employees and customers, where everyone is willing to help and exceed customer expectations. The company recently received a Best Employer award from Via Bona Slovakia. For more information email: [arturgevorkyan@gevorkyan.sk](mailto:arturgevorkyan@gevorkyan.sk) [www.gevorkyan.sk](http://www.gevorkyan.sk) ■

## Summer 2015 issue of Metal Additive Manufacturing magazine now available to download



The latest issue of *Metal Additive Manufacturing*, the quarterly magazine for the metal AM industry, is now available to download from the publication's website. Available in both print (ISSN 2057-3014) and digital (ISSN 2055-7183) formats, *Metal Additive Manufacturing* magazine brings together industry news and articles on technical and commercial developments in the industry. In addition to a comprehensive 31 page industry news section, this 72 page issue includes the following articles and reports:

- Additive Industries: Moving towards automation and integration in metal Additive Manufacturing

- Advances in aerospace applications: MTU produces Airbus A320neo borescope bosses with Additive Manufacturing
- AMPM2015 conference report: Innovative materials, powder characterisation and metallographic testing
- Rapid.Tech 2015: Germany's conference and exhibition on AM targets an international audience
- Concept Laser's QMmeltpool 3D: In-situ quality assurance with real-time monitoring down to the micron level.

A free digital issue in PDF format is available to download from the magazine's website.

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## UK MIM producer CMG Technologies targets users of metal 3D printing technology

CMG Technologies, the UK's leading Metal Injection Moulding specialist, states that it has trademarked the term 'CMG Technologies 3D Metal Moulding®' as part of the company's efforts to raise awareness of the scope of opportunities offered by MIM. The company believes that whilst more and more manufacturers are becoming familiar with the capabilities of metal 3D printing/ Additive Manufacturing, the process and benefits offered by Metal Injection Moulding are less well-known in the UK.

CMG's Technical Sales and Marketing Director, Rachel Garrett, stated, "Although 3D printing has been around for more than 30 years, it is only in recent years that it has become widely recognised in the

manufacturing industry. The term 3D printing has captured the attention of both industry and the general public and this, we believe, coupled with significant developments in technology which reduced both the size and costs of 3D printing machines, has played a vital role in the process gaining widespread understanding."

CMG is hoping to replicate the success of this transition for Metal Injection Moulding. MIM is still a relatively new industry and a significant number of companies that need to procure complex metal components may not be aware of its existence, let alone the savings they could make in terms of cost, lead times and environmental impact.

"The phrase '3D metal moulding' paints an immediate picture of what



Scalpel handles manufactured by CMG Technologies prior to debinding and sintering

it is that CMG Technologies actually does," explained Garrett. "Whilst the term Metal Injection Moulding describes the process well, it is not always exactly clear what the outcome is - 3D parts moulded from metal. To those involved in this industry like we are, this sounds obvious, but to people unfamiliar with MIM hopefully this term makes it easier to understand what we do."

CMG believes that whilst 3D metal printing technologies are developing at an extraordinary rate, there are currently still limitations as to their capabilities. The technology is still predominantly used for prototype production as the technology is not yet fast enough to cope with mass production. 3D metal printing can also only be used to produce components from a relatively small selection of materials.

"Rather than being seen as a competitor to 3D metal printing, MIM can actually provide the stepping stone needed between prototype and volume production. Once the initial tooling is complete we are able to deliver huge volumes of parts in a far wider variety of materials than is currently possible with 3D printing," Garrett stated. CMG added that its MIM facility is most effective for small, complex components in annual volumes of 1000 or more, although if the part is particularly complex and expensive the process could still be cost effective for volumes of 500 or more. Globally, MIM technology providers can deliver extremely high volumes of a complex 3D part, with some components being produced at a rate of several million parts per month for sectors such as consumer electronics. Parts can be produced from a wide range of materials including titanium and precious metals, making the process particularly attractive for manufacturing parts used in the aerospace and medical devices sectors.

[www.cmgtechnologies.co.uk](http://www.cmgtechnologies.co.uk) ■

## India's SRPM begins MIM grade powder production

Shree Rajeshwaranand Paper Mills Ltd (SRPM), located in Gujarat, India, has announced a move into metal powder production. In addition to being one of India's leading paper products manufacturers, the company has diversified into manufacturing resin bond, ceramic bond and metal bond grinding wheels and a range of metal powders.

SRPM announced that it has established a state of the art manufacturing plant with the necessary quality control systems including AAS, FSSS, SEM, laser particle analyser, etc. The company began production in March 2015 and offers special grades of pre-alloyed powders suitable for Metal Injection Moulding, Diamond Tools and other Powder Metallurgy applications. The range also includes cobalt, tungsten, iron and nickel metal powders.

SRPM was established in 1995 and employs around 250 staff. The company's Managing Director, Prakash Vora, added that further expansion of all its current activities will begin in the near future.

[shreerajeshwaranandgroup.com](http://shreerajeshwaranandgroup.com) ■

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## Arburg to focus on part quality at Euro PM2015

In a presentation at the Euro PM2015 Congress and Exhibition, Reims, France, October 4-7 2015, Marko Maetzig, who is responsible for applications development at injection moulding machine producer Arburg GmbH + Co KG, will examine the relationship between mould temperature and PIM part quality.

"Fluctuations in quality often arise during powder processing, leading to variations in part properties," stated Maetzig. "A constant green compact density should be strived for in order to avoid time-consuming tests and costly mould modifications." Congress participants will discover how this can be achieved through complete pressure compensation in the moulded part and how the company's Variotherm process can contribute to this.

Arburg, headquartered in Lossburg, Germany, has an extremely well

equipped laboratory in which new application areas for the PIM process are continuously being developed. All upstream and downstream production steps such as material preparation, debinding, sintering and part analyses can also be tested there. Equipment includes a shear roll extruder, debinding systems, sintering ovens and a device for simultaneous thermal analysis. This comprehensive range is complemented by automation solutions, application-specific consulting and special PIM training courses.

Arburg states that fundamentally, the same machines used in plastics processing are also used for Powder Injection Moulding. However, the Allrounder machines for PIM processing feature special equipment such as a highly wear resistant cylinder module, a special screw geometry adapted to Powder Injection



Experts Marko Maetzig, Hartmut Walcher and Uwe Haupt (left to right) at Arburg's modern PIM laboratory.

Moulding and a position-regulated screw.

The products produced on the company's Allrounder injection moulding machines range from micro gear wheels with an external diameter of 1.4 mm to ceramic cores for stationary gas turbines weighing up to two kilograms. New materials, mould and machine technology are opening the door to a growing number of innovative PIM applications in a diverse range of industries.

www.arburg.com ■

## Bodycote's Jane LaGoy recognised with ASTM President's Leadership Award

Jane LaGoy, Technical Services Manager at Bodycote in Andover, Massachusetts, USA, has received the 2015 ASTM International President's Leadership Award in recognition of her work for ASTM's Metal Powders Committee. The President's Leadership Award recognises individuals early in their ASTM career who have significantly advanced our mission through extraordinary accomplishment, example and vision. It is presented annually to two deserving ASTM members. This year's other recipient is Kent Lowry, M.D., an orthopedic surgeon at Northland Orthopedic Associates in Rhinelander, Wisconsin.

LaGoy, who joined ASTM in 2010, is a very active member of Committee B09 on Metal Powders and Metal Powder Products. She currently serves as B09 secretary and chairs Subcommittee B09.05 on Structural Parts. She has been instrumental in the development of and revisions to several key ASTM standards for the Powder Metallurgy industry during her short tenure. LaGoy also works on Committees A01 on Steel, Stainless Steel and Related Alloys, B07 on Light Metals and Alloys, and F42 on Additive Manufacturing Technologies in addition to B09.

A graduate of Rensselaer Polytechnic Institute, Troy, New York, where she received a bachelor's degree in materials engineering, LaGoy earned her MBA from Rivier College in Nashua, New Hampshire. She has worked at Bodycote since 1992 as an engineer, metallurgist and lab manager, assuming her current role in 2013. LaGoy specialises in Hot Isostatic Pressing (HIP), materials characterisation and Powder Metallurgy.

www.astm.org ■

## APMA International Conference on Powder Metallurgy: Technical Programme now available

The Technical Programme has been published for APMA 2015, the 3rd International Conference on Powder Metallurgy in Asia. The event will take place in Kyoto, Japan, November 8-10, 2015.

Organised by the Japan Society of Powder and Powder Metallurgy (JSPM) and the Japan Powder

Metallurgy Association (JPMA), the event includes both oral and poster presentations along with an international exhibition.

The official language of the conference is English and early registration discounted offers are available until September 30.

www.apma2015.jp ■



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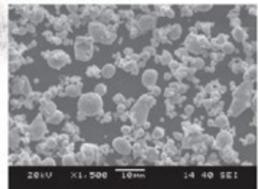
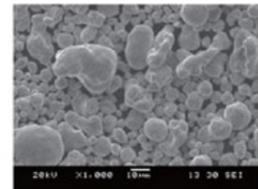
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## Hagen Symposium to focus on process efficiencies and special properties of PM materials

The Fachverband Pulvermetallurgie (FPM), the German trade association for Powder Metallurgy, has published the technical programme for the 34th Hagen Symposium scheduled to take place in Hagen, Germany, November 26-27, 2015.

The programme will embrace a wide range of PM processes such as rapid sintering, Additive Manufacturing, trends in hardmetal cutting tools, advanced PM aerospace alloys, ferrous structural parts, powder forged steels, magnetic materials and ceramics for filtration applications. Around 60 organisations from industry and research are expected to take part in the exhibition which will run alongside the symposium.

The symposium will include the presentation of the Skaupy Prize to Austrian scientist Professor Wolf-Dieter Schubert who heads the Research Group on the Metallurgy of the Less Common Metals at the Institute of Chemical Technology and Analytics, Vienna University of Technology.

Professor Schubert, who has devoted much of his working life to researching metallurgical aspects relating to tungsten carbide based hardmetals, will present his Skaupy Lecture on 'Ultra-fine grain hardmetals – from powder to sintered tools'. The lecture will cover powder production, the selection of grain growth inhibitors, alternative consolidation processes for ultra-fine grain hardmetals, and examples of applications.

[www.pulvermetallurgie.com](http://www.pulvermetallurgie.com) ■

## Powder Metallurgy Day at Ceramitec to focus on Additive Manufacturing

Ceramitec 2015, the international trade show for the entire ceramics industry, ranging from conventional ceramics and raw materials to technical ceramics and Powder Metallurgy, will take place at Messe München, Munich, Germany, from 20 to 23 October 2015.

The Supporting Programme will provide a platform for the transfer of knowledge and expertise in research and development. Attendance to the specialist lectures and panel discussions will be free of charge and simultaneous translation in German and English will be offered for all lectures. This year's programme will begin with a panel discussion themed "ceramitec goes digital" on the opening day, Tuesday 20 October 2015. Experts from ceramics and Powder Metallurgy will report on progress in Additive Manufacturing with regard to industrial realisation and further needs for research and development.

[www.ceramitec.de](http://www.ceramitec.de) ■

## Copper-polymer bullets made by injection moulding

The firing of small arms ammunition for training, sport, law enforcement, and military purposes is a significant source of environmental pollution. The ammunition for small arms is normally made from high density metals such as lead and its alloys, which are traditionally shaped by casting or by cold forming where the metal is shaped in dies to create the projectile shape. Powder Metallurgy has also been used to produce non-sintered lead-free bullets where the frangible, or "soft", rounds are designed to break apart when they hit walls or other hard surfaces to prevent ricochets during close-quarters combat.

Early in 2015 PolyCase Ammunition of Savannah, Georgia, USA, introduced a range of patent-pending lead-free projectiles using a copper alloy powder combined with a high-

strength polymer binder which the company has designated Cu/PTM. The Cu/P projectiles are lighter and faster than lead, are environmentally safe and range compliant. They are produced by PolyCase using a fully automated injection-moulding process and loaded to SAAMI specifications in high quality brass cases.

PolyCase founder and CEO Paul Lemke stated that by using injection moulding of the copper-polymer mixture, the company has been able to produce bullets with a number of advantages. For example, the Cu/P bullets can weigh up to 30% less than lead bullets with similar profiles and being lead free they are more environmentally friendly. Lighter bullet weight also means higher velocity, less recoil and low ricochet – all important qualities for indoor or



*INCPT- Ammo6-CU1-5784 ammunition made by injection moulding of a copper-polymer compound by PolyCase Ammunition*

close range training.

The injection moulding process used by PolyCase is said to have fewer weight variations compared with cast lead bullets and ensures excellent concentricity. The process also allows unique shapes to be produced which would not be possible with conventional processes.

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## MIM industry recognised in the 2015 MPIF Design Excellence Awards competition

The international Metal Injection Moulding industry once again accounted for a significant proportion of the winning parts in the Metal Powder Industry Federation's 2015 Powder Metallurgy Design Excellence Awards competition. The winners were announced at POWDERMET2015, San Diego, USA, May 17-20.

### Grand Prize Awards

#### Automotive - Engine

Advanced Materials Technologies Pte Ltd, Singapore, earned the Grand Prize in the Automotive - Engine Category for a MIM bus nozzle used in a selective catalytic reduction (SCR) system of European commercial vehicles (Fig. 1). The nozzle performs the SCR function using urea and compressed air to reduce the NO<sub>x</sub> produced during combustion to N<sub>2</sub> and H<sub>2</sub>O.

Made of an austenitic stainless steel (AISI HK30), the part employs a patented technology that uses a removable polymeric insert to form the highly complex internal undercut channel, a feature the fabricator deems



Fig. 1 A MIM bus nozzle manufactured by Advanced Materials Technologies Pte Ltd, Singapore, and used in a selective catalytic reduction system

impossible to achieve using conventional machining. All internal channels are achieved through MIM, while the threads on top are machined and Micro TIG welding is used to seal the small openings left by the polymeric insert; tip flatness is achieved through a grinding operation. It is estimated that fabricating the nozzle through the welding/brazing of multiple parts would have increased its cost by more than 200%.



Fig. 2 Four MIM components made by Indo-US MIM Tec Pvt. Ltd and used in proportional valves found in hydraulic circuits of off-highway and farm equipment

#### Lawn & Garden/Off-Highway

Four MIM components made by Indo-US MIM Tec Pvt. Ltd, Bangalore, India, namely a catcher, tension bar, base cap and body, earned the Grand Prize in the Lawn & Garden/Off-Highway Category (Fig. 2). The components, made for Danfoss, Denmark, are used in proportional valves found in hydraulic circuits of off-highway and farm equipment.

The two parts forming the new base are made of MIM 17-4 PH stainless steel, while the catcher and tension bar are formed of 4605 low-alloy steel. All dimensions of the two parts forming the new base are achieved in the as-MIM condition, including the internal thread in the body.

The catcher undergoes a grinding and burnishing process to attain the OD tolerance and surface finish, while the tension bar needs only a turning operation to form an external thread without a parting line. The parts were formerly produced via machining, welding, conventional PM, and fastening. By completely re-designing the parts to maximize the advantages MIM offers, the customer obtained savings estimated at 65%, with annual production of 350,000.

#### Hand Tools/Recreation

Advanced Forming Technology, An ARCMIM Company, Longmont, Colorado, USA, was awarded the Grand Prize in the Hand Tools/Recreation Category for a breech block made for Smith & Wesson (Fig. 3). Fabricated via MIM from 4605 low-alloy steel, the block is inserted into the slide body of a .22 caliber pistol, creating the breech face and other critical functions.

The unique geometry of the part, composed of two large masses separated by a channel running axially down the length of the part, creating two distinct bodies connected by very small ribs, presented a significant challenge to keeping the part together during sintering and maintaining tight final tolerances. The component was manufactured net-shape with no secondary machining operations; it is heat treated to a hardness range of

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Fig. 3 A breech block made for Smith & Wesson by Advanced Forming Technology (AFT)



Fig. 4 A MIM 4605 low-alloy steel top plate made for Multimatic Inmet, Canada, Indo-US MIM Tec Pvt. Ltd.

37-45 HRC, receives a black oxide finish and then undergoes a coining operation to qualify the width. The fabricator believes this is the first breech block fabricated using MIM, an indication that it is possible to expand the application boundaries for MIM even in a mature arena such as firearms.

### Awards of Distinction

#### Automotive - Chassis

Indo-US MIM Tec Pvt. Ltd. received the Award of Distinction in the Automotive - Chassis Category for a MIM 4605 low-alloy steel top plate (Fig. 4) made for Multimatic Inmet, Canada. The part goes into shock absorbers of Chevrolet Camaro automobiles. The complexity of the part, with its 18 holes and six thin ribs that connect to a ring around a central hole, presented a challenge to complete filling.

The gating and venting system played a key role in producing this part defect free. It is produced close to net shape, with surface grinding to achieve flatness and a facing operation to achieve height tolerance being the only secondary operations performed. This application is a new design for MIM and delivers increased repeatability/accuracy of the shock absorber over the previous machined version, with estimated savings of 25%.

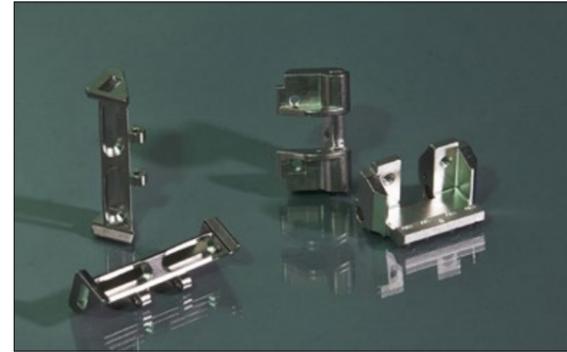


Fig. 5 MIM lock parts made for Rutherford Controls Inc by Indo-US MIM Tec Pvt. Ltd.



Fig. 6 A MIM bolt manufactured by Indo-US MIM Tec Pvt. Ltd. and used in a Keystone Sporting Arms, LLC rifle

### Hardware/Appliances

Indo-US MIM Tec Pvt. Ltd received the Award of Distinction in the Hardware/Appliances Category for two MIM 17-4 PH stainless steel parts, front and rear keepers that go into industrial electrical locks (Fig. 5), made for Rutherford Controls Inc. The parts' complexity, with many cross holes and sharp knurl features, required the use of multiple slides, some moving at different angles. The parts are made close to net shape requiring only a final coining to adjust a small distortion and a tapping operation on the front keeper. Annual quantities are 20,000 per part.

### Hand Tools/Recreation

Indo-US MIM Tec Pvt. Ltd. received the second Award of Distinction in the Hand Tools/Recreation Category for a 4605 low-alloy steel bolt (Fig. 6) used in the Crickett 22LR rifle made by Keystone Sporting Arms, LLC. Made via MIM, the part replaced one produced by brazing together three machined parts, delivering an estimated 35% cost saving in the process. The part's design features many cross holes and undercuts, requiring complex side core matchings in the molding cavity. The fabricator delivers 60,000 pieces annually.

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Fig. 7 A MIM shell manufactured by Indo-US MIM Tec Pvt. Ltd. for Hirose Korea Co. Ltd.



Fig. 8 MIM connectors made by Indo-US MIM Tec Pvt. Ltd. for Amphenol Air LB, France

**Electronic/Electrical**

Indo-US MIM Tec Pvt. Ltd. received two Awards of Distinction in the Electronic/Electrical Category. The first award was given for a MIM 17-4 PH stainless steel shell (Fig. 7) made for Hirose Korea Co. Ltd. The part goes into a charging assembly for mobile phones. The complex geometry of the part, with thin cross sections and internal undercuts, was achieved with a slide-in-slide mechanism moving on specially designed cam tracks. The MIM part replaced an earlier design that used sheet metal processing and welding, with an estimated 20% cost savings. Two million of the parts are delivered each month.

The second Award of Distinction in the Electronic/Electrical Category earned by Indo-US MIM Tec Pvt. Ltd. was for two parts, a male fool-proof device and a female polarizer (Fig. 8), made for Amphenol Air LB France. Made via MIM from 4340 low-alloy steel, the parts go into an electric connector assembly made for Airbus. Both parts are fabricated to net shape, with all dimensions achieved in the as-MIM condition; this includes the threads, which are formed through auto unwinding in the tooling. The fabricator delivers 320,000 pieces annually.

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## Carbon nanotube reinforced iron-based MMCs produced by Powder Injection Moulding

Metal Matrix Composites (MMCs) using carbon nanotubes (CNTs) as the reinforcing material have generated significant interest in recent years through their ability to produce lightweight components with high stiffness and strength. Researchers at Veermata Jijabai Technological Institute in Matunga, Mumbai, India, have been investigating the use of the Powder Injection Moulding (PIM) process to produce value added MMC products with iron powder derived from grinding waste as the base metal. On its own this type of iron powder would have inadequate properties but when CNTs are added significantly better properties can be achieved. V.J. Pillewan and co-researchers published their findings on the use of PIM to produce Fe-CNT MMCs in the International Journal of Emerging Technology and Advanced Engineering (Vol. 5, No. 5, May 2015).

They stated that carbon nanotubes were added to iron powder in fractions ranging from 1% to 6% by weight. The feedstock

for the MMCs was prepared by first mixing the iron powder with stearic acid at 60°C followed by the addition of paraffin wax and CNTs with mixing at 90°C, and finally adding HDPE and mixing at 160°C. The proportions of the various materials used to produce the PIM samples are shown in the Table. Solvent debinding was preferred to thermal debinding because of more complete binder removal. Sintering was done in an inert atmosphere at 1150°C for 2 hr which was found to be sufficient to provide bonding between the iron and CNTs.

Some porosity was found in the sintered PIM samples, but the researchers state that work is underway to improve sintered density and hence to improve strength properties. As the percentage of CNTs increases in the samples it improves the hardness of the Fe-CNT composite. However, adding more CNTs may lead to fracture of these samples, and further optimisation is required. Hardness values ranged from 55 to 87 HRB. Wear tests on the PIM Fe-CNT MMCs are reported. ■

Material	Sample A (86-14)	Sample B (88-12)	Sample C (90-10)	Sample D (91-09)
Iron powder 85%	75 g	75 g	75 g	75 g
CNT	0.9 g (1%)	2.65 g (3%)	4.41 g (5%)	5.29 g (6%)
Binder				
Paraffin wax (70%)	8.65 g	7.41 g	6.18 g	5.56 g
HDPE (12%)	1.48 g	1.27 g	1.06 g	0.96 g
Stearic acid (18%)	2.22 g	1.91 g	1.59 g	1.43 g
<b>Total</b>	<b>88.25 g</b>	<b>88.24 g</b>	<b>88.24 g</b>	<b>88.24 g</b>

Table 1 Mixing of proportions of Fe-CNT MMCs (From paper by V.J. Pillewan, et al. published in the International Journal of Emerging Technology and Advanced Engineering (Vol. 5, No. 5, May 2015))

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## Expansion of powder production operations at Atomising Systems

Atomising Systems Ltd, Sheffield, UK, has reported that it has expanded its workforce to cope with a major influx of orders for metal powder. Staff numbers at its Darnall facility have increased from 35 in September 2014 to over 60 in June 2015 to allow the company's two 500 kW melting furnaces to operate at maximum output to service an order book stretching into 2016.

The company, which as well as manufacturing metal powders is a major supplier of equipment for the atomisation of metals, has taken on four new apprentices to join several that were hired two years ago and who are now finishing their training.

As well as hiring enough staff to operate the existing plant at maximum output, ASL is investing heavily in new equipment. A £100,000 major upgrade of the gas atomiser is in hand, while a large new sieving station of similar cost has just been put into production on special steel grades.

Further investments include a new instrument using laser diffraction to measure particle sizes as fine as one micron and the QC laboratory is being expanded with the addition of an oxygen analysing system.

Simon Dunkley, who took over management of the company from his father, Dr John Dunkley, in 2010 stated, "After some tough times, it is very gratifying to see our investment in plant and research at last paying dividends. It is particularly pleasing to be able to offer so many young people the opportunity to work in a leading-edge technical environment that allows so much scope for them to develop their talents. Our recent investments should further allow us to fulfil the exacting demands of leading metal powder users around the world."

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The company's new apprentices with Simon Dunkley (centre). Left to right: Ashley Coe, Steven Parker, Natalie Galt and Ben Twomey

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## Engel signs exclusive Liquidmetal agreement

Injection moulding machine producer Engel Holding GmbH, Schertberg, Austria, has signed an agreement with Liquidmetal Technologies Inc., Rancho Santa Margarita, California, USA, whereby Engel will be the exclusive machine manufacturing partner for Liquidmetal's unique patented process for producing components from amorphous alloys by Liquidmetal injection moulding.

The exclusive agreement was announced at the 2015 Engel Symposium held in St Valentin, Austria, June 16-18, where a special all-electric E-Motion injection moulding production cell (Fig. 1) was demonstrated for Liquidmetal injection moulded medical forceps (Fig. 2) from an amorphous alloy based on zirconium.

The amorphous alloys are available in the form of slugs cut from round rods. These slugs are automatically fed into a melting chamber where the material is induction melted under high vacuum. Instead of a screw normally used in injection moulding, the special Engel machine has a piston with which the molten metal alloy is injected into a thermo-regulated mould. Very rapid cooling leads to the formation of the amorphous structure in the zirconium alloy which gives the material its unique characteristics. Robots are used to remove the finished parts. The sprue



Fig. 1 Engel's special Liquidmetal all-electric E-Motion injection moulding machine

can, for example, be removed with the help of a water-jet cutting machine or mechanical shears.

Dr Gerhard Dimmler, Engel Vice-President Research & Development Products, stated these amorphous materials are extremely hard, but at the same time highly elastic, with an elasticity of 2%, compared to 0.2% for steel and 1% for titanium. Liquidmetal amorphous zirconium alloys also have a low specific weight and excellent corrosion resistance, which make these alloys particularly suitable for moulding precision components. Additionally, the shrinkage rate during rapid solidification is extremely low (0.4%) which allows injection moulding to net shape.

Engel sees good potential for components produced by Liquidmetal injection moulding in the field of medical technology, with the parts for the forceps being moulded at the Symposium being a good example, while endoprostheses such as hip joints or stents are also conceivable. Thanks to the excellent mechanical properties of the material, very robust components can also be achieved with very low wall thicknesses.

www.engelglobal.com | www.liquidmetal.com ■



Fig. 2 Parts for medical forceps were produced from a Liquidmetal alloy at the 2015 Engel Symposium held in St Valentin, Austria

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## ATI expand nickel based superalloy powder capability

Allegheny Technologies Incorporated (ATI) is investing around \$70 million to expand its nickel-based superalloy powder production to satisfy strong demand from the aerospace jet engine market. The development is expected to take two years to complete and will be located at its Specialty Materials business unit in North Carolina, USA.

Nickel-based superalloy powders provide extreme alloy compositions and a refined microstructure that offer increased performance and longer useful lives in high-temperature and highly corrosive environments. "This strategic growth project will strengthen ATI's position in the production of technically demanding superalloy powders used to produce advanced mill products and forgings, primarily for next-generation jet engines," stated Rich Harshman, ATI's Chairman, President and CEO.

"A significant portion of the powders to be produced from this expansion are needed to meet requirements of existing long-term agreements with jet engine OEMs that run well into the next decade. The expansion also better positions ATI to continue as a leading innovator supplying advanced powders to the new and rapidly growing Additive Manufacturing industry," added Harshman.

www.atimetals.com ■

## EURO PM2015 Technical Programme now available

The Euro PM2015 Congress & Exhibition Technical Programme, including papers, tour information and exhibitor listing is now available from the European Powder Metallurgy Association (EPMA). The annual Powder Metallurgy event will this year be held in Reims, France, October 4 - 7, 2015. Euro PM2015 will cover all the aspects of Powder Metallurgy including:

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M4	256.81	3.74	0.9588	378	510
C1	645.72	1.43	0.9833	346	510
C7	244.98	1.34	0.9924	335	510
CS13	1024.23	1.29	0.9190	367	510



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## Japanese MIM production data shows further decline in sales

The Japan Powder Metallurgy Association (JPMA) recently published a market report which showed that sales of Metal Injection Moulded products declined for the fifth successive year in 2014 (Fig. 1). Sales of Yen 10.566 billion (\$84.3 million) were recorded in 2014, an 11.6% fall on sales for 2010, and a 34% drop on sales of Yen 13.981 billion achieved in 2008.

The JPMA figures were based on the returns of 21 companies in Japan active in Metal Injection Moulding production. The JPMA is forecasting a small recovery in MIM sales in 2015 and 2016. MIM producers believe that more efforts are needed to promote the benefits of the technology particularly in terms of quality and dimensional accuracy of complex MIM components.

The JPMA reports that the main application sectors for MIM parts in Japan remain industrial machinery at 23.6% (20.5% in 2013), followed by medical instruments at 18.3%, and automotive at 17.3% - down from 19.3% in 2013. Fig. 2 gives a breakdown of all the application sectors for MIM and Fig. 3 gives a breakdown of the materials used

in MIM production. Stainless steels remain by far the largest group at 62.2% followed by Fe-Ni base alloy, and magnetic materials at 8.7%.

www.jpma.gr.jp ■

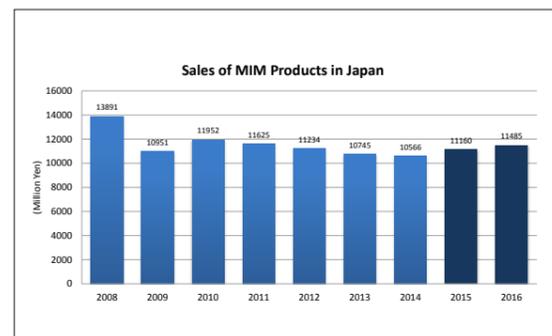


Fig. 1 Sales of MIM Products in Japan (FY2008-2014: results, FY2015-2016: estimate) [Courtesy JPMA]

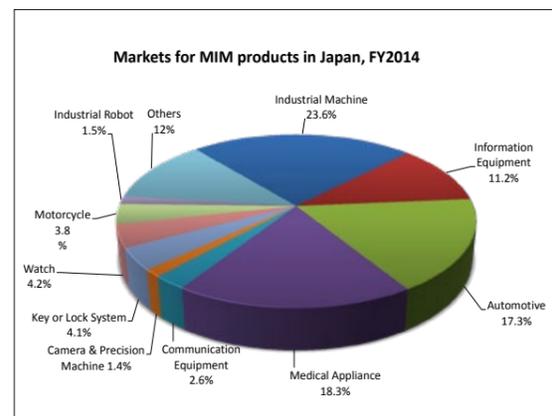


Fig. 2 Distribution ratio of MIM markets in Japan (FY2014) [Courtesy JPMA]

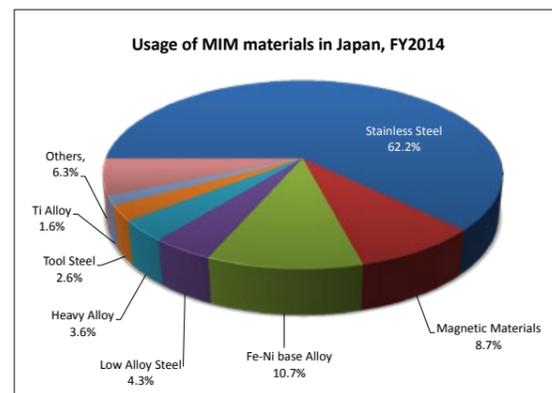


Fig. 3 Distribution ratio of MIM materials in Japan (FY2014) [Courtesy JPMA]

## Ceramic Injection Moulding and gas-pressure sintering used to process ultra-fine silicon nitride powder

Silicon nitride (Si<sub>3</sub>N<sub>4</sub>) ceramics have been produced by a number of manufacturing processes for use as bearings, gas turbine components, and other high temperature environment applications. Compared with other oxide ceramics such as Al<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub> is less dense and exhibits lower thermal expansion co-efficient, a greater strength retention at high temperature, and good corrosion resistance.

Yian-Feng Yang and colleagues at the Changsa University of Science & Technology, Changsa, and Tsinghua University in Beijing, have been investigating the use of Ceramic Injection Moulding (CIM) to produce high precision and complex functional shapes having high surface quality from ultra-fine Si<sub>3</sub>N<sub>4</sub> powder. The authors report the results of their investigation in the *International Journal of Minerals, Metallurgy and Materials* (Vol. 22, No. 6, June 2015). They state that although CIM is successfully used for some oxide ceramics, it has not yet been extensively used for Si<sub>3</sub>N<sub>4</sub> because of the difficulty in producing a CIM feedstock with a sufficiently high powder loading and low viscosity. This is especially the case for sub-micron Si<sub>3</sub>N<sub>4</sub> powders.

The researchers used Si<sub>3</sub>N<sub>4</sub> powder having a median particle size (d50) of 0.34 micron where the α-Si<sub>3</sub>N<sub>4</sub> phase content was greater than 92 wt%. This was ball milled in ethanol together with a sintering additive (6 wt% Y<sub>2</sub>O<sub>3</sub> + 2 wt% La<sub>2</sub>O<sub>3</sub>) and the resulting composite slurry was dried for use in the CIM feedstock. The organic binders used for the feedstock included polypropylene (PP), polyethylene (PE) and wax (PP:PE:W = 10:10:80 by mass). Additionally, stearic acid was used as a surfactant. Spherical parts and test bars were produced from the feedstock by injection moulding followed by solvent debinding at 40°C, thermal debinding at 900°C, and gas pressure sintering at 1795°C for 3 hrs under N<sub>2</sub> pressure of 8 MPa.

The results show that a solid loading of less than 50vol% and the surfactant mass fraction of 6wt% gave perfect flowability of the Si<sub>3</sub>N<sub>4</sub> feedstock making it suitable for CIM. Defects detected during production are traced to improper moulding parameters, mould design, debinding parameters, residual stress, or inhomogeneous composition distribution in the green parts. Gas pressure sintered properties of Si<sub>3</sub>N<sub>4</sub> included density of 3.2 g/cm<sup>3</sup>, Vickers hardness of 16.5 GPa, and fracture toughness of 7.2 MPa.m<sup>1/2</sup>.

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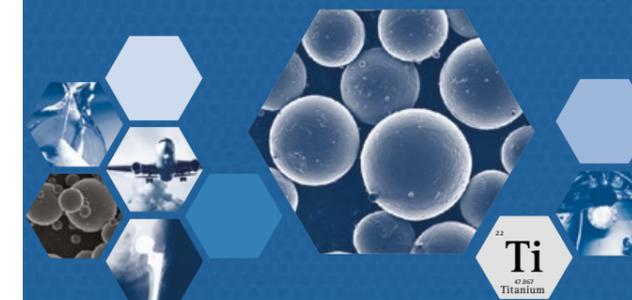
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## University Carlos III studies PIM of zirconium silicate

A recently completed ECOPIIM research project undertaken by the University Carlos III de Madrid in collaboration with Spanish partners Guzman Global, based in Nules, and MIM-Tech Alfa, based in Eibar, has resulted in a new Powder Injection Moulding processing route to produce components from zirconium silicate (ZrSiO<sub>4</sub>).

The new process developed under the ECOPIIM project is said to use a feedstock binder system with a high percentage of soluble polymer which is more eco-friendly than conventional binders. The results of the project, which had the objective of finding new applications for zircon silicate in sectors such as automotive, aerospace, jewellery, telecommunications and machinery, were published in *Boletín de la Sociedad Española de Cerámica y Vidrio* (Vol. 54, June 2015, pp

93-100) by authors Carolina Abayo, A. Jimenez-Morales, and J. M. Torralba.

The authors stated the raw ZrSiO<sub>4</sub> powder, grade ARMIN-05 supplied by Guzman Global, was obtained from mineral sands zircon silicate. The powder had a particle size distribution of 1.94 µm (D50) and 5.49 µm (D90) respectively; apparent density is 19.05%. The irregular morphology (Fig. 1) of the ZrSiO<sub>4</sub> powder contrasts with the normally spherical powder morphology used in Powder Injection Moulding. They studied PIM binder systems composed of Cellulose Acetate Butyrate (CAB), which is an alternative to polyolefin, and Polyethylene Glycol (PEG), a water-soluble polymer, to which was added stearic acid as surfactant and antioxidant phenothiazine.

Different grades of molecular weights (Mw) were used to study the influence of binder composition

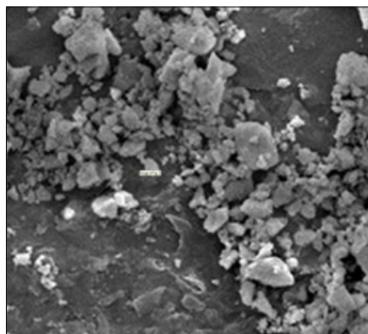


Fig. 1 Morphology of raw zircon powders obtained by SEM (From paper by C. Abajoa, et al. *Boletín de la Sociedad Española de Cerámica y Vidrio*, June 2015)

on the PIM process; PEG between 1.5 K and 20 K Mw and CAB between 20 K and 30 K Mw. It was stated that optimising binder system design is extremely important to avoid defects in the PIM parts at different production stages. It was established that a binder system based on high-Mw of CAB and low-Mw of PEG (binder B3 in Table 1) with a suitable rheological behaviour and successful solvent

debinding has the potential to be cost efficient for PIM production of ZrSiO<sub>4</sub> components. This binder system is also eco-friendly through the use of a high percentage of soluble polymers and CAB which brings about zero balance of CO<sub>2</sub> emissions through its thermal degradation.

To fully test the B3 binder system, green parts having dimensions of 60 mm x 8 mm x 4 mm were injection moulded. Water solvent debinding was carried out from room

temperature to 60°C followed by thermal debinding at up to 500°C, and sintering at 1500°C for 3 hr. The PIM processed ZrSiO<sub>4</sub> parts were found to be defect free. The sintered parts were tested for dimensional change, relative density by water absorption method and flexural strength obtained on a three-point bending testing machine. A competitive flexural strength was reported for the sintered PIM ZrSiO<sub>4</sub> test pieces.

www.uc3m.es ■

	CAB 30K	CAB 20K	PEG 20K	PEG 10K	PEG 4K	PEG 1.5K	SA	PTZ
B1		30	70					
B2	30		33.5	33.5			2.5	0.5
B3	30				33.5	33.5	2.5	0.5
B4		30	33.5	33.5			2.5	0.5
B5		30			33.5	33.5	2.5	0.5
B6		30	50	5	5	10		
B7		40	60					

Table 1 Binder compositions in vol% of the binder with different grades of molecular weights (From paper by C. Abajoa, et al. *Boletín de la Sociedad Española de Cerámica y Vidrio*, June 2015)

## POWDERMET2016 Call for Papers issued

The Metal Powder Industries Federation (MPIF) has issued a Call for Papers for its POWDERMET2016 International Conference on Powder Metallurgy and Particulate Materials, Boston, Massachusetts, USA, June 5-8, 2016.

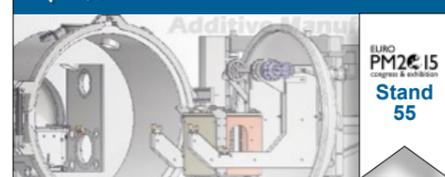
Authors are requested to submit abstracts for papers to be presented in the general technical sessions and poster programmes no later than September 18, 2015. Abstracts can address all aspects of Powder Metallurgy and particulate materials technology. The technical sessions will include the following categories:

- Design & Modelling of PM Materials, Components & Processes
- Particulate Production
- General Compaction & Forming Processes

- Powder Injection Moulding (Metals & Ceramics)
- Pre-Sintering & Sintering
- Secondary Operations
- Materials
- Refractory Metals, Carbides & Ceramics
- Advanced Particulate Materials & Processes
- Material Properties
- Test & Evaluation
- Applications
- Management Issues

The MPIF's Additive Manufacturing with Powder Metallurgy conference (AMPM2016) will be held in conjunction with POWDERMET2016.

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## Carpenter on course to supply aerospace superalloy powder market

Carpenter Technology has stated that it remains confident that operations will begin soon at its new superalloy powder facility in Alabama, USA, as part of a multi-level agreement with United Technologies Corporation's (UTC) Pratt & Whitney Division. The agreement with UTC was signed in October 2013 and includes Carpenter supplying Pratt & Whitney with superalloy powder for up to 20 years.

Gregory A Pratt, Carpenter's Chairman, President and Chief Executive Officer stated, "We're extremely excited to produce an aerospace superalloy powder with demand volumes directly linked to the move by engine manufacturers to develop better fuel-efficient engines. Carpenter is also optimistic that demand for superalloy powder will begin to grow in other key markets we serve."

Pratt added, "Our relationship with UTC has allowed us to expand our position as a key supplier in the fast-growing aerospace market. With the recent opening of our technologically advanced lean manufacturing facility in Alabama, which is adjacent to the superalloy powder facility, our ability to supply ultra-premium products for the aerospace market has never been better."

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## PM Review: Autumn/Fall 2015 issue out now

The latest issue of *PM Review*, the magazine for the PM industry, has just been published and is available to download free of charge from [www.ipmd.net](http://www.ipmd.net).

This 84 page issue features the following articles and technical reports:

- An introduction to Powder Metallurgy Soft Magnetic components: Materials and applications
- Sintering in PM: Selection of sintering tray material for improved part quality
- POWDERMET2015: North America's PM industry continues on growth track; MPIF PM Design Excellence Awards 2015
- POWDERMET2015: Further developments in Hot Isostatic Pressing technology

*PM Review* is available in both print (ISSN 2050-9693) and digital (ISSN 2050-9707) formats. Current and past issues are available to download free-of-charge.

[www.ipmd.net/pmreview](http://www.ipmd.net/pmreview) ■



## Profits increase at GKN's PM division

UK-based GKN plc has reported a solid performance in its half-year sales figures ending 30 June 2015. Overall six-month sales were reported as £3,853 million, up 1% on the same period in 2014. GKN's Powder Metallurgy division, which comprises GKN Sinter Metals and metal powder producer Hoeganaes, achieved first half organic sales of £474 million, £6 million higher (1%), after the £8 million pass through to customers of lower scrap steel prices. There was no impact from currency translation and there was a £3 million decline as a result of a disposal.

It was stated that good growth was achieved in North America, China and Europe but sales in Brazil fell due to weaker automotive and industrial markets. The company's PM division includes one of Europe's largest MIM operations, located in Bad Langensalza, Germany.

The organic increase in profit was £2 million and there was a £1 million gain on currency translation. The divisional trading margin was 11.8% (2014: 11.3%) reflecting the move towards higher value "design for Powder Metallurgy" parts and a small margin benefit from lower raw material prices passed through to customers. Return on average invested capital was 22.0% (2014: 21.0%), reflecting the improvement in profitability.

During the period, GKN Powder Metallurgy continued to achieve a number of important milestones, which included continuing strong product and development activities in engines and transmissions and a strengthening of its position in China following the announcement of a new joint venture to produce metal powders. The company has also developed a new range of technically enhanced titanium powders at its Powder Innovation Centre in the US.

Commenting on the results, Nigel Stein, Chief Executive of GKN, stated, "This was another solid performance, particularly in our automotive businesses, with GKN Driveline delivering 4% organic sales growth and an 8.3% trading margin while GKN Powder Metallurgy achieved an 11.8% margin. GKN Aerospace delivered in line with expectations and won some important new contracts for the future. We have continued to perform well against our key markets and report good results in spite of some end market weakness, particularly in GKN Land Systems. We expect these trends to continue in the second half and for 2015 to be another year of growth. The acquisition of Fokker Technologies, also announced today, supports our strategy, brings excellent technology, an expanded geographic footprint and additional content on high growth aerospace programmes."

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# Inmatec Technologies GmbH: A feedstock supplier at the forefront of CIM technology

Ceramic Injection Moulding has come a long way in recent years, with the industry blossoming from the manufacture of simple engineering components to the production of advanced dental implants and beautiful watch components for the world's most prestigious luxury brands. As Dr Georg Schlieper reveals for *PIM International*, behind this success are specialist feedstock producers such as Germany's Inmatec Technologies GmbH.

Inmatec Technologies GmbH was founded in 1998 as a specialist producer of thermoplastic ceramic feedstocks. The company is located in Rheinbach, a medieval town with a population of around 28,000 located close to Bonn, the seat of government of the former West Germany. Situated in the middle of a fertile agricultural region, the town has a tradition of glass manufacturing following the arrival of Bohemian glass manufacturers who were forced to leave their homeland after World War II. Today, Rheinbach houses a sectoral campus of the Bonn-Rhein-Sieg University of Applied Sciences. Inmatec cultivates close contacts with this university, as well as the Koblenz University of Applied Science's Department of Material Science in Höhr-Grenzhausen, where regular seminars are held about Ceramic Injection Moulding technology.

Founder and CEO of Inmatec Technologies is Dr Moritz von Witzleben, a mineralogist who during his PhD thesis work came in contact with the PIM group at specialist injection moulding machine

manufacturer Arburg GmbH + Co KG. There he realised that there was a need within the PIM industry for high quality feedstocks and decided to establish a business in this field. He started with a commercially available binder system that he compounded with ceramic powder. The requirements of his very first customer, who is still a customer today, increased much faster than initially planned and the company

grew rapidly. The commercial binder system used for this initial order has since proven so successful in such a wide range of applications that it is now commonly used in the company's standard PIM grades as well as in customer-specific feedstocks. Today, Inmatec is the leading supplier of thermoplastic ceramic feedstocks in Europe with customers all over the world. In addition to his work at Inmatec, Dr von Witzleben is CFO of



Fig. 1 The home of Inmatec Technologies GmbH (Courtesy Inmatec)

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Hönbacher Str. 10  
D-96515 Sonneberg

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Fig. 2 Ceramic powder and binders (Courtesy Inmatec)



Fig. 3 Compounded CIM feedstock (Courtesy Inmatec)

the German Ceramic Society (DKG) and President Elect of the European Ceramic Society (ECERS) for the period 2017-2019.

The company's capital comes from a venture capital investor in combination with private shareholders, including the CEO. A further strategic investor has secured its raw material source by buying into the company, taking advantage of Inmatec's materials development capabilities. High-end products for the luxury products market, in which aesthetic as well as technical qualities are essential, are manufactured from feedstock of the highest possible purity and quality. The knowledge and expertise

to produce this high quality is one of Inmatec's key assets.

Inmatec's factory is housed in a plain functional building (Fig. 1) that has a production floor space of roughly 1000 m<sup>2</sup> plus additional space for raw materials and feedstock storage. Facilities for processing both metal and ceramic feedstocks are installed, but the focus is, to a large extent, on ceramic feedstocks. This is where Inmatec's technical expertise is greatest. Inmatec's ceramic feedstocks are used not only for CIM, but also for thermoplastic extrusion. Approximately 70% of production is exported, mainly to other European countries, but also to a growing market in South and North America.

The company's market share in Asia is still relatively small.

Of Inmatec's staff of 25 employees, nine work in R&D and eleven work in feedstock production. Five production shear roll machines operate in two shifts. Further, up to four students are supervised and guided in their Bachelor, Master or Diploma thesis work as well as carrying out their practical work at Inmatec. This cooperation is a win-win situation for the students and for Inmatec. The students gain insight into the everyday work of the industry and Inmatec takes advantage of their work to meet technical challenges. Inmatec also regularly offers two apprenticeship places, one in business administration and one for a plastics and rubber process technician. Seminars held by the company at the University in Höhr-Grenzhausen aim to inspire an interest in CIM amongst young students. This is regarded as an important activity to secure the availability of skilled specialists in the future.

### Developing customised feedstocks

When an enquiry is received for a new CIM product, the Project Manager initially analyses the application and decides whether a standard feedstock can be used. If a customised feedstock has to be developed, Inmatec's significant experience and close relationships with raw material suppliers are essential in selecting suitable raw materials and determining if additional powder processing steps are required.

The choice of binder composition is equally important. A polymer that can withstand the temperatures required for plasticising, exhibits a good flowability during injection moulding, adheres to the powder particles and coats them with a homogeneous layer is ideal. Binder removal should be fast without damaging the shape of the components, environmentally friendly and easy to handle.

"New ceramic products often require longer development cycles than other materials," stated Daniela

Raab, Sales and Project Manager at Inmatec, "because the manufacturing process is more complex and each step must be optimised separately and in detail."

In recent years, the market demands on the ceramic powders have become more specific and Inmatec has reacted to these requests with a build-up of expertise in powder processing. Particle size distribution, specific surface and chemical composition are the main characteristics of ceramic powders. CIM generally uses powders with particle sizes in the range between 200 and 1000 nanometres and a specific surface between 5 and 20 m<sup>2</sup> per gram. For the control of the shrinkage, however, these characteristics must be specified within much closer limits.

Exceptions, however, prove the rule. If needed for a special application, nano powders as well as ceramic powders with particles sizes of more than 20 microns have been processed successfully into ceramic feedstock. Inmatec also creates special alloy compositions by blending powders like zirconia and alumina in various proportions. Coloured zirconia feedstocks are produced by adding pigments to zirconia powder.

When the powder blend has been determined, the next step is the selection of the most suitable binder for the designated application. Each binder is made up of several components; a basic binder, a backbone binder and additives (Fig. 2). Inmatec has a variety of binder components available that can influence the flow characteristics of the feedstock as well as strength and ductility of the green compact within wide limits, as required by injection moulding and subsequent processing.

The feedstock production process is, of course, at the heart of Inmatec's expertise. The shear roll machines at Inmatec have been modified according to the specific requirements of the Inmatec process. Powder blend and binder are first mixed mechanically and then plasticised and homogenised by a pair of heated shear rolls. The objective



Fig. 4 Shear roll used for feedstock homogenisation (Courtesy Inmatec)



Fig. 5 A shear roll machine at Inmatec (Courtesy Inmatec)

of shear roll processing is to remove agglomerates and coat each powder particle with a polymer layer so as to make injection possible and reduce abrasion in the injection moulding machine. Because of the fine particle size of the powder, a very strong shearing action is required to produce a high quality feedstock and the total processing time is dependent on powder properties.

Fig. 4 shows the set of two parallel shear rolls. Between the rolls there is a small gap. The powder-binder mix, which is fed into the gap at one end of the shear rolls, sticks on one roll and the other roll is clean. The surface of the rolls is coated to avoid abrasion and their profile is designed in such

a way that, with each turn of the roll, the material is transported a small distance towards the other end of the roll, where it is cut off by a knife. This produces uniform feedstock granules that are ideal for injection moulding.

A shear roll machine operating at Inmatec is shown in Fig. 5. On the platform behind the machine, one can see the pre-mix material feeder.

When feedstock samples have been produced and tests on Inmatec's in-house injection moulding facility have been successful, the customer also tests the injection moulding performance of the feedstock at their own facility using the tooling for the new product. Based on the feedback from the customer,

modifications of the feedstock are, if necessary, made at this stage. The next step is the definition and optimisation of the debinding process. In general, debinding is carried out in two steps, solvent and thermal debinding. Depending on the binder composition, solvent debinding can be performed in water or an organic solvent such as acetone. The solvent bath is usually heated from 25 to 60°C in order to accelerate the diffusion processes. The purpose of solvent debinding is to remove the basic binder and create a network of open porosity in the CIM compacts that later allows the evaporating backbone binder to escape during thermal debinding without causing internal pressures that might destroy the parts. The time and temperatures required for solvent debinding depend on the wall thickness of the parts and the particle size of the powder.

The time-temperature profile for thermal debinding is then determined. Based on past experience, a general profile is applied as a starting point, but modifications are often required depending on the particle size, binder content and wall thickness of the part. Daniela Raab stressed the point that the interaction between inorganic powders and organic binders must be accounted for. Heating ramps and dwell times may differ, not only for different binder systems, but also for different inorganic powders. Moreover, having started with a

general and safe profile in terms of time and temperature settings, ramps and dwell times are improved and adjusted during volume production for the specific ceramic component to reduce the overall debinding time.

The sintering cycle requires considerations on how to place the products in the furnace. Can they stand alone or do they need support by a powder bed or other means? Supports may be used to avoid distortion, but then the size and shape of supports have to be defined. For new powders, the sintering cycle and the final maximum temperature need to be defined. These parameters may be different from the general recommendations given by the powder suppliers because CIM shaped parts need more, and different, energy input to achieve the desired final microstructure.

Inmatec, therefore, not only delivers the customised feedstock, but also develops a complete set of processing parameters from injection moulding to the sintered component in close cooperation with the customer. This comprehensive service is unique in the CIM industry and Inmatec is convinced that this is the way to secure and extend its strong business position in a high-wage country like Germany.

With all these steps, the development of a new product can be time-consuming and tedious. In some cases, it takes one and a half to two years. Then, the end user may require further tests to ensure the reliability

and durability of the products, as is common, for example, in the automotive industry. The reward is that these products, once established, are produced for many years in high volumes and secure the regular utilisation of Inmatec's production facilities.

### Standard feedstocks

Of course, such long development cycles are not economically feasible for every product. Inmatec, therefore, also offers standard feedstocks for the most common materials, which are held in stock and can be delivered at short notice. These are listed in Table 1. All standard feedstocks are designed for solvent debinding in water. The optimum processing temperature is approximately 150-160°C and the mould should be heated to 50 to 65°C. According to Dr Karin Hajek, Inmatec's Sales Director, customers who have used standard feedstocks successfully for some time show a clear preference for the standard grades. The present annual consumption of standard grade alumina powder by Inmatec is 30 metric tons.

It is not easy for a feedstock manufacturer to maintain consistent feedstock quality even for standard grades, because of the variations in the characteristics of commercial powders. Ceramic powder suppliers generally show little interest in complying with the specific requirements of the CIM industry since

	Ceramic powder	Colour	Particle size d <sub>50</sub>	Theoretical density	Sintering
INMAFEED K1008	Al <sub>2</sub> O <sub>3</sub> , 96%	white	1.8 -2.2 µm	3.80 g/cm <sup>3</sup>	1620°C/air
INMAFEED K1010	Al <sub>2</sub> O <sub>3</sub> , 99.7%	white	1.0 -1.5 µm	3.86 g/cm <sup>3</sup>	1680°C/air
INMAFEED K1013	Al <sub>2</sub> O <sub>3</sub> , 99.8%	ivory	0.5 µm	3.94 g/cm <sup>3</sup>	1600 -1650°C/air
INMAFEED K1021	Al <sub>2</sub> O <sub>3</sub> , 99.99%	white / translucent	0.4 -0.6 µm	3.99 g/cm <sup>3</sup>	approx. 1800°C/H <sub>2</sub> for translucent appl.
INMAFEED K1009	ZrO <sub>2</sub> 3Y (Tosoh)	white	0.6 µm	6.05 g/cm <sup>3</sup>	1400 -1450°C/air
INMAFEED K1012	ZrO <sub>2</sub> 3Y (Tosoh)	white	0.6 µm	6.05 g/cm <sup>3</sup>	1500°C/air
INMAFEED K1015	ZrO <sub>2</sub> 3Y (Tosoh)	black	0.4 µm	6.00 g/cm <sup>3</sup>	1400 -1500°C/air
INMAFEED K1016	ZrO <sub>2</sub> 3Y (DK)	black	0.5 - 0,7 µm	5.90 g/cm <sup>3</sup>	1400°C/air

Table 1 Standard feedstock grades offered by Inmatec

volumes are much lower than in the die pressed ceramics industry. For cost reasons, Inmatec has to rely on commercially available powders and it modifies them according to its requirements if necessary. "We expend immense efforts to make sure that our processes are constant and reproducible," stated Karin Hajek. "A Quality Management commissioner has been appointed whose exclusive task it is to supervise, maintain and improve the QM system and carry out regular quality audits."

Hajek also announced that Inmatec will present a new binder system at this year's Ceramitec exhibition. Feedstocks, based on this proprietary binder system, will be marketed under the trade name INMAFLOW. They will be characterised by excellent shape retention, a wider processing window and shorter debinding times than the standard grades available today. Technical innovation is regarded as the best way to secure future business and the new binder system is a significant step in this direction. Since the processing of these feedstocks is more robust than before, more users and new applications are anticipated in the future.

### Quality management

The Quality Management (QM) system of Inmatec is certified according to the ISO TS 16949 standard. This is currently the strictest requirement that qualifies for deliveries to the automotive industry. The ISO TS 16949 standard ensures that reference samples are retained after each processing step and kept for ten years and requires that all processes are efficiently controlled and documented. A large amount of processing parameter data are recorded electronically and continuously; other data are typed into the system by the operators.

The most important test during the incoming inspection of powder, according to Daniela Raab, is the weight loss of the powders by annealing in air at 1025°C, according to ISO 26845. The weight loss determines the content of organic

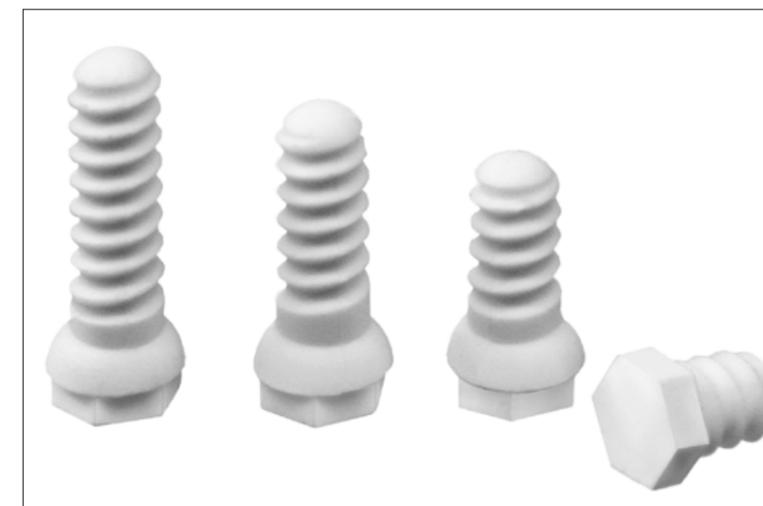


Fig. 6 Biocompatible alumina bone screws (Courtesy Kläger Spritzguss)

substances in the powder. After feedstock processing, the weight loss of the feedstock is measured for a reverse cross check of the powder-binder ratio. Each feedstock batch is injected with reference parameters and a standard mould to monitor batch-to-batch consistency. Injection moulded parts are also retained as reference samples.

The standard quality assurance procedure can be extended to an entire incoming goods inspection that is conducted for the customer at Inmatec. Each delivered product is accompanied by a quality certificate with the data determined by quality control on the respective production lot. "For some customised feedstocks, the shrinkage and the mould oversize factor are specified within very close limits," stated Raab, "and we are able to reproduce the mould factor within the third decimal. We think that rheometer measurements are not very meaningful for our purposes, but, if a customer asks for them, we will comply. We can perform most tests in-house and rely on accredited laboratories for special tests."

### Customer support

The relationship between a feedstock supplier and the consumer is much closer than between a parts producer and their customer. If a quality problem arises in the production

process of a CIM parts manufacturer, the feedstock supplier is directly involved and must support the parts manufacturer in the analysis of the cause of trouble, beginning with the verification of its own raw materials and processes and continuing with the detailed analysis of the customer's production. This is only possible if a trusting relationship has been established and if the customer is truthful and provides deep insight into its processes and internal procedures.

Inmatec sees the consulting services that are provided to customers for the development of new Ceramic Injection Moulded products as a part of its business, which is largely independent from the daily production of standard and customised feedstocks. Customers are welcome at Inmatec, independent of their level of expertise or equipment for CIM production.

Complete beginners are advised about the equipment required and trained in handling the feedstocks properly. Inmatec can order the design and manufacture of the tooling, if requested by the customer, and has facilities for first sample manufacturing, but the regular production of CIM parts is not considered by Inmatec because it would interfere with the interests of its customers.



Fig. 7 A CIM functional ceramic heating element for the inset cordless glue gun (Courtesy Steinel)

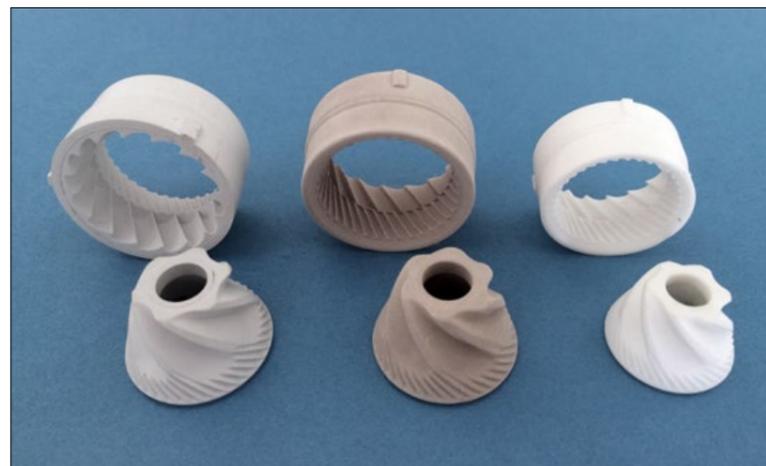


Fig. 8 Salt and pepper mill grinders (Courtesy Inmatec)

## Research & development

Product development is strictly customer-oriented. Based on customer enquiries, special materials, feedstocks, tools and processes are developed for new applications. Trends in product development are both the miniaturisation of mechanical systems and the manufacture of larger CIM components. New products made from functional ceramics are also under development.

Inmatec has also significant activities in relation to long-term CIM research. This is mainly done through cooperation in national and international research projects. Inmatec qualifies for such projects as an SME, which is at the very start of the CIM process chain.

Research is very much a third business area for Inmatec. For all research projects relating to the development of ceramic products with complex shapes by injection moulding, co-injection moulding, extrusion or other new shaping technologies, Inmatec is often the first port of call for ceramic feedstock. "We can only support our customer's new developments successfully if we ourselves are at the forefront of CIM technology development," stated Karin Hajek. "This is not limited to the raw materials aspect of the process, but also includes a full understanding of, for example, advanced tooling, debinding and sintering technologies." Currently one international EU project and

five national research projects are under way with the participation of Inmatec.

## Engineering ceramics on the rise

Numerous examples of innovative CIM applications demonstrate the growing demand for complex products made of engineering ceramics.

### Medical applications

Ceramics are increasingly used in medical technology for implants. An example of ceramic screws used for fixing implants to the bone is shown in Fig. 6. The screws combine high mechanical strength and durability with biocompatibility. Other applications of CIM products in medical technology are tools for micro-invasive surgery. Currently, one of the most widely used materials for structural applications in medical technology is alumina. In the future, many more applications for functional ceramics are expected in medical technology for innovative methods of surgical intervention, medical diagnostics, therapy and patient monitoring.

### Battery powdered glue guns

Consumer product manufacturers can also take advantage of ceramic products, as shown in Fig. 7. The first glue gun powered by a rechargeable battery was made possible with a heating element made of a functional PTC ceramic. The initial design is shown here which has a base diameter of 10 mm, an opening of 3 mm diameter and a wall thickness of 1 mm. The wall thickness has since been reduced. This element heats extremely quickly and reduces power consumption by 90%. Ceramic Injection Moulding technology enabled the production of an element with minimal wall thickness. More innovative ceramic applications in consumer products have the potential to contribute to a significant reduction in energy consumption by households.

### Salt and pepper mills

Salt and pepper mills are equipped with grinders of fairly complex design. Fig. 8 shows examples of this product in the green (left), brown (middle) and sintered (right) states. These components were initially developed as MIM products. However, these are now partially being replaced by ceramic parts.

### Coffee machines

Many modern household coffee machines use coffee pods whose aluminium capsules are pierced with a ceramic needle (Fig. 9). The alumina part is resistant to wear and abrasion, stays sharp and does not corrode which is of particular importance in food and beverage processing.

### Translucent dental brackets

An example of a translucent alumina product, namely dental brackets, is shown in Fig. 10. These brackets are almost invisible and therefore aesthetically much more attractive than metal brackets.

## Opportunities and obstacles for CIM growth

Karin Hajek outlined the business opportunities of CIM technology and obstacles to a faster growth from the perspective of a ceramic feedstock supplier. She regretted that many design engineers are not yet fully aware of the numerous advantages of engineering ceramics and, therefore, do not exploit CIM technology as much as they could. The misconception of ceramic materials, as being brittle like glass and therefore posing an incalculable risk in structural applications, has been disproven many times, but is still present in the minds of many engineers. "Ceramic materials cover a wide range of properties," she stated, "and CIM has proven to be a cost-efficient mass production technology that can be used to process almost any ceramic powder."

Standard ceramics are characterised by extremely high hardness and wear resistance,



Fig. 9 Ceramic Injection Moulded coffee pod needle (Courtesy Kläger Spritzguss)



Fig. 10 Dental brackets made from translucent alumina (Courtesy Inmatec)

combined with corrosion and high temperature resistance. They are electrically isolating, biocompatible and resist most chemicals. However, the world of engineering ceramics is indeed much more diverse than the average engineer with a general interest in materials science would imagine. In addition to the most widely known structural ceramics, such as alumina, zirconia, silicon carbide, silicon nitride and boron nitride, there is a wide variety of functional ceramics with special properties for high-tech applications. These include piezo ceramics and materials that change their electrical resistivity and other properties with temperature, the so-called positive and negative temperature coefficient ceramics (PTCs and NTCs).

Most ceramic materials are difficult to machine due to their high hardness. Ceramic Injection Moulding, however, allows the production of complex ceramic components, not only from standard ceramic materials, but also from functional ceramics. CIM, as a net shape technology, offers almost 100% raw material utilisation and can help to reduce the amount of machining dramatically. Many complex shapes are only viable with CIM technology. "This potential," stated Hajek, "is not fully recognised by major end-users of these materials who continue to design only for simple shapes such as platelets or rods, although a more complex design with other shapes would be technically advantageous and commercially attractive."



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Fig. 11 CIM zirconia watch parts for the luxury market (Courtesy Inmatec)

Besides exhibiting at the forthcoming Ceramitec exhibition in Munich, Germany, from October 20-23, which is considered as the most important event for the European ceramics industry, networking in professional organisations is another important source of new projects. Germany's DKG trade association, Europe's ECERS and the German language MIM and CIM Expert Groups are highly valued as places where Inmatec can meet and network with materials and equipment suppliers, research organisations and parts manufacturers. Such groups enable a pooling of resources to move CIM technology forward, as a company such as Inmatec does not, by itself, have sufficient resources for widespread technology marketing activities.

#### Aesthetic ceramics

The focus of Inmatec is, instead, on further improving feedstock quality and technical innovation. Aesthetic ceramics, with white zirconia leading the way, are of significant interest in this respect (Fig. 11). Since the ceramic powder and binder inevitably come into contact with metallic components in the compounding process, the chances are that abrasion of metal particles will

contaminate the feedstock. This can result in undesired dark spots on aesthetic parts, which leads to rejection. Inmatec has therefore modified all of its equipment so that contact of the ceramic powder and feedstock with metal is minimised. Karin Hajek stated, "We have made great progress in reducing the reject rates of aesthetic ceramics produced by our customers with our feedstocks and are still working on further improvement. Today, Inmatec probably delivers the whitest zirconia feedstock in the industry."

#### Translucent ceramics

Translucent alumina is at least as demanding on feedstock purity as white zirconia. The feedstock for translucent alumina is made from ultra-pure alumina powder of minimum 99.99% purity and an average particle size D50 of 0.4 µm. The entire processing route must be strictly controlled so that any contamination with metallic particles is absolutely prevented. The sintered products must be free of porosity and the grain size should be smaller than the wavelength of visible light, i.e. 400 nanometres. When these conditions are met, alumina can be almost as transparent as glass.

#### Future trends

The diversity of ceramic materials, covering a broad range of properties and novel processing technologies for complex shapes that could not be imagined before, opens vast opportunities for the future. Functional ceramics are expected to find significantly more applications in the next five to ten years. The development of new products will be accompanied by steadily growing demands on ceramic powders with tighter technical specifications. Integrating two or more different materials in one product by multi-component injection moulding, or the combination of extrusion and injection moulding, will become standard processes. Additive Manufacturing is just beginning to be used on an industrial scale and its future potential can hardly be estimated currently.

According to Karin Hajek, ceramics can provide a significant contribution to meeting the challenges of the growing world population, environmental pollution, global warming and demographic change in industrialised countries. "Not only is CIM a raw material and energy saving manufacturing technology itself, but CIM products with their unique combination of properties are also suitable for technical innovation that helps solve many of the challenges of our time," she concluded.

#### Contacts

Inmatec Technologies GmbH  
Dr Karin Hajek  
Dipl.-Ing. Daniela Raab  
Heerstrassenbenden 10  
D-53359 Rheinbach, Germany  
Tel: +49 2226 9087 41  
[karin.hajek@inmatec-gmbh.com](mailto:karin.hajek@inmatec-gmbh.com)  
[daniela.raab@inmatec-gmbh.com](mailto:daniela.raab@inmatec-gmbh.com)

#### Author

Dr Georg Schlieper  
Gammatec Engineering GmbH  
Mermbacher Strasse 28  
D-42477 Radevormwald, Germany  
[info@gammatec.com](mailto:info@gammatec.com)

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## Ceramic Injection Moulding adds to the allure of luxury watches

Recent years have seen an increasing number of leading luxury watchmakers embracing Ceramic Injection Moulding (CIM) for watchcases, bezels, bracelet segments and even movement parts, adding a significant premium to their already successful metal based timepieces. The development and application of new types of ceramic powders, binders, and processing equipment for CIM watch components has also added to the sector's success. Bernard Williams, Consulting Editor of *PIM International*, reports on recent CIM applications from the luxury market.

Ceramic Injection Moulded zirconium oxide ( $ZrO_2$ ) was first developed for luxury watchcases and bracelet segments in the mid-1980s by Rado Watches of Lengnau, Switzerland (today part of the Swatch Group) and IWC Schaffhausen, also based in Switzerland. The work that went into developing ceramic watches paved the way for the introduction of the first series CIM production models for IWC and Rado in 1994. IWC introduced the Da Vinci, said to be the most complicated wristwatch ever produced in ceramic at that time, and Rado introduced the Integral.

Since then the luxury watch industry has fully embraced all that Ceramic Injection Moulding has to offer, whilst, at the same time, making significant technical advances in terms of quality, performance and the diversity of surface finishes and colours that can be achieved.

CIM watch components are, in the main, produced using ultrafine tetragonal zirconia powders although other types of ceramic powders such as silicon nitride, titanium nitride

and boron carbide, have also been introduced. The ceramic powders are blended with a binder, homogenised and granulated to produce the CIM feedstock. Following injection moulding, the components go through binder removal stages and are then sintered in dedicated sintering furnaces. After sintering, the CIM

parts can be polished to give a satin or gloss finish.

The chemically derived ultrafine  $ZrO_2$  powder, first produced commercially by Tosoh Corporation in Japan in 1983, shows very good sintering activity and, with appropriate powder processing, it can be readily sintered to practically theoretical



Fig. 1 IWC Schaffhausen's Da Vinci Chronograph Ceramic uses a combination of high-tech ceramic zirconium oxide and titanium (Photo IWC Schaffhausen)

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Fig. 2 IWC Schaffhausen's Ingenieur Constant Force Tourbillon combining platinum and ceramics (Photo IWC Schaffhausen)

density. The sintered material exhibits an unusual combination of several favourable properties; it shows high strength, high toughness, high hardness, low thermal conductivity, a high volumetric density and a high refractive index [1]. The high strength of sintered zirconia is said to be associated with the very fine crystal-

*“The sintered material exhibits an unusual combination of several favourable properties; it shows high strength, high toughness, high hardness, low thermal conductivity, a high volumetric density and a high refractive index”*

lite size of the sintered material, usually not more than 0.3 - 0.5 µm, allowing a four-point bend strength of 1000 MPa or more.

Significant progress has been made in debinding technology for ceramic watch parts in recent years. In addition to the catalytic debinding approach developed by BASF, which shows excellent performance in terms of extremely short debinding



Fig. 3 Brown ceramics used by IWC Schaffhausen for its Pilot's watch Chronograph Antoine de Saint-Exupery (Photo IWC Schaffhausen)

times, thermal debinding and water or solvent debinding are also used. The solvent debinding process has been widely adopted in the CIM sector in recent years where a portion of the binder is chemically removed using solvents such as acetone, trichloroethane, heptane and even water. The open porosity created in the ceramic

component after solvent debinding allows the degraded binder products to diffuse easily to the surface of the parts.

LÖMI of Aschaffenburg, Germany, an equipment manufacturer specialising in solvent debinding, states that it has seen a significant increase in demand for its solvent debinding furnaces over the past few years including CIM manufacturers

specialising in producing parts for watches. The company states that these new furnaces offer manufacturers the flexibility to use various types of feedstock systems, making it easier to test and use new CIM feedstocks without having to invest in a new debinding furnace [2].

Black zirconia parts have become standard for many CIM watches. However, a number of higher end watch producers are turning their attention to incorporating other colours such as red, blue, orange, white, grey, brown etc. For coloured CIM zirconia parts, the material has to be pigmented, with pigments being mixtures of several transition metal oxides such as iron, cobalt, nickel, manganese and chromium. The key to the selection of colouring pigments is that they must survive the high sintering temperature without evaporating or melting

**Recent and noteworthy CIM watch developments**

This review highlights just a few recent developments by various manufacturers that illustrate the state of the art in CIM watch applications.

**IWC Schaffhausen**

IWC Schaffhausen has, over the past twenty years, significantly expanded its range of ceramic watch cases and bracelet parts. The latest Da Vinci ceramic watch is housed in a distinctive 44 mm wide tonneau-shaped case (Fig. 1). IWC Schaffhausen also uses CIM cases for the Ingenieur Constant Force Tourbillon (Fig. 2), the Ingenieur AMG and the top-of-the-range Luminor 1950 brands.

Recently, a novel brown ceramic watch case was introduced for IWC Schaffhausen's special limited edition Pilot's Watch Chronograph Antoine de Saint-Exupery (Fig. 3). Here the watch case is made from a mixture of silicon nitride and titanium nitride powders with the material sintered at 1800°C in a nitrogen inert gas atmosphere, using overpressure during sintering to attain full density. The combination of the two ceramic materials is said to make the silicon nitride even harder and more shatterproof than most other ceramics, but its increased hardness does require more effort in subsequent finishing steps such as grinding and polishing.

**Rado**

Since Rado released its first mass-produced ceramic watch, the Integral, in 1994 (Fig. 4) Ceramic Injection Moulding has become a proprietary feature of all Rado watches. In addition to featuring the inaugural black zirconia, the company now includes ceramic watches in pink, turquoise, burgundy, blue, purple and metallic colours. Surface treatment can give the ceramic material a metallic brilliance or particular colour such as platinum.

To achieve this, Rado has developed and patented a plasma carburising process where gases activated at 20,000°C alter the surface composition and surface colour of the ceramic without affecting its essential properties such as scratch resistance. The result is a striking metallic brilliance without the use of any metal (Fig. 5).

In 2014, Rado introduced a world first to the watchmaking world, a high tech injection moulded ceramic



Fig. 4 The Integral was the first Rado ceramic watch to be produced in series (Photo Rado Watch Co. Ltd.)



Fig. 5 The Rado HyperChrome XXL Chronograph uses plasma surface treatment to achieve the platinum colour effect on ceramics (Photo Rado Watch Co. Ltd.)



Fig. 6 Rado's Esenza Ceramic Touch is controlled by the press and sweep of a finger alongside of the case (Photo Rado Watch Co. Ltd.)

watch with touch functions. The Rado Esenza Ceramic Touch (Fig. 6) is controlled by the touch and sweep of a finger alongside the ceramic case, left for hours and right for minutes. Another application of CIM at Rado is the Diamaster Skeleton with black ceramic case and strap which has been produced in a limited edition of just 499 pieces.

In 2015, Rado turned to brown for its new range of HyperChrome timepieces. The company uses both polished and matt chocolate brown coloured ceramic for the cases and bracelet parts using Ceramic Injection Moulded ultralight silicon nitride (Fig. 7). For the Limited Edition Rado HyperChrome Automatic Chronograph Tachymeter, the company's



Fig. 7 Rado turned to silicon nitride for the ceramic case and bracelet used on the new limited edition HyperChrome Automatic Chronograph introduced in 2015 (Photo Rado Watch Co. Ltd.)

designers have contrasted the matt brown high tech ceramic case with a polished rich brown bezel, engraved with a tachymetric scale and filled with white Super-Luminova.

**Rolex**

Rolex, which has workshops at Plan-les-Ouates on the outskirts of Geneva, adopted black ceramic bezels in 2005 and introduced its exclusive



Fig. 8 A world first for Rolex, a red and blue ceramic bezel on its Oyster Perpetual GMT-Master II (Photo Rolex SA)

Cerachrom bezel inserts in blue and green in addition to black in 2007. In 2010, Rolex developed a monobloc Cerachrom bezel, rather than a bezel insert, for the Cosmograph Daytona timepiece. In 2014 the company introduced a red and blue Cerachrom bezel (Fig. 8) for its Oyster Perpetual GMT-Master II, echoing the first GMT-Master bezel of 1955.

The red and blue bezel is claimed

to be a significant technical achievement on two counts. Red is a difficult colour to obtain by Ceramic Injection Moulding and, having succeeded in doing so, Rolex then found a way to locally modify the chemical composition of each grain right to the core of the ceramic. In this way, colour changes from red to blue from one half to the next ensuring a sharp delineation between the two colours.



Fig. 9 Omega's Seamaster Planet Ocean Orange Ceramic in a brushed and polished 43.5 millimeter case in 950 grade platinum (Photo Omega SA)



Fig. 10 Omega's Speedmaster Grey Side of the Moon has a ceramic case, matt black ceramic dials and ceramic clasp (Photo Omega SA)



Fig. 11 Hublot's 'Magic Gold' watches are produced from porous boron carbide impregnated with gold (Photo Hublot SA)

This is done before sintering so that, during the course of the sintering step, the added compounds react with the basic elements of the red Cerachrom insert to produce the final blue colour.

**Omega**

Another breakthrough with coloured ceramics in 2014 came from Bienne-based Omega (part of the Swatch Group) with the introduction of the company's Seamaster Planet Orange Ceramic (Fig. 9). This features a bi-directional rotating bezel with 24 hour gradations on a polished ring made from orange ceramic. Technical ceramics have in the main been restricted to black, white or pastel colours with bright colours such as orange missing. Omega stated that it took two years of development and no less than 3,500 hours of stability tests to obtain the orange colour. This was primarily due to problems with the pigmentation of intense colours, but the company persevered and the desired result was achieved using thermal treatments.

Omega produces its Speedmaster Dark Side of the Moon in black zirconia ceramic and the Speedmaster Grey Side of the Moon watch



Fig. 12 The Chanel J12-365 in black ceramic with steel accents and diamond inlay (Photo Chanel SA)

uses a white ceramic, which, after plasma treatment, attains a unique metallic grey look (Fig. 10). A feature of the Grey Side of the Moon is its Super-LumiNova tachymetric scale on the brushed ceramic bezel.

**Hublot**

Hublot, Nyon, Switzerland, has been using Ceramic Injection Moulding since its Big Bang watches were introduced in 2005, featuring polished and satin black and white zirconia as well as developing new colours such as bright red and yellow. In 2012, the company introduced a new patented 'Magic Gold' alloy on its Big Bang Ferrari Limited Edition timepiece.

The new material was developed in partnership with the Swiss Federal Institute of Technology (EPFC) in Lausanne and is said to be a fusion of 24 carat gold and boron carbide. The boron carbide powder is injection moulded to the required preform shape which is then debound and sintered to create a rigid porous structure. After sintering, 24 carat gold alloyed with 3% molten liquid gold is injected under high pressure with inert gas at high temperature into the porous boron carbide allowing the metal to fill the ceramic



Fig. 13 Gucci uses black ceramic for its G-Chrono range (Photo Guccio Gucci S.p.A)

pores and creating a 'fusion' of the two to produce Magic Gold (Fig. 11).

**Chanel**

Fashion house Chanel began using ceramics in 2000 and the company has been credited with making ceramic watches fashionable through the use of black zirconia in the iconic J12 watch. White ceramic was introduced by Chanel in 2002. Currently the fashion brand uses Ceramic Injection Moulding not only for watch cases and bracelet parts, but also in the mechanisms. Some Chanel models use a ceramic rotor and ceramic tourbillions, the latter placed on a ceramic plate instead of brass.

The latest J12-365 watch case is available in either black or white, depending on the version, and these are accented with steel or beige gold, with or without diamond inlay (Fig. 12). The new shade of 18 carat gold is obtained through an alloy development at Chanel. For its Chromatic line of timepieces, Chanel developed a cermet material based on titanium and ceramic, which, when polished with diamond powder, makes the surface shine in an unique way.



Fig. 14 Blancpain uses brushed ceramics for the case and bezel of its Bathyscaphe Flyback watch (Photo Blancpain SA)



Fig. 15 The Royal Oak Offshore Diver collection showcases Audemars Piguet's use of white ceramics (Photo Audemars Piguet)



Fig. 16 Officine Panerai's Radiomir 1940 3 days Ceramica watch (Photo Officine Panerai)



Fig. 17 Corum's latest Golden Bridge watches feature slender black ceramic curved cases (Photo Corum)



Fig. 18 The first Bell & Ross watch with a ceramic case, the BR01, introduced in 2014 (Photo Bell & Ross)

**Gucci**

Gucci uses a black ceramic case for its G-Chrono range using a smaller dial, measuring 38 mm rather than the original 44 mm (Fig.13).

**Ralph Lauren**

The Ralph Lauren Sporting Chronograph Black Ceramic was the first Ralph Lauren watch with a case made of ceramic zirconia and which contains a mechanical movement from Jaeger-LeCoultre.

**Blancpain**

Blancpain, also part of the Swatch Group and located in Le Brassus in the heart of the Swiss Jura, uses a 43 mm black satin brushed ceramic case and bezel for its Bathyscaphe Flyback watch (Fig. 14). The ceramic unidirectional rotating bezel bears hour-markers filled with Liquid-Metal®, enabling a perfect bond with the ceramic bezel. This combination offers exceptional scratch resistance.

**Audemars Piguet**

The family owned company Audemars Piguet, also located in Le Brassus, uses black and white 'Super

Ceramics' for the latest in its range of Royal Oak Offshore Diver (Fig. 15) and Chronograph collections. White CIM parts include the band, bezel, case middle, pushers and crown. The case and band parts are polished and satin-brush finished.

**Officine Panerai**

The latest version of the Radiomir watch, from Officine Panerai, Neuchatel, Switzerland, is its Radiomir 1940 3 Days Ceramica, which has a matt black ceramic case and a minimalist dial inspired by the brand's history as a supplier of timepieces to the Royal Italian Navy. The case and bracelet are made from matt black zirconia, and the case measures 48 mm in diameter, and offers water resistance to 100 metres (Fig. 16).

**Corum**

Corum, La Chaux-de-Fonds, Switzerland, is celebrating the 35th year since it began producing the now emblematic Golden Bridge timepiece, and the 10th year since the company re-designed the watch. The latest Golden Bridge and Miss Golden

Bridge timepieces now incorporate black zirconia curved tonneau shaped cases (Fig.17) combined with a unique caseback treatment introduced in 2014. The caseback becomes opaque when worn but appears clear when off the wrist.

**Bell & Ross**

Bell & Ross introduced its first watch with a ceramic case, the BR01 Ceramic, in 2014 (Fig. 18). The black ceramic case is 42 mm in diameter and is close to 50 mm in length with relatively short lugs. The case design is based on the older Bell & Ross classic pilot timepieces. It is also available in white ceramic for ladies' timepieces.

**Other brands**

Several other leading brands have developed ceramic watches. Fendi's Ceramic Chronograph comes in various hues including blue, white, brown and black, whilst TechnoMarine offers its Cruise Ceramic in white or black ceramic with the option of rose gold or white diamonds on the bezel. Modus also offers fashion ceramic watches in white or black.

Roger Dubois, Geneva, uses black ceramics for the bezel on its Easy Diver Black Swan range and Ulysee Nardin recently introduced ceramics into its Executive Dual Time range of timepieces.

**CIM producers play key role in watch industry**

CIM producers have, of course, played a key role in the development of CIM parts for watches. Key among European CIM producers are Comadur, Hardex, Chatelain and Formatec, all of which manufacture for the leading Swiss watch brands.

**Comadur**

Comadur, based in Col-les-Roches, Switzerland, is part of the Swatch group. Swatch states that Comadur has played an important role over the past twenty years in the development and manufacture of new CIM watch parts for models including the Omega Speedmaster 'Dark Side of the Moon' and 'Grey Side of the Moon' models, the Rado DiaMaster and the new Rado True collection, the Esenza Ceramic Touch, the HyperChrome Court

Collection, the D-Star and the launch of the all ceramic Fifty Fathoms Bathyscaphe chronographs.

Comadur also produces sapphire crystals and watch stones for the movements manufacturer ETA, rare earth sintered micro magnets for quartz watches and micro motors and polycrystalline micro bearings used in watch movements or in a wide range of micro motors.

Comadur saw a marked increase in CIM production as a result of increased demand and is stated to have completed the internalisation of raw material (feedstock) production for Ceramic Injection Moulding. The company is now said to be completely independent in every phase of CIM production.

It has also established a production line to meet the growing demands for smaller volumes and ever-more demanding requests for new colours. The company will continue to upgrade production methods to allow faster set-up times, also optimising flows between the various manufacturing operations and improving product quality, particularly in the ceramic sector, with invest-

ment in new machines and renewal of production lines. The company already has over 20 Arburg Allrounder Powder Injection Moulding machines.

**Hardex**

Hardex, part of France's IMI Group, is based in Besançon, Franche-Comte, which borders the key watchmaking region of Switzerland. The company was established in 2006 to access the emerging markets for CIM components used in watches, as well as luxury consumer products such as pens and jewellery, drawing on more than 25 years' experience in the manufacture of ceramic parts under the name Cheval Freres.

Hardex operates a complete CIM production line, which includes post sintering and assembly operations. CIM is mostly used for medium volume production, whilst larger volume simple shapes are produced by die pressing and small batches by machining. The company buys in its CIM feedstock from external sources.

For post-sintering operations, Hardex is able to offer surface finishing such as high gloss polishing, brushed finishes, sandblasting, laser

engraving and PVD coating. These operations are essential for high-end luxury watch products where a perfect surface finish is essential. Examples of CIM parts produced by Hardex for the luxury Swiss watch market include black ceramic top ring and pushers for the Executive Dual Time

one of the most important suppliers of external parts to Switzerland's luxury watchmakers. The company was established in 1947, but has been wholly owned by Chanel since 1993. This, however, does not prevent it from working with a dozen or so other clients including Rado.

Today, the company's CIM production is finely honed and R&D continues on the development of new finishing processes to create new surface renditions such as matt ceramics.

White, black and grey are currently the most widespread grades used by G&F Chatelain given the difficulties inherent in finding a colour that will withstand the high sintering temperatures.

*"G&F Chatelain began using Ceramic Injection Moulding in 2001 for the Chanel J12 watch and volume production of CIM components followed six years later"*

watch by Ulysse Nardin, the CIM zirconia top ring and back ring for the Black Swan watch by Roger Dubois and the rear case of the Easy Diver watch also by Roger Dubois [3].

**G&F Chatelain SA**

Despite its low profile, G&F Chatelain SA, based in La Chaux de Fonds in the Swiss canton of Neuchatel, is

G&F Chatelain began using Ceramic Injection Moulding in 2001 for the Chanel J12 watch and volume production of CIM components followed six years later. It now also includes movement parts such as ceramic ball bearings for the barrel, external watch parts, including cases, bezels and bracelet links, and ceramic jewellery rings.

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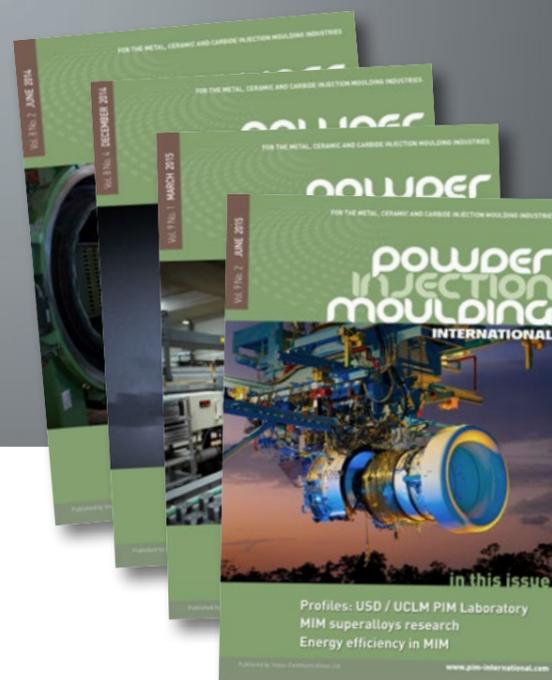


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## Micro Powder Injection Moulding: Processes, materials and applications

Micro Powder Injection Moulding (MicroPIM) continues to attract attention as a unique process by which to manufacture metal and ceramic micro components in medium to high volumes. In the following exclusive review for *Powder Injection Moulding International* magazine Dr Volker Piotter and Dr Tassilo Moritz present a status report on the technology, from binder developments to best practice in process simulation, injection moulding and sintering. A number of innovative application areas are also reviewed.

In recent years micro component manufacture by means of a replication process has become an important economic and technical field. Injection moulding in particular is used for the production of both individual micro components with typical dimensions of  $\leq 1$  mm as well as microstructured components, that is to say micro or even nanoscale structures on larger carrier surfaces. This is primarily due to the technology's high efficiency in medium or large series fabrication, the ability to create complex shaped pieces, and the wide range of processable materials. The latter issue in particular corresponds very closely to the trend towards further diversification in micro manufacture, most notably a tendency towards using more varieties of functional or high-strength materials. This trend can be attributed to the current extremely wide range of application areas for microsystems technology products [1, 2, 3, 4, 5]. From this

diversity, in turn, a wide range of requirements arise with regard to the materials to be used.

Silicon and thermoplastics are still used most frequently in microsystems technology. The demand for processes that allow the production

of large quantities of ceramic and metallic materials is therefore increasing. In this context, Powder Injection Moulding is a very attractive option as it has already been firmly established in macroscopic production [6, 7, 8, 9, 10].

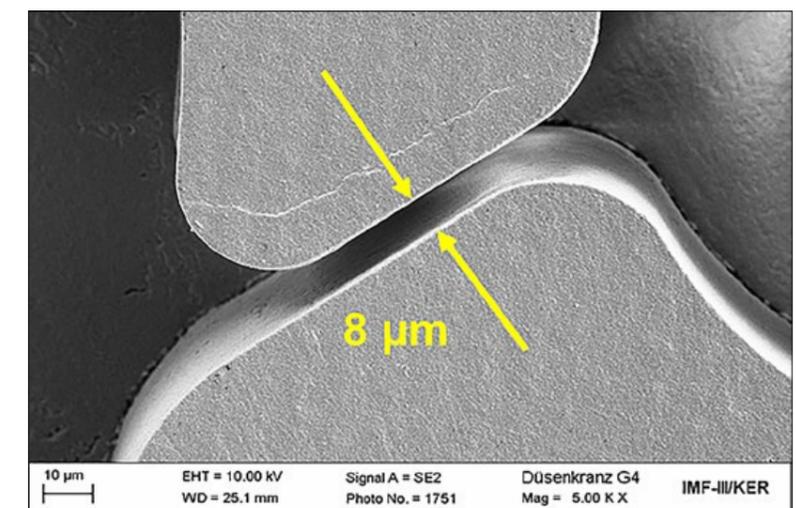


Fig. 1 A CIM microturbine stator manufactured from  $ZrO_2$ , demonstrating what can be achieved with MicroPIM technology [Courtesy KIT]

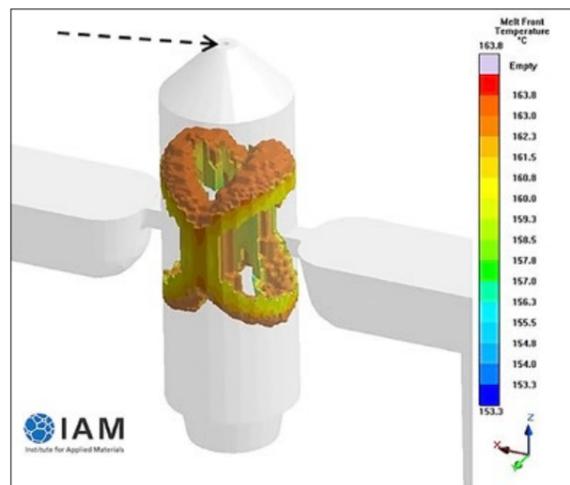


Fig. 2 Mould filling simulation of CFEL micro ceramic nozzle (outer tube with guiding features inside) showing prediction of flow front temperature to avoid distinct weld lines. Hole diameter at tip  $< 100\mu\text{m}$  (see dashed arrow)

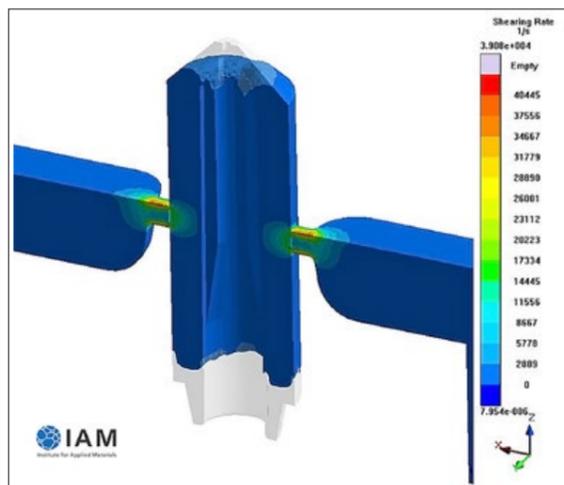


Fig. 3 Calculation of maximum shear rates to identify sections of probable powder-binder segregations

When moving from macro to micro dimensions, however, some points have to be considered. Micro component manufacture is characterised by:

- Higher surface-to-volume ratios
- Faster cooling and freezing of feedstocks
- Higher demoulding forces relative to the load-bearing cross-section
- Higher shear rates than in macroscopic runners
- Abrupt changes of runner sections.

### Unique aspects of Micro Powder Injection Moulding

#### Feedstocks

Micro Powder Injection Moulding (MicroPIM) makes great demands on both binder ingredients and feedstock composition. For example, particles in the micrometre or even sub-micrometre range are required in order to fulfil demands of MicroPIM component surface roughness, feature detail and mechanical properties. This extraordinarily small powder grain size, in turn, influences the composition of the binder systems. As a first consideration, feedstocks showing a very good flowability are required. Binder

systems influence this flowability and thus play an important role in the moulding process.

Additionally, the strength of green parts must be considered with MicroPIM. Due to their relatively large surface-to-volume ratios but small load-bearing cross-sections, the components are subjected to high forces during debinding.

As in macroscopic PIM technology, further factors of significance are, for instance, feedstock homogeneity, shelf-life and recyclability, easy and ecologically safe debinding, and controlled shrinkage behavior.

The most widely used binder systems are based on polyolefines and paraffin wax. Adaptation to the specific demands of MicroPIM is performed by optimisation of composition of binders and appropriate adjustment of the powder-binder ratio.

Moreover, the addition of surface-active substances (dispersants) to binders allows for a significant viscosity reduction and a much better feedstock homogeneity. To achieve total coverage of the powder particles with dispersant [11], the portion of additives ideally corresponds to the entire feedstock powder surface.

A major drawback of the polyolefine/paraffin system, however, is the fact that preliminary chemical

debinding in hexane is required in most cases. Due to its high flammability and environmental risks, hexane is poorly suited for use outside a laboratory environment.

Therefore, a new two-component binder has been developed at Karlsruhe Institute of Technology (KIT), Germany, that contains high-molecular polymethylmethacrylate (PMMA) as the structural polymer, low-molecular polyethylene glycol (PEG) as the plasticiser and dispersants as additives [12]. Since PEG dissolves readily in cold water, this binder is environmentally friendly. The approach was developed as an extrudable thermoplastic mixture (PMMA/PEG4000). In the meantime, the new binder concept has also been successfully implemented with polyvinylbutyral/PEG [13].

#### Powders

With reference to ceramic materials, zirconium and aluminium oxide powders are most commonly used in MicroPIM, with well established applications being fibre-optic ferrules and wire bonding nozzles. Nitride ceramics, such as silicon nitride, are still at an early stage of development. The primary metals utilised are PM stainless steels, such as 17-4PH and 316L, and

nonferrous metals, such as copper, titanium, tungsten and tungsten alloys.

MicroPIM mostly uses powders with ultrafine or low particle diameters. In the case of ceramic powders, typical  $D_{50}$  values are in the range of 500 - 800 nm for aluminium oxide and 300 - 500 nm for zirconium oxide. Moreover, several experiments have been performed to manufacture bimodal mixtures from conventional ceramic powders and extremely fine nanopowders to achieve higher powder contents and better contour moulding [14].

In contrast, steels have typical  $D_{50}$  values in the range of 1.5  $\mu\text{m}$  to 4.5  $\mu\text{m}$  and these values can also often extend into the range up to 10  $\mu\text{m}$  or above. For the major MicroMIM stainless steels 316L and 17-4PH, the finest fractions available on the market are  $< 5\mu\text{m}$ .

To ensure a sufficiently high powder content and acceptable feedstock flowability, powder particles are preferably globular. The spherical shape, moreover, largely prevents orientation effects in spite of the shear rates, which are generally higher in MicroPIM.

#### The shaping step

The rheological and thermodynamic properties of PIM feedstocks clearly differ from each other with regard to viscosity and thermal conductivity. These characteristics, among others, generate particular technological MicroPIM features, which, in certain cases, even differ from the micro injection moulding of polymers, which, in parallel, is also undergoing steady progress [15].

Importance has to be attached, for example, to the appropriate design of sprues and runners, gate sizes, numbers of cavities and the positioning of ejectors. In micromoulding, the general PIM design guidelines [16, 17] have to be observed at least as strictly as in the macroscopic world.

The accelerated formation of a cooled surface layer and the premature freezing of the gate that can be observed during injection, reduce the holding pressure time



Fig. 4 Micro-switch for development of flexible joints of single and multi-material combinations (left: ATZ/zirconia; right: steel 17-4 PH/zirconia)

with corresponding consequences in terms of part quality. To a certain extent, this can be avoided by choosing larger gate dimensions or by using hot runners for the sprue and runner system. In this context, it is important to note the so-called variothermal tempering, which, prior to injection, heats the mould core to temperatures near to the feedstock melting point and cools it down again

tools. A flexible tool design, ensuring an easy exchange of wear-relevant components, can improve this situation. In view of the use of rather soft tool materials, this applies to MicroPIM in particular.

A further important issue concerns dimensional accuracy, which is determined, among other factors, by the sintering procedure. To keep sintering shrinkage low, maximum

*“zirconium and aluminium oxide powders are most commonly used in MicroPIM, with well established applications being fiber-optic ferrules and wire bonding nozzles”*

after the filling step to enable ejection of a dimensionally stable part. A detailed description of this processing methodology is given in [18, 19]. The relevant micro-specific aspects, such as the variothermal tempering or the often applied tool evacuation, can be implemented using upgraded systems offered by some equipment manufacturers.

Surprisingly little is found in the literature with regard to the obvious fact that highly filled ceramic feedstocks induce higher wear on

powder content is sought. This is, however, limited by the consequently increasing viscosity of the feedstock. Therefore, the precise control of the physical effects occurring during sintering determines the exact dimensional tolerances. This issue presents a basic challenge for all sintering methods and, thus, also for MicroPIM [20].

To roughly outline the technical performance of MicroPIM, Table 1 lists several critical dimensions for different classes of materials. As is

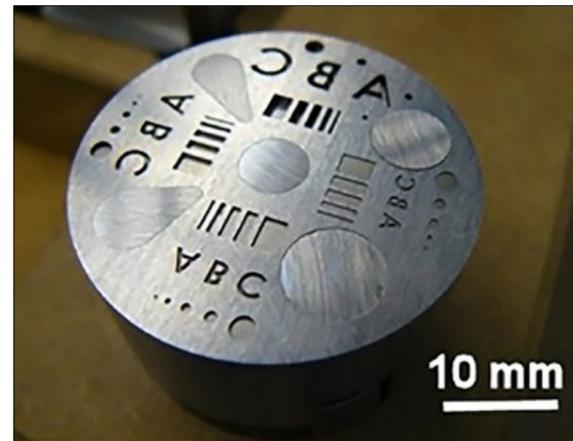


Fig. 5 Tool insert for micro in-mould embossing

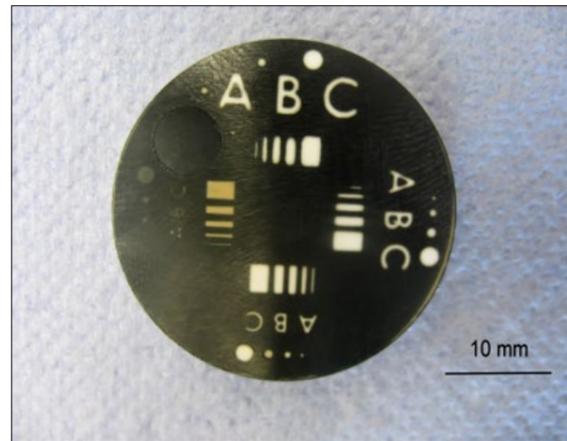


Fig. 6 Sintered zirconia disc consisting of a black zirconia tape embossed by injecting a white zirconia feedstock

evident from the parameters, the low particle sizes of the ceramic materials compared with those of the metal powders have a positive effect. The best moulding results, however, are achieved with pure polymers.

#### Simulation

Computer-aided simulation of manufacturing processes allows determination, prior to production, of the general applicability of certain methods and of the process parameters to be expected. It is due to this fact that simulation has already been used for many years in macroscopic engineering [21]. When applied to micro systems technology, it is important to consider that subsequent modifications of, for instance, LIGA moulding tools are virtually impossible, so design errors must be resolved at an early stage.

Several commercial software programs are available for the simulation of plastic processing, with leading examples being MOLDFLOW<sup>®</sup>,

Moldex 3D<sup>®</sup>, Sigmasoft<sup>®</sup>, and PIMSolver<sup>®</sup>. These programs all use the finite-element method, mainly applying volume model networks. However, it should be emphasised that the reliability of such simulations completely depends on an adequate materials model. Here the first difficulty occurs; although PIM feedstocks consist of at least two phases, most commercial programs use one-phase models. One consequence of this is that typical PIM effects, such as powder-binder segregation, cannot always be predicted adequately. As the launch of entirely new programs is costly, software developers usually modify their existing systems. An interesting approach in this context is to operate with a model which takes into account collision-induced flux and viscosity-induced flux. Such an approach has just been introduced by Sigmasoft and initial results are very promising, as typical PIM effects, such as plug-flow, can be predicted [22].

The entire modelling of all physical and chemical effects occurring during the different PIM steps, however, was and is still a matter of research [23, 24, 25]. Nevertheless, if the limitations described above are taken into account, the use of simulation tools allows mould filling processes to be analysed in advance to an acceptable degree. Additionally, important physical parameters, such as shear stress and shear rate, can be determined, leading to useful predictions as to the sections of the parts where calamities might occur.

A good example is given in a collaborative project between the Center for Free-Electron Laser Science (CFEL) at DESY, Hamburg and KIT, which was aimed at developing twin-tube ceramic nozzles to be used in experiments with X-ray free-electron lasers. Such nozzles are required to generate a strongly focused liquid jet of the analysis sample perpendicular to the beam of intense femtosecond pulses of X-rays (Figs. 2, 3).

Material	Minimum lateral dimensions [µm]	Min. details [µm]	Aspect Ratio <sup>a</sup> sunken structures	Tolerances <sup>b</sup> [%]	Surface roughness <sup>c</sup> R <sub>max</sub> /R <sub>a</sub> [µm]
Metals	25	10	> 10	≤ ±0.4	7/0.8
Ceramics	≤ 10	< 3	15	± 0.1 - ±0.3	< 3/0.2
Polymers	10	≤ 0.08	25	±0.05	0.05/0.04

<sup>a</sup> = AR, corresponds to flow path-to-wall thickness ratio    <sup>b</sup> = reproducibility of nominal size  
<sup>c</sup> = dependent on the type of mould insert

Table 1 Important parameters reflecting the present state of MicroPIM. The corresponding values for micro injection moulding with pure polymers are added for comparison

## Multi-component PIM

It is clear to see that the assembly of different individual pieces into complete systems is an elaborately complex, and thus costly, process. This is an especially important consideration in micro dimensions. If, in addition, one considers the fact that in microsystems engineering multifunctional components consisting of several components are also increasingly needed, it becomes evident that new process variants have to be developed.

Probably the most prominent example is the so-called two-component micro injection moulding process [26], where at least two different feedstocks are injected simultaneously, or at different times, to form a composite component during moulding. The compound parts obtained in this way consist mainly of a combination of materials with different properties that may well be opposed to one another, with examples being electrically conducting/insulating or magnetic/non-magnetic [27, 28]. Combining multi component injection moulding with MicroPIM, one obtains the 2C micro Powder Injection Moulding method (2C-MicroPIM).

The process procedure can be carried out in at least two principally different variants, depending on whether flexible or permanent joints are required. Permanent joints must have defined bonding surfaces, for instance, between gear wheels and shafts. To ensure that stresses and strain in the two components are reduced to a minimum, the material selection and process procedures must be adapted in such a way that the partial components are sintered almost simultaneously. This requires that all components are largely identical as regards the degree of shrinkage and the sintering kinetics.

For flexible joints, conditions are almost the reverse. The process parameters and material combinations must be selected in such a way that sintering of the inner section starts earlier than sintering of the outer section and that, in addition,



Fig. 7 Ceramic micro-caps with pressure sensitive ceramic membrane in the as-fired state prior to electrical conducting steps

the degree of shrinkage of the inner section is higher. Only under such conditions, and with an appropriate tool design, can a gap enabling sufficient rotary motion be formed between the gear wheel and the shaft [29].

For the development of flexible joints of micro components, a special testing tool has been constructed at Fraunhofer IKTS. The so-called micro-switch tool allows the

Fig. 4 shows a micro-switch made of the material combinations steel 17-4 PH/zirconia and ATZ/zirconia, respectively.

Another promising option for the combination of different materials by micro PIM is given by micro in-mould labelling (micro IML). For this approach, a thin ceramic or metallic green tape, made by tape casting, is inserted into the cavity of the injection moulding tool. Next,

**“By using this tool, flexible joints of a single material may be developed by combining either feedstocks with different shrinkages, or different materials, such as stainless steel and zirconia”**

sequential injection moulding of an outer C-shaped guiding component and an inner flexible micro-disc. The inner diameter of the guiding component is 2.5 mm. By using this tool, either flexible joints of a single material may be developed by combining feedstocks with different shrinkages, or different materials, such as stainless steel and zirconia, electrically conductive and electrically insulating ceramics or differently coloured ceramics can be combined.

the ceramic or metallic feedstock is injected and combined with the green tape. The injection step also offers the possibility to attain a microstructuring of the green tape by using the applied injection pressure of the feedstock and by a certain inner tool surface microstructure. This additional option of microstructuring is named in-mould labelling. A testing tool for this technology has been designed and constructed by Fraunhofer IKTS within a project funded by the German



Fig. 8 Testing components for injection moulded microstructures made of coloured sintered glass powders

Federal Ministry for Economic Affairs and Energy (KF2087326AG1). In this case, the green tape is embossed by the injection pressure of the feedstock and by a tool insert showing different alphabetic characters and geometrical shapes with different depths (100, 300, 500, 1000 microns) as shown in Figs. 5 and 6.

### Applications and case studies

Within the project CeraHOT (No. 100118509), funded by the Free State of Saxony, a pressure sensor has been developed which was covered by a ceramic micro-cap with a pressure sensitive ceramic membrane. This micro-cap was made of alumina with an outer diameter of 5 mm consisting of a supporting ring with an inner diameter of 1.25 mm and the pressure sensitive ceramic membrane with a thickness of only 250 microns. In further processing steps, this membrane was equipped with a thin electrical conductive platinum layer in an appropriate geometry for transforming the mechanical deformation of the membrane due to the pressure difference into electrical signals. The component was made by micro injection moulding and weighed only 85 mg. Fig. 7 shows the as-sintered micro-cap prior to grinding and platinum layer coating.

As well as ceramics, other materials, such as glasses, can be processed by micro injection moulding. As an example, micro-structured testing components made of coloured glasses are shown in Fig. 8. These components have been developed by IKTS and Inmatec Technologies GmbH in a project called Colorglass (KF2087345CK3, KF2166905CK3), funded by the German Federal Ministry for Economic Affairs and Energy.

### Outlook

MicroPIM provides the possibility of combining the material properties of metal and ceramic materials with the high economic efficiency of the increasingly established micro injection moulding method. The attractiveness of this symbiosis is mainly thanks to:

- A very high economic efficiency in the case of medium and large series production
- The current and future availability of a wide range of non-polymeric materials
- The possibility of using functional materials such as hard/ductile or insulating/conductive materials
- The fact that components with complex geometries can be

manufactured with little or no post-sintering treatment

- The opportunity of combining injection moulding with semi-products produced by other shaping techniques such as tape casting
- The commercial availability of production equipment.

The different binder systems available today for the development of feedstocks enable both the manufacture of ceramic and metallic microstructured components. Newly developed binders such as PMMA/PEG or PVB/PEG allow the use of water as a solvent for the chemical preliminary debinding and, hence, can be considered environmentally friendly.

Among the major trends in further micro Powder Injection Moulding development is the optimisation of process control at all process stages, particularly with regard to the reduction of demixing effects that are considered the main cause of deformations and dimensional imprecisions. In addition, efforts are being made to improve the dimensional accuracy of the sintered parts to below  $\pm 0.1\%$ . In this context, it is important, moreover, to mention the upgrading of existing software programs through implementation of MicroPIM-specific effects.

Another essential trend is the development of finer powders for MicroPIM, by providing feedstocks with additives of ultrafine or nanodisperse fractions, to obtain still smaller details and higher surface qualities.

Last but not least, there are different approaches to manufacturing multi-component micro parts, by for example conventional two-component PIM or methods such as in-mould labelling with PIM feedstocks (IML-MicroPIM) [30, 31], and sinter joining [32]. These approaches are not only economically efficient thanks to a reduction in assembly steps, but also provide multifunctional components which otherwise cannot easily, or in some cases cannot at all, be made by any other process.

### Authors

Volker Piotter  
Karlsruhe Institute of Technology (KIT)  
Institute for Applied Materials (IAM-WK)  
Hermann-von-Helmholtz-Platz 1  
76344 Eggenstein-Leopoldshafen  
Germany  
volker.piotter@kit.edu

Tassilo Moritz  
Fraunhofer Institute for Ceramic Technologies and Systems (IKTS)  
Winterbergstr. 28  
01277 Dresden  
Germany  
Tassilo.Moritz@ikts.fraunhofer.de

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## Additive Manufacturing of Ceramics: Opportunities and solutions for the CIM industry

The Additive Manufacturing of ceramics has the potential to not only transform the way the ceramic industry addresses prototype development, but also presents an opportunity to open up new applications that until now were impossible to produce with conventional ceramic technology. In this extensive review, Dr Johannes Homa and Dr Martin Schwentenwein from Austria's Lithoz GmbH present the state of the art in ceramic AM and review the technical and commercial considerations with specific relevance to the CIM community.

Additive Manufacturing (AM) is now well known amongst those involved in industrial manufacturing, as well as wider society, thanks to numerous magazine cover stories and huge media interest. A wide variety of applications has been reported, from weapons to organs and even entire buildings. While some of these applications might need a few more years to become a reality, the question is always raised as to whether AM will be the standard manufacturing process of the future.

AM technology has already had an enormous impact on a number of areas such as dental, medical, and aerospace technology. Additionally, budget 3D printers are available for the mass consumer market and their use could turn the traditional ways of manufacturing upside down. Consumers are now in the position to produce their own products, which some suggest could lead to a decentralisation of production. Some commentators claim that a new age has arrived and we will see a democratisation

of industrial production, where huge factories disappear and everybody manufactures the required goods at home. Will this really happen or is this scenario just a newspaper hoax or an excessively enthusiastic outlook of the future? Can AM replace all traditional

manufacturing technologies, which have been used and optimised over many decades and even centuries? The ceramic industry could perceive this as a threat because its traditional processes are so complex and elaborate.

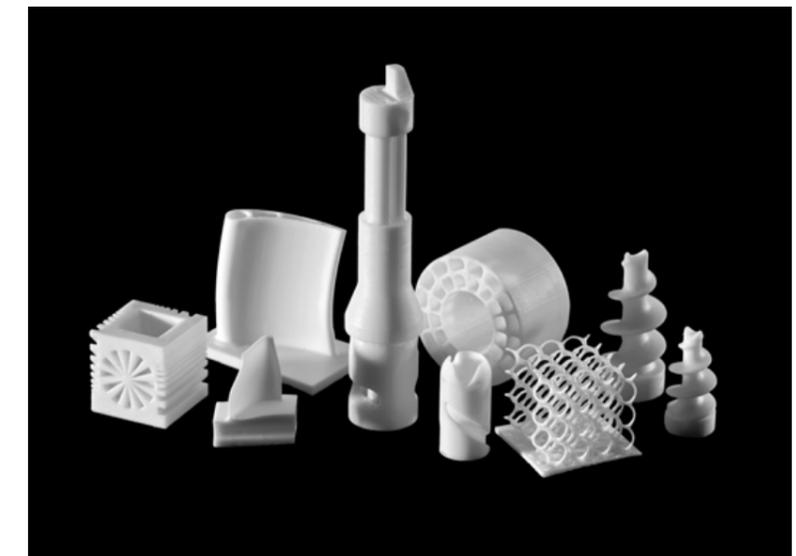


Fig. 1 A range of additively manufactured ceramic components produced on Lithoz's Cerafab system

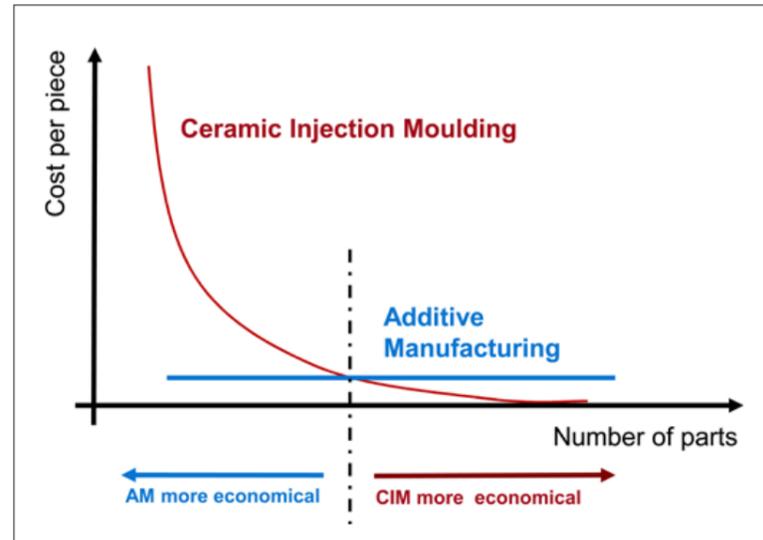


Fig. 2 Cost effectiveness for prototypes and small scale series

### The advantages of Additive Manufacturing

What is beyond dispute is that AM has a huge potential for the future and has many advantages compared to traditional ceramic forming

no mould is required to create the desired shape and the part is directly created from a CAD file. The machining process on the other hand doesn't require the usage of product-specific moulds, but it does take time to prepare the tool path for the CNC

*“What is beyond dispute is that AM has a huge potential for the future and has many advantages compared to traditional ceramic forming processes”*

processes. The major advantage of AM is that it is based on a tool-less production process. Unlike other ceramic forming processes such as injection moulding or slip casting,

machining and critically not every design can be machined.

A tool-less manufacturing process is well suited to prototype and small-scale series production because the

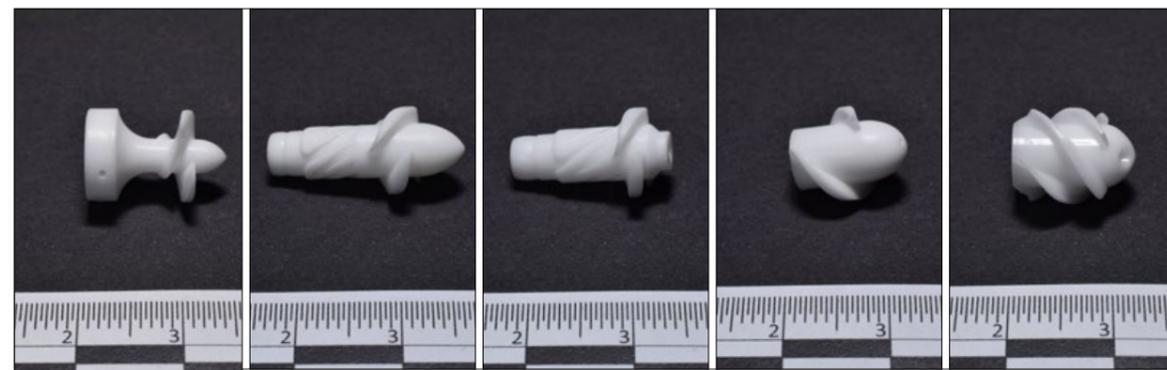


Fig. 3 Design alteration of the ceramic blood pump developed by Vienna University of Technology (Scale 2:1)

time and costs for the production of the tool can be saved. In particular, tools for Ceramic Injection Moulding are very expensive and have very long lead times. Consequently, in traditional forming, CIM is typically used when complex parts are required in large quantities, but it is very difficult for the process to make cost-effective prototypes or small scale series. Sometimes, however, it is necessary to have just a few parts in order to test an idea or for iterations to achieve an optimised design. In such cases, AM could be the method of choice and act as a supplement and 'door opener' for the CIM industry. As shown schematically in Fig. 2, the costs for AM parts are independent of their quantity and the costs for CIM parts decrease with increasing numbers. Consequentially, there is a break-even point up to which AM is more cost effective than CIM. This is, of course, an oversimplification and a number of different parameters need to be taken into account. However, it gives a first-order indication of the potential of AM. Additionally, the time for producing the mould, which might be up to ten weeks depending on design complexity and special requirements, could be saved. This increase in efficiency has the potential to significantly speed up the development and production cycle of a new product.

It is very difficult to determine this break-even point in general. There are two main drivers that play a crucial role. For CIM, it is the complexity of the part, which in turn determines the costs of the tool. For AM, it is the

size of the part. This determines how many parts can be built in parallel and how long it takes to finish one run. Different case studies have shown that the break-even point varies and might range from a few dozen to tens of thousands of parts, depending on the application.

However, all these considerations become irrelevant if the material properties of the AM parts do not comply with the characteristics of conventionally formed parts. Thus, the ultimate key requirement is to meet the quality criteria of conventional manufacturing processes. It only makes sense to use AM as an addition to traditional forming technologies if the material properties and the geometrical tolerances are sufficient. The technology could then support the development of new products because designs can be easily altered and the parts can be evaluated under real-life conditions. The first tests can be performed using different designs and feedback loops and new iterations can be implemented very efficiently. For example, the Vienna University of Technology (VUT) is currently developing a ceramic blood pump, where the design was subjected to more than 15 alterations [1]. As shown in Fig. 3, it is not only major design iterations that have been made, but also minor changes that were necessary to meet specific requirements during the different development stages.

### Embracing complexity

As already mentioned, another limitation of tool-based manufacturing technologies is the increasing cost with increasing design complexity. If the mould design is more complicated and consists of multiple individual parts, it will consequently become more expensive. As shown in Fig. 4, the costs per unit for AM, in contrast to CIM, are independent of the complexity of the part. Again, this statement is an oversimplification, because the costs are dependent on many different parameters such as the addition of support structures

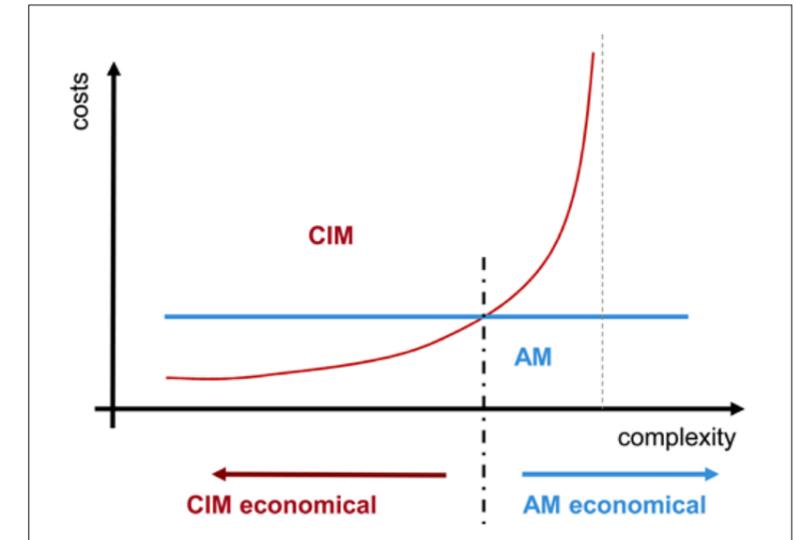


Fig. 4 Cost versus complexity for AM and CIM

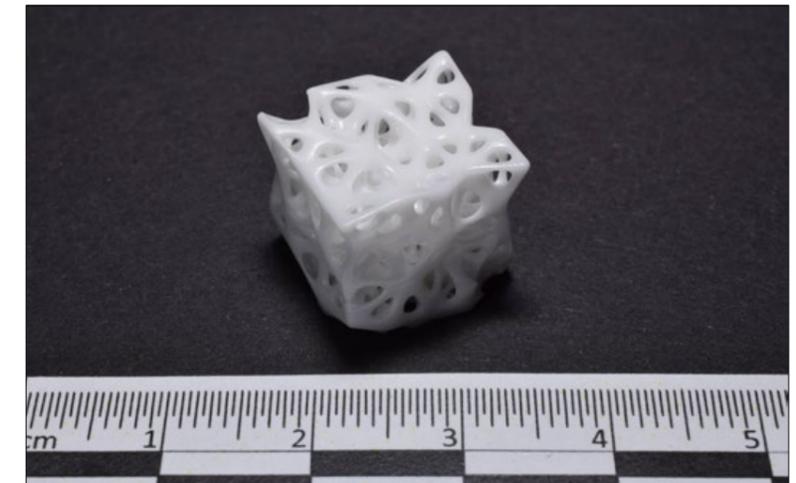


Fig. 5 Cellular structure made by AM

and the effort required in cleaning. However, in most cases, the influence of complexity on the costs of a given AM component is negligible when compared to conventional manufacturing techniques. Hence, there is again a break-even point in this model where the choice of using AM over CIM becomes more cost effective. This statement on its own is already an important finding, but much more interesting is the asymptote which can be observed for a certain complexity for CIM produced parts. It indicates that some designs are simply not feasible with tool-based manufacturing processes because of the unavoidable step of removing the part from the mould.

Even with subtractive methods, such as turning or milling, it is not possible to fabricate every required design.

A part such as the one shown in Fig. 5 cannot be produced by CIM or by machining, but only by AM. It is impossible to demould this part or to create the pore structure with a tool. Such a structure can however be used for scaffolds with defined pore geometry and size, catalyst carriers or lightweight structures. These are only the obvious applications, but there is much more potential in this new design freedom. Prevailing design limitations with conventional manufacturing technologies can be overcome and the design process can be focused on functionality.



Fig. 6 Blood pump with two bearing points which can only be produced by AM

Generally speaking, AM enables a paradigm shift from manufacturing-oriented design towards design-oriented manufacturing. This does not mean that AM has no design limitations at all, but it does allow the engineer to design differently. Products that in the past required a labour-intensive assembly can now be manufactured in a single part. Additional functionalities can also be integrated in a single part and thus new ideas and designs can be realised. Unfortunately, few examples exist in the ceramic industry. Ceramic engineers have been taught in the past to avoid complex structures and to design their products to be as simple as possible. It will be a difficult

task for these engineers to move beyond the conventional design rules and embrace the new options.

It must, however, be emphasised that the traditional manufacturing technologies will in no way become obsolete. The majority of functional parts will still be produced by traditional forming processes, but there will be some applications where AM will be advantageous, and some where AM will be the only viable option. Consequently, AM will succeed where the additional benefits that can be achieved with the new design freedom outperform traditional forming technologies. This does not only include manufacturing costs, but also the savings due to the elimina-

tion of assembly costs or indirect savings because of the better performance of an AM produced part.

As long as traditional forming technologies can be easily applied for mass production, AM will in most cases be more expensive, slower and not as accurate. AM should therefore be seen as a supplement for traditional forming technologies and as a means of overcoming current manufacturing limitations - both from an economical and a technological point of view. Fig. 6 is an example of a design that cannot be manufactured using traditional forming technologies. The part is a blood pump with two bearing points. In this case, it is necessary to use AM to produce this geometry since this design gives additional benefits such as increased operational stability.

As has been demonstrated, CIM or any other traditional forming technology will not be replaced by AM, but AM could bring additional benefits for the production of prototypes or small scale series. Moreover, AM could be implemented as a tool to support the development and sales of new ceramic mass products by providing the first prototypes in a quick and cost effective manner. However, much more important in the long term is the opportunity to develop completely new applications with the newly gained design freedom.

AM is of course the latest in a long line of ceramic forming technologies and as with other processes it has its own pros and cons. With the advent of CIM in the 1940s, other ceramic forming processes did not suddenly cease to be used. Simple geometries are still dry pressed, but CIM opened up a completely new field of complementary applications which had not been possible before. Notably these had, in most cases, to be developed from scratch.

Of course, there are some borderline cases where the limits become blurred, but in most situations it becomes very clear which technology should be used. Furthermore, no



Fig. 7 Lithoz's CTO Dr Johannes Patzer with the CeraFab 7500

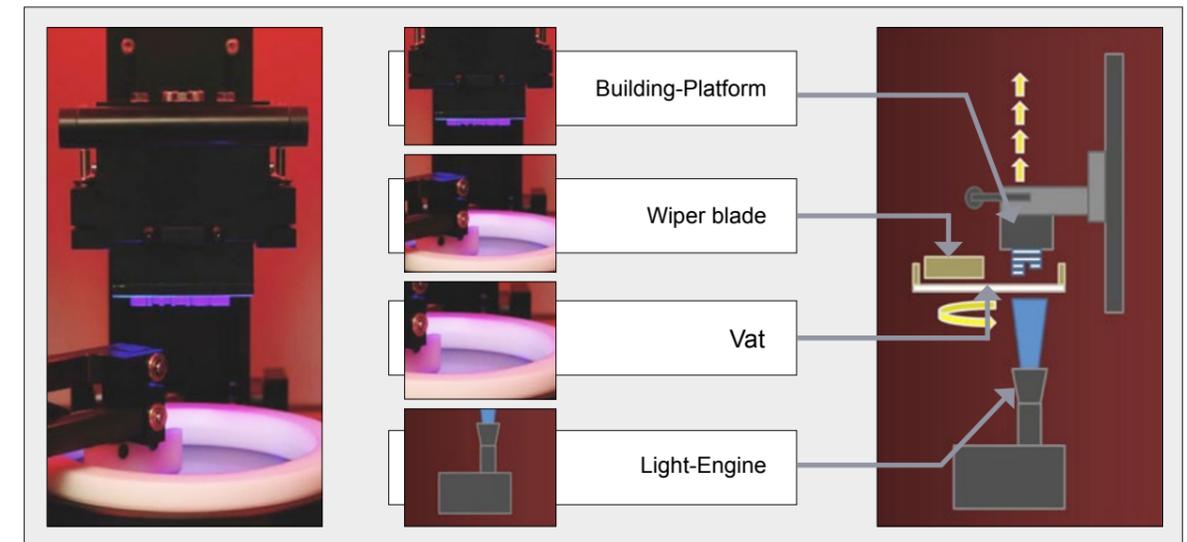


Fig. 8 Building chamber of the CeraFab 7500 (left) and schematic structure (right)

one will believe that one can use a design developed for dry pressed parts and injection mould it without encountering any problems. It will take some time to understand a new forming technology and to identify its advantages and disadvantages.

Curiously, AM is seen completely differently by many people. It is expected to be the miracle technology, where the above mentioned considerations should no longer be valid. This can be mostly attributed to the media hype surrounding this new technology. AM has been billed as the solution to every manufacturing problem; a disruptive technology which turns the production landscape of the next century upside down. The ceramic industry will need to understand the potential of AM and think about new applications. The industry has the opportunity to define the competitive advantage to others in dealing and using this new technology. AM should be seen as a door opener for new applications with the potential to extend the use of ceramics into new markets. There are already a number of success stories in metal AM where AM has generated huge benefits for the aerospace, dental, medical and tooling sectors, to name just a few. It is therefore critical for the ceramics industry to gain experience with AM and develop new applications.

### Additive Manufacturing of ceramics

Before AM is accepted by the ceramic industry, however, another very important condition has to be fulfilled. The material properties of the AM parts should be comparable to those made by conventional ceramic forming technologies. Focusing on structural ceramic parts, strength and density are the most important properties. There are several AM technologies using ceramic materials. It is obvious that every technology has its respective pros and cons and different applications also require different technologies. If high densities and good precision are required, which is in most cases very important for parts produced by CIM, many AM technologies have difficulties in meeting the high standards of the ceramic industry.

Processes such as laser sintering, 3D printing or Fused Deposition Modelling (FDM) have already been adapted to process ceramic materials. However, they have difficulties in reaching the required material properties for dense and precise parts. Direct Laser Sintering (or, more accurately, laser melting because it is actually a melting process) is not really favourable for ceramic materials because the material is not sintered but molten and this is

in most cases not really desirable because of a detrimental microstructure. Furthermore, high stresses are generated through the melting of each layer on top of the existing structure and ceramics, in contrast to metals, cannot be annealed. The indirect approach, where ceramic powders are coated with a polymer and only this polymeric binder is fused together has problems in achieving dense parts, because green density is not sufficient because of the limited compaction of the powder bed. This is very similar to 3D printing, where a binder is jetted into a powder bed.

All powder-based technologies have to use specific powders, which can be spread easily. Due to the relatively coarse powders (20-30 µm), which are very often used, the resolution is restricted and densification is very difficult. FDM or Robocasting have rather anisotropic material properties due to the extrusion of the material. Very thin nozzles could be used to achieve good resolution, but the building times would consequently become very long.

The Lithography-based Ceramic Manufacturing (LCM) technology, developed by Lithoz, attempts to follow a traditional ceramic approach to achieve the required properties [2]. Lithoz is currently offering two

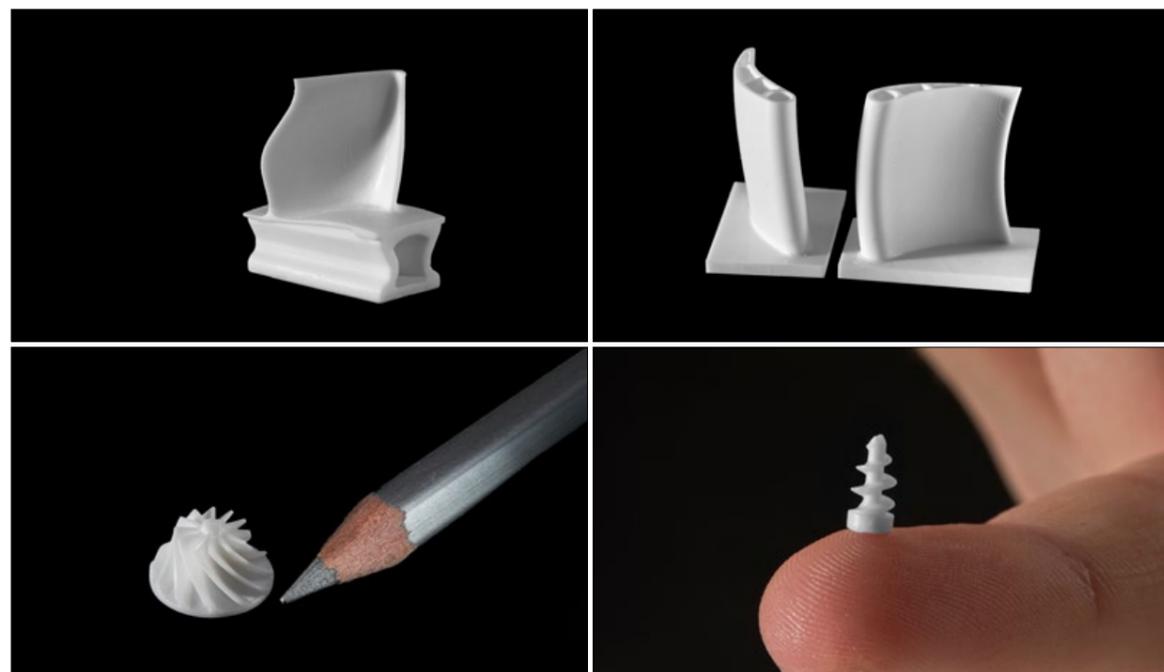


Fig. 9 Different parts made by LCM technology

dedicated AM systems for the production of high performance ceramic parts. The CeraFab 7500 (Fig. 7) has a build envelope of 76 mm x 43 mm x 150 mm, whilst the CeraFab 8500 has a build envelope of 115 mm x 64 mm x 150 mm.

As with any other AM process, LCM starts with a CAD file of the required part. The part is virtually sliced into very thin layers (down to 25 µm) and then the physical part is built up by adding the individual cross-sections in a layer-by-layer manner. LCM is a slurry-based process, where the

ceramic particles are homogeneously dispersed in a photosensitive resin which is mainly composed of monomers, dispersants and photo-initiators. The achievable solid content is to a great extent dependent on the ceramic powder used and ranges typically from 40 to 60 vol%. Since the slurry is photosensitive, the cross-sections are selectively solidified by light.

Unlike most AM technologies, LCM builds the part upside down. This method significantly reduces the required amount of slurry. Moreover,

almost 100 % of the material that is fed into the process is solidified, which clearly renders this technique very attractive in terms of costs and resource efficiency. The schematic layout of the CeraFab system is shown in Fig. 8, with the light engine at the bottom of the build chamber. Each cross-section is exposed through the transparent vat. Since the slurry has a relatively high viscosity of between 1 and 100 Pas, a recoating system is needed to spread a fresh layer of slurry before each curing sequence. The recoating system consists of a

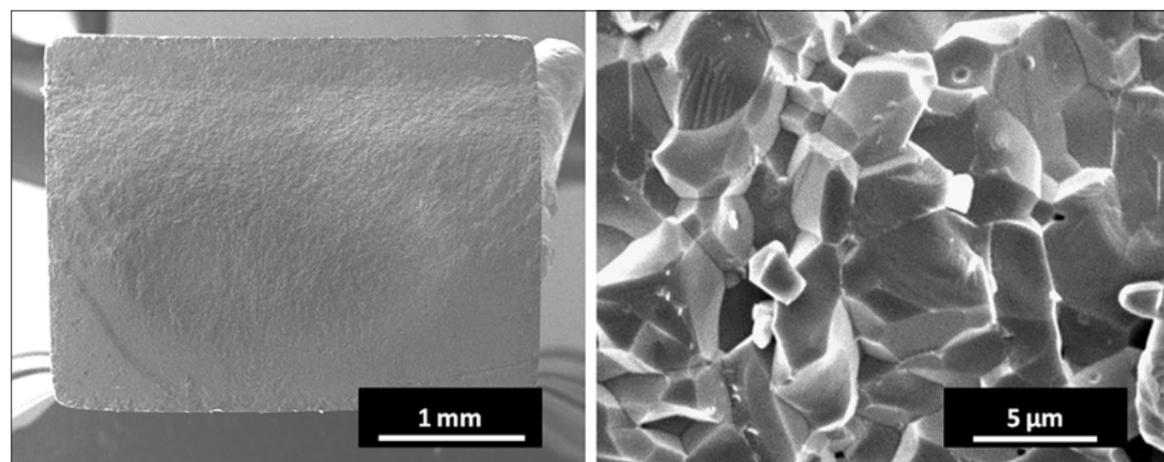


Fig. 10 Microstructure of alumina processed with the LCM technology at different magnifications

fixed wiper blade in combination with a rotating vat. After solidification of each layer, the building platform is moved upwards, the layer is renewed by the recoating system and then the building platform is lowered again. The building platform forms a gap together with the surface of the vat, which corresponds to the thickness of one layer. The slurry in the gap is selectively exposed to visible light to solidify the next cross section. After finishing the whole run by creating the part in a layer-by-layer approach, the part is removed from the platform and cleaned.

Since a binder is used, the produced part consists of both ceramic particles and an organic photopolymer network. This green part therefore needs to be debound and sintered, as in conventional ceramic forming technologies. The debinding is done by heating the part according to a specific debinding schedule. Subsequently, the part has to be sintered according to the requirements of the powder used. This process is well known from traditional ceramic processing and the material properties are the same or, at least, very similar to conventionally formed parts when the same powder has been used.

Since the choice of powder is linked to the final properties of the ceramic part, LCM follows the approach of adapting the technology to a given powder and not vice-versa. This means that the technology can process almost any sinterable powder. So far, many different powders have been processed using LCM, with particle sizes ranging from approximately from 100 nm to 100 µm. There is no definite limitation for the particle size of the powder used, but, as in any other slurry-based process, nanopowders are much more difficult to disperse and the viscosity rises with increasing surface area. Thus, the required minimum solids content has to be met. At the upper end, the layer thickness is the limiting factor as the particle size should not exceed the thickness of an individual layer.

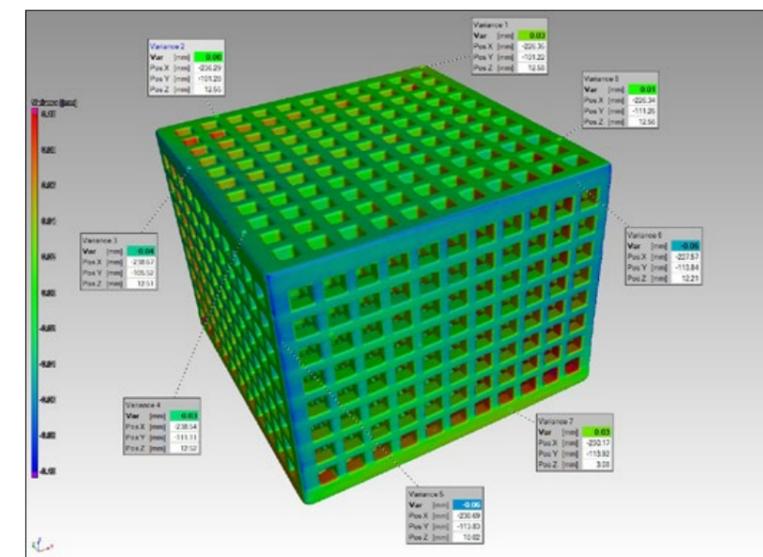


Fig. 11. A graphical target-actual comparison between a LCM produced structure after debinding and sintering and its underlying CAD design

Materials that have already been successfully processed by LCM range from alumina, silica, zirconia, cordierite and glass ceramics to tri-calcium-phosphate and hydroxyl-apatite for biomedical applications. Ceramic parts of different materials are shown in Fig. 9.

It should be noted that dark materials are more difficult to process due to their higher light absorbance. Since the light needs to penetrate through the ceramic slurry to the equivalent of at least one layer thickness, the usage of dark materials is challenging. However, it has already been shown at Vienna University of Technology that silicon carbide can be processed by LCM [3]. Further work is currently being undertaken by Lithoz to expand the range of processable materials for LCM to additional oxides as well as to nitrides and carbides.

Alumina is one of the most widespread materials in the ceramic industry and Lithoz has focussed in recent years on this material. With its LithaLox HP 500, Lithoz provides a high purity alumina with good material properties. Densities of over 3.96 g/cm<sup>3</sup> (99.4 % TD) can be achieved, resulting in a four point bending strength of around 430 MPa with a Weibull-modulus of 11. The microstructure of an alumina

fracture surface can be seen in Fig. 10 at various magnifications. No difference can be seen in comparison to traditionally formed alumina and it is no longer possible to tell that this part has been manufactured in a layer-wise manner.

Surface roughness after sintering, without any polishing or grinding, is well below 1 µm, which is acceptable for most applications. However, the parts can also be post-machined in cases where smoother surfaces are required.

Fig. 11 shows a graphical target-actual comparison between a LCM produced structure after debinding and sintering and its underlying CAD design, conducted at the Technical University of Kosice's, Department of Biomedical Engineering and Measurement in Slovakia. The outer dimensions of the test geometry were 13 mm x 13 mm x 10 mm in the sintered state. As can be seen from this measurement, any point on the part showed a deviation of less than 60 µm from the target dimensions. On the one hand, this clearly shows the highly isotropic shrinkage behaviour of the LCM produced part and, on the other hand, it also confirms that it is possible to compensate for shrinkage during sintering without sacrificing precision or dimensional accuracy in the final part.

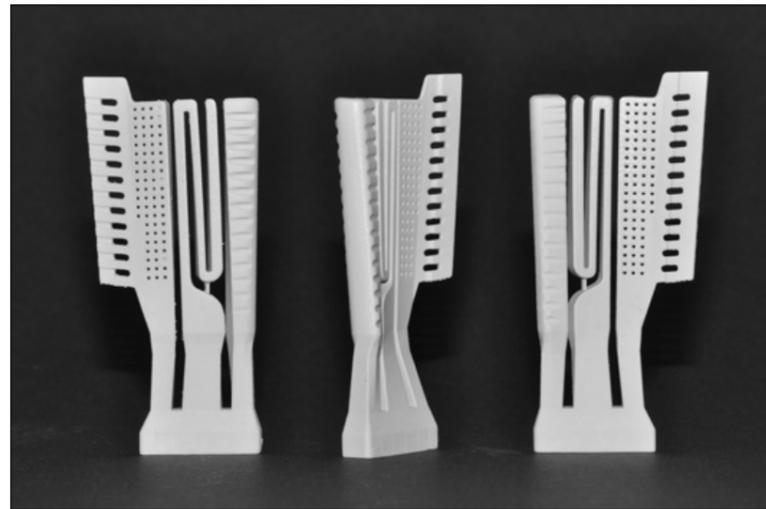


Fig. 12 Ceramic casting cores made by LCM

### Conclusion

AM today offers the ceramics industry a new tool for prototyping and for developing new applications. With LCM, it is possible to achieve the same, or at least very similar, properties to conventional ceramic manufacturing technologies. This point is inevitably a basic condition for the final acceptance of AM as a forming technology for structural parts. If the necessary tolerances and surface qualities are not reached, grinding and polishing can be applied to achieve the desired values.

AM is not a threat to the ceramics industry, but much more an opportunity to move into new fields and develop new applications. AM has a huge potential to become a 'game changer' for the industry with its new freedom in design. This new way of designing has to be fully understood and it will take some years to convey the new limits in design to the industry. Therefore, it is very important to start working with AM as this is the easiest way of learning.

AM should not be compared with conventional forming technologies in terms of cost and performance, because AM is not intended to replace traditional forming technologies. AM is a new forming technology and it will push the current limits of manufacturing, thus allowing the ceramic industry to move forward. AM should

be seen as a new forming method, which opens up new opportunities and opportunities. It is not a substitute for traditional forming processes, but rather an addition to the industry to open up new markets.

One such promising market is ceramic casting cores for turbines made from superalloys. The cooling channels designed into these products are becoming ever more complex and Ceramic Injection Moulding is reaching its limits. There is a high probability that some special core designs will be made in the future only by AM. Other products in mechanical or biomedical engineering will also emerge. Current limitations are only in our minds and we should tear down these barriers to reach new markets. AM could provide an enormous opportunity for the ceramic industry to extend its business. It just remains for the application of AM to be developed.

### Authors

Dr Johannes Homa and  
Dr Martin Schwentenwein

Lithoz GmbH  
Mollardgasse 85a/2/64-69  
1060 Vienna  
Austria  
Tel: +43 1 9346612 201  
Email: jhoma@lithoz.com  
www.lithoz.com

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### Further resources

Videos showing the LCM process are available to view at [www.lithoz.com/en/news/press/lithoz-videos/](http://www.lithoz.com/en/news/press/lithoz-videos/)

## POWDERMET2015: Advances in MIM materials and processing highlighted in San Diego

The POWDERMET2015 Conference and Exhibition, which took place in San Diego, California, from May 17-20, 2015, continues to encourage MIM producers, industry suppliers and researchers from around the world to share their latest research and developments. In this exclusive report for *PIM International* Dr David Whittaker reviews papers from Japan, India, Germany, the UK and the USA that reflect a number of key areas for MIM process enhancement and industry growth.

There was much to interest devotees of Metal Injection Moulding at the POWDERMET2015 conference, with four technical sessions specifically dedicated to MIM and a number of relevant papers in other sessions. In the exhibit hall, the industry was also well represented with a host of materials and technology suppliers from around the world participating. As reported elsewhere in this issue, MIM also scored a large number of successes in the Metal Powder Industries Federation's 2015 Design Excellence awards.

### Improving the fatigue strength of MIM titanium alloys

A number of technical papers related to material developments in particular MIM-processed alloy types, notably titanium alloys, stainless steels and high speed steels. A paper by Hideshi Miura, Choe Jungho,

Toshiko Osada, Kentaro Kudo and Fujio Tsumori (Kyushu University, Japan) addressed the improvement of the fatigue strength of titanium alloy (Ti-6Al-4V) compacts processed by MIM [1].

MIM processing is potentially advantageous for Ti-6Al-4V, not least

because this alloy has poor workability in conventional processing. However, previous work by this group had revealed a potential 'Achilles's heel' for MIM Ti-6Al-4V, in that it delivers low fatigue strength compared with the wrought counterpart.



Fig. 1 POWDERMET2015 took place in San Diego, USA

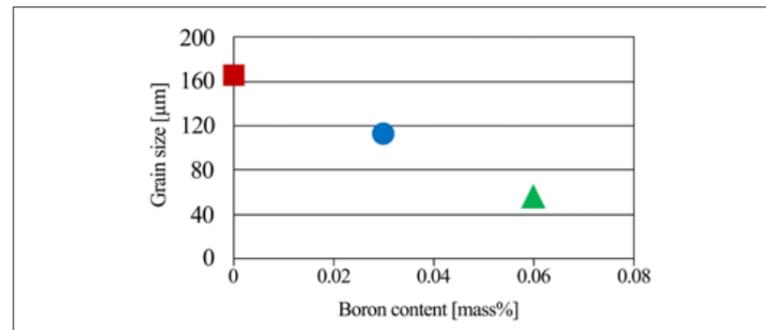


Fig. 2 Grain sizes measured by EBSD [1]

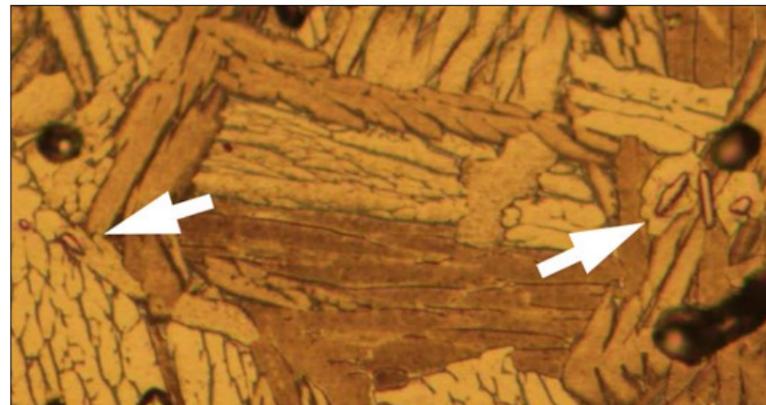


Fig. 3 Optical micrograph of TiB in 0.6%B compacts [1]

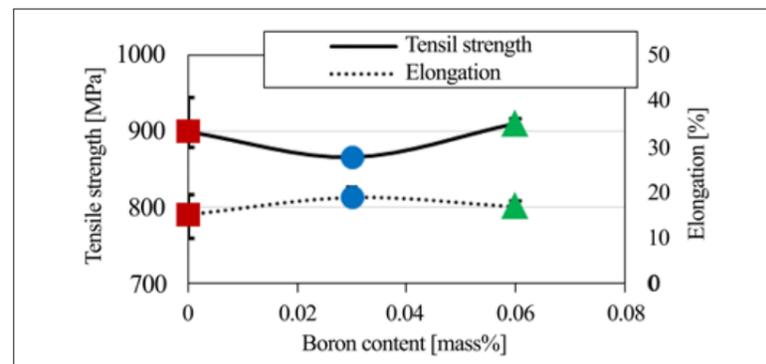


Fig. 4 Tensile test results [1]

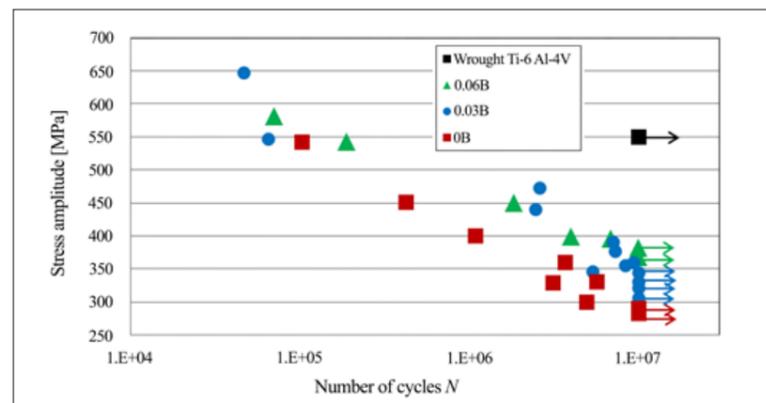


Fig. 5 Rotating bend fatigue test results [1]

The reported study involved the addition of small amounts of boron to the MIM alloy composition, using a  $\text{TiB}_2$  powder. The Ti-6Al-4V powder used was spherical and was classified to be less than  $45 \mu\text{m}$  diameter with a mean diameter of  $31.43 \mu\text{m}$ , while the  $\text{TiB}_2$  addition was much finer with a mean diameter of  $1.36 \mu\text{m}$ .

Powder mixes containing 0, 0.3 and 0.6% boron were processed to MIM feedstock, moulded into tensile and fatigue test-pieces and debound and sintered. The as-sintered tensile and fatigue properties were then assessed.

Fig. 2 show that the prior  $\beta$  grain sizes of boron added compacts, measured using electron back scattering diffraction (EBSD), were finer than that of boron free compacts. However, in the case of the 0.03 % boron addition, some of the prior  $\beta$  grain sizes remained unaffected and a few prior large  $\beta$  grains, which were almost the same size as in the boron free samples, were still present. This resulted in an increase in the average grain size of the 0.03% B sample. Boron was shown to be present as titanium boride in the sintered parts, with the boride particles pinning the grain boundaries and preventing grain growth. Fig. 3 points out these titanium boride particles using arrows in an optical micrograph. The titanium boride particles have a sharp needle like shape with length under  $10 \mu\text{m}$ .

Fig. 4 shows the tensile test results. In all cases, the tensile strength is over 850 MPa. The tensile strength seems not to have been affected by the grain size or the boron content. The ASTM B348 Gr5 standard for wrought Ti-6Al-4V quotes a value of 895 MPa for tensile strength and a minimum elongation of 10%. With the exception of the tensile strength for the 0.03% B sample, all the requirements of the ASTM standard were therefore met.

The fatigue tests clearly showed the effect of the grain size refinement. Fig. 5 shows the results of rotational bending fatigue tests. The fatigue limit for the boron free samples was 291 MPa, that for the 0.03 % B samples was 345 MPa and that for

Sl#	Powder	Fe	Ni	Cr	Mo	Co	Mn	Nb	Si	Cu	C	N	O	S	W	P
1	SS 174PH	Bal	4.1	16.8	0.11	nil	0.70	0.2	0.67	4.2	0.026	0.10	0.13	0.004	nil	0.024
2	SS 316	Bal	11.2	17.5	2.4	nil	1.19	nil	0.59	nil	0.02			0.009	nil	0.014
3	SS 420	Bal	0.3	13.5	0.5	nil	0.8	nil	0.75	nil	0.6	nil	0.35	0.03	nil	0.03
4	Nitronic 60	Bal	8.6	17.2	nil	nil	7.4	nil	4.5	nil	0.018	0.16	0.09	0.006	nil	0.01
5	1.4882	Bal	4.1	21.5	nil	nil	9.3	2.2	0.17	nil	0.55	0.42	nil	nil	1.3	nil
6	440 C	Bal	0.12	17	0.64	nil	0.74	nil	0.64	nil	1.11	nil	nil	0.008	nil	0.016
7	Ni free SS	Bal	0.12	17	3.3	nil	10.4	nil	0.89	nil	0.018	0.32	nil	nil	nil	nil

Table 1 Chemical composition of Stainless steel (SS) powders chosen in the study [2]

the 0.06 % B samples was 382 MPa. However, no effect of grain refinement was seen for the high stress, low cycle tests.

A data point from a wrought Ti-6Al-4V sample, mill annealed and with an average grain size of  $70 \mu\text{m}$ , was included in Fig. 5 for comparison. This fatigue test was, however, carried in a pull-pull mode at an  $R = 0.1$  in the load control mode and with a frequency of 10 Hz. It is therefore highly questionable whether such a direct comparison of properties is valid. Notwithstanding this, however, the authors concluded that the study had shown that boron addition increased the fatigue limit of MIM Ti-6Al-4V, though the fatigue limit was still lower than that for the wrought alloy.

### MIM of stainless steels for applications with critical surface finishing requirements

A paper by B N Mukund, T S Shivashankar and M Sachin (Indo-US MIM tec. Pvt. Ltd., India) turned attention to stainless steels and reported on MIM of stainless steels for surface finishing applications [2].

The recent high growth rate in consumer electronic applications with the growth of smartphones, smart-watches, wearable electronic devices, laptops and tablets has demonstrated the advantage of MIM technology in manufacturing highly complex designed parts with tight tolerances at competitive price compared to alternative manufacturing technologies. Most of these applications in consumer electronic devices also require a high gloss metallic texture or mirror finish and appropriate strength for protection.

In the reported research work, MIM stainless steel materials such as 17-4PH, 316, 420, Nitronic 60, 1.4882, 440C and nickel-free stainless steels were used to evaluate their polishing and lustre properties. The powders used were made by gas atomisation and their chemical compositions were as shown in Table 1.

Components were injection moulded in a machine with a hardened screw and barrel and the parts were subsequently sintered in an argon atmosphere. The sintered components were polished with the aim of obtaining a consistent mirror finish on the surface of the

MIM components. Material characteristics such as sintered density, surface porosity and hardness were analysed and correlated to the mirror finished products.

The levels of porosity in the sintered products were analysed for all the materials chosen using light microscopy. The surface of the watch case product was mounted on an acrylic material and polished on the surface of the component to obtain a uniform surface without any roughness. The as-polished acrylic metallographic mount was analysed to study the percentage surface porosity on a selected area of the MIM watchcase, as shown in Fig. 6.

All of the MIM sintered watch case products were polished to understand the mirror finish with the materials. Polishing consisted of lapping on the surface of the sintered component just before polishing as a preliminary step, the lapping process being performed in different steps depending on the roughness or burrs present on the sintered metal product (normally two or three stages) using different grit sizes ranging from 100 to 600 grit size. After lapping of the parts, the next step involved rough polishing in two different stages. The polishing was carried out using a cotton wheel with a rough buffing compound



Fig. 6 Selected area on the surface of the MIM watchcase component chosen to analyse and interpret % surface porosity [2]

Polishability Rating	Lustre rating
Rating 1: Least number of pores / No pores	Rating 1: Bright finish
Rating 2: Medium number of pores	Rating 2: Matte finish
Rating 3: More number of pores	Rating 3: Dull finish

Table 2 Polishability and lustre ratings [2]

S1#	Material	% Surface Porosity on the MIM sintered product analyzed through light microscope	Polishability rating after mirror polishing	Lustre rating after mirror polishing
1	SS 174PH	0.50	1	2
2	SS 316	0.38	1	1
3	SS 420	1.16	2	2
4	Nitronic 60	2.05	3	2
5	1.4882	1.43	3	3
6	440 C	0.39	1	3
7	Ni free SS	1.95	2	1

Table 3 Polishability and lustre level rating on the MIM watch case products after mirror polishing [2]

used to obtain a good lustre and mirror finish. The polished components were ultrasonically cleaned to remove the buffed compound during polishing.

The mirror polishing inspection was carried out using a natural white light providing 1500 +/- 200 lux at the working level. The surface of

the polished product was rotated at different angles in order to check for defects, if any, including the surface porosity. The distance between the eyes and product was maintained between 30-40 cm. Inspection time was fixed for 10 seconds and, if no defects were observed, the product was acceptable.

In the present study, the materials, which had the least level of or absence of surface porosity without any surface defects, were reported to be given a polishability rating 1. Polishability and lustre level ratings were given on a scale of 1-3 as defined in Table 2. The reported study evaluated the polishability rating by examining the intensity of pores on the surface of the polished MIM product visually. The lustre rating was also quoted, based on visual observations and the materials / products, which had a bright finish, were reported to be given lustre rating 1.

Table 3 shows the after-polishing observations of the MIM watch case sintered product in terms of polishability and lustre level rating. It was observed that 17-4PH, 316 and 440C were found to have the least / no pores on the surface (Polishability rating level 1) of the mirror polished sample in comparison to the other materials. This could be due to the lower porosity achieved after sintering (or higher density) as shown. Nitronic 60 and alloy 1.4882 were found to have the highest number of pores, whereas 420 and Ni free stainless steel were found to have a medium number of pores.

The porosity levels observed after mirror polishing were given ratings from level 1 to level 3 depending on the number of pores visually observed

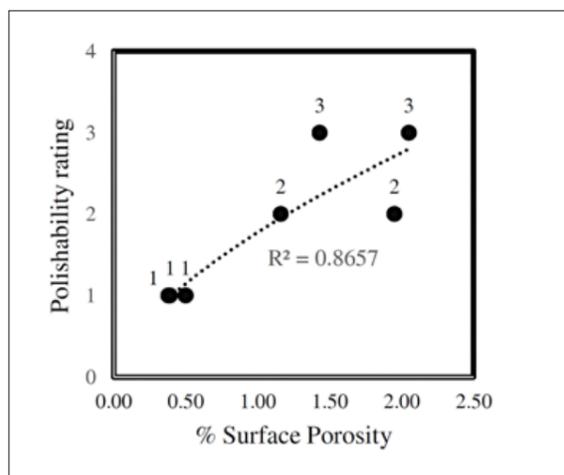


Fig. 7 Relationship between polishability rating and % surface porosity measured through light microscope showing positive correlation [2]

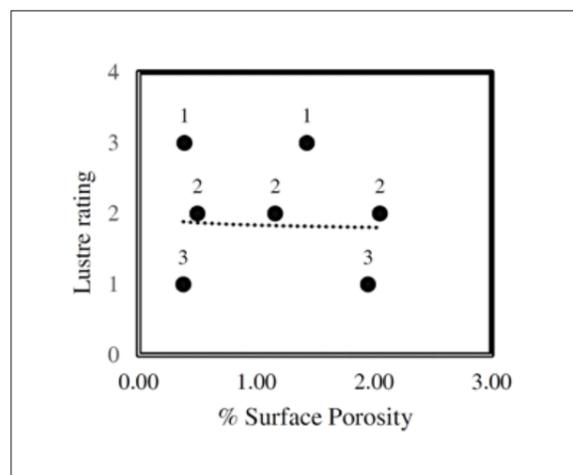


Fig. 8 Relationship between lustre rating and % surface porosity showing no correlation [2]

S1#	Material	Polishability rating	Lustre rating	Experimental Hardness (Average), HV1	Potential Applications
1	SS 174PH	1	2	340.12	Consumer electronics components like cell phones, smart watches, wearable devices & aesthetic applications
2	SS 316	1	1	145.20	
3	Ni free SS	2	1	292.90	
4	SS 420	2	2	390.41	Applications which need moderate wear resistance and strength which includes hand tools, power tool and medical instrumentation
5	Nitronic 60	3	2	186.73	
6	1.4882	3	3	342.02	High wear resistant applications where cosmetic requirements are not mandatory
7	440 C	1	3	550.57	

Table 4 Hardness of the MIM watch case product with different materials and the potential applications [2]

and the results were correlated to the % surface porosity measured through light microscopy, as reported in Fig. 7. As can be noted from Fig. 7, there was a positive correlation between the polishability rating and the % surface porosity measured, which indicated that the porous surface on the polished MIM product is due to the pores present in the sintered product prior to the mirror polishing process. Hence, the sintered product should have the least level or no pores on the surface to obtain a highly aesthetic mirror-finished surface after polishing.

As shown in Table 3, 316 and Ni free stainless steel were found to have excellent lustre in comparison to the other materials evaluated. As can be seen in Fig. 8, there is no correlation found between the lustre rating and the % surface porosity measured, which indicates that the lustre on the polished MIM product is no longer due to the pores present in the sintered product prior to the mirror polishing process. The lustre is mainly dependent on the chemical composition of the material, especially the presence of molybdenum, which is beneficial for lustre.

Table 4 reports the hardness of the MIM sintered product measured for the various stainless steel materials chosen. As shown in this table, 440 C was found to have the highest hardness whereas 316 was found to have the lowest hardness. It was also observed that both 440C and 316 materials were found to have the

highest polishability rating (Rating level 1). Hence, it can be concluded that polishability is independent of hardness.

The overall conclusions drawn from the study were that 17-4PH, 316 and 440 C were found to be the best in terms of polishability rating, because sintered density was found to be greater than 99% of the theoretical density. However 420, Ni free stainless steel, 1.4882 and Nitronic 60 were found to have density lower than 99% of the theoretical density and hence may need some post-sintering operation, such as HIPping, to improve density.

It was found that the material which had a better polishability rating with least or no porosity need not necessarily show good lustre after polishing and vice versa, as both are independent characteristics of the material. An example of this was 440C, where the lustre was not good. It is evident from the reported study that 316 is the most suitable material to provide a combination of an excellent mirror finish with the best lustre on MIM components. However, 17-4PH and Ni free stainless steels can also be used where the application demands higher hardness than 316 can provide, along with the best mirror finish and lustre in MIM products.

Further studies are planned to evaluate the polishability and lustre rating of Ni free stainless steel, 420, Nitronic 60 and 1.4882 alloying compositions after performing HIPping on the sintered products.

### MIM M2 high speed steel produced by pre-alloy and master alloy routes

A paper by Keith Murray, Martin Kearns, Mary-Kate Johnston and Paul Davies (Sandvik Osprey, UK) and Viacheslav Ryabinin and Erainy Gonzalez (TCK, Dominican Republic) considered the sintering and properties of MIM M2 high speed steel produced by pre-alloy and master alloy routes [3].

The reported study assessed gas atomised pre-alloyed M2 powders in two different particle size fractions (90% -22 µm and 80% -22 µm) and compared sintering response and achieved mechanical properties with those of high and low carbon M2 master alloy grades mixed at a ratio of 1:2 with fine carbonyl iron powders of two different carbon contents (0.81 and 0.005 wt%). The chemical analyses of the powders used are shown in Table 5.

It was found that sintering the 90% -22 µm gas atomised pre-alloyed powder product at 1220°C was effective in achieving full density, whereas the coarser 80% -22 µm powder fraction required a temperature of 1240°C for full densification (Fig. 9). Microstructural coarsening of pre-alloyed M2 was observed with a sintering temperature of 1240°C (Fig. 10), with the observation of grain coarsening and the precipitation of coarse grain boundary carbides, associated with precipitate-free zones adjacent to grain boundaries.

Alloy	Fe	Cr	W	Mo	V	Mn	Si	N	C	O
M2 PA 031912-1	Bal	4.1	6.1	4.8	1.9	0.39	0.31	0.04	0.84	0.074
M2 PA 121514-1	Bal	4.0	6.0	4.6	1.9	0.28	0.25	N.A.	0.83	N.A.
M2 MA 121514-2	Bal	13.3	17.9	15.3	5.8	1.0	1.2	N.A.	2.9	N.A.
M2 MA low C 121514-3	Bal	13.3	18.4	15.0	6.1	1.0	0.9	N.A.	1.14	N.A.
Sintez HC 669	Bal	-	-	-	-	-	-	0.81	0.78	0.25
Sintez BC 477	Bal	-	-	-	-	-	-	0.005	0.02	0.43

P, S both <= 0.01% in all cases

Table 5 Chemical analyses of the powders used in the study [3]

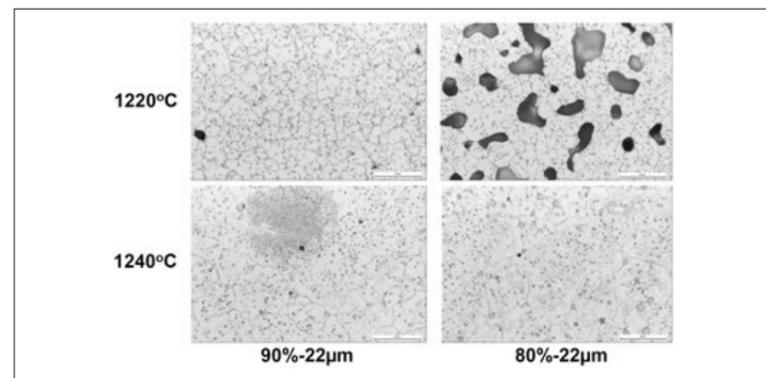


Fig. 9 Sintering response of products from the two pre-alloyed M2 particle size fractions [3]

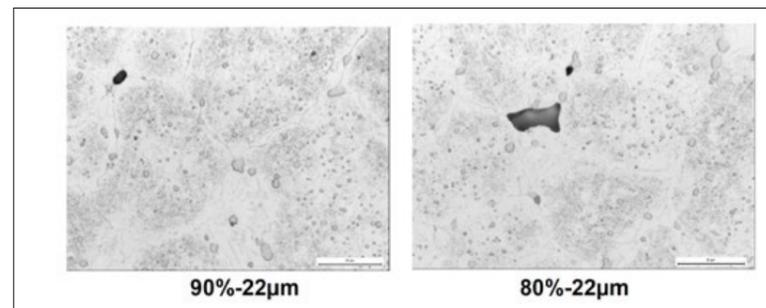


Fig. 10 Microstructure of pre-alloyed M2 materials sintered at 1250°C [3]

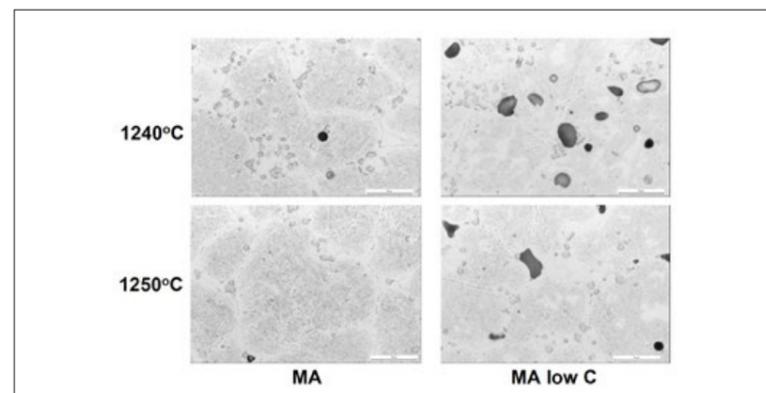


Fig. 11 Effect of temperature on sintering of M2 master alloy route materials [3]

The higher carbon master alloy and carbonyl iron powder mixture required a sintering temperature of 1240°C for full densification, but the achieved microstructures were inhomogeneous, whereas the low carbon master alloy and carbonyl iron powder mixture showed lower density at this sintering temperature (Fig. 11), because of a higher solidus temperature. In all cases, measured carbon losses in sintering were modest and were predictable on the basis of initial carbon and oxygen levels (Table 6).

The highest hardness and strength levels were achieved in the sintered condition with the pre-alloyed variants (see Table 7). Work is currently ongoing to determine whether a solutionising and tempering heat treatment of the M2 produced through the master alloy route can lead to homogenised microstructures and enhanced mechanical properties.

On the other hand, the master alloy route offers significant benefits in terms of sintering distortion levels, with distortion being observed to be much lower than with the pre-alloyed material route. All of the pre-alloyed material samples (80% -22 µm and 90% -22 µm) failed cantilever and slump tests. The deflections observed in cantilever tests on M2 master alloy samples were found to be in line with the expectations of beam theory (Fig. 12). The master alloy mixtures, because of the addition of the finer carbonyl iron powders, had a particle size distribution more equivalent to a 90% -12 µm pre-alloyed powder and so it is anticipated that, to achieve equivalent rigidity in a pre-alloyed

Alloy, Lot #	%C (M2)	%O (M2)	%C (CIP)	%O (CIP)	%C (final part)			
					1220° C	1240° C	1250° C	ΔC
M2 PA, 031912-1, 90%-22	0.84	0.074	-	-	0.82	0.81	0.81	-0.03
M2 PA, 121514-1, 80%-22	0.83	N.A.	-	-	0.80	0.79	0.79	-0.04
M2 MA, 121514-2	2.90	N.A.	0.02	0.43	-	0.89	0.90	-0.07
M2 MA low C, 121514-3	1.14	N.A.	0.78	0.25	-	0.85	0.87	-0.04

ΔC in PA is relatively small & increases with rising temperature  
ΔC in MA is higher and related to % oxygen in the starting mix

Table 6 Changes in carbon content during sintering [3]

variant, a 90% - 12 µm product would be required with consequent cost penalties.

### The influence of hydrogen partial pressure on the debinding and sintering response of MIM stainless steels

A suite of papers at POWDERMET2015 considered the optimisation of a range of processing parameters in MIM. The paper by Satyajit Banerjee (DSH Technologies LLC, USA) and Claus Joens (Elnik Systems LLC, USA) assessed the influence of hydrogen partial pressure on the debinding and sintering response of MIM stainless steels [4].

The debinding and sintering of MIM parts under a 100% hydrogen atmosphere has been reported, but in furnaces that can only run at 15 mbar partial pressure i.e. below the lower explosion limit for hydrogen. Because such furnaces do not need to incorporate safeguards to meet NFPA safety requirements, they are of relatively low cost compared with standard hydrogen furnaces. Standard furnaces, with all of the relevant safeguards, would, on the other hand, operate typically at partial pressures of 400 and/or 800 mbar.

The question addressed in this paper was whether the extra cost involved in debinding and sintering in hydrogen at a partial pressure of 400 mbar as opposed to 15 mbar is justified in terms of improved properties and performance in the final MIM product. 17-4 PH and 316L stainless

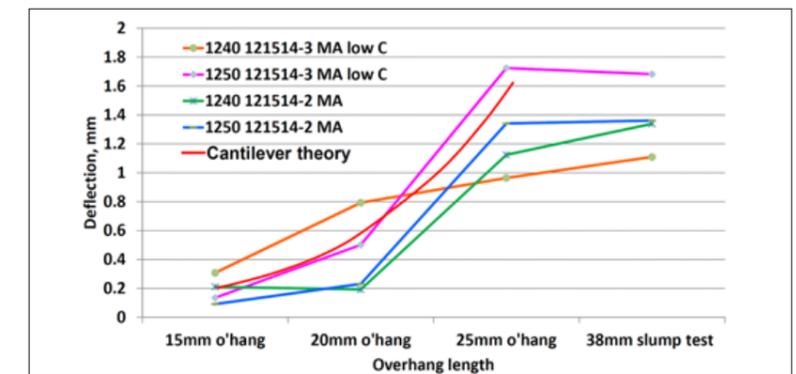


Fig. 12 Results of drape tests for M2 master alloy route materials – deflection vs. overhang [3]

Sintering Temp, °C	M2 source	0.2%PS (MPa)	UTS (MPa)	%EI	HRC
1220	PA	978	1499	-	57.5
1240	PA	1040	1418	-	57.5
	MA	988	1377	0.5	55
1250	MA LC	776	1329	1	54
	MA	969	1141	1.5	56
	MA LC	1106	1386	0.8	55

Table 7 As sintered mechanical properties for the M2 material variants [3]

steel feedstocks were therefore injection moulded into ISO tensile bars and then debound and sintered in hydrogen at either 15 or 400 mbar. The achieved as-sintered tensile properties for 17-4PH are shown in Table 8 and are compared with the values quoted for this grade in MPIF Standard 35 (Table 9). It can be seen that the values for the 15 mbar runs were above the minimum property values specified in MPIF 35, but not better than the typical values mentioned. The values for the 400 mbar sintered products were

consistently better than the typical values from the MPIF 35 specification. Corrosion testing was also carried out by immersing the tensile bars in a 1% saline solution at 62°C for 5 days. The 1% saline corresponds to the saline content of most body fluids, whereas every 5°C above body temperature (37°C) corresponds to a doubling effect of the time of immersion in the test. The parts were immersed for 5 days at 62°C; this would correspond to the equivalent of immersion for 5 x 25 (i.e. 160 days) at body temperature.

Atmosphere	Density	TS (MPa)	0.2% YS (MPa)	% Elong.	% Cr	% Cu	% C
15 mbar (A/S)	7.69	875	687	78	16.25	3.45	0.007
H900		1196	1067	7.3			
400 mbar (A/S)	7.68	995	778	4.5	16.21	4.04	0.013
H900		1266	1104	6.1			

Table 8 Tensile properties of as-sintered 17-4 PH [4]

	Density	TS (MPa)	YS (MPa)	% Elong.
Min. A/S	7.5	790	650	4
H900		1070	970	4
Typical A/S	7.5	900	730	6
H900		1190	1090	6

Table 9 MPIF Standard 35 values for as-sintered 17-4 PH [4]

In the case of both stainless steel alloys, the parts sintered at 400 mbar showed no corrosion and a much better corrosion resistance. For both alloys, the parts sintered at 15 mbar started showing signs of corrosion after one day. In the case of the 15 mbar corrosion samples one of the bars showed corrosion within the first day and was removed after 24 hours.

The underlying reasons for these differences in properties and performance were then investigated

and were found to relate to differences in the populations of inclusions in sintered parts processed at the two different partial pressures. The scanning electron micrograph in Fig. 14 shows a spheroidal inclusion seen in a part sintered at 15 mbar. The fracture itself is a mixture of brittle and ductile fractures. The energy dispersive X-ray analysis (EDXA) of the inclusion shows it to comprise mainly silicon and oxygen suggesting the presence of silica with other oxides of

chromium and iron. The comparative assessment of inclusions in parts sintered at 400 mbar (Fig. 15) did show some oxides but the oxides seen in this analysis had a peak intensity of more than 200 times weaker than those seen in Fig. 14.

SEM/EDXA assessments were also carried out on a corroded and cracked sample that had been debound and sintered at 15 mbar. The SEM micrograph in Fig. 16 shows that bending had caused the part to fracture further along grain boundaries. An EDAX analysis of the region showed chlorides and sodium from the saline, oxygen and silicon from the silica inclusions, calcium, probably from the tap water, and chromium and iron. This picture probably verifies the theory that the presence of the silica type inclusions in the presence of the chloride ions results in the formation of galvanic cells that enhance the rate of corrosion of the alloy sintered at 15 mbar. When the same alloy is sintered at 400 mbar, there are no oxide inclusions to form the galvanic cells that enhance the corrosion rate.

Overall, the authors concluded that, although the achieved tensile properties do not show a large difference between sintering at 15 mbar and 400 mbar, the overall effect of sintering at 400 mbar is a superior product with optimum corrosion resistance and optimum properties comparable to wrought products.

### Controlling furnace Carbon Potential in continuous MIM production

A paper by Akin Malas (Linde AG, Germany), Eric Will (Linde LLC, USA) and Soren Wiburg (AGA Gas AB, Sweden) addressed the cost savings and quality improvement that can accrue from enhanced Carbon Potential (CP) control in the sintering furnace atmosphere [5].

During the sintering, especially of ferrous materials, the Carbon Potential of the atmosphere is a variable that needs to be accurately monitored and controlled to correct for carburisation and decarburisation of the parts being processed. The

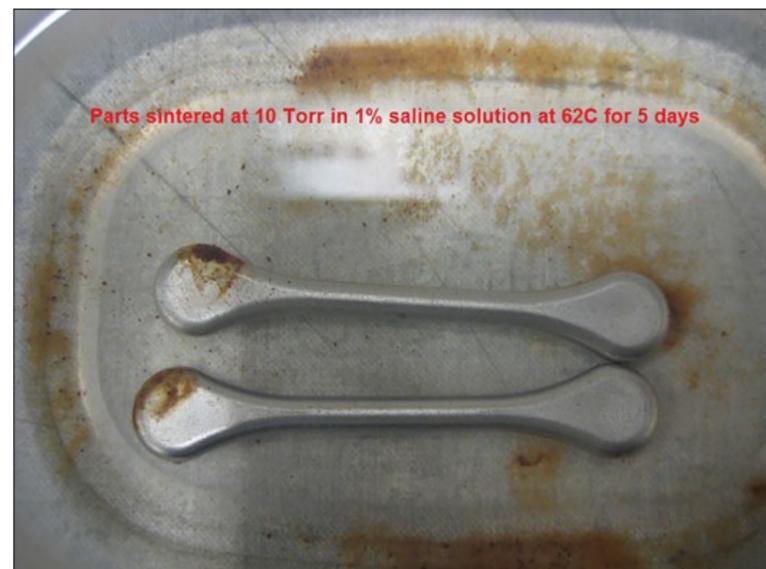


Fig. 13 316L parts sintered at 15 mbar after 5 days in 1% saline solution [4]

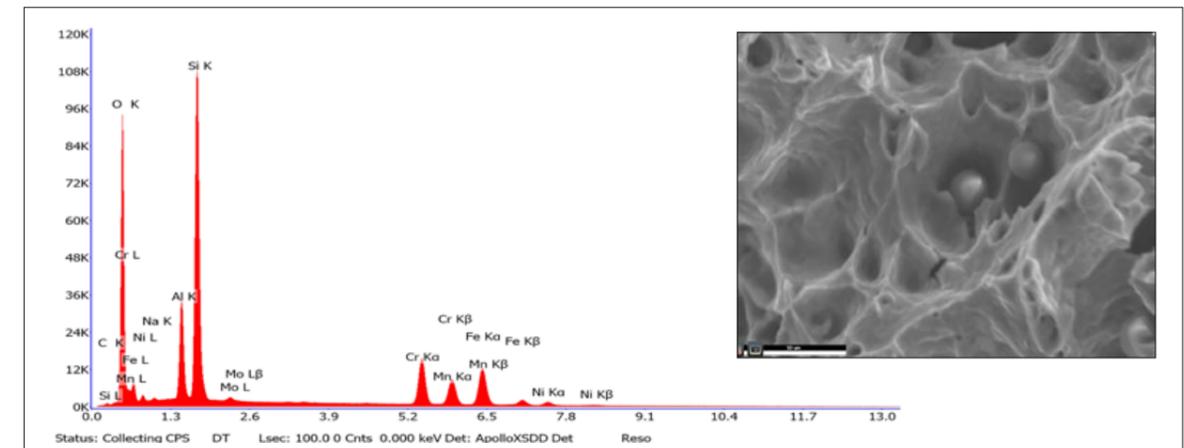


Fig. 14 EDXA of the inset scanning electron micrograph of inclusions seen in a 17-4 PH part sintered at 15 mbar [4]

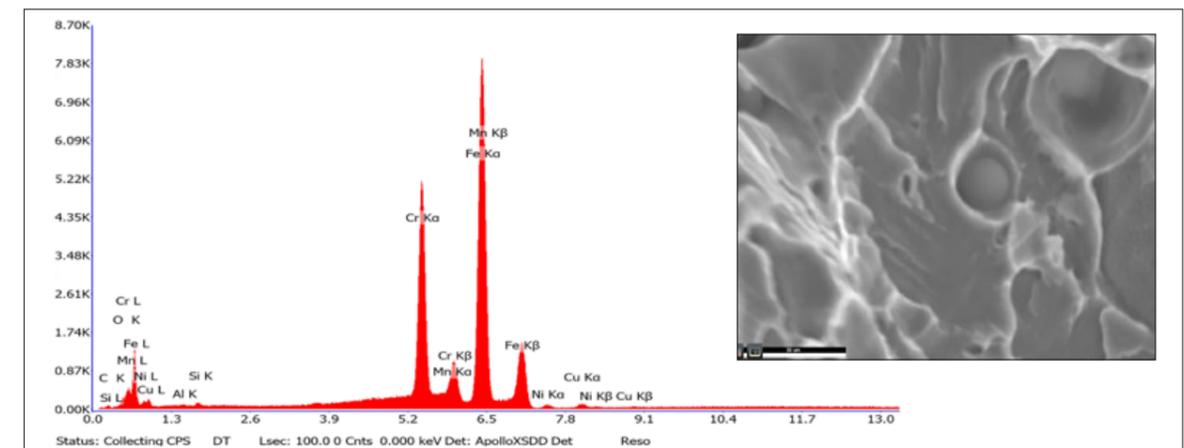


Fig. 15 EDXA of the inset scanning electron micrograph of inclusions seen in a 17-4 PH part sintered at 400 mbar [4]

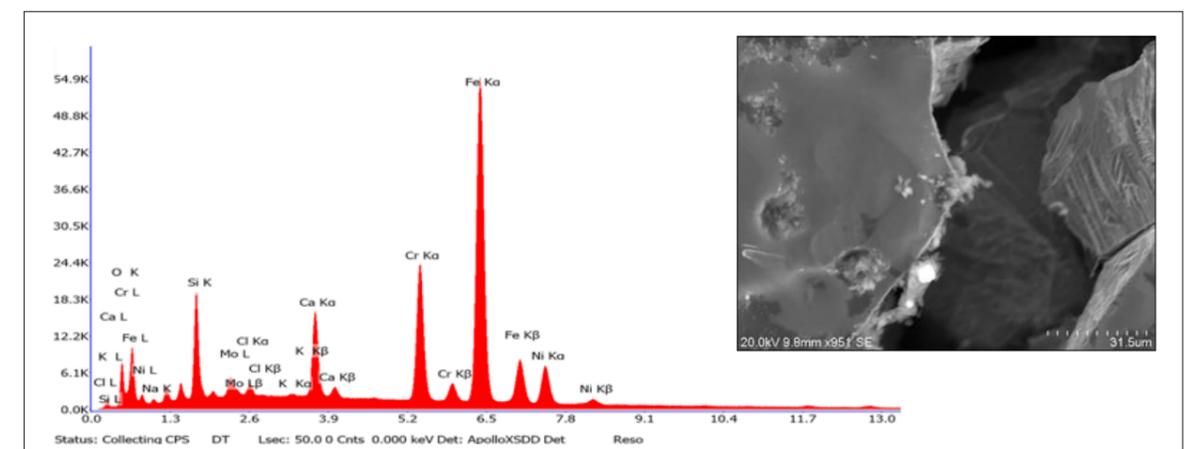


Fig. 16 EDXA of the inset corrosion sample after 1 day, 15 mbar, cracked area [4]

varying carbon content of the parts coming into the furnace is addressed and corrected/adjusted for with on-line monitoring and control feedback.

To achieve these objectives, Linde has developed and commercialised

a new way of monitoring and correcting carbon control issues with a technology called Sinterflex®. This system incorporates an oxygen probe as well as a carbon monoxide and hydrogen analyser for proper carbon potential determination and control.

The system has been deployed in the control of sintering of both die pressed PM compacts and MIM products.

Carbon control is a challenging task in MIM sintering processes and is currently most commonly achieved

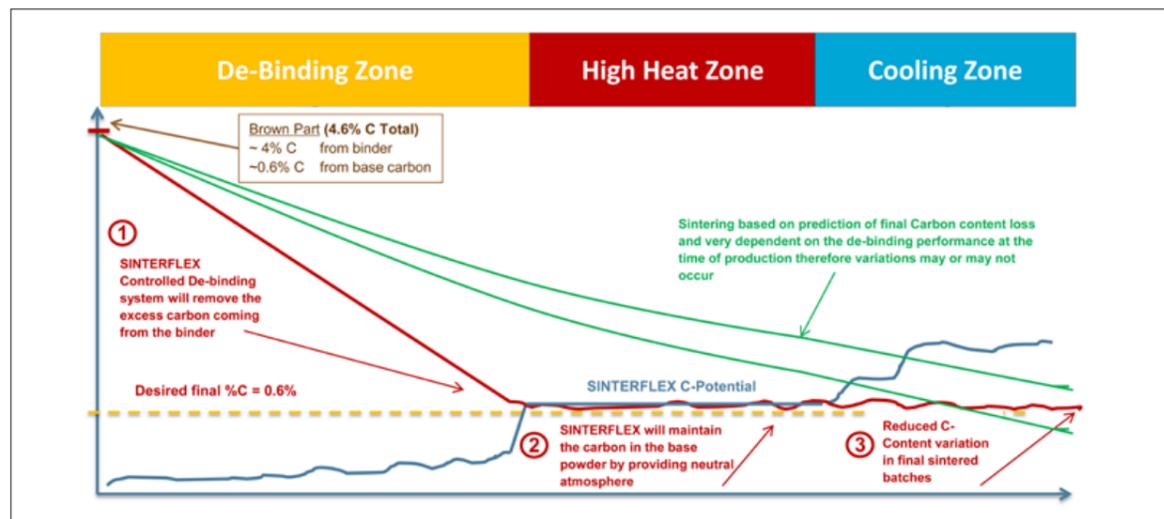


Fig. 17 Comparison of current practice versus SINTERFLEX Atmosphere Control System in the MIM sintering process [5]

by prediction of process conditions in almost all of the companies operating a continuous MIM furnace. The control of the furnace process is carried out only by controlling the input gases and predicting the changes based on the final carbon results. Therefore, when part analysis results come from the quality control laboratory, the parts either pass the requirements or are rejected based on best guesses, leading to high scrap rates. These scrap rates, in mainly continuous operations, can be up to 25 percent in cases where complex alloys are processed, such as stainless steels.

Fig. 17 shows a graph demonstrating the current practice in green versus an operation handled by the Sinterflex Atmosphere Control System in red. Existing practice, as shown by the green line, is to predict the total decarburisation of the 4.8% carbon in the part down to the specification during the whole sintering process involving all zones of the furnace. The brown part starts with 4.8% carbon and enters the thermal de-binding zone, where the binder is supposed to be removed from the MIM mass. However, the carbon will be carried through to the high heat zone (probably in the outer sections of the

parts) causing a source for carbon in the atmosphere and hence providing a protection for decarburising. However, the measurements show that the atmosphere during this time is extremely decarburising and therefore the de-binding continues until the sintering is complete, where the parts reach their final carbon levels. Because of this uncontrolled decarburisation, it is always difficult to predict the final results as well as the variation of the carbon from one production run to another due to an unstable furnace atmosphere.

On the other hand, the Sinterflex Atmosphere Control System (ACS) does not rely on the availability of the binders (hydrocarbons) as the source of carbon in the high heat zone. The Sinterflex system starts with the complete removal of the binders in the pre-heat / de-binding zone of the furnace, as shown in the red line.

Therefore, the brown body no longer contains any binders in the high heat zone. The system creates a very decarburising atmosphere to make sure the binder is removed at this point. However, the high heat zone is already very low in carbon, at approximately 0.05% C potential. Therefore, the second section of the Sinterflex system starts to create a neutral atmosphere, as in most industrial hardening furnaces regardless of the final part requirement, to make sure that the carbon

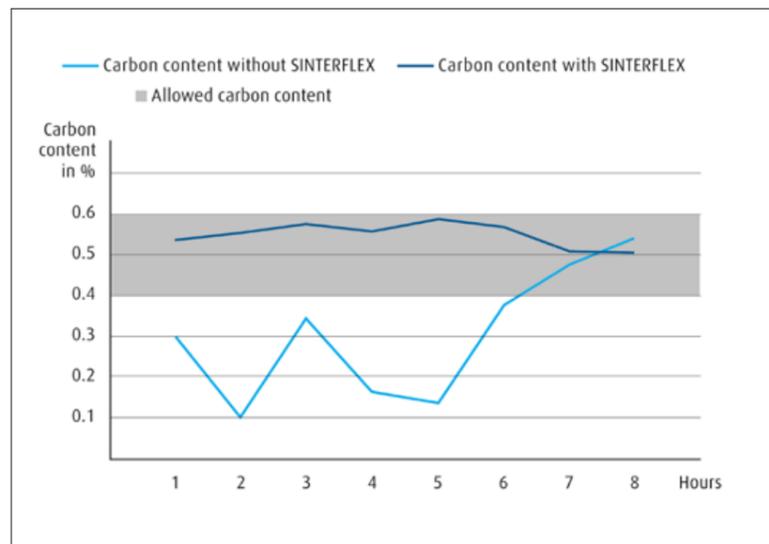


Fig. 18 Carbon content of sintered parts over an 8-hour period [5]

in the matrix left after de-binding is protected in a neutral atmosphere. The parts then enter the cooling zone where there is still a slight carbon containing atmosphere present. This allows a final protection of the carbon layer from the surface.

As a means of quantifying the benefits of deploying the system in MIM sintering, the results of collaborative trials with Megamet Solid Metals (now Ruger Precision Metals, LLC) were then discussed. Since December 2013, tests have been running to investigate carbon percentage, hydrogen percentage, mV of oxygen, as well as other characteristics that are monitored and recorded. Homogeneity and consistency in the furnace atmosphere are pivotal to the success of MIM manufacturing. In order to characterise how Sinterflex ACS affects these attributes within the process, the part rejection statistics and standard deviation of carbon content within parts over periods of time have been recorded. Results have been classified into three categories; parts in specification immediately after sintering, workable parts that could be remediated with additional processing and parts that needed to be scrapped due to the severe difference in carbon between specification and actual final carbon content after processing.

Carbon content in the absence of monitoring and control can often differ by +/- 0.3% on just one boat. These differences can result in manufacturers having to do multiple post treatments. In Fig. 18, the carbon content over a one day period of production is shown in conjunction with its specified upper and lower limit requirements. Both graphs are based on the same part constructed of the same material. There is a visible difference between the two graphs, as the part when controlled with the Sinterflex ACS is continuously processed within specifications for the entire time and has less variance in carbon content trends. This can be further proven by the standard deviation of the carbon content in the parts throughout that day. Without

the Sinterflex system, the standard deviation is approximately 0.15%, while with the Sinterflex system the standard deviation is reduced to approximately 0.03%.

Sinterflex ACS also allowed Megamet to keep the hydrogen flows at fixed values, leading to simplified operation as well as reduced operator interference with production, and to reduce hydrogen flows, resulting in lower atmosphere costs.

The other metric that has been studied in deeming the project a success has been the impact on rejection and reprocessing statistics. Ultimately being able to control the atmosphere should result in less post processing and less rejected parts. Megamet characterised its rejection statistics in three categories:

**In spec**

In spec means that the part is within specification and needs no more post processing to adhere to the customer stipulations.

**Unusable**

Unusable implies that the parts are rejected and officially scrapped.

**Workable**

Workable indicates a more complicated aggregation of parts that can further sectioned into "planned post-processing" and "un-planned

post-processing". A fraction of this tier is purposely processed to end in this category, because it needs further heat treatment by either nitriding or other post-processes and this is therefore described as planned post-processing. The other portion of this group is "workable" due to the carburisation potential in the atmosphere causing this section to be out of the "in spec" category, but not far enough from specification where the part is completely in the "unusable" category. These parts can undergo unplanned post-processing that will move the parts into the "in spec" category, therefore resulting in experiencing unplanned post-processing costs.

Fig. 19 shows a comparison of results over a two month period based on the categorisation above. By using Sinterflex technology, the number of unusable parts decreased by 15%, resulting in significant savings.

As the Sinterflex system continues to run, the testing process becomes less necessary because the carbon results are more reliable. Instead of testing parts every three hours, it has become acceptable to extend testing of parts to once or twice a day. In addition to the favourable reduction in unusable parts, the volume of parts in specification increased by 30%. This was effectively double the amount of parts that were within

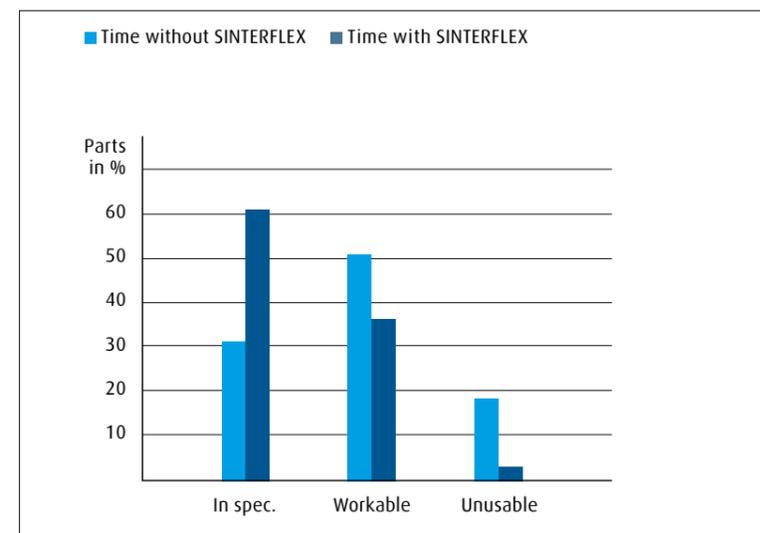


Fig. 19 Ratio of parts within and out of specification over two-month period [5]

Cycle Time Productivity per Week					
Cycle Time	40 hrs per week				Single Molder
	WK 1	WK 2	WK 3	WK 4	1 Yr
25 sec	11520	23040	34560	46080	599040
24 sec	12000	24000	36000	48000	624000
23 sec	12521	25042	27563	50084	651092
22 sec	13090	26180	39270	52360	680680
21 sec	13714	27428	41142	54856	713128
20 sec	14400	28800	43200	57600	748800

Table 10 Comparison of parts produced at various cycle times [6]

specification before Sinterflex was implemented.

When parts are out of specification, a decision is made as to whether the part can be post processed back into specification by heat treatment. Sometimes, parts can undergo this post processing and still not be in specification. The quantity of parts categorised as 'workable parts' also decreased by 15%, demonstrating that the Sinterflex system has tempered this ambiguity in the post processing category.

Finally, the ability of the Sinterflex unit to monitor, record and ultimately adjust the process parameters of the sintering operation has identified other issues from a product and atmosphere standpoint that were not readily apparent prior to installing the unit. By tracking the pre-heat section dew point, hot zone carbon monoxide and oxygen content, as well as hydrogen content, the Sinterflex system allowed Linde and Megamet to gain a greater insight into the complex workings of the different zones of a continuous furnace. Troubleshooting those periods of process problems is greatly reduced and the net amount of rejected parts goes down accordingly.

Overall, it has been concluded that active control of the furnace atmosphere is important in the optimisation of business performance, but that there are secondary and tertiary benefits to be gained in operations from the accurate monitoring and data acquisition of the Sinterflex system.

### Robotics for improved quality and productivity

Finally, a paper by Jonathan Newman (Kinetics Dynacast Inc., USA) considered the impact of robotic integration on the improvement of quality and productivity in MIM [6]. This paper underlined the benefits of robotic integration in responding to the challenge of simultaneously improving both productivity and product quality.

Robots have the advantage of being predictable. They can take repeatable tasks and perform them time and again with extreme accuracy and speed. One of their biggest advantages is that they remove the variation caused by humans that can plague manufacturing in quality control and productivity. Because of this, they can reduce lag time that can rob productivity and can also eliminate damage that can be caused by human handling.

The productivity gains that can be delivered by a stable automated process are illustrated in Table 10. This table shows how a one shift operation can make significant gains in the number of parts produced per year by reducing cycle time by just a few seconds. Table 11 demonstrates how the reduction of yield loss from quality issues caused by human variation can make a measurable difference throughout the year. This information is derived from a two cavity tool with a cycle time of 25 seconds from Table 10.

The starting point for the definition of an integrated robotic system is, of

course, the choice of robot. This is dependent on the task to be undertaken. Available robot types divide between the "dumb" and "smart" categories.

Dumb robots are non-programmable. They are simple in nature, lack complicated programming and could be mechanically operated. An example would be a 2-axis robot that would reach into a mould. The parts would drop into a small cup and, upon retraction, would drop the parts onto a conveyor.

Smart robots are programmable, and (depending on the style) can be very capable of complex movements. They can range from 3 to 6 axis and host a variety of end of arms (EOAs) to accomplish a wide variety of tasks. This category of robots could be any of the following:

#### Gantry robots

Gantry robots are 3 axis (X, Y, and Z) robots and travel in linear motions. Many are attached to the top of moulders, but larger ones can span the machines for wider applications. These robots can also be given a wrist to give them rotational movement at the EOA. These can be ideal for pulling parts from a mould and then placing them on a conveyor or setter. With an articulating wrist, they can be capable of even more complex placement or of presenting parts to an inspection system.

#### Articulated multi axis robots

Articulated, multi axis robots can have up to 10 joints, but most commonly are 6-axis. These joints give them a very wide range of motion within a work cell. They can reach multiple angles with a variety of tools or EOAs that enable them to perform more complicated tasks in an industrial environment. Whether a primary or secondary robot, they are capable of picking, placing, presenting, handing off, assembling, etc.

#### SCARA robots

SCARA robots generally have two joints (one on a fixed cylinder), and operate in a single plane. These lend themselves well to applications such

Quality Yield per Week by % of Loss					
% loss	40 hrs per week				Single Molder
	WK 1	WK 2	WK 2	WK 4	1 Yr
25	8640	17280	25920	34560	449280
15	9792	19584	29376	39168	509184
10	10368	20736	31104	41472	539136
5	10944	21888	32832	43776	569088
2	11290	22579	33869	45158	587059

Table 11 Value of product yielded based on % quality loss [6]

as component assembly or replacing parts from one location to another. In a moulding environment, SCARA robots may have limited application. However, these applications could aid in inspection, placement or assembly, depending on the operation.

#### Delta robots

Delta robots resemble a spider with their multiple legs and operate in a circular/domed work area. They are extremely accurate and capable of precise movements. These capabilities make them widely used

in the electronic and food industries. Like the SCARA robots, Delta robots may have limited application in MIM. Secondary placement that requires precision may be the key to this robot.

Next, the End of Arms (EOAs) are chosen. EOAs give the robot a purpose. In the MIM context, the EOA is used to pick parts from the tool in the moulder. After the part is picked, it is placed directly on a conveyor for human placement, into a basket for dewaxing or directly onto a setter for further processing.

Conveyors are commonplace and can be used as a simple transfer unit or a complicated indexing system that can move multiple parts on tiles or shelves.

Another method of moving parts after robotic placement is the use of Shuttle or Index Tables. These are generally more complicated than a conveyor system. The robot can place multiple parts onto a setter. Once that setter is full, the table will index. The robot can now place parts on a secondary setter that was set in place prior to the first becoming full. The first setter can then be placed on a cart to transfer to the next operation or an inspection station.

MIM inspection systems are visual in nature (camera systems). The capabilities of these systems vary and can look for visual defects in parts or take actual measurements on parts. In some cases, this type of system could be capable of both tasks.

In constructing a robotic cell, safety considerations are very important. Some robots can be programmed for pressure (touch), but this is generally not done in a

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METAL ADDITIVE MANUFACTURING



manufacturing environment. So, if a person is physically contacted by a robot, they have the potential to be seriously injured or worse. To protect against such hazards, the robotic cell needs to be properly guarded. To help make cells safer, standards for robot guarding have been established by ANSI. For MIM purposes, the guarding should follow a few rules. It must encompass the entire envelope of the robot (and possible auxiliary equipment) range of motion. It must also have interlocks, whether mechanical or electrical, that will shut the system down in the case of a breach. The guarding should also be a physical barrier when applicable.

The tasks to be accomplished in integration of a robotic cell were then outlined by the author. The design of the cell starts with the non-operator side of the machine (opposite to the control panel). The cell should be set to this side for safety reasons and so that the operator will not have it impeding their work.

The next step is the installation, beginning with the robot and EOA. Once the set-up is complete, the EOAs can be changed to meet the needs of the parts being produced. Then, the conveying system should be set up (either an actual conveyor or some type of indexing table), the inspection system mounted, the loading components (shelves, tiles or baskets) incorporated, logistics for movement of the parts from the moulder to the next operation considered (generally in the form of a cart) and, finally, the guarding installed.

Now that the cell is set up, it still needs to be programmed. This is a delicate but necessary step to make sure that quality and productivity do not suffer. Done correctly, production speed and overall yield should increase.

In operation of such an integrated cell, when the robot is holding the parts, it cycles to the inspection station. Here, the parts are presented to a vision system and then a scale. These automatically determine if the parts are in specification to move on. If the parts do not pass this

inspection, the robot's programming allows it to drop them. Good parts will move on.

From the inspection station, the robot moves to placement. Company and part requirements will determine the style of setters that are needed. The robot places the exact pattern that has been programmed. Because of the robotic placement, the parts are protected from human handling and setting damage.

Once the parts have been placed, the robot's tasks in the cycle are complete and the programmable index system takes over. When the system receives the signal that the placement is complete and the setter(s) are full, it moves the setter out of the robotic envelope. Keeping in line with the concept of full integration, the setter can be automatically placed into a cart. When the cart is full, the operator that started the process is signalled and then pulls out the cart, installs a new cart for loading and transfers the full cart to the next operation.

### Author

Dr David Whittaker  
231 Coalway Road  
Wolverhampton  
WV3 7NG, UK  
Tel: 01902 338498  
Email: whittakerd4@gmail.com

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### 2015

#### Euromold 2015

September 20-25,  
Dusseldorf, Germany  
www.euromold2015.com

#### Euro PM2015

October 4-7,  
Reims, France  
www.epma.com

#### Ceramitec 2015

October 20-23,  
Munich, Germany  
www.ceramitec.de

#### APMA 2015 3rd International Conference on Powder Metallurgy in Asia

November 8-10  
Kyoto, Japan  
www.apma.asia

#### formnext

November 17-20,  
Frankfurt, Germany  
www.mesago.de/en/formnext

### 2016

#### PM16 International Conference

February 18-20,  
Pune, India  
pmai.in/pm16

#### MIM2016 International Conference on Injection Moulding of Metals, Ceramics and Carbides

March 7-9, Irvine, USA  
www.mpif.org

#### AMPM2016 Additive Manufacturing with Powder Metallurgy

June 5-7,  
Boston, USA  
www.mpif.org/Meetings/2016/AMPM2016

#### POWDERMET2016 International Conference on Powder Metallurgy & Particulate Materials

June 5-8,  
Boston, USA  
www.mpif.org

#### World PM2016 Congress & Exhibition

October 9-13,  
Hamburg, Germany  
www.epma.com/world-pm2016

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Congress & Exhibition**

**4 - 7 October 2015**

**Reims Congress Centre, France**



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