

FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

Vol. 7 No. 1 MARCH 2013

# powder injection moulding

**INTERNATIONAL**



**in this issue**

**PIM trends and forecasts**  
**Profile: Polymer Technologies Inc.**  
**MIM superalloys**

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For the metal, ceramic and carbide injection moulding industries

# PIM high performance materials open up new opportunities

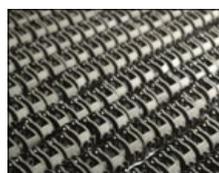
Welcome to our first issue of 2013. As shown in our statistical review of the industry ([page 35](#)), PIM has enjoyed tremendous growth in recent years thanks to its ability to deliver high volume, high precision components to a wide range of both new and well established markets. Some major applications, however, are inevitably cyclic in nature, making industry forecasting an uncertain business. As far as today's cycles are concerned, political factors in the USA are driving tremendous growth for MIM firearm components, and the smartphone and tablet market in Asia is driving the industry forward at an unprecedented pace and putting a strain on capacity at many producers.

However, it is not only high volume applications where PIM can deliver. The PIM of advanced materials for high performance applications is opening up ever more opportunities for the industry. Whether for high temperature jet engine applications, PIM parts that operate in highly corrosive environments, or the next generation of automotive turbocharger systems, interest in the advantages that PIM processing offers continues to grow.

One producer that has chosen to focus on these materials is Polymer Technologies Inc., based in New Jersey, USA. We profile the company and report on its unique and extensive experience of working with the aerospace industry ([page 45](#)).

The aerospace sector has of course long recognised the benefits of the near net shape manufacturing of difficult to process materials such as superalloys. The processing, application and properties of such materials by aero-engine manufacturers has however remained one of the industry's few remaining secrets. In this issue we publish a comprehensive review of what literature has been published on the MIM of superalloys and consider what opportunities this sector of the industry holds ([page 53](#)).

Nick Williams  
Managing Director and Editor



## Cover image

*MIM parts being processed at Polymer Technologies Inc.*

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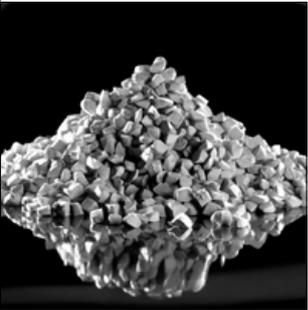
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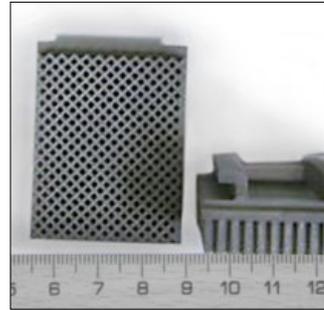
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## In this issue

- 35 Powder Injection Moulding: Statistical trends and forward forecasts for the industry**  
The Powder Injection Moulding (PIM) industry has come a long way since the early 1980s, when sales reports were first published. In this exclusive report for *PIM International*, Prof Randall German presents a statistical review of industry trends, including both metal and ceramic injection moulding, and considers what the future prospects are for an industry that serves a diverse range of end-user markets and has evolved to have such striking regional variations.
  
- 45 Polymer Technologies Inc. looks to the aerospace industry for new PIM applications**  
In 2012 Polymer Technologies Inc., based in Clifton, New Jersey, USA, celebrated 25 years as a specialist producer of precision injection moulded components. Since the mid 1990s, when the company expanded from advanced plastics to Metal Injection Moulding, it has successfully targeted niche markets such as aerospace with a focus on high performance materials. *PIM International's* Nick Williams reports on recent developments.
  
- 53 The processing and properties of Metal Injection Moulded Superalloys**  
The production of MIM superalloys is one of the few remaining secretive areas of our technology, with information on applications in the aerospace sector, and the necessary properties achieved, kept confidential in order to protect decades of privately funded research and a

competitive advantage. There remains, however, a significant volume of data available on what can be achieved via MIM processing. In this extensive review, Burghardt Klöden, Thomas Weissgärber, Bernd Kieback and Ingolf Langer present a detailed analysis of published work to-date and consider the potential for this area of MIM technology.

## Technical papers

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- 72 Development of the Ultrasonic Spray Pyrolysis (USP) process for nanopowder production**  
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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)

## Dynacast International adds variant of the MIM process to its manufacturing technologies

Dynacast International, a global die casting manufacturer headquartered in Charlotte, North Carolina, USA, announced in February that it is adding Metal Injection Moulding (MIM) to its service offerings. The addition of MIM as a manufacturing process, states the company, means that it will be expanding its ability to produce small, complex components using a wider variety of metals.

Dynacast has been manufacturing small and medium sized die cast components for more than 70 years using

aluminium, magnesium and zinc alloys. By adding MIM as a new service, the company believes that it is strengthening its commitment to producing the highest quality, precision-engineered components for its customers.

"We are delighted to be adding MIM to our service offerings," stated Simon Newman, President and CEO of Dynacast. "This process is a natural fit for Dynacast, as it only sharpens our focus on providing small, complex components to our customers and it opens up a whole new market for us," Newman added.

Dynacast states that it has developed a new variant on the MIM process which increases productivity while reducing variation and costs.

According to the company, the new MIM system gets rid of injection moulding equipment and conventional tooling altogether. Instead, it takes its cues from precision die casting technology. The company modified its proprietary A2 die-casting machine to make it compatible with MIM feedstocks. Modifications included the installation of a custom feeding system - a new hopper, gooseneck and feeding controls - optimised for the powder flow and thermal characteristics of MIM feedstocks.

In addition to the A2 machine itself, the new MIM platform makes use of the company's multi-slide tooling. Well known in the die casting industry for its ability to produce precision components at high production volumes, this tooling technology employs a set of sliders that converge in the die block to create the cores, cavity and runner system. Conventional MIM tools, by contrast, arrange the cores, cavities and runners within two opposing mould halves. The result, states Dynacast, is an increase in cycle times and productivity with improved part-to-part consistency and tighter tolerances.

Dynacast states that for the manufacture of a precision firearms component, its MIM process achieved a 1.67 Cpk on a key print tolerance of +/- 0.001 inches, equivalent to just one out of tolerance event per million parts.

The debinding and sintering steps used at Dynacast are the same as for conventional MIM processing. The parts shrink at the same rate as other MIM parts, and they end up with a final density that typically exceeds 98%, just as it does with a well controlled traditional MIM process. Dynacast states that its MIM process can produce small, precision components from steel, copper and titanium alloys.

Dynacast operates 22 manufacturing facilities in 16 countries around the world and has some 3000 employees. It serves a variety of industries including automotive, consumer electronics, healthcare, hardware, computers and peripherals and many others.

[www.dynacastmim.com](http://www.dynacastmim.com)



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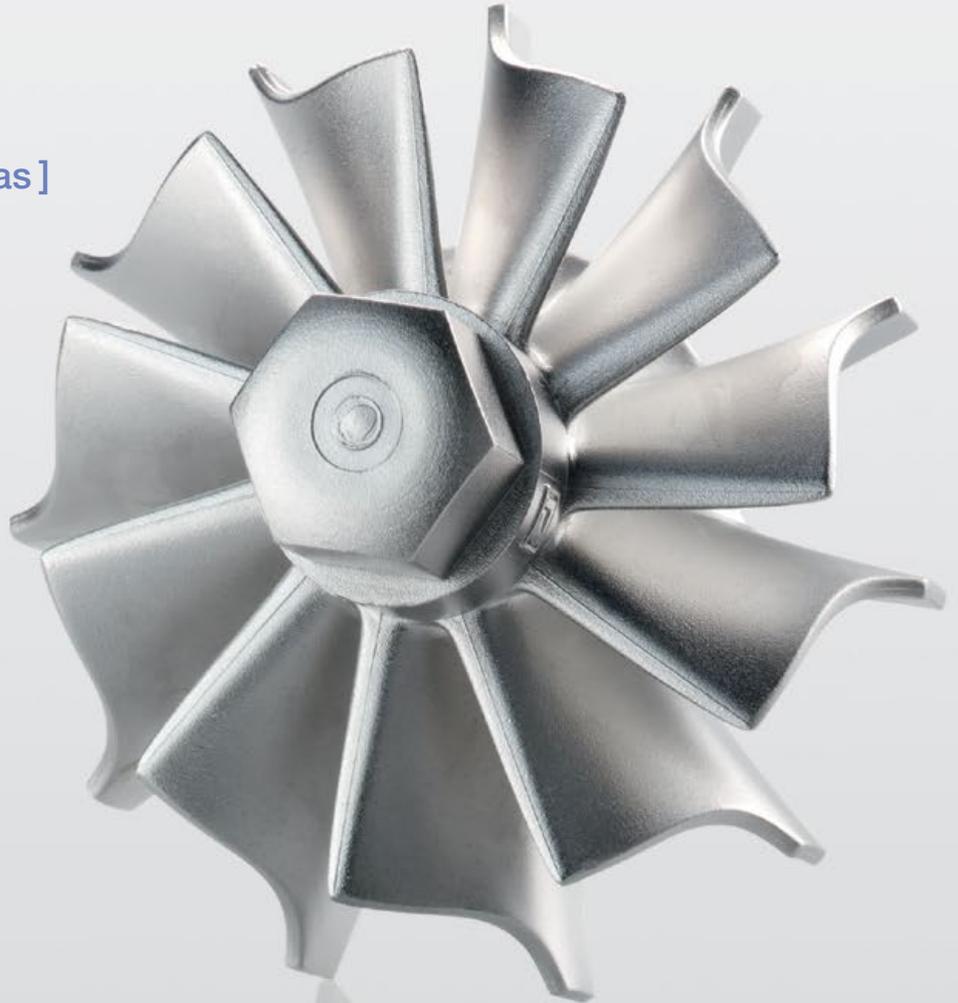
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 **BASF**

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## New BASF facilities in Asia support the growth of the Metal Injection Moulding industry

BASF is strengthening its activities in the field of Metal Injection Moulding (MIM) in the Asia Pacific region with two new facilities for Catamold®, its ready-to-mold feedstock for MIM. BASF will set up a new Catamold® production facility at its Kuanyin site in Taiwan. The new plant will have an annual production capacity of more than 5,000 tons and will start-up in the second half of 2013.

In addition, BASF has opened a new technical service lab for the company's MIM feedstock business in Shanghai, China. The new technical service lab for Catamold® is located within BASF's Innovation Campus Asia Pacific in Pudong, providing technical support as well as customer training.

Currently, the region Asia Pacific represents approximately 50% share of the global MIM market. "At BASF, we expect that this share will increase to 60% by the year 2020," said Dr. Stefan Koser, Vice President of BASF's Metal Systems Business Unit. "The Asian

market is therefore one of the major growth drivers for our Catamold® business. The investment shows our strong commitment towards the Metal Injection Moulding Industry in Asia and will enable further substantial growth potential for this technology," Koser added.

"With the new lab we are now able to better and faster serve our customers" said Steven Hung, Head of Regional Business Management Metal Systems Asia Pacific.

Catamold® is BASF's ready-to-mould feedstock for metal injection moulding (MIM) and ceramic injection moulding (CIM), offering a diverse portfolio of low-alloy steels, stainless steels, special alloys and ceramics. Catamold® is successfully used in various applications, from the automotive industry to consumer goods, from the construction industry to medical or computer and communication technology.

[www.basf.com/catamold](http://www.basf.com/catamold) ■

## Titanium PM conference to be held in New Zealand

New Zealand's Titanium Industry Development Association (TiDA) has announced that it is hosting the 2013 International Titanium Powder Processing, Consolidation and Metallurgy Conference, to be held on 2-4 December 2013.

This conference follows on from the success of the inaugural Ti Powder Conference held at the University of Queensland in 2011. The Conference, taking place at the University of Waikato, will attract leaders in the titanium industry, including academics and manufacturers and international delegates.

The conference is supported by the Japan Society of Powder and Powder Metallurgy (JSPM), Chinese Society for Metals (CSM), University of Queensland, CSIRO, IRL and the University of Waikato, plus leading Ti manufacturers.

[www.tida.co.nz](http://www.tida.co.nz) ■

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## 3M completes the acquisition of CIM specialist Ceradyne

3M completed its acquisition of Technical ceramics and Ceramic Injection Moulding (CIM) specialist Ceradyne, Inc., headquartered in Costa Mesa, California, USA, on November 28, 2012. The completion follows the previously announced merger agreement among Ceradyne, 3M, and 3M's wholly owned subsidiary, Cyborg Acquisition Corporation, through the merger of Cyborg Acquisition Corporation with and into Ceradyne. As a result of the merger, Ceradyne is now a wholly owned subsidiary of 3M.

The combination of Ceradyne and 3M, it is stated, will enable new technologies and innovation for uniquely tailored materials requiring advanced ceramics. Ceradyne will join the 3M Advanced Materials Division within 3M's Industrial Business Group. The 3M Advanced Materials Division provides materials for lightweight solutions and materials for performance in harsh environments to customers in a broad array of growth industries.

Ceradyne Inc. develops and manufactures advanced technical ceramic products and components for the defence, industrial, nuclear power, oil and gas, solar energy, electronic, automotive/transportation and medical markets.

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

## Inmatec Technologies expands ceramic feedstock production

Inmatec Technologies GmbH, a producer and developer of feedstocks for ceramic injection moulding based in Rheinbach, Germany, has announced the commissioning of a fourth feedstock production unit.

Inmatec told *PIM International* that the feedstocks are produced on shear roll extruders featuring a special wear-resistant coating and commented that, "Utmost cleanliness is required during feedstock production and thorough quality control, ensuring the consistently high quality of the feedstocks produced."

The current focus of feedstock development at Inmatec is on high purity alumina materials for final translucent and transparent

components. The processing of powders for the production of glasses, stated the company, is a rapidly growing field of activity.

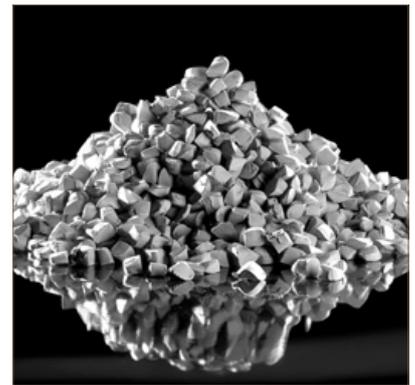
"In the meantime the demand for zirconia based feedstocks is also increasing. Moreover various other raw materials are requested which are processed into very specific products," added Inmatec.

With the new production unit Inmatec Technologies GmbH states that it is able to compensate production peaks. "Our already high flexibility is increased even more and our production units can be used specifically for certain types of materials."

[www.inmatec-gmbh.com](http://www.inmatec-gmbh.com) ■



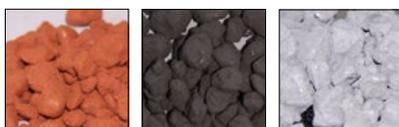
*Inmatec's CIM feedstock production is based in Rheinbach, Germany*



*Inmatec's CIM feedstock*

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## 2013 Fellow Award recipients announced by APMI International

Dr Olle Gröndler, a highly respected powder metallurgist who has actively contributed to the advancement of PM technology in Europe, Asia, and North America, and Roger Lawcock, Director, Product & Process Development, Stackpole International, regarded as one of the pioneers of the roll-densification process for gears, have been selected to receive the 2013 Fellow Award from APMI International. The award is APMI's most prestigious award and the presentation will take place at PowderMet2013, Chicago.

Gröndler has over 42 years of PM experience and has published 60 reports in international journals or conference proceedings. He has made unparalleled contributions to fully dense PM technology, and is one of the leading experts on the subject of hot isostatic pressing (HIP).

A recipient of a PhD in metallurgy and material science from the Royal Institute of Technology, Sweden,

in 1977, he has made significant contributions in the sintering of high alloys, including tool steels, stainless steels and cemented carbides.

Lawcock has promoted the advancement of PM as a science by disseminating and exchanging information through his many publication efforts. He received his MSc in metallurgy from the University of Manchester, UK, in 1987. During more than 30 years in PM he has focused on high-volume, high-performance automotive applications based on lean alloys, high-temperature sintering, and improvements to core and surface density. His work in the roll-densification process for gears is credited with converting numerous gear applications from wrought to PM. He has been instrumental during the material and design development phases of several award-winning components.

[www.mpif.org](http://www.mpif.org) ■

## Jayesh Industries expands fine powder production

Jayesh Industries Ltd., based in Mumbai, India, is an established manufacturer and exporter of ferro alloy powders. Now, as part of a major expansion programme, the company is starting fine powder manufacturing at a unit in Navi, Mumbai.

The company is commencing with the production of cobalt, nickel, tungsten, tungsten carbide and prealloyed powders. Jayesh Industries' Ashok K Gosavi told ipmd.net, "The above metal powder products will be introduced in the domestic and international markets from January 2013. The project is at the last stage of its completion. Our metal powder manufacturing unit is well equipped with all advanced production technology and quality systems and our intention is to supply our metal powder products to the hardmetal, diamond tools, PM and MIM sectors."

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

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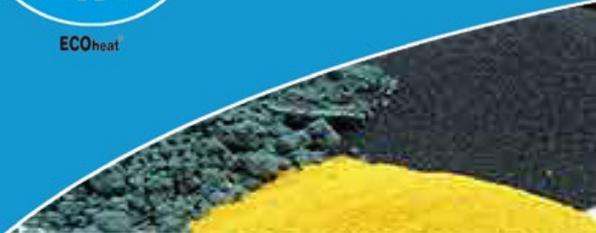
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Continues MIM Pusher Furnace

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## MIM pavilion set to debut at China PM Expo 2013

PM CHINA 2013, the 2013 China (Shanghai) International PM Exhibition & Conference, takes place from April 26-28 at the Shanghai Everbright Convention & Exhibition Center. Since the first PM CHINA event in 2008, PM CHINA has expanded into a major exhibition that attracts both domestic and international participants.

The 2013 event sees the debut of a new Metal Injection Moulding (MIM) pavilion, helping to strengthen industry recognition in China. The organisers state that the MIM pavilion will attract a wide range of visitors from a more diverse range of industries than conventional Powder Metallurgy.

The organisers state that a number of key industry suppliers including BASF, Atmix, Arburg, Sandvik Osprey, Cremer and Ametek are scheduled to participate.

A MIM symposium will also be held in parallel with the exhibition.

[www.cn-pmexpo.com](http://www.cn-pmexpo.com) ■

## EPMA 2013 PM Summer School to take place in Trento

The European Powder Metallurgy Association (EPMA) has announced that its 2013 PM Summer School will take place in Trento, Italy, from 8 - 12 July 2013. The Summer School is specifically designed for young graduate designers, engineers and scientists drawn from a wide range of disciplines such as materials science, design, engineering, manufacturing or metallurgy.

The five day residential event, consisting of a range of lectures given by PM experts drawn from both industry and academia, will be hosted by Professor Alberto Molinari and his team from the University of Trento.

Topics to be covered will include the manufacture of metal powders, powder compaction, MIM, modelling, sintering and Hot Isostatic Pressing. Participants will be able to discuss and solve problems as well as get hands on experience of various PM processes in the University's laboratories.

There will also be the opportunity to

visit a PM component manufacturer.

The Summer School, which is coordinated by Professor José Torralba from the University Carlos III in Madrid and Joan Hallward from the EPMA, is designed for young graduate designers, engineers and scientists from disciplines such as materials science, design, engineering, manufacturing or metallurgy. Graduates under 35 and who have obtained their degree from a European institution are eligible to apply. A fee of €575 covers tuition, course materials and accommodation.

[www.epma.com/summerschool](http://www.epma.com/summerschool) ■



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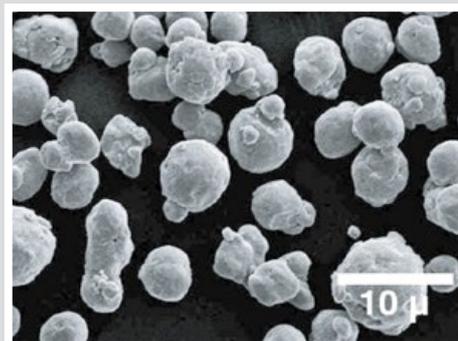
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	d10	d50	d90	Tapped Density
SNP-400	4.4	11.4	25.2	5.57
4SP-10	3.0	6.3	11.2	5.48
SNP-20+ 10	7.2	11.4	17.1	5.45
SNP+20	12.6	20.8	34.6	5.37



## LÖMI introduces new debinding furnace for debinding with organic solvents and water in one system

German debinding furnace manufacturer LÖMI has presented a new furnace that is capable of debinding with both organic solvents and water in one system. The new model, EDA-50LW, is the first of its kind in the industry and meets the increasing demand from new PIM producers wishing to easily enter the market as well as from established producers wishing to diversify their processes.

LÖMI's Ralf Wegemann told *PIM International*, "Our new furnace is available as a test or rental system so that PIM producers can test their processes at the LÖMI pilot plant stations or in their own company with a rental furnace. In this way, PIM producers can try out new kinds of feedstock and/or binder systems or optimise their existing processes, using both organic solvents and water to debind their green parts.

Should a PIM producer later decide to purchase a new debinding furnace, LÖMI will customise the furnace to the producer's process, based on the test results. The costs for the rental system can be credited to the new investment."

The company has many years of experience in the PIM industry and all their furnaces are developed in close cooperation with feedstock manufacturers and PIM part producers. The new furnace, states the company, is technically mature and robust and comes in a stainless steel design. It offers a closed system, making it environmentally beneficial. The furnace features an integrated drying of parts, saving time and costs, as no additional handling of the brown parts is required between debinding and drying. The integrated solvent recovery ensures a fresh debinding agent at all times. The furnace is



The new LÖMI LEDA-50LW debinding furnace is capable of debinding with both organic solvents and water

ergonomically designed as a front-end loader, and a large variety of trays and loading carriages are available in order to save time during the production process.

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



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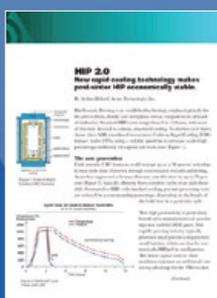
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The white paper describes current and future trends in hot isostatic pressing. You'll also receive a data sheet on uniform rapid cooling.

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## Indian consultancy to focus on MIM

Reflecting the growing interest in MIM in India, a new consultancy, LechTech Materials Pvt. Ltd (LechTech), has been formed. LechTech's Dr. Ravi Bollina told *PIM International* that his team offers in-depth technical know-how, financial knowledge and turnkey solutions for the complete range of PM industries, including MIM.

Bollina stated that current projects include setting up a MIM line and a recycling unit as well as business consulting for Mergers and Acquisitions and JV partners. "The MIM industry in India is nascent, except for the global giant Indo-US MIM. In comparison to China with 130 MIM plants, India has maybe one or two small ones, and now the industry is realising the potential of MIM there is more interest. In 2013, there will be two more MIM companies starting up in India. We see a growth in the number of companies coming up each year with specialised products for all industrial markets."

[bollina@lechtechmaterials.com](mailto:bollina@lechtechmaterials.com) ■

## Dr Lorenz Sigl receives Skaupy Prize at the 2012 Hagen Symposium

Dr Lorenz Sigl, Head of Innovation Services at Austria's Plansee SE, was presented with the 2012 Skaupy Prize at the Hagen Symposium, November 29-30. Organised by the Fachverband Pulvermetallurgie, the symposium is a key annual meeting for the German-speaking PM community.

In keeping with the theme of the symposium, "Powder Metallurgy: from raw material to the application" Dr Sigl's Skaupy presentation explored PM's potential as a source of innovation in green energy engineering. In his very broad approach he included both powder metal and ceramic materials.

New powder metal applications were identified in photovoltaic solar cells which transform sunlight directly into electric power without any negative side effects, in fuel cells and in membranes for the separation of hydrogen and carbon dioxide. These



*Presentation of the Skaupy Award to Dr Lorenz Sigl by Fachverband officials. From left: Prof Herbert Danninger, Dr Lorenz Sigl, Prof Bernd Kieback and Hans Kolaska (Photo courtesy Fachverband Pulvermetallurgie)*

applications have a great potential for growth in the future.

Dr Sigl grew up in the Austrian province of Styria and studied mechanical engineering at Leoben. His professional career led him to the Max-Planck Institute Stuttgart, Germany, the University of California Santa Barbara, USA, Elektroschmelzwerk Kempten, Germany, and finally Metallwerk Plansee, Austria.

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

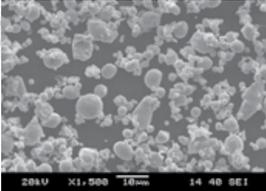




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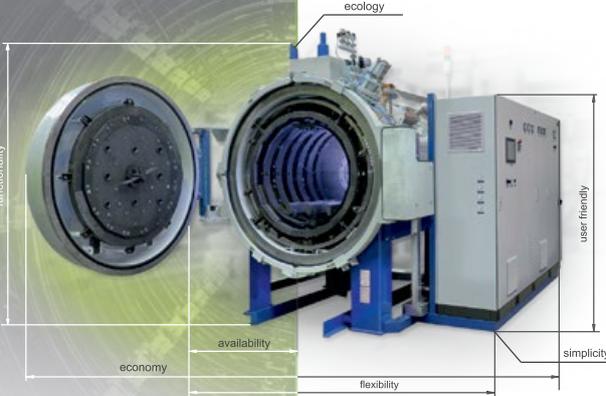


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## Potential for powder injection moulded ceramics in UAV engine components

Silicon nitride has long been seen as a potential ceramic material for manufacturing high-efficiency engine components for transportation and in portable power generators due to its high temperature stability, good wear resistance, excellent corrosion resist-



Fig. 1 Internal combustion engine for UAVs produced by Northwest UAV Propulsion Systems Inc. (From PhD Thesis by J. Lenz, March 2012)

ance, thermal shock resistance and low density (weight). However, the use of silicon nitride in engine components greatly depends on the ability to produce the near net-shape components required economically.

A research project at Oregon State University in Corvallis, USA, led by Juergen Lenz has been investigating the use of powder injection moulded silicon nitride for components used in engines for Unmanned Aerial Vehicles (UAVs) such as drones (Fig.1) where the target was to develop an energy efficient powertrain capable of operating in remote mission locations and with very low maintenance requirements.

The project for the UAV engine used a new material system consisting of a mixture of nanoscale and micro-scale particles of silicon nitride. Magnesia and yttria were used as sintering additives. The powders were mixed with a multi-component polymer binder system based on paraffin wax. The binder-powder (60% vol% solids loading) was analysed for its properties and moulding attributes. The study involved several steps of development and processing. These steps include torque rheometry analysis, mixing scale-up, property measurements of binder-powder, injection moulding with an Arburg 221M moulder, binder removal, sintering, scanning electron microscopy analysis and mechanical property measurements.

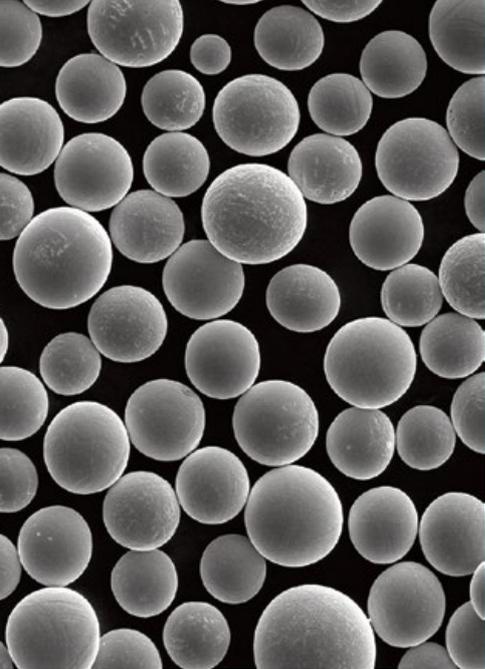
Simulations of the injection moulding process were conducted to assess the feasibility of manufacturing a ceramic engine and to determine its optimal process parameters. The study resulted in the successful development of design parameters that will enable fabrication of silicon nitride engine components by powder injection moulding.

Results from the research were recently published in the *Journal of Minerals, Metals and Materials Society (JOM)*, Vol 64, issue 3, 2012, 388-392.

[www.oregonstate.edu](http://www.oregonstate.edu) ■

### Submitting News

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



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

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

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

## HDH Powders: Greater Control of PSD

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

## PS Powders: Free Flowing

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

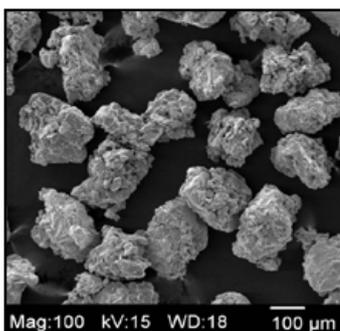
## Powder Metallurgy Applications

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

## Markets Served

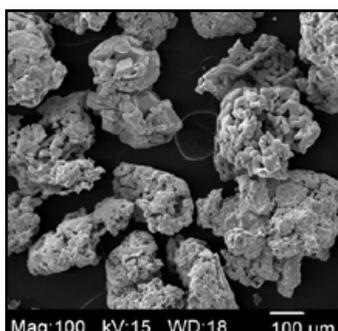
- Medical Device
- Thin Film (Sputtering Target)
- Feedstock
- Electronics
- Net Shape

Morphology: HDH Magnesium Reduced Titanium Sponge Powder



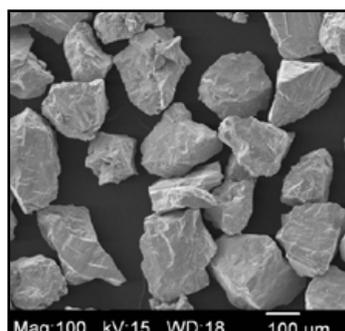
SEM image (x100) of HDH Magnesium Reduced Ti Sponge Powder, 70 mesh (212µm) x 100 mesh (150µm)

Morphology: HDH Sodium Reduced Titanium Sponge Powder



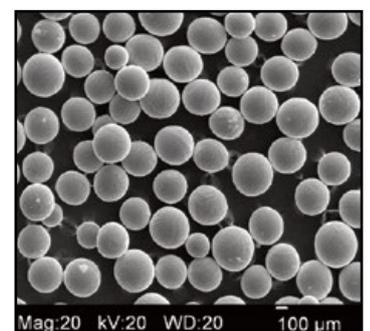
SEM image (x100) of HDH Sodium Reduced Ti Sponge Powder, 70 mesh (212µm) x 100 mesh (150µm)

Morphology: HDH CP Titanium Powder



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

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



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



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# Induction-heated quick-sinter system

patent pending

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

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

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

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

**For more information please contact us.**

## Bid for Swedish metal powder producer Höganäs AB

H Intressenter AB, a company jointly owned by Lindengruppen AB and Foundation Asset Management Sweden AB (FAM), announced on February 11 a public offer to purchase all shares in Swedish metal powder producer Höganäs AB.

As well as being a leading producer of iron powders for structural Powder Metallurgy components, Höganäs has been developing a new coarse powder feedstock system that offers the potential to economically produce larger components using the MIM process.

H Intressenter is offering 320 kronor a share in cash, according to a statement issued by the company, valuing Höganäs at around 11.5 billion kronor (\$1.79 billion). "Lindengruppen's ambition is to develop the businesses we own in a private context", stated Lindengruppen's Chairman Jenny Lindén Urnes. "We have therefore made a very attractive offer to the other shareholders."

Lindengruppen has been a long-standing owner of Höganäs through its full ownership from 1987-1994 and as the largest shareholder since the public listing in 1994. Lindengruppen has undertaken to transfer all of its shares in Höganäs to H Intressenter, provided that the offer is completed. Including Lindengruppen's 21.8% of shares and following H Intressenter's recent purchase of Industrivarden AB's 12.6% share stake, H Intressenter has 34.4% of the stock and 47.9% of votes in Höganäs.

Foundation Asset Management (FAM) states that it has significant experience from owning and developing industrial companies and a strong global network in sectors that are relevant to Höganäs. "This is an attractive opportunity for FAM to become a partner in a well-run business with a strong market position," stated FAM's CEO, Lars Wedenborn. "Höganäs is an interesting addition to FAM's portfolio which will create long term value and returns for the Wallenberg foundations. Lindengruppen's and FAM's competences complement each other well, which creates a solid platform for long term development of the company."

Höganäs has reported that its management board will announce its opinion of the offer and the reasons for its opinion, as well as an opinion as to the fairness of the offer from an independent expert, no later than two weeks prior to the expiry of the acceptance period. The company announced that Handelsbanken Capital Markets has been appointed as financial advisor and provider of the fairness opinion.

The acceptance period for the offer is expected to commence on 15 March 2013 and end on 12 April 2013, with an expected settlement date of 17 April 2013.

"Höganäs' employees have made the company the well-run business that we see today," stated Erik Urnes, CEO of Lindengruppen. "We have great confidence in the management team and the company's existing strategies, and will continue to make investments in the company, especially in research and innovation."

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



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## Hybrid PM-MIM process produces ultra-micro gears

Hitachi Powdered Metals, part of Hitachi Chemicals Ltd, located in Matsudo-shi, Chiba, Japan, sees considerable potential for Powder Metallurgy (PM) in the manufacture of micro and ultra-micro PM parts for multifunctional home appliances and advanced medical equipment.

However, until now PM processing of such parts has not been possible because of restrictions in the use of ultrafine metal powders in conventional powder compaction. The filling of such ultrafine powders into complex dies by gravity would be difficult due to a number of factors resulting in inhomogeneous pressure distribution during forming and the resulting density variations within the green compacts. To overcome these problems the company has developed a new 'flow forming' process which uses the same feedstock as used in Metal Injection Moulding (MIM).

A paper presented by Narutoshi Murasugi and Zenzo Ishijima at the PM2012 World Congress in Yokohama last October described the new flow forming process which the authors stated had been successfully used to produce sintered micro cup-shaped components having a wall thickness of just 0.05 mm, a record for PM, and also sintered micro gears with 10 teeth that are smaller than a grain of rice (Fig. 1). Murasugi stated that the feedstock for the new process was produced by mixing a polyacetal resin (POM) and paraffin wax (PW) binder with SUS440C stainless steel powder, having a mean particle size of 2 µm, in a dual axis kneader. The kneaded feedstock is supplied to the compacting die cavity using a specially developed cylindrical discharge device similar to a plunger in a MIM moulding machine. This ensures accurate weighing of the feedstock material into the die. The feedstock is heated to 433 K and a relatively low pressing force (<9N) is applied using a servomechanism at a punch stroke speed of 0.05 mm/s. The process can also produce two-stepped gears with a complex axis.

Debinding of the formed parts was done at 773 K x 7.2 ks, followed by sintering of the debound stainless steel parts at 1373 K x 3.6 ks in a dissociated ammonia atmosphere. The sintered parts were said to reach a relative density of 95% with a grain size of less than 5 µm. It was

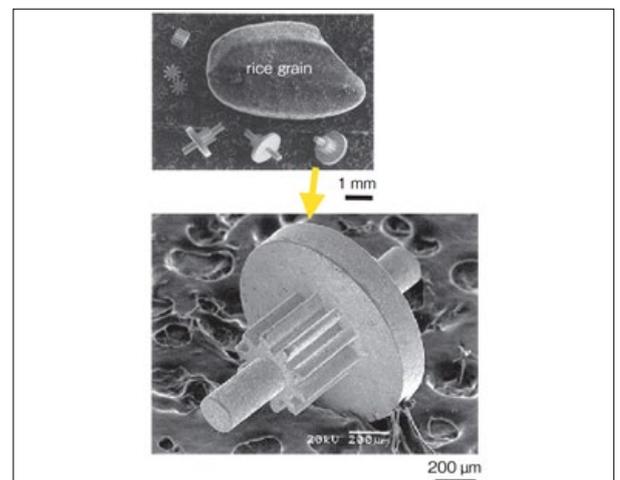


Fig. 1 Sintered micro gears produced under optimum flow forming conditions with grains of rice shown for comparison

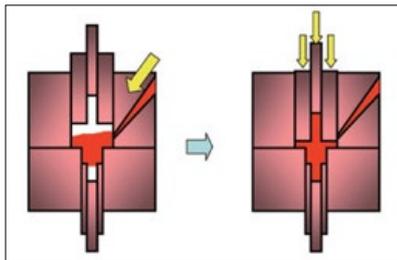


Fig. 2 Die structure and forming process using MIM feedstock

found that the parts could be sinter hardened to achieve a hardness of 680 - 730 HV.

Dimensional accuracy of the sintered micro gears was measured using a laser probe non-contact 3D measuring instrument, and the authors reported that the single pitch and total accumulative pitch error of the sintered micro gears was less than 2 µm. Total profile error was less than 8 µm, radial run-out less than 7 µm, coaxiality of the gear and shaft is less than 7 µm, and surface roughness less than Ra 0.31 µm. Improvement of the machining accuracy of the forming die is expected to lead to a better tooth profile and tooth run-out.

[www.hitachi-pm.co.jp](http://www.hitachi-pm.co.jp) ■

## International Porous and Powder Materials Symposium Call for Papers

The International Porous and Powder Materials Symposium and Exhibition, PPM 2013, will be held at the Sheraton Cesme Hotel Resort & Spa in Izmir, Turkey, from September 3-6, 2013. The deadline for submission of papers is March 15.

The symposium will focus on the fundamental aspects of characterisation, manipulation, production and usage of porous and powder materials. The organisers anticipate the symposium to be one of the largest gatherings of its kind, with a broad attendance from academia, research organisations, and industry. The event will also include an exhibition for displaying products, characterisation and production equipment.

For more information and to submit an abstract visit [www.ppm2013.org](http://www.ppm2013.org) ■

## Changes at Seco/Warwick

The Seco/Warwick Group has acquired Nespi International GmbH, a German furnace engineering company specialising in retrofits, repairs, service and spare parts for many types of furnaces. The acquisition will see Nespi International GmbH renamed Seco/Warwick Service GmbH.

Pawel Wyrzykowski, Group CEO, stated, "Our new company in Germany, in combination with our engineer teams and factory in Swiebodzin, will be an attractive proposal for many customers in Germany, Austria and Switzerland."

It has also been announced that control of the manufacturing sites Seco/Warwick Europe (Poland), Seco/Warwick Corporation and Retech (both USA), Seco/Warwick Retech (China), Seco/Warwick Allied (India) and the service sites Seco/Warwick Service (Germany) and Seco/Warwick Russia (Russia) have been consolidated under the Seco/Warwick Group.

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

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## New atomisation technology produces fine amorphous metal powders

The Research Centre for Metallic Glasses at the Institute of Materials Research, Tohoku University, in conjunction with the Department of Mechanical Engineering at Iwate University and Hard Industry Ltd which is based in Hachinohe, Aomori Pref., Japan, have announced the development of new atomisation technology aimed at the volume production of fine iron-based amorphous alloy powders having particle size in the single micron range. The new amorphous powders are expected to find applications in soft magnetic materials, thermal sprayed coatings, and because of their fine size and spherical particle shape, also powder injection moulding and powder metallurgy applications.

Hard Industry Ltd, which produces equipment and materials for hardfacing and thermal spraying as well as a range of machine tools, stated that the new atomisation technology, for which a patent has been applied, involves the use of a high-velocity combustion flame with kerosene combining with air to create the combustion flame. This compares with conventional gas and water atomisation processes with the former using high-pressure gas as the atomising medium, and the latter using high-pressure water. Each is said to have its drawbacks in the production of ultra-fine,

spherical shaped metal powders such as safety regulations in the use of high-pressure gas (e.g. argon), and water atomisation requiring expensive high pressure pumps. In contrast, the high-velocity combustion flame atomisation process overcomes these safety requirements, and is said to require only equipment to produce the high-speed flame, thereby cutting equipment and operation costs compared with conventional atomisation processes. The combustion flame is said to have a speed of 1600 m/s and temperature of 1600°C.

The developers of the new technology state that in the first stage of the simplified atomisation mechanism, molten metal is spun at high relative velocity. In the second stage, the surface tension divides the spinning molten metal into small particles. It is, therefore, essential that both the surface tension and viscosity of the molten metal be maintained at a high temperature in order to produce the fine powder, and this is achieved by using the high-velocity combustion flame. This compares with conventional atomisation processes where conventional gas and water atomisation methods use lower-temperature media, which causes the atomisation temperature to decrease during atomisation and this suppresses the atomisation mechanism for fine powders. If the temperature of the feeding molten alloy should be increased excessively in conventional atomisation then the lifetime of the crucible and nozzle would be considerably reduced.

Hard Industry states that the newly developed atomisation equipment can be categorised into two groups, which use multiple high-velocity combustion flame burners and ring-slit-shaped high-velocity combustion flame burners, respectively. The entire process is referred to as the counter-flame jet atomisation (CFJA) method. A schematic diagram of the CFJA method using four high-velocity combustion flame burners is

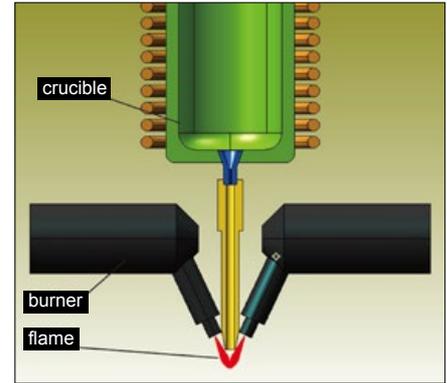


Fig. 1 Schematic diagram of the newly developed counter-flame jet atomisation (CFJA) process

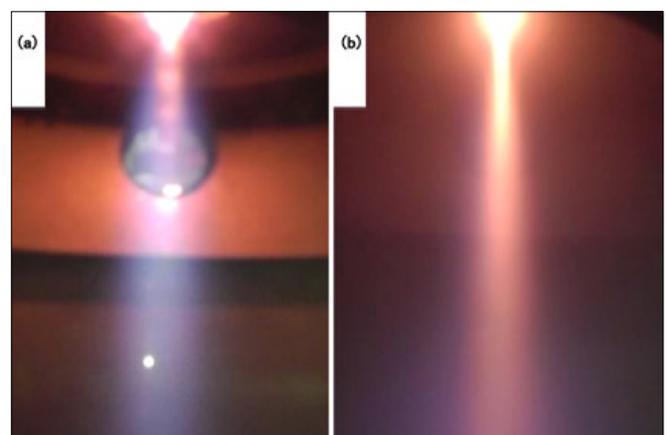


Fig. 2 (a) Combined flame from four individual burners; (b) appearance of molten metal atomisation using the combined four flames

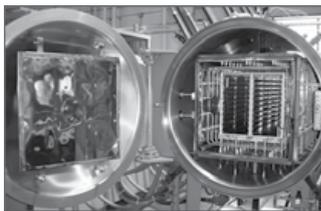


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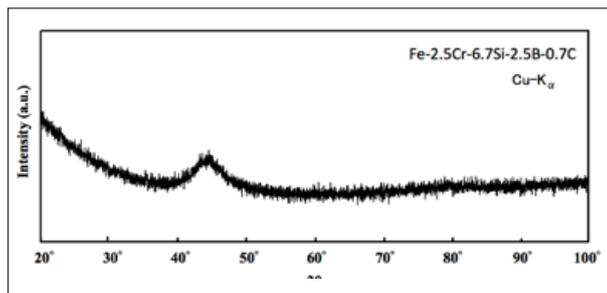


Fig. 3 X-ray diffraction of  $Fe_{73.2}Cr_{2.2}Si_{11.1}B_{10.8}C_{2.7}$  (Fe-2.5Cr-6.7Si-2.5B-0.7C in wt.%) amorphous alloy powder using a specific rapid cooling system

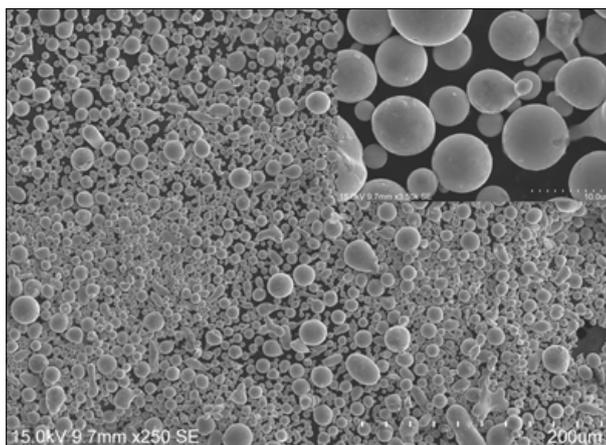


Fig. 4 SEM image of  $Fe_{73.2}Cr_{2.2}Si_{11.1}B_{10.8}C_{2.7}$  (Fe-2.5Cr-6.7Si-2.5B-0.7C in wt.%) amorphous alloy powder using a specific rapid cooling system

shown in Fig. 1. The four L-shaped burners create a cross-shaped intersection of the combustion flame by setting the four flames at a vertex angle of about 50°. Fig. 2(a) shows the combined flame after the four flames intersect. Furthermore, the combined flames at the intersection exhibit a feature called 'shock diamonds', which are usually observed in high-velocity flames with velocities greater than that of sonic waves. The combustion conditions of the four individual flames and the spinning speed are automatically controlled by computer. Fig. 2(b) shows the atomisation process using the four counter-crossed flames shown in Fig. 2(a). The colour of the molten alloy darkens in the area outside of the combined combustion flames.

Adding a developed specific rapid cooling system to the new atomisation process allowed the atomised molten alloy powder to be quenched in a dry state in the CFJA process, to produce an amorphous  $Fe_{73.2}Cr_{2.2}Si_{11.1}B_{10.8}C_{2.7}$  (Fe-2.5Cr-6.7Si-2.5B-0.7C in wt.%) alloy powder. The phases were characterised by X ray diffraction, as shown in Fig. 3. The spectra exhibit only a broad halo pattern of amorphous structure without any distinct Bragg peaks.

A scanning electron microscopy image of the amorphous atomised iron-based powder is shown in Fig. 4. Because it is difficult to classify such fine powder particle sizes, a wide dispersion in powder size can be seen. However, the smallest particle sizes are identified as being in the single micron range. The atomised powders are spherical in shape, have good flowability with good densification expected for sintered powder injection moulded and powder metallurgy processed components. ■



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## Eli노 develops new Eco-MIM batch debinding and sintering system

Eli노 Industrie-Ofenbau GmbH, based in Düren, Germany, has supplied custom-heat treatment and sintering equipment since 1933 and more than 4,000 furnaces have been delivered worldwide. The company has recently developed its new "Eco-MIM" batch furnace range for the complete processing of MIM parts. This new system, which the company claims is very energy efficient, has recently been validated and approved.

The approach that Eli노 has taken with its new system is to develop an Eco-MIM one-step complete debinding furnace, which is designed to work alongside a new Eco-MIM batch sintering furnace. The one-step debinding unit offers catalytic debinding, thermal debinding and pre-sintering up to 900°C with a special convection system offering temperature homogeneity of ± 5°C. A special off gas burner system is installed and different atmospheres (Ar, N<sub>2</sub>, H<sub>2</sub>, N<sub>2</sub>-H<sub>2</sub>, and air) can be used during the thermal debinding process, depending on materials selection. Ceramic materials can also be thermally debound in pure air. If required, the system is able to switch to a different protective atmosphere during processing and prior to pre-sintering.

Water or solvent debound parts can be dried in the Eco-MIM debinding furnace at between 80 and 90°C. Different temperature-time profiles can also be defined for the



The new Eli노 Eco-MIM debinding furnace



The Eli노 Eco-MIM sintering furnace

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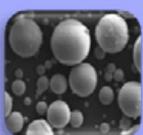
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various debinding steps with different atmospheres. Part production, states Elino, can be increased up to three cycles per 24 hours with an air cooling system.

The company states that separate thermal debinding is beneficial for the complete removal of residual binder and results in fewer hydrocarbons contaminating the sintering furnace.

The company's "Eco-MIM" batch sintering furnace is also claimed to offer cost effective processing and Elino states that the sintering cycle can be shortened by using a fast heating ramp, increasing production to three complete cycles in 24 hours. "Our system results in no condensation, no contamination, no blockages in the vacuum pump and longer muffle life in the vacuum sintering furnace," stated Elino.

Charge carriers have been designed to fit exactly the same way in both the sintering furnace as in the debinding furnace making the transfer of parts easier.

The company also states that reactive materials such as Ti and Ti alloys can be sintered with fewer impurities under vacuum levels from  $10^{-2}$  to  $10^{-5}$  with temperature accuracy of  $\pm 3^{\circ}\text{C}$  at a maximum  $1450^{\circ}\text{C}$ . Process gases such as Ar,  $\text{N}_2$ ,  $\text{H}_2$  and a  $\text{H}_2\text{-N}_2$  mixture can also be used in the sintering furnace.

Elino has been a manufacturer of continuous debinding and sintering furnaces for MIM for a number of years, with its systems used in a number of countries. The company claims that its continuous MIM furnaces consume less process gas than alternative systems and are amongst the most cost-effective to operate.

[www.elino.de](http://www.elino.de) | [www.elino-us.com](http://www.elino-us.com) ■

## MIM set for more gains in North America in 2013

Following growth years in 2011 and 2012, the Metal Injection Moulding (MIM) industry is expecting further gains in 2013, states Peter K Johnson in his 'State of the North American MIM Industry' report, published in the *International Journal of Powder Metallurgy* (Vol. 49, No. 1, 2013, 17-19).

Metal powder shipments to the MIM industry are estimated to have increased to a range of 900 to 1350 tonnes in 2012 with stainless steel powders making up around 65% of the total. Low alloy steel powders made up 36.5%, soft magnetic powders 6.8%, tungsten 1.3% and other powders 4.2%.

MIM parts for the firearms industry continued to dominate the applications for MIM components, stated Johnson, making up 42.3% of production by weight. This is followed by the medical/dental sector (14%), and the automotive sector at 13%.

Johnson also reported that many MIM parts are now being hot isostatically pressed (HIPed) to produce pore-free, or close-to-pore free density components. It is conservatively estimated that today the HIPing of MIM parts represents 6-7% of the HIP processing business. One MIM producer reports that HIPing can provide a 10% improvement in mechanical properties.

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## Fully automatic PIM multi-component tool kit enables the replication and joining of parts without brazing for nuclear fusion power plants

At Karlsruhe Institute of Technology (KIT), Germany, divertor design concepts for future nuclear fusion power plants beyond ITER (International Thermonuclear Experimental Reactor) are being investigated, as well as manufacturing methods for the mass production of such parts. The divertor is one of the most important plasma-facing components of the reactor. The component must remove impurities from the fusion plasma and has to withstand high surface heat loads.

Preferable materials that are able to withstand such extreme conditions are tungsten and tungsten alloys. One of the most promising divertor design concepts developed at KIT for the future nuclear fusion power reactor DEMO (DEMOstration Power Plant) is based on modular He-cooled finger units. Each 1-finger module consists of many individual parts, for example a tungsten tile and a tungsten alloy thimble. For the whole divertor system more than 250,000 individual parts are needed.

The manufacturing of such tungsten parts by mechanical machining such as milling and turning is extremely cost and time intensive as the material is very hard and brittle. Powder Injection Moulding (PIM) promises to enable the large-scale production of parts with high near-net-shape precision, hence cost-saving when compared to conventional machining. The PIM process was adapted and developed at KIT for one- and two component tungsten PIM and promising results have already been achieved.

The most recent challenge was to apply a suitable joining process to a mass production technology such as PIM. The motivation for this work was therefore the development and investigation of an alternative joining method for two different materials by using 2-Component-Powder Injection Moulding (2C-PIM). Based on the previous results of the tungsten the PIM divertor part, a newly developed



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Fig. 1 Green parts (top left), finished parts after heat-treatment (top right) and cut views (bottom) of the produced 1C-PIM and 2C-PIM mockups

fully automatic 2C-PIM tool allows the replication of fusion relevant components such as the tungsten tile and the tungsten alloy thimble in one step without additional brazing. The microstructure of the finished samples, and the quality of the joining zone, were characterised and found to be remarkably fine.

Green parts, finished parts and cut views of the finished mock-ups are shown in Fig. 1. The material connection of the 2C-PIM combination W + W-2La<sub>2</sub>O<sub>3</sub> (Fig. 1, bottom right) are successful. No cracks or gaps in the seam of the joining zone between the W tile and the W-alloy thimble are visible and a solid bond of the material interface was achieved. In comparison, the cut view and the resulting microstructure of the 1C-PIM mock-up consisting of pure tungsten is shown in Fig. 1, bottom left. An interesting detail is the boundary line between the tile and the thimble (marked by the white arrows), which is still visible only for the 2C-PIM parts. Further steps are the investigation of material properties via mechanical characterisation and HHF-tests.

This work, states KIT, has demonstrated that PIM is a powerful and viable process for mass production even when the joining of complex shaped parts is required and is an ideal tool for scientific investigations on prototype materials. For more information please contact Dr. Steffen Antusch, email [steffen.antusch@kit.edu](mailto:steffen.antusch@kit.edu)

[www.kit.edu](http://www.kit.edu) ■

## Powder Metallurgy Review: Spring 2013 issue out now

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

The issue includes an exclusive ten page report on the current state of Japan's PM industry by Dr Yoshinobu Takeda, Höganäs Japan KK. Dr Takeda explores how a lack of growth in domestic demand has seen the country's PM companies establish overseas subsidiaries that are able to thrive in the booming Asian automotive market.

This latest issue also offers a unique insight into the views of GKN Powder Metallurgy's recently appointed CEO, Peter Oberparleiter, as the division continues to go from strength to strength.

Paul Whittaker, Editor of *Powder Metallurgy Review*, stated, "Following the very positive feedback received for our launch edition at the Euro PM2012 Congress, Switzerland, and the PM2012 World Congress, Japan, we are very pleased to present this much expanded 72 page issue."

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## New method for cost effective classification of ultrafine 316L stainless steel powders

Stainless steel powders make up more than half of all the various alloy powders used in the global Powder Injection Moulding (PIM) industry, and there is increasing pressure on powder producers to supply the finest size fractions at competitive prices for the production of complex PIM components. Stainless steel powder for PIM is generally produced by gas atomisation using argon or nitrogen, or high pressure water atomisation, in order to achieve the desired spherical particle shape needed for PIM processing. However, both atomisation routes produce a relatively coarse powder from which the desired very fine powder needed by PIM producers (<40 µm and finer) have to be sieved out using air separation methods. This can make the required ultrafine stainless steel powders very expensive.

Now an interdisciplinary research

project at the Division of Surface and Corrosion Science at the Royal Institute of Technology, Stockholm, Sweden, has developed technology whereby ultrafine 316L stainless steel powder particles lower than 20 µm can be economically separated from the bulk of the atomised stainless powder using magnetic separation. Dr Yolanda Hedberg and her colleagues in the paper "Ultrafine 316 L stainless steel particles with frozen-in magnetic structures characterised by means of electron backscattered diffraction" (*Materials Letters* 2011;65(14):2089-2092) found that using electron backscatter diffraction (EBSD), a technique traditionally used to assess structural information for massive materials, the smallest particle size fraction of gas atomised AISI 316L stainless steel powder (<4 µm) had a different crystallographic structure

compared with larger size fractions of the same powder (<45 µm).

Despite similar chemical compositions, as shown in Table 1, the researchers found significant differences in crystallographic structure between the two particle size fractions of gas atomised 316L stainless steel powders (Fig. 1). Most of the ultrafine particles revealed a predominantly ferritic bcc structure (red) which existed as single crystals, whereas the larger size fractions generally showed particles having slightly smaller grain sizes compared with massive 316 L, but with the same austenitic crystallographic fcc structure (green) as in massive 316 L. As expected, ferrite was present to a very low extent (less than 10%) in the larger sized particle fraction and in massive sheet.

To explain why the thermodynamically unstable ferritic phase could be formed and predominate in most of the finest sized particles of the ultrafine 316L powder, particles of both size fractions were embedded, polished and etched



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to obtain further microstructural information via SEM imaging. The larger sized particles (<45 µm) revealed a dendritic microstructure, whereas the more rapidly cooled ultrafine sized particles (<4 µm) showed a cellular microstructure. The theoretical cooling rate of the larger sized particles was calculated based on the secondary dendrite arm spacing (DAS) value, which was estimated to be approximately 1 µm, which according to these calculations would correspond to a cooling rate in the order of 10,000 K s<sup>-1</sup>, or even some orders of magnitude higher than previously suggested in the literature. The cellular microstructure of the ultrafine sized powder suggests an even faster cooling rate, also indicated by the fact that most individual particles in fact existed as single crystals. Thermodynamic considerations suggest that the melt must be undercooled by at least 900 K to form ferrite in preference to austenite.

The researchers concluded that

the differences in crystallographic structure and hence magnetic properties of the ultrafine metastable ferrite 316L stainless steel powder opens up the possibility for magnetic separation, without any expensive sieving or air classification steps, of the smallest particle size fraction obtained during gas atomisation of austenitic 316L stainless steel particles. They believe that the technology will help to lower the cost of producing PIM grade 316L powders, and potentially other ultrafine high-alloyed gas atomised powders, which in turn will help manufacturers achieve cost effective production of complex PIM components.

This research work has led to an application for a World Patent (WO 2012/125113) and the research team is keen to further exploit the invention with any interested parties.

Further information is available from Dr Yolanda Hedberg, email: yolanda@kth.se ■

	Fe (wt.%)	Cr (wt.%)	Ni (wt.%)	Mo (wt.%)	Mn (wt.%)	Si (wt.%)	C (wt.%)	S (wt.%)
<45 µm	68.9	16.8	10.3	2.1	1.4	0.5	0.03	0.01
<4 µm	65.5	18.5	11.6	2.3	1.4	0.65	0.05	0.008

Table 1 Nominal composition of the 316 L powder particles based on supplier information

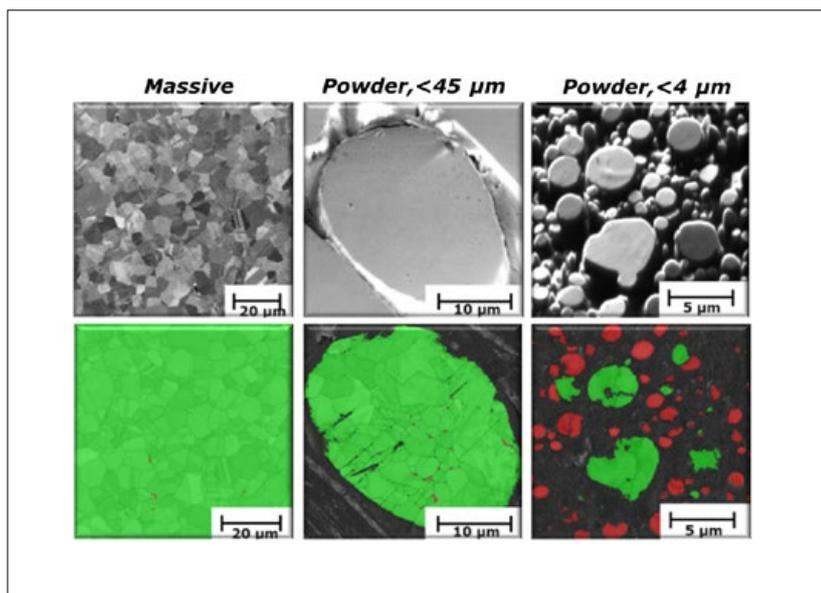


Fig. 1 Differences in microstructure (grain sizes) and crystallographic structure of 316 L as massive sheet (left), gas-atomised particles <45 µm (middle) and particles <4 µm (right), by means of SEM and EBSD showing the orientation of the different grains and different phases (green: austenitic and red: ferritic)

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## Formatec promotes innovations in technical ceramics technology to Dutch industry

Dutch Ceramic Injection Moulding producer Formatec Ceramics, based in Goirle, organised a seminar on December 13 2012 promoting the latest advances in technical ceramics, including MicroCIM, silicon nitride CIM and 3D ceramic printing. Close to 100 preregistered guests attended the seminar, which included guided tours of Formatec's plant and product demonstrations.

Michiel de Bruijcker, Managing Director of Formatec, told *Powder Injection Moulding International*, "This day was our opportunity to share the latest developments in technical ceramics with Dutch industry and the interest generated by the event was greatly above expectation. The showcasing of our newest capability, 3D printing with technical ceramics,

certainly caught the attention of delegates."

After a welcome by de Bruijcker, the first speaker was Joep Brouwers, Vice Director of Brainport Development. Brouwers presented all the details about the technology region, known as 'Brainport', located in and around the city of Eindhoven. This region is currently listed as one of the top technology centres in the world, with its success primarily driven by an 'Open Supply Chain' philosophy. The local mindset between sub-supplier, supplier and OEM companies, combined with a no-nonsense mentality, has positively affected business relations.

The application of silicon nitride was illustrated with a short movie featuring a model airplane with a

functioning jet-engine. The turbine wheel of the demonstration jet engine was fabricated from silicon nitride. During this part of the presentation, Formatec commented on the feedstock development for silicon nitride and the material properties, such as high fracture toughness, extreme wear resistance and great thermal shock resistance, were discussed.

Formatec explained the limited shaping capabilities of the material to-date, such as hot pressing, extrusion and grinding, however Formatec has now added the possibility of injection moulding silicon nitride so that more complex shapes can be achieved.

Current feedstock development is targeted at three visual appearances for the material, grey, dark grey and gold bronze. Such innovations, state Formatec, are attracting attention for the aesthetic as well as functional use of this material. It was stated that this is supported by the excellent finishes



Participants at the Formatec seminar in Goirle



Tour of an injection moulding area at Formatec



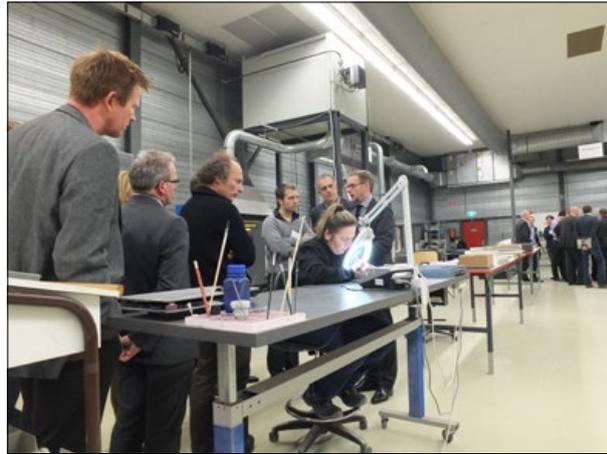
Explaining the DLP printing principles as applied to ceramics



The tour of Formatec proved popular with participants



Production facilities at Formatec



Guided tours through production were part of the CIM seminar

that can be achieved by polishing.

The seminar also paid close attention to Micro-CIM technology. A case study of a fibre optic micro connector featuring 64 holes on a surface of 2x2 mm was chosen to explain the potential of MicroCIM. Each hole is 125 micron and is directly formed in the mould cavity.

Formatec stated that the future of Micro-CIM depends not only on smaller and more precise tooling, but on an integrated strategy covering powders, tooling, injection moulding equipment and measuring technologies.

The presentation was concluded with the announcement that Formatec is a partner in the Hi-Micro EU project (High Precision Micro Production Technologies), initiated by the Technical University of Leuven. The Hi-Micro project is a three year project officially launched in October 2012 and, stated Formatec, guarantees Micro-CIM research funding for the coming years.

The final on-stage presentation was reserved for the day's highlight, the printing of ceramics via the Digital Light Process (DLP). 3D printing, or Additive Manufacturing, covers many technologies and numerous types of materials. Formatec presented, following an in-house market study, what these technologies can mean for shaping ceramics. It was quickly concluded that printing ceramics by use of the DLP-technology could be a significant addition to Formatec's product portfolio. Shortly after this conclusion Formatec started production with its first DLP printer, which was demonstrated during the CIM seminar.

The DLP process is based on a layer-by-layer curing of a photopolymer. This polymer is cured by light projection that shapes the product layer. A z-axis lifts the product and the curing process starts again. After the process is finished a 'green body' remains, similar to an injection moulded green body. This is then thermally processed via the company's debinding and sintering systems and the final product is a fully functional ceramic body with all the typical ceramic properties.

The presentation reviewed the new shaping possibilities that this technology offers. The technology will be applied to prototype production, but it was predicted that series production will also be performed with this technology, especially for small sized, highly accurate complex shaped products.

de Bruijcker commented to *Powder Injection Moulding International*, "The interest at the seminar was great and the post-event reaction was very positive. Overall the audience rated this seminar with 8.4 on a scale of 1-10 and highlighted the 3D printing presentation and the guided tour through production as most interesting. I believe that Formatec presented itself as a high-tech, innovative manufacturer that is not afraid of challenges, and that we can use these challenges to become more successful."

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# Powder Injection Moulding: Statistical trends and forward forecasts for the industry

The Powder Injection Moulding (PIM) industry has come a long way since the early 1980s, when sales reports were first published. In this exclusive report for *Powder Injection Moulding International*, Prof Randall German presents a statistical review of industry trends, including both metal and ceramic injection moulding, and considers what the future prospects are for an industry that serves a diverse range of end-user markets and has evolved to have such striking regional variations.

Almost everyone in Powder Injection Moulding (PIM), especially the readers of *Powder Injection Moulding International*, knows that PIM recently took some large leaps forward. For 2012 PIM technology accounted for about \$1.45 billion in sales. This activity was supported by almost 450 production firms and 200 key suppliers. It is an impressive performance, rising

from modest sales of \$6 million in 1986 when there were about 30 active operations.

This sales growth did not come as fast as projected, largely due to the cycles associated with large projects in computers, consumer products, cellular telephones, and jet engines; indeed most materials-intensive businesses always seem to grow

slower than projected. This review on industry trends relies on data collected over several years to document the changes. The trend lines are used to suggest future directions. Although PIM is growing, at the same time significant markets have passed away, such as parts for hard disk drives. It would seem small trends and small regional differences become amplified over time. This profile includes data on sales, employment, capacity, productivity, materials, and geographic variations to highlight future trends.

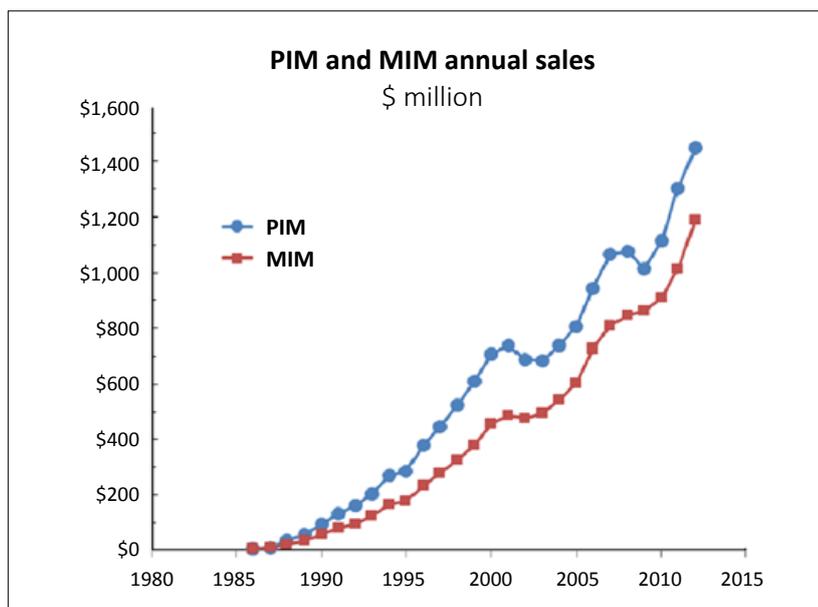


Fig. 1 Sales history for Powder Injection Moulding (PIM) and the subset of Metal Injection Moulding (MIM) plotted from earliest recorded values to 2012

## Overview

First sales reports on PIM came out in 1986, based on surveys of the approximately 30 firms initially active in the technology. This was about ten years after the first patents and pilot efforts arose. In 1986 the best guess on annual sales was about \$6 million. But a significant issue was the cost of those sales, estimated at about \$45 million. In other words, PIM was not profitable and most of the efforts were subsidised pilot projects. Although many firms watched, attended the conferences, and joined consortia, these observers were reluctant to build plants for an unprofitable industry. For obvious reasons, the early actors exited or married with larger firms – examples

The global PIM industry in 1997	
Number of PIM operations	234
Global sales for PIM	\$446 million
Global sales for MIM	\$277 million
Global employment in PIM	4075
Annual sales for largest PIM firm	\$21 million
Median company annual sales	\$0.5 million
Number of installed moulding machines	699
Number of installed sintering furnaces	540
Number of installed mixers	296
Percent of industry primarily captive	28%

Table 1 Glimpse of the global PIM industry in 1997

include Cabot, IBM, Rocketdyne, Brunswick, AMP, Degussa, Remington Arms, Deloro Stellite, Maya Technologies, Briggs-Stratton, Quest Technology, Multi-Material Molding,

tion Moulding) versus year are traced in Fig. 1. There have been some bumps in the sales growth, but the overall trajectory gives 22 to 24% per year compound increase for both PIM and

*'large projects come and go, leading to rapid gains during the upswing, but excess capacity and unprofitable operations on the downswing'*

DuPont, Form Physics, New Industrial Techniques, Injectamax, ES&P, and R&W Ceramics.

The estimate for 2012 global powder injection moulding (PIM) sales is \$1.45 billion. For perspective, the sales growth for PIM and MIM (Metal Injec-

MIM. Even with currency fluctuations taken into account, on the whole PIM simply grew with time.

After the year 2000 sales growth slowed and in three out of the most recent twelve years PIM growth was negative, twice declining by 6%.

Besides economic slowdown, the largest factor was due to aerospace ceramic casting core cycles tied to commercial aviation engine purchases. On the other hand, carbides remain a small but steady activity.

As evident in Fig. 1, today metals dominate the sales. In the 1980s and 1990s ceramic casting cores for aerospace applications grew quickly to account for almost 35% of the market. Indeed, when Certech was sold it had annual sales of \$91 million. However, since the turn of the millennium MIM has shown more growth and now accounts for 82% of annual PIM sales.

Large metal and ceramic projects tend to be cyclic. For example the large projects in recent years included automotive turbochargers, cellular telephone hinges, audio earphone jacks, computer hinges, watch cases and watch bands, hard disk drive latches and balances, jet engine linkages, and a host of related applications. Today MIM is dominated by buttons, plugs, switches, and connectors for tablet computers and cellular telephones. These large projects come and go, leading to rapid gains during the upswing, but excess capacity and unprofitable operations on the downswing.

In reviewing the industry, trends emerge that help to understand these cycles and the future. An unresolved question is whether these trends have value in predicting the future for PIM.

### A glimpse at PIM and MIM

The first complete benchmark statistical review on PIM and MIM was performed in 1996, when global PIM sales totalled about \$378 million distributed over 219 companies. This was followed by a more sophisticated review that captured details on the industry in 1997. Some highlights of that review are given in Table 1. Note the mean (or the average) company sales is \$1.9 million (\$446 million divided by 234), while the median is the 50% point (half are large and half are smaller) showing an abundance of small firms at that time.

In 1997 only about half of the industry was operating profitably, up significantly from the early 1990s. Although sales were increasing 22% per year, the parallel increase in the number of firms was 12% per year. Thus, many of the early MIM firms complained that growth reports were

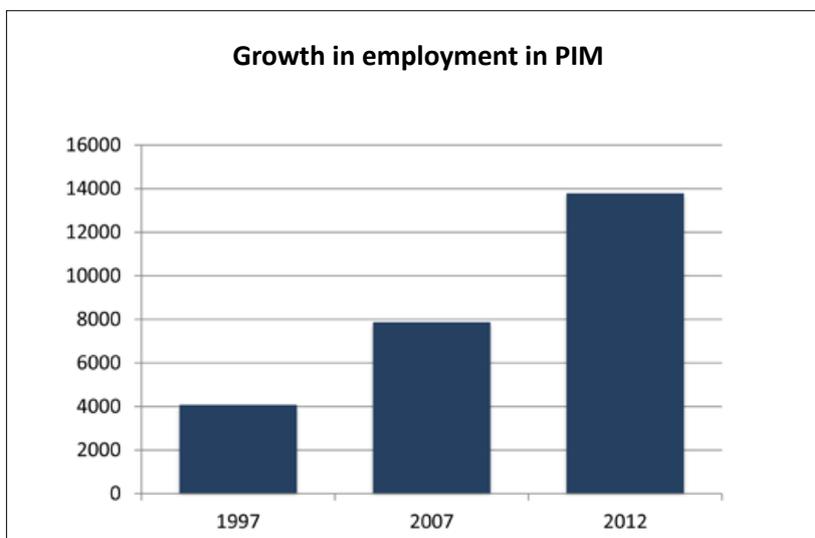


Fig. 2 Growth in PIM industry employment 1997-2012

too generous. Individual firms were realising less than the announced 22% growth rate, largely due to the increase in the number of competitors, many of which were operating at a loss. This spread sales over more and more firms. Such a trend continued into the early 2000s, when the number of firms with production or pilot operations peaked at 570, and included some large company actors such as Xerox, Kodak, Dow Chemical, Tyco, Panasonic, Olympus, Osram, T&N Technology, Sumitomo Cement, Nippon Tungsten, Kawasaki, and Cannon. Today a similar growth burst is recurring, with over 150 MIM operations in China. Most of these are smaller facilities, often set up with government grants, but still one company in China reportedly has over 200 installed moulders. If they track the usual trajectory in MIM, a few will grow to become significant actors in the field, but many will fade away.

For the past 26 years MIM averaged more than 20% growth per year, but for the past 10 years that has tempered to an average 9% per year. Only 2001 showed negative growth for MIM.

In retrospect, metal and ceramic powder injection moulding followed a classic technological chasm curve.

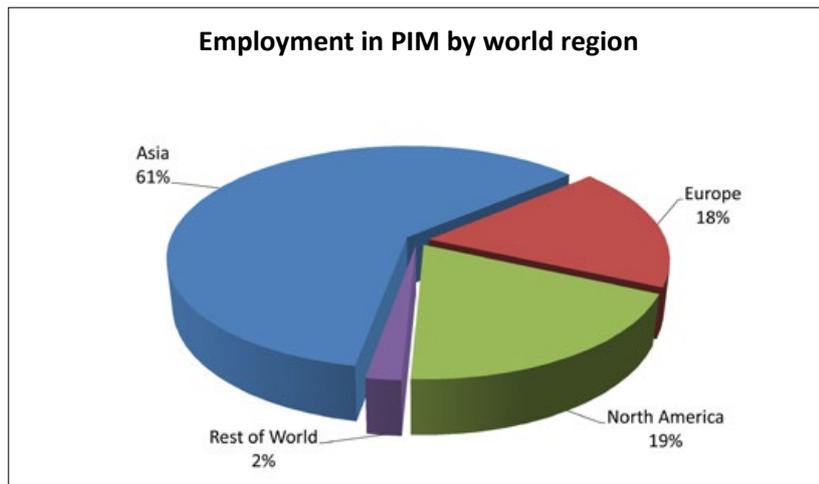


Fig. 3 Employment in the PIM industry by world region 2012

Innovation arose in the 1970s (reviving concepts from the advent of plastic moulding from the 1930-1940 time frame). MIM products won the first "Part of the Year" awards in the late 1970s, but had trivial sales for the next 10 years (the chasm), then rocketed at more than 30% per year growth, as several new firms entered the technology and the industry discovered profitability.

For the past decade PIM has generally experienced growth, mostly in the 8 to 14% range. The North American

variant is on the lower side and the Asian variant is on the higher side of this range. Unexpected events impeded sales growth, such as terrorist attacks in 2001 leading to the collapse of the aerospace industry.

The 15 years of statistical surveys shows an early identification of the unit manufacturing cell for PIM. Early facilities were typically small, with 10 to 20 people, batch mixing, two to three moulding machines, and two to three batch sintering furnaces. As growth occurred, the number of manufac-

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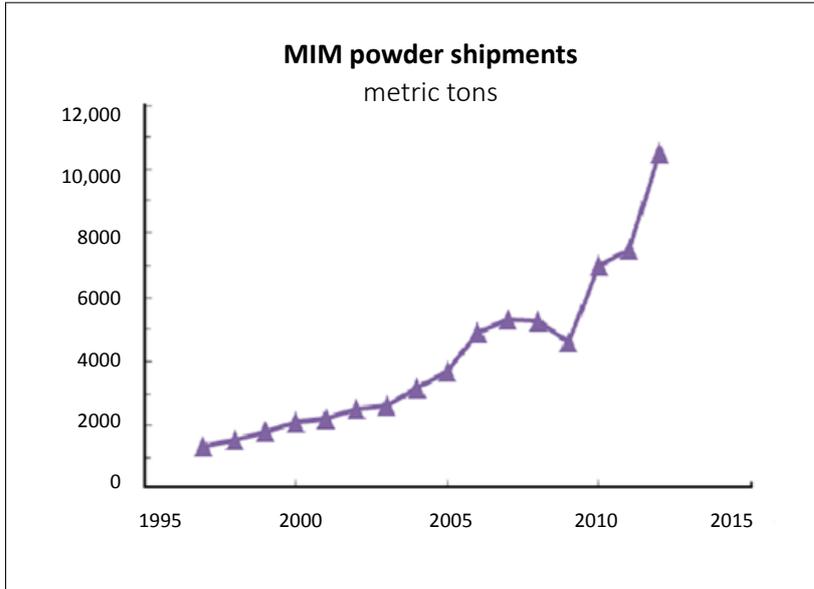


Fig. 4 Powder shipments to the MIM industry versus year. Realise that powder shipments and MIM part sales are not in synchronisation because of long transport and processing times

Region	Share of PIM firms	Share of PIM sales	Share of PIM employment	Share of MIM powder usage
Asia	49%	48%	61%	53%
Europe	27%	26%	18%	23%
North America	21%	24%	19%	21%
Rest of World	3%	2%	2%	3%

Table 2 Contrast and comparison of global sectors in Powder Injection Moulding

	1997	2012
Number of companies	234	445
Number of employees	4,075	13,800
Total annual industry sales	\$446 million	\$1,450 million
Installed number of moulders	699	2614
Installed number of mixers	296	462
Installed number of furnaces	540	1,167
Mean employees per company	17	31
Mean annual sales per company	\$1.9 million	\$3.3 million
Mean number of moulders per company	3.0	5.9
Mean number of furnaces per company	2.3	2.6
Mean number of employees per moulder	5.8	5.3
Mean number of employees per furnace	7.5	11.8
Mean annual sales per moulder	\$638,000	\$554,000
Mean annual sales per furnace	\$825,000	\$1,243,000
Mean annual sales per kg of powder	\$332	\$138

Table 3 Comparison of industry statistics and productivity ratio changes

turing cells grew, but the production ratios remained similar. Today, a typical facility has multiples of the same early production ratios. The unit manufacturing cell seems to be about:

- \$2.5 million in sales
- 20 employees per cell
- 1 mixer per cell
- 5 moulders per cell
- 2 furnaces per cell
- 10 million parts per year per cell.

There are many exceptions, including firms that purchase feed-stock (no mixers), to firms with large continuous mixers, to firms with 20 moulders and only one large batch furnace. But as a statistical profile of the industry, these values help define typical production ratios.

**PIM materials**

In terms of materials, the largest is stainless steel, a trend that started in the 1990s, as watch case and orthodontic bracket production switched to MIM. After stainless steels the next most popular materials are ferrous (steel) alloys largely based on carbonyl iron. In ceramics, alumina is the most common material, followed by silica, zirconia, and tungsten carbide.

The balance of MIM is applied to high performance superalloys, titanium, tungsten alloys, copper, tool steels, and electronic alloys. These latter materials have been the subject of much research attention, but so far account for less than 20% of sales.

**PIM applications**

In terms of applications, the most consistent market is industrial components. Automotive and consumer applications (mostly in electronic devices such as tablets, computers, and cellular telephones) are probably over 40% of the current market. Regional differences make firearm and medical-dental dominant in North America, electronic-computer-cell phone dominant in Asia, and consumer-automotive applications dominant in Europe.

A common denominator is widespread reliance on industrial components, such as those used in robots, plumbing fixtures, hand tools, speciality fittings, valves, sensor bodies, locks, solenoids, thermocouples, metal cutting tools, milling inserts, wire drawing dies, stone cutting beads, lighting devices, plumbing hand tools, and so on.

Regional trends were evident early in PIM. Ceramic applications are large in North America, while most of the rest of the world tends to focus on metals. As a brief summary, Table 2 compares four geographic regions with respect to several parameters – number of firms, sales, employment, and powder consumption. Each of the values is a percentage of the total.

Essentially this shows the Asian firms make smaller parts with more labour. The recent surge in MIM products for Apple, such as buttons, plugs, and connectors are aligned with this trend.

### Time trends

A significant change in PIM is how widespread the technology has become. The list of countries that have at least one PIM facility includes Australia, Austria, Belgium, Brazil, Bulgaria, Canada, China, Denmark, France, Germany, Hungary, India, Indonesia, Israel, Italy, Japan, Korea, Luxembourg, Malaysia, Mexico, Netherlands, New Zealand, Norway, Poland, Puerto Rico, Russia, Singapore, Slovakia, South Africa, Spain, Sweden, Switzerland, Taiwan, Thailand, Turkey,

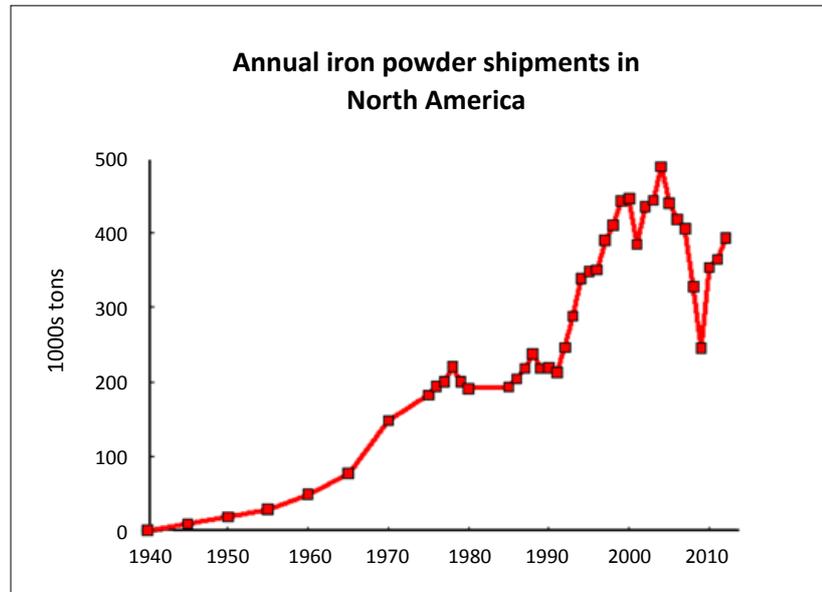


Fig. 5 Besides PIM, other powder-based manufacturing technologies have large demand changes as evident by the iron powder consumption for powder metallurgy in North America tracing back to 1940

United Kingdom, and the United States of America. Of these China, USA, Germany, and Japan have the largest number of firms, followed by Taiwan, Korea, and Switzerland.

One of the more telling sales trends has been the slower growth in North

America versus Asia. In 1997, North America accounted for 48% of PIM sales, but is now at 24%. Likewise, installed capacity in North America fell from 57% of the industry in 1997 to now about 21%. European sales have not changed as much and have ranged

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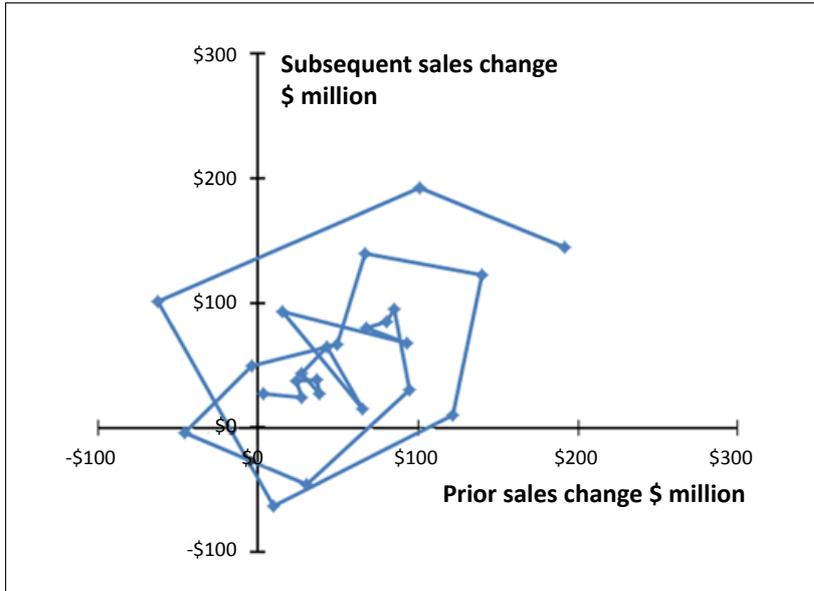


Fig. 6 A plot of the chaotic sales change for PIM over its history. This plot shows the change in PIM sales year to year where consecutive years are plotted against one another. For 2012 the gain was \$145 million and that makes up the y-axis value while the prior year's gain was \$192 million to give the x-axis value for the last point. The diagram would suggest that for the 2012-2013 pair, with now an x-axis value of \$145 million, would give a y-axis value near zero. Thus, due to high sensitivity to swings in large orders, PIM for 2013 might be a flat year. Regression analysis on the other hand would predict a 22% sales gain for 2013 to give a \$319 million sales gain

from 25 to 31% of global activities over the past 15 years, but Asia has grown dramatically, with several new and large MIM facilities in Korea, Taiwan, Thailand, and China to compliment the previously established facilities in India, Japan, and Singapore. Today, the largest firms employ upwards of 1500 people and have 200 installed

properly factor part-time head count). This increased in 10 years to 7870 and now for 2012 is at 13,800 people (Fig. 2). The majority of these positions are in Asia (Fig. 3). Overall sales per employee peaked in the 2007 to 2009 range and now are down to \$114,000 per year. This probably reflects some of the rapid growth in Asia.

***‘What is evident is a technology generally dominated by smaller firms. The typical PIM company is probably too small to provide for significant gains in research, marketing, or production innovation.’***

moulders, an impressive scale of production. Curiously the number of firms in North America peaked in the year 2000 and via merger, acquisition, or closure has fallen to under 95 today.

**Employment trends**

Employment in PIM has tracked the sales increases. In 1997 the total industry employment was 4075 people (based on full-time equivalents to

**Powders and feedstock**

Earlier a plot was given on the sales trend. Fortunately, data have been collected on a few other aspects to help understand the trends. Powder shipments to the industry are plotted in Fig. 4. In some cases the purchases were out of synchronisation with component sales, due to the time between powder shipment and component shipment, but the overall surge

in powder demand is evident in the past few years. This has coordinated with increased production capacity at all of the major powder fabricators and contributed to the qualification of several new producers.

In terms of feedstock purchase versus self-mixing, the data show a relatively stable ratio of 72 to 76% of the companies doing self-mixing, accounting for 77% of PIM sales. Smaller firms are slightly more likely to purchase feedstock. For the firms with internal mixing capabilities, the typical sales per mixer are over \$2 million per year and this statistic has been level for about 10 years.

**Production technology**

Moulding capacity has expanded with sales. Curiously, the global sales per moulder has not increased much, and remains near \$554,000 per moulder per year, down from a peak of \$643,000 in 2007. The 2012 figure is based on an installed capacity of 2614 moulding machines; about 10% are low pressure moulders as favoured for forming ceramics and carbides.

Debinding technologies once included a wide variety of options – chlorinated solvents, water, vacuum sublimation, air drying, freeze-drying, wicking, catalytic, oxidation, and thermal pyrolysis. In 1997 almost half the industry relied on thermal debinding and 25% relied on some form of solvent debinding. Today the industry has largely converged to three options – thermal pyrolysis (43%), debinding in either water or other solvent (35%), and catalytic debinding (21%).

Sintering furnace trends show about 1167 installed devices, with a significant number of continuous furnaces. Tracking of furnace capacity data is poor, so some of these are small tube furnaces for orthodontic bracket sintering and others are double-wide continuous furnaces capable of sintering four million parts per day. Overall the annual sales per furnace are constant for the past five years at about \$1.2 million per year.

**Productivity ratios**

In 1997, the annual sales were \$1.9 million per firm and today that ratio is \$3.3 million. To see where major changes arose in the technology, Table 3 compares the productivity ratios from 1997 and 2012.

Some impressive statistics are evident here, but also a decline in sales per employee and per unit mass of powder are troublesome.

What is evident is a technology generally dominated by smaller firms. The typical PIM company is probably too small to provide for significant gains in research, marketing, or production innovation. A few larger firms have significant investments, but the increase in employment and expanded capital investment in recent years (furnaces, mixers, and moulders) should have increased sales productivity – sales per employee, per moulder, per furnace, or per kg of material. Unfortunately, the productivity changes are inconsistent. It appears many of the production and sales gains are traced to lower costs and not to improved capabilities. Thus, as mentioned earlier, the unit manufacturing cell seems to be about the same for several years. The largest production change is probably via continuous sintering, reflected by the increase in sales per furnace.

### Major trends

Some trends become evident, and as a forewarning not all of the crystal ball view is good news. The positive news is in the sales growth, and increased capacity, powder consumption, and new products. Yet for some time the materials mix in PIM has remained stagnant with a dominance by stainless steel. Most of this business is coming from investment casting and machining. After stainless steels, next are the several ferrous alloys (Fe, Fe-Ni, Fe-Ni-C, and low alloy steels), then alumina. These three material groups are probably 80% of the PIM products. Other compositions remain as special materials – cemented carbide, copper, nickel, silica, superalloys, titanium, tool steel, tungsten, zirconia, and various electronic alloys.

Geographic trends are evident by the substantial growth taking place in China and India, but with degraded productivity figures based on sales per employee or sales per unit mass. In the past 15 years the sales growth rate in Asia has been double that of North America and Europe. This has supported significant gains in MIM, but these are tied to a few large projects and more labour intensity. A problem with large orders is in the pendulum swings; what is a boom now can quickly turn to a bust next year. All it takes is a new design that no longer includes MIM. For example, what if the stainless steel buttons on cell phones, computers, and similar devices are replaced by soft buttons on touch pads? All of a sudden the demand switches away from MIM with resulting idle capacity.

It is useful to view the possible MIM cycles using historical data. One up and down perspective comes from North American iron powder shipments. As plotted in Fig. 5, the cycle has had large bumps as the field grew, leading to a dramatic fall over a short period of time. The same thing happens in PIM as the projects increase in size. Some of the new, large scale products are “fashion” and have a production life of just six months.

In terms of binders, the wax-polymer systems remain dominant with over 30 years of history. Variants are designed for water immersion debinding, but mixing and moulding remain similar to early paraffin wax – carnuba wax – polypropylene variants that required chlorinated or flammable solvents for debinding. Water soluble binders based on polyethylene glycol, polyvinyl alcohol, and similar polymers emerged in the early 1990s, at about the same time as catalytic debinding, and both have achieved good market acceptance in the intervening 20 years. Even so, thermal pyrolysis (thermal debinding) is widely

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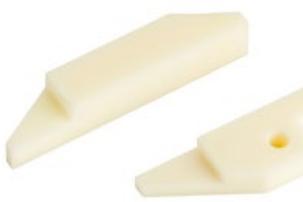
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employed since it is similar to the well-known delubrication and dewaxing treatments used in powder metallurgy, ceramics, and cemented carbides. In the past 15 years the number of binder and debinding options has declined to essentially three variants. Efforts to introduce new binders are hindered by a reluctance to change.

Self-mixing remains the dominant feedstock route. The majority of PIM firms enjoy the lower cost and production flexibility from self-mixing. No changes are evident in that situation, especially as patents expire and cost pressures arise. Self-mixing will sustain as the dominant approach.

The basic mix-mould-debind-sinter manufacturing cell was isolated years ago and seems to be unchanged. Continuous furnaces are applied to large projects, but still the bulk of the industry prefers the flexibility that comes with batch furnaces. Early PIM firms found a mix of batch and continuous furnaces allowed for a balance between cost and flexibility, and that seems to remain true.

### Future forecast

The large question comes with regard to the sales growth for PIM. Trend analysis shows that certain applications come and then go away, often rapidly – disk drive latches, cell phone hinges, watch cases, locks and latches, logos, and ammunition. Historically the ranking of the most stable to the least stable application is as follows:

- **Industrial:** sensor housings, robot components, pumps, valves, handles, lock components
- **Medical-Dental:** surgical tools, staplers, orthodontic brackets, endodontic tips, biopsy tools, orthoscopic fittings
- **Aerospace:** casting cores, inlet valves, latches, connectors, linkages, flow bodies
- **Firearm-Military-Law Enforcement-Defence:** trigger guards, handcuffs, triggers, firing mechanisms, spindles, projectiles, penetrators, sights, fragmentation projectiles, arming fuses
- **Automotive-Truck:** turbochargers, rocker arms, shift levers, interior mounts, lock and key systems, fuel injectors
- **Computer-Telecommunications:** glass-metal seal packages, heat sinks, wire bonding tools, hinges, connectors
- **Consumer:** arrowheads, luggage buckles, computer logos, cell phone hinges, golf club inserts, fishing weights, knives, camera components, musical instrument parts, lighting
- **Jewellery-Watches:** watch cases, winding mechanisms, wedding rings, cuff links.

The industrial components tend to have longer production runs, so once a component is qualified for PIM it remains in production for considerable time. Medical and dental applications are safe havens for production, although products may be difficult to qualify. They remain in production for considerable time and profitability has consistently been excellent. Companies that remain focused on industrial and medical-dental areas have a very stable base. In North America the firearm business remains solid, although the quantities are low; still the field supports several firms, including firms in Austria, Germany, China,

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for titan MIM parts

- Keralpor Y (dimensions up to 300 x 300 mm possible, standard thickness 1,1,5/2 mm)

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Russia, and Brazil. In some cases these same products are related to military, defence, or law enforcement customers. Automotive use of PIM has grown considerably. For applications such as turbochargers, the production quantities are large, until the vehicle is redesigned. More risky are the telecommunication, consumer, and jewellery and watch products. These

**'Automotive use of PIM has grown considerably. For applications such as turbochargers, the production quantities are large, until the vehicle is redesigned'**

products disappear quickly and leave idle capacity. Cell phones are redesigned every six months, so it is hard to keep up with that pace. Likewise, the logos for computers and luggage are a low technology application and should the fashion shift, production ends rapidly – it is easy to qualify for production, but likewise easy to be replaced.

Finally, a few comments on the five year forecast. The PIM growth curve seems smooth, but on close examination there is a chaotic characteristic. In examining the sales gains year over year, what emerges is a spiral of increasing amplitude. This is shown in Fig. 6, where the sequential year to year sales gains are plotted against each other. The PIM sales gain for one year is plotted against the sales gain for the next year, over 25 years. The growth pattern increases in amplitude, but goes around in a cycle of booms and busts. For 2012 the sales gain was \$145 million (y-value) over the prior year which had a sales gain of \$192 million (x-value). This gives the last point on the plot. The next point would then use \$145 million as the x-value. Following the prior cycle, then the projected y-value would be \$0, tracing out a spiral. Thus, 2013 would be a flat year with no sales growth. From a view based on chaos theory, PIM sales growth looks flat for the next few years.

An alternative view would be to project with the 22% annual growth. This is an unrealistic forecast, although everyone would like to see this happen. Such high growth would be difficult to sustain in terms of human resources, capital investment, tool design, and

product qualification. It is unclear if there would be a sufficient customer base willing to accept PIM to sustain such a high growth.

Looking forward, two options emerge – nearly flat growth giving 2017 sales of about \$1.8 billion versus sustained high growth giving 2017 sales of about \$3.9 billion. This is a considerable difference. The flat

growth is depressing, but reflects the fact that PIM is bidding on a few large projects and will likewise lose large projects. The final situation is probably between the \$1.8 and \$3.9 billion projections, giving 2017 global PIM sales near \$2.6 billion. It is a target anyhow. As with any portfolio, it is best to diversify.

### Acknowledgements

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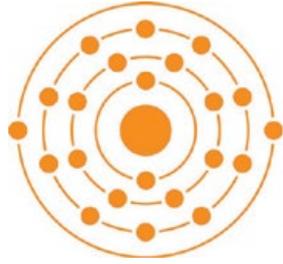
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Powder metallurgy is leading the way.



# Polymer Technologies Inc. looks to the aerospace industry for new PIM applications

In 2012 Polymer Technologies Inc., based in Clifton, New Jersey, USA, celebrated 25 years as a specialist producer of precision injection moulded components. Since the mid-1990s, when the company expanded from advanced plastics to Metal Injection Moulding, it has successfully targeted niche markets such as aerospace with a focus on high performance materials. *PIM International's* Nick Williams reports on recent developments.

Polymer Technologies Inc. (PTI) has, since the company's foundation in 1987, been focussed on the injection moulding of advanced materials for demanding specialist applications. Founded by Mel Goldenberg, now PTI's Chairman and Chief Technology Officer, the company quickly developed a reputation for the injection moulding of challenging and exotic polymer materials. Today the list of plastics processed includes advanced materials such as carbon filled polyphenylsulfone (PPSU) and nylon with multiple fillers including metal powders. Such expertise resulted in the company building a strong relationship with US military institutions and aerospace companies, as well as high value industrial customers in the oil, gas and medical industries.

It was in the mid 1990s that PTI first became involved in Metal Injection Moulding, initially through process development work with Allied Signal, later to become Honeywell. Sensing the potential in the technology, PTI took an opportunity to acquire Honeywell's MIM technology, including production equipment and key personnel.

In the subsequent years the MIM operation at PTI has grown in both scale and expertise. This has been in

large part thanks to the combination of the stability and continuity that is provided by a family owned and managed business, with Mel's son Neal joining the firm in 1997 and becoming President in 2011, and the depth of technical knowledge provided by key members of the original team at Honeywell who continued to expand their knowledge of MIM processing as PTI evolved.

## PIM production at PTI today

In 2013 PIM is expected to account for around 65% of the business generated out of PTI's 100,000 ft<sup>2</sup> plant in Clifton, New Jersey. The proportion of business generated by PIM has increased by 10% over the last 12 months, and the company expects this upward trend for its metal and ceramics business to



Fig. 1 The entrance to Polymer Technologies Inc.'s 100,000ft<sup>2</sup> facility in Clifton, New Jersey



Fig. 2 Neal Goldenberg (left), President of PTI with his father Mel (right), the company's Chairman and CTO

continue. Plastic injection moulding and toll sintering services account for the balance of the company's business. The plant, which operates 24 hours a day, five days a week, employs a staff of more than 70 and is currently operating at 50-55% capacity.

The United States accounts for approximately 80% of PTI's business, with military, defence, aerospace, firearms, medical, surgical, orthopaedic and general industry being the primary end-user markets served. Whilst PTI chooses not to compete with the largest MIM firms in the United States or worldwide on



Fig. 3 The 65 ft CM Furnaces 300 series continuous debinding and sintering furnace at Polymer Technologies Inc.

many commodity-type applications with little or no added value, it does believe that it stands out when it comes to specialist materials, hard to process parts and demanding end-user markets.

PTI's plant operates 24 injection moulding machines from Arburg and Engel, of which eight are dedicated

hot zone and is reserved for the sintering of short runs and specialist materials such as high temperature alloys.

PTI also offers toll sintering services which can be scheduled as needed to make the most efficient use of furnace capacity. "PTI's corporate strategy is to leverage its reputation,

*'The criteria for feedstock selection at PTI include an evaluation of the properties necessary to achieve the geometries, tolerances and strength specifications, as well as ease of production'*



Fig. 4 A large MIM aerospace component being removed from a mould at PTI

to PIM. A new 38-ton Arburg machine is the latest addition to the facility. The production area features lean-manufacturing based work cells, allowing for maximum efficiency on the production lines.

For the sintering of PIM parts PTI uses both continuous and batch furnaces. Continuous sintering is undertaken using a 65 ft. 300 Series pusher furnace from CM Furnaces Inc., a furnace manufacturer with a long history of expertise in MIM conveniently located only a few miles away in Bloomfield. The CM furnace can operate at up to 1650°C and has six debind/pre-heat zones and three individually controlled sintering zones. The company's batch vacuum furnace, manufactured by AVS Inc., has a 10ft<sup>3</sup>

expertise, technology, capital equipment and intellectual property to continually grow as a company and expand as needed to become the known innovator and leader in the injection moulding industry," Neal Goldenberg told *PIM International*.

### Binders and feedstock

Despite the company's experience in manufacturing feedstock in-house, PTI today uses feedstock systems from external suppliers as well as its own propriety binder technology, a trend that has been observed at a number of PIM part producers in recent years. Feedstocks processed include thermal, catalytic, and solvent or aqueous systems.



Fig. 5 Green MIM parts entering the continuous debinding and sintering furnace at PTI

"The choice of feedstock system is considered at the development stage of a product. It is essential when working with MIM applications that both client and supplier work as a team with the design of the component for MIM-ability, functionality, and processability. The choice of feedstock system is integral to these requirements. The criteria for feedstock selection at PTI include an evaluation of the properties necessary to achieve the geometries, tolerances and strength specifications, as well as ease of production so that we can meet our own efficiency standards as well as guarantee consistency from lot to lot. All of this is a part of understanding the requirements of the customer, in terms of product specifications, application and deadlines. Building a toolbox with the right tools for the job - it's all in the tools!" stated Don Olson, Director of Engineering at PTI.

"For aerospace components we will frequently use our own in-house feedstock. For many applications, however, we are happy to rely on external suppliers who we trust to provide consistent high quality feedstock, allowing our company to place the experts where they are needed most, in development of the component."

PTI manufactures its in-house feedstock via batch (sigma blade) or twin-screw compounders, giving it the flexibility to produce large or small volumes as required.

### Recognising the importance of tooling

In February 2012 PTI announced its acquisition of Polmold, a New Jersey based manufacturer and repair shop for injection moulds, tools, dies and fixtures. Polmold manufactures tooling for both plastic and MIM programmes and has for many years been a key supplier to PTI. The purchase, states PTI, strengthened its position as a 'one-stop shop' custom manufacturing solutions provider.

"The acquisition of Polmold complemented our existing product offering and continues our strategic plan to offer streamlined custom manufacturing services to our customers in the USA and globally," commented Neal Goldenberg. "Our vertical integration of this critical technology into our operation truly makes us a complete solutions provider for any engineered injection moulding application."



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Chrome Cobalt Alloys: F75  
Steels: 4605, 4140, M2, FN0205, FN08  
Heavy Metals: Tungsten, W-Cu  
Ceramic: Alumina, Zirconia



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Fig. 6 MIM parts being loaded into PTI's batch vacuum furnace manufactured by AVS Inc. The furnace has a 10ft<sup>3</sup> hot zone

Polmold, which has retained its own name and identity, was founded by Henry Marzec in 1980 and Marzec continues to play an important role as General Manager. The company's extensive facilities include five CAD/CAM work stations, four recent computer numerical control (CNC) machining centres, seven CNC milling machines, two lathe machines, two wire and three "sinker" electric discharge machines (EDM), six grinders and QC inspection equipment to guarantee the quality of the tools supplied.

There is of course only so much that can be done with tooling before costs become prohibitive, so PTI uses its experience to apply a number of innovative solutions to efficiently manufacture complex parts such as sinter-bonding two separately moulded

parts or machining green parts on an in-house multi-axis CNC machine.

Polmold continues to manufacture moulds for external companies and Neal Goldenberg stated that tooling sales have thrived. "We have had approximately 20-24 new tools for R&D and production, purchased over the past 24 months."

Given the critical importance of tooling in the MIM process, in terms of component development time and the impact on production in the case of tooling wear or failure, the advantages of having an experienced in-house tool manufacturing facility were clear to see for PTI's President, Mel Goldenberg. Commenting at the time of the acquisition, he stated, "We have been pleased to see a strong resurgence of tooling work being done here in the United States. Much of

this is due to the realisation that the cost-savings from work done overseas in places such as China didn't pan out the way people had hoped it would when variables such as lost time and additional costs of rework were not a part of the original equation. Further, given the size of some of the tooling, transportation and import fees, outsourcing has become very cost prohibitive."

### Product development and quality control

PTI has for many years made use of simulation software to identify problems at an early stage in a component's development. Don Olson stated, "Moldflow has become essential to achieve success with product and tool design, as well as with the production of tools and components to required geometries and tolerances. It assists in identifying the most effective area for the gates and parting lines, as well as providing the necessary information to prevent the trapping of air that results in voids. Such systems also help to avoid high stress areas that can develop during moulding, which result in quality issues later in the process or for the customer. They are an integral component to our tool box."

"Part development after tooling has been produced can range anywhere from 4 -12 weeks, depending on the difficulty, complexity of geometries and tolerances required. Development time is typically unaffected by the type of material specified. Part size and weight has the potential to have a larger effect on development time as larger



Fig. 7 Top: A 316L Segmented Ring, approx. 4" OD (20 g). Bottom: A 17-4PH Missile Wing, approx. 6" in length (120 g as sintered and 110 g after machining)

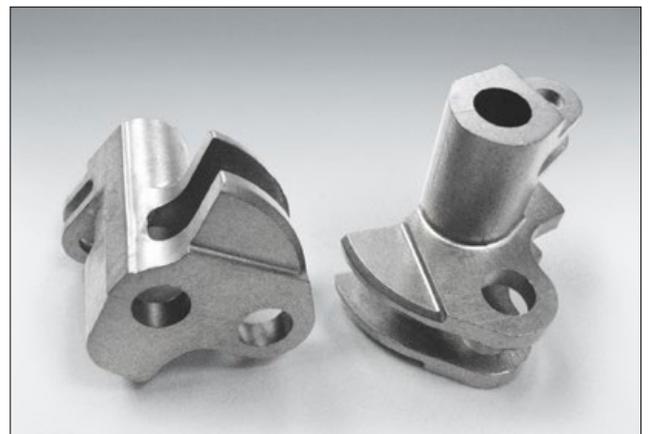


Fig. 8 A 17-4PH Connecting Latch for an aerospace application weighing 90 g



Fig. 9 Top: An Inconel Coupler (70 g) with a 2" OD. This replaced three parts which were machined and welded. Below: A tungsten "Fuse weight"



Fig. 10 Stainless steel aerospace components, clockwise from the top: Connector (5 g), Shuttle (3 g) and Projectile Fin (4 g)



Fig. 11 A MIM surgical implant component manufactured from titanium

components take longer to debind, sinter and heat treat," stated Dorrie Myhre, PTI's Sales and Marketing Specialist.

Efficient volumes for non-aerospace MIM components at PTI begin at 20,000 parts per year. "MIM is an economy of scale production process and the higher the volume the more cost effective part pricing. Part sizes average at 0.25 g, with wall thicknesses as low as 0.010" and lengths up to 6". The smaller the part, the better the process works," added Dorrie Myhre.

PTI maintains strict quality control measures throughout the entire lifespan of a project, utilising a complete systems approach with evaluations at the raw material, work-in-progress and finished goods levels. The testing and measuring technologies in PTI's quality control department include first piece inspection, Statistical Process Control (SPC), Coordinate Measuring Machines (CMM), optical comparators and digital optical metallography, as well as a range of mechanical test equipment.

### Opportunities for MIM in the aerospace sector

PTI has enjoyed a relatively long relationship with the aerospace industry and is one of only a limited number of MIM suppliers to serve this sector. The strength of this relationship links back to the fact that so many of PTI's employees came from Allied-Signal/

Honeywell, where they were well versed in aerospace guidelines and procedures.

MIM's advantages, such as lower costs compared to alternative processes such as casting and machining, material properties which can exceed those of traditional processes, and a design freedom that offers the potential to combine multiple components in to one, are amplified when it comes to many hard to machine aerospace materials. In addition, MIM's credentials as a green and sustainable process are highly desirable within the aerospace industry, which itself is under extreme pressure to reduce its carbon footprint.

When considering the challenges of supplying MIM products to the aerospace industry, Don Olson, told

ISO. Aerospace companies are also starting to place more responsibility on Tier 1 and Tier 2 suppliers for design and development, production and assembly. As such, MIM houses need to be able to adjust to these new demands and capability requirements."

PTI has for some years been certified to the SAE AS9100B and ISO 9001:2008 aerospace quality standards and in 2012 the company was certified to the most current version, AS9100C, making it compliant to the most current requirements. This certification is regarded by PTI as a reflection of the company's on-going efforts to further advance the expansion and acceptability of its MIM technology for use in aircraft parts and engine components. Steve Sesny, Vice-President of Operations at PTI stated,

***'Aerospace companies are also starting to place more responsibility on Tier 1 and Tier 2 suppliers for design and development, production and assembly'***

*PIM International*, "The complexity of the aerospace companies' supply chain is in itself a challenge, but above all it is the extremely complex and stringent quality requirements, specifically SAE AS9100, which has expectations well above and beyond those for

"MIM offers reduced part weights, increased throughput and significant cost reductions when compared to competing technologies such as investment casting and machining. With AS9100 our aerospace clients can be assured that the MIM parts and

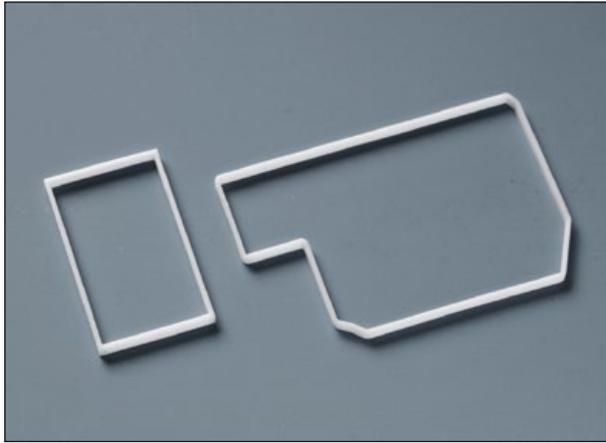


Fig. 12 CIM zirconia "Insulating Rings" weighing less than 1 g. Left: 1" length, right: 1.5" length



Fig. 13 CIM zirconia surgical instrument components, OD ranging from 0.080" to 0.150", length from 0.250" to 0.500"



Fig. 14 CIM alumina components, approx. 3" in length and 1" across, weighing 4 and 8 g



Fig. 15 CIM alumina aerospace parts with outstanding strength to weight ratios and high temperature properties

engineering services provided meet all the technical requirements and process specifications to the most rigid of standards."

Despite the opportunities that exist as a result of current growth in aerospace production, there are some significant barriers to entry.

competitive market, where there is a flow down demand to the suppliers for cost savings. With lower volume requirements and highly engineered components, and highly stringent quality guidelines, MIM houses must meet the challenges of lean/sustainable manufacturing to survive."

risk they perceive with MIM."

For those who are able to overcome these challenges, there is undoubted potential in the aerospace sector. Specific current and target applications include gas turbine and jet engine components, parts for combusive environments, fuel air mixer components, anti-aircraft countermeasure devices, and electronic components. A range of MIM materials are used in the aerospace sector, from Inconel 718 and Hastelloy X superalloys to 316L austenitic stainless steel, 410 and 420 martensitic stainless steels and 17-4 PH and 13-8 PH precipitation hardened stainless steels.

Dorrie Myhre added, "There is great potential for companies who are already established within this sector as well as new suppliers who can meet the critical deadlines and diverse capability requirements. The increase in the popularity of air taxis is a factor resulting in increased supplier demand, with some forecasters

***'There is great potential for companies who are already established within this sector as well as new suppliers who can meet the critical deadlines and diverse capability requirements'***

Dorrie Myhre stated, "With an increase in the production of aerospace components, MIM lead times for tooling and component development can be a hindrance when attempting to enter this market. There are also the economic barriers that exist in a highly

"Whilst there appears to be a gradually increasing acceptance of MIM by the aerospace industry, new technologies have high transfer costs and with tight constraints on unnecessary spending, most companies are concerned about the



Fig. 16 An aerospace engine component manufactured from zirconia for its high temperature characteristics and chemical resistance to jet fuel, diameter 1.5"

predicting that production rates will increase by 45% by 2015. Taking this into account, with the cost saving strategies that are being executed by companies in the industry, MIM will be an effective and efficient option for many of the components. Typical annual production volumes are, however, lower with aerospace applications. Annual production can be as low as 500 parts at the lowest end of the scale, rising to around 10,000 parts per year at the higher end."

The technology and skillsets that PTI has developed to serve the aerospace sector are also finding application in other markets, including products for corrosive environments, turbine and compressor systems, satellites, rocket components, military and defence applications and firearms.

## Ceramic Injection Moulding at PTI

PTI recently added Ceramic Injection Moulding (CIM) to its portfolio of services, and currently produces CIM components for the medical market. It is expected that the introduction of CIM will open up new opportunities in terms of markets and applications.

Neal Goldenberg stated, "We see opportunities for CIM in a number of our key markets. In the medical sector, the biocompatibility of ceramics is a tremendous advantage, as are the thermal/electrical insulating properties of CIM materials, offering the ability to isolate heat sources at the end of medical devices thereby reducing the risk of injury to patients. In the aerospace industry ceramics provide significant compressive strength, high

wear and low coefficient of friction for parts that rotate against one another, such as hub bushings, and their extreme heat resistance makes them suitable for engine applications. There are also opportunities for the use of ceramics in aggressive environments such as jet fuel systems."

"Bringing in this technology was a critical component in our strategic vision and plan for the company to be

Additionally, having to identify and use another type of feedstock presented further risk as there was no true way to know how the modified material would react to current processes."

"MIM houses were required to re-think their purchasing strategies, as well as how to finance larger purchases to ensure that they were able to maintain necessary inventories to continue production. Larger, stable

*'We see opportunities for CIM in a number of our key markets. In the medical sector, the biocompatibility of ceramics is a tremendous advantage'*

the single source solutions provider for injection moulding applications. We believe diversity in the industry is one of the options to obtain and maintain a competitive advantage and we have evolved to that platform."

"We have the capabilities to provide a diverse range of solutions for our current customers, especially for those companies that are placing a great deal of effort to reduce their supplier base. This diversity will also extend to new customers who are also seeking out key suppliers, willing to be flexible and provide options as well as multiple services and products that will play an important role in the achievement of their strategic goals for a cost efficient and lean manufacturing supply chain."

## Outlook

When considering the challenges that lie ahead for the PIM industry, one of the most frequently raised concerns among part producers is the availability of MIM grade powders.

Commenting on PTI's view of the situation, Dorrie Myhre stated, "We do see challenges in this area. An example was in 2009 when the United Steel Workers Union members voted to go on strike at the Sudbury Mine, in Ontario Canada. The mine was owned by Vale Inco, a Brazilian company and one of the three largest producers of nickel in the world. The strike put the US and many of its manufactures in an extremely vulnerable position, threatening production shut downs, major delays in deliveries and increased backorders, especially for the MIM houses that used pre-alloyed powders.

and financially secure companies were able to react, providing them with a competitive advantage over smaller firms who could not afford to stock-pile material."

PTI also commented that the rising price of raw materials was a cause for concern, with the uncertainty of powder prices potentially undermining the MIM industry's competitive advantage.

In relation to the potential for PIM in the aerospace industry, PTI sees a number of issues that need to be addressed by both end-users and the PIM industry.

Neal Goldenberg concluded, "The aerospace industry needs to recognise that while MIM is equal in properties to cast and machined components, it has small variances with process parameters, SPC and quality requirements, which need to be designed to work effectively with the technology. Education and training is required to bring a better understanding of PIM technology, its capabilities, its value and benefits. Our industry, on the other hand, needs to offer ever greater flexibility and diversity, strong quality controls such as certification to AS9100, and an ability to meet and exceed the aerospace industry supply chain challenges."

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# The processing and properties of Metal Injection Moulded Superalloys

The production of MIM superalloys is one of the few remaining secretive areas of our technology, with information on applications in the aerospace sector, and the necessary properties achieved, kept confidential in order to protect decades of privately funded research and a competitive advantage. There remains, however, a significant volume of data available on what can be achieved via MIM processing. In this extensive review, Burghardt Klöden, Thomas Weissgärber, Bernd Kieback and Ingolf Langer present a detailed analysis of published work to-date and consider the potential for this area of MIM technology.

Superalloys have been known for nearly a century, although the systematic development of superalloys and their production technologies started about 70 years ago. Their excellent combination of high-temperature properties, namely corrosion resistance and mechanical strength, has led to a wide range of applications. Today the main application areas are in aviation/aerospace and power plants. Extensive reviews on superalloys can be found in [1] and [2].

In terms of manufacturing technologies, the first superalloys were forged. Later this changed to casting, which resulted in significant material improvements. For even higher thermo-mechanical loads, several generations of single-crystalline Ni-base superalloys were developed. Fig. 1 summarises the development of mechanical strength over the decades. Clearly the single crystalline superalloys have the highest strength levels and therefore the highest application temperatures. However, their production is expensive and application is therefore restricted to the most severe conditions.

With respect to Powder Metallurgy (PM) in general [1], Ni-base

superalloys play a key role in parts for aero engines. As a result of operational conditions, namely temperature and operating speed, becoming ever more demanding, alloy compositions became increasingly complex in order to achieve both mechanical strength (static and dynamic) and corrosion resistance. At the same time, a high degree of homogeneity with respect to element distribution and grain size needed to be maintained.

This led to the development of PM processing, particularly for parts that cannot be made by ingot metallurgy, for example because of the high amount of certain alloying elements, or where a PM part has significantly better properties, for example in terms of alloy homogeneity, density and grain size. Furthermore, cost savings in terms of the number of processing steps and material input weight compared to ingot metallurgy

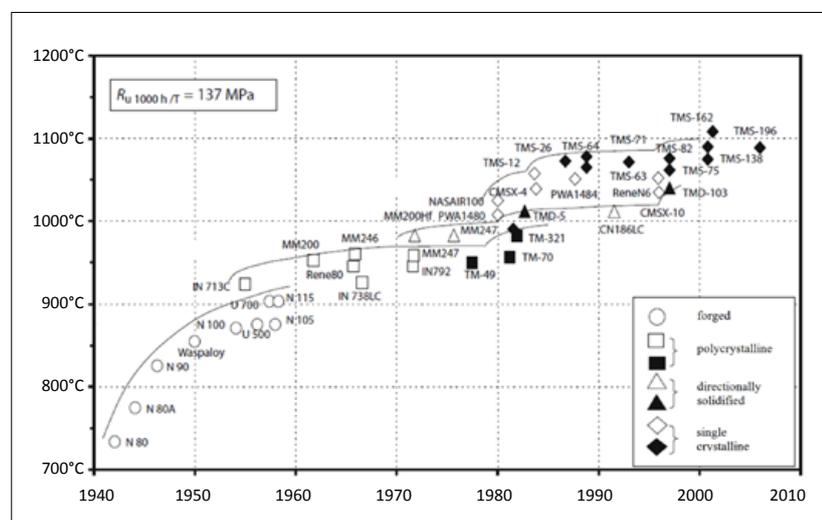


Fig. 1 The development of high-temperature strength for Ni-base superalloys [3]

Alloy	Ni	Fe	Cr	Al	Mo	Co	Nb	W	Si	Mn	Ti	Ta
<b>1st generation</b>												
IN 718	Bal.	18.5	19	0.5	3		5.1		0.2	0.2	0.9	
IN 625	Bal.	5*	21	0.4*	9	1*	3.7		0.5*	0.5*	0.4*	
IN HX	Bal.	18.5	22		9	1.5		0.6	0.5	0.5		
U 700	Bal.		14 - 16	3.85 - 4.15	4.5 - 5.5	16 - 18					3.35 - 3.65	
<b>2nd generation</b>												
Nimonic 90	Bal.	1.5	19.5	1.5		16.5			0.3	0.3	2.5	
IN 713C	Bal.		13.5	6.0	4.5		2.3				0.9	
IN 713LC	Bal.		12.0	6.0	4.5		2				0.7	
U 720	Bal.		18.0		3.0	14.8	2.5	1.25			5.0	
U 720Li	Bal.		16.0			15.0	2.5	1.25			5.0	
IN 100	Bal.		12.5	5	3.2	18.5					5	
GMR-235	Bal.	11	15	3.8	4.8				0.4	0.3	2	
MAR-M 247	Bal.		8.6	5.5	0.7	10		10			1.2	3.1
N18	Bal.	11.5		4.35	6.5	15.7					4.35	

Table 1 MIM superalloy compositions (main elements, all values in wt%; \* maximum value)

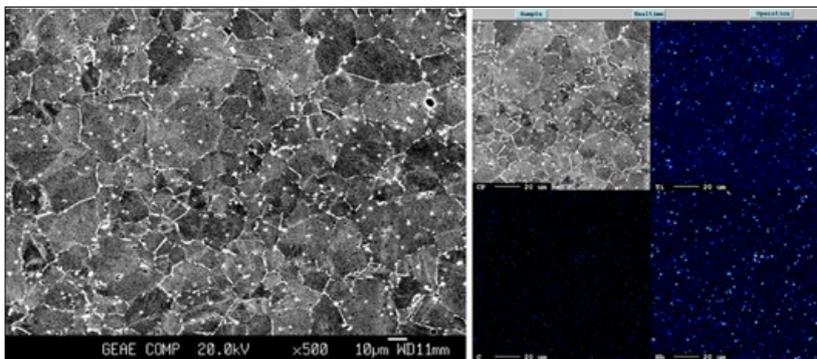


Fig. 2 Microstructure of MIM IN 718 after sintering and heat treatment [23]

can be considered, especially for (near-) net-shape processing routes. The main processing routes are [1, 4]:

- HIP
- Hot extrusion
- A combination of HIP and extrusion
- + Isothermal or superplastic forging.

HIP and/or extrusion of canned powders, or preconsolidated billets, lead to full density parts, while impurities are minimised. In HIP particularly, the grain size and precipitate content can be controlled by temperature and time. In the case of extrusion or non-direct HIP the forging step turns the billet into the component.

There are a variety of PM superalloys available. The most recent results of alloy development together with application examples are summarised in [4] and [5].

## Superalloys by MIM

According to a review by German *et al.* [47] the feasibility of processing superalloys by MIM was demonstrated around 30 years ago and today 2% of worldwide sales in MIM are superalloy parts. Superalloys are therefore still rather niche within MIM. Table 1 gives a summary of Ni-base superalloys, which have been processed by MIM. The "1<sup>st</sup> generation", which was a focus for research and development for nearly two decades, consisted mainly of two alloy systems (IN718 + IN625), while the "2<sup>nd</sup> generation", contains an increasing number of  $\gamma$ - $\gamma'$  alloys. As for PM superalloys in general, the development of MIM superalloys has also been driven primarily by the aerospace industry during the 1<sup>st</sup> generation, while the 2<sup>nd</sup> generation is being additionally driven by the automotive sector for, as an example, turbine wheels for turbochargers. Both MIM superalloy generations will be reviewed in detail.

## The first generation of MIM superalloys

The alloy systems covered in this report are summarised in the following tables:

- Table 7: Powder and processing
- Table 8: Sintering and density
- Table 9: Impurities

Additional heat treatments and tensile properties will be reviewed in the respective sections.

### Udimet 700

Two of the earliest papers dealt with the production and properties of Udimet 700 [6, 7]. With the chosen sintering parameters, >94% TD density as achieved (an additional HIP treatment increased the density to nearly 100%). In terms of impurities, carbon and oxygen pickup was observed during processing. The tensile properties are comparable with a reference cast sample. The creep strength is comparable to cast samples and better than HIPed and vacuum plasma sprayed samples.

### Inconel 718

This alloy is hardened by two types of precipitates:  $\gamma'$  ( $\text{Ni}_3\text{Nb}$ ) and to some extent  $\gamma''$  ( $[(\text{Ni}, \text{Ti})_3\text{Al}]$ ). In order to tailor the size and morphology of precipitates the alloy is usually supplied in the heat-treated condition, which involves solution annealing and aging. Properties are summarised in [8] and [9].

A typical microstructure of IN 718 after MIM, sintering and heat treatment (HIP + solution annealing +

aging) is shown in Fig. 2. The sample has been consolidated to full density. Furthermore, according to the element mappings the main precipitates are Ni<sub>3</sub>Nb and fine carbides.

In [10 and 11] different sintering parameters were tested. Furthermore, thermal analysis was performed to analyse phase changes with Laves phase formation being observed at 1180°C. In terms of atmosphere, only sintering under vacuum lead to acceptable densities, while hydrogen was excluded. In terms of temperature, holding times and heat ramp, tensile test properties comparable to AMS 5662 were achieved for certain parameters (for example T = 1275°C, t = 8h, ΔT = 1°C/min).

In [12-14] fully heat-treated samples were analysed with respect to mechanical properties and compared to existing standards. The results of tensile and creep tests were within the specification of ASM 5596. For stress rupture testing, two sample batches were prepared (with and without additional machining, surface ground and polished gage section). Values of both batches were below the requirements (rupture life 23 h and elongation 4% at T = 648°C, σ = 690 MPa); however, the machining improved stress rupture life and elongation. With an alternative heat treatment the minimum required elongation was also reached. HCF and LCF testing were performed as well, although only with a limited number of samples. Sample preparation proved to be critical in terms of mechanical performance for these tests as well.

In [15] the relationship between sintering parameters and mechanical properties was investigated. The results are summarised in Table 3. Pre-sintering and holding, respectively, at temperatures below the sintering temperature lead to different mechanical performance. However, all values fell short of those of cast and wrought samples. Tensile tests in the heat-treated condition lead to a significantly higher strength and an elongation, which fulfil minimum requirements according to AMS 5596.

In [18] a master alloy route was used to produce test samples. The heat treatment improved material properties to a significant extent (Table 2). Values compare well with cast / wrought material and only the ductility is significantly lower. The authors note that this might be due to minor surface flaws and the sintered surface texture.

State	T [°C]	YS [MPa]	UTS [MPa]	Elong. [%]	Ref.
Cast + heat treated	20	915	1090	11	[18]
Wrought + heat-treated	20	1185	1435	21	[18]
MIM sintered + heat-treated (HIP + SA + AG)	20	> 1034	> 1241	> 6	AMS 5917
	649	> 827	> 931	> 6	AMS 5917
Sintered + HIP	20	824	1009	39.3	[21]
Sintered + heat-treated (HIP + SA + AG)	20	794 - 995	812 - 1218	5 - 17.4	[10] values depend on sintering parameters
	20	1123	1330	14.8	[14]
	482	990	1108	16.6	[14]
	538	988	1105	14	[14]
	593	1013	1088	12.5	[14]
	648	912	1039	10.2	[14]
	20	908	1340	23	[17]
	20	1078	1250	21.7	[21]
	650	1050	1177	16.6	[21]
	20	1054	1379	29	[24]
Sintered + heat-treated (SA + AG)	20	1061-1088	1206-1297	9.4 - 13.4	[15]
	20	840	1259	21	[17]
	20	1046	1211	6	[18]
	540	895	1027	4	[18]
		900	1065	4	[19]
	20	1124	1304	8.7	[21]
	20	≈ 960 - 1100 ≈ 1030 - 1120 ≈ 1160		≈ 16 - 26 ≈ 2.5 - 5 ≈ 15	[23] Values estimated from figures
As-sintered	20	503	936	20	[18]
	20		800 (WA) 1000 (GA)	15 (WA) 8 (GA)	[20]
	20	876	1054	25.4	[21]

Table 2 Mechanical properties of alloy IN 718 (HIP = hot isostatic pressing, SA = solution annealing, AG = aging; for heat treatment parameters see Table 4; GA = gas atomised powder, WA = water atomised powder)

In [20] different water atomised (WA) and gas atomised (GA) powders were compared. While the GA powder samples showed the highest strength and hardness, the WA powder samples yielded the highest elongation.

In [21] samples in different states were analysed and the influence of sintering temperature on alloy properties was evaluated. In the sintered

state, differences in properties were concluded to be due to a transition of mainly solid-state to liquid-phase sintering with increasing temperature. The HIP treatment increases elongation, while reducing strength and hardness due to grain growth. The additional heat treatment (AMS 5663) increases strength and hardness in the sintered and the sintered + HIP state,

Alloy 718 sample	Sintering conditions	0.2% Proof stress (Mpa)	Ultimate tensile strength (Mpa)	Elong. (%)
C1026-1	1285°C 40 minutes		913	20.5
C1026-2	1285°C 40 minutes		915	20.0
C1064-A	1285°C 40 minutes	449	932	17
C1064-B	Pre-sintered at 1090°C in H <sub>2</sub> 1285°C 40 minutes	458	891	15
C1068-A	1287°C 60 minutes	574	931	19
C1068-B	Pre-sintered at 1090°C in H <sub>2</sub> 1287°C 60 minutes	526	844	13
C1069	1287°C 60 minutes Held at 620°C for 240 minutes	661	980	11
C1071	285°C 60 minutes	497	933	19
C1073	285°C 60 minutes	509	939	21
Alloy 718	Cast	414	862	51
Alloy 718	Cast and Annealed	462	935	41
Alloy 718 wrought bar <sup>a</sup>	Heat treated Condition <sup>b</sup>	1185	1435	21.0

<sup>a</sup> ASM International Handbook Vol. 1

<sup>b</sup> ASM 5663 specification [solution heat treatment 1 hour in Ar at 950°C then air cooled, followed by precipitation heat treatment at 718°C for 8 hours, furnace cooled at 38°C/min. to 620°C held for 8 hours and finally air cooled]

Table 3 Sintering conditions and mechanical properties of IN 718 [15]

while elongation is reduced. Values are compared with wrought samples and are comparable or superior.

Contreras *et al.* [22] analysed IN 718 powders with different particle size distributions (PSD) and compositions. Blends with different kinds of particle size distributions were produced from four initial powders. It was observed that the blend with a wide PSD shows the highest critical solids loading. Furthermore, different thermal debinding regimes were tested and analysed with respect to residual impurity content (C, O). After this analysis, argon was chosen as the debinding atmosphere. Different sintering regimes were also evaluated with respect to density and shrinkage. It is stated that the particle size distribution doesn't influence the density (all parameters lead to densities  $\geq 94\%$ ), but does influence shrinkage; the blend with a wide PSD showing the lowest shrinkage values.

In [23] samples of powders from different vendors were compared in terms of mechanical properties. The oxygen content of the initial powder significantly influenced tensile elongation, while strength was not influenced. Further tests at elevated temperatures

were also undertaken, with the minimum values of all parameters according to AMS 5383 being met. LCF testing shows comparable behaviour to wrought samples. Furthermore it is noted that strength and ductility depend on surface finish and geometry.

The following conclusions are therefore drawn:

- In terms of powder and process parameters (Table 7) the powders are commonly gas atomised with a particle size  $< 22 \mu\text{m}$ . The feedstock contains typically 60 – 70 vol% of solids. The majority of binders (which have been disclosed) are wax-polymer-based (for example polyethylene + paraffin wax). Debinding is usually a two-step process involving a solvent and thermal stage.
- In terms of heat treatment (Table 8) the only possible atmosphere is vacuum. The minimum sintering temperature, which is needed to achieve sufficient densities, is in the range of 1260°C with a minimum time of 2 h.
- With respect to the tensile mechanical properties (Table 2) as-sintered samples yield the lowest values in terms of strength.

HIP	Solution anneal	Aging	Ref.
1190°C, 4 h	980°C, 1 h, Ar	720°C, 8 h + 620°C, 8 h	[14]
1190°C, 4 h	950°C, 1 h, Ar	718°C, 8 h + 620°C, 8 h	[10] [13]
	950°C, 1 h, Ar	718°C, 8 h + 620°C, 8 h	[15]
1160°C, 3 h	980°C, 1 h, Ar	720°C, 8 h + 620°C, 8 h	[17]
	980°C, 1 h, HV	720°C, 8 h + 620°C, 8 h	[18]
	980°C, 1 h, H <sub>2</sub>	720°C, 8 h + 620°C, 8 h	[19]
1190°C, 4 h	950°C, 1 h, Ar	720°C, 8 h + 620°C, 8 h	[21]

Table 4 Heat treatment parameters of IN 718

This is why additional heat treatment is imperative. Comparing the parameters of different sources (Table 4), these are very similar and mostly based on existing specifications for IN 718 (for example AMS 5662, 5663). Applying solution annealing + aging heat treatment generally increases tensile strength to values, which are comparable to cast/wrought material. Inserting a HIP step prior to the rest of heat treatment keeps tensile strength at the same level, while generally the elongation increases. Furthermore, some results on high-temperature mechanical properties were obtained. However, a general trend can't be concluded from this data.

- Comparing the available data to the recently published industry standard AMS 5917, most of the data fulfils the norm for tensile strength and elongation at room temperature as well as at elevated temperature.

#### Inconel 625

IN 625 is a solid-solution-strengthened alloy, which may derive additional strength due to the precipitation of Mo/Nb-carbides. Properties are summarised in [25]. The microstructure after sintering and an additional solution annealing treatment is shown in Fig. 3. The element mappings show a homogeneous distribution of elements, while in the sintered state precipitates were observed.

In [10] different sintering and furnace parameters were tested. Densification was not influenced, as all temperatures were above the solidus line. The furnace parameters influenced the thermal gradient and hence the microstructure, which lead to a spread in mechanical properties. Generally, the more uniform and fine-grained samples had the highest strength values.

In [19] sintered and annealed samples (1150°C, 2 h) were analysed. With respect to mechanical properties, strength values are comparable with cast alloys, but the strength is considerably lower than that of a wrought sample. The elongation is considerably lower than cast/wrought.

In [26] sintered and solution-annealed (1177°C, 15 min, vacuum) samples were analysed. It is noted that carbides and the Laves phase are already detected in debound samples. However, after sintering these phases have dissolved and re-homogenisation of elements has taken place.

In an overview article [27] samples were analysed in the as-sintered and heat-treated condition (solution anneal: 1150°C, 2 h + aging 750°C at different times). Hardness values between 208 HV<sub>0.1</sub> (sintered), 250 HV<sub>0.1</sub> (solution annealed) and a maximum value of 313 HV<sub>0.1</sub> (solution anneal + aging) were measured. Furthermore, tensile tests in all conditions were performed. The results show the same tendency, notably that the aging step increases the tensile strength. Values are comparable with cast alloys, but the strength is considerably lower than that of a wrought sample. It is noted that the heat treatment most likely leads to the formation of  $\gamma''$  and carbide precipitates as well as TCP phases. Microstructural changes are analysed by SEM and EDS, indicating some NbC formation in the sintered state and dissolution of carbides after solution annealing. Precipitates in the aged state could not be observed due to their very small size. The additional TEM analysis remained inconclusive, as only NbC was detected again.

The following conclusions are drawn:

- In terms of powder and process parameters (Table 7) the powders are commonly gas atomised with a particle size < 27  $\mu\text{m}$ . The feedstock contains typically 60 – 65 vol% of solids. The majority

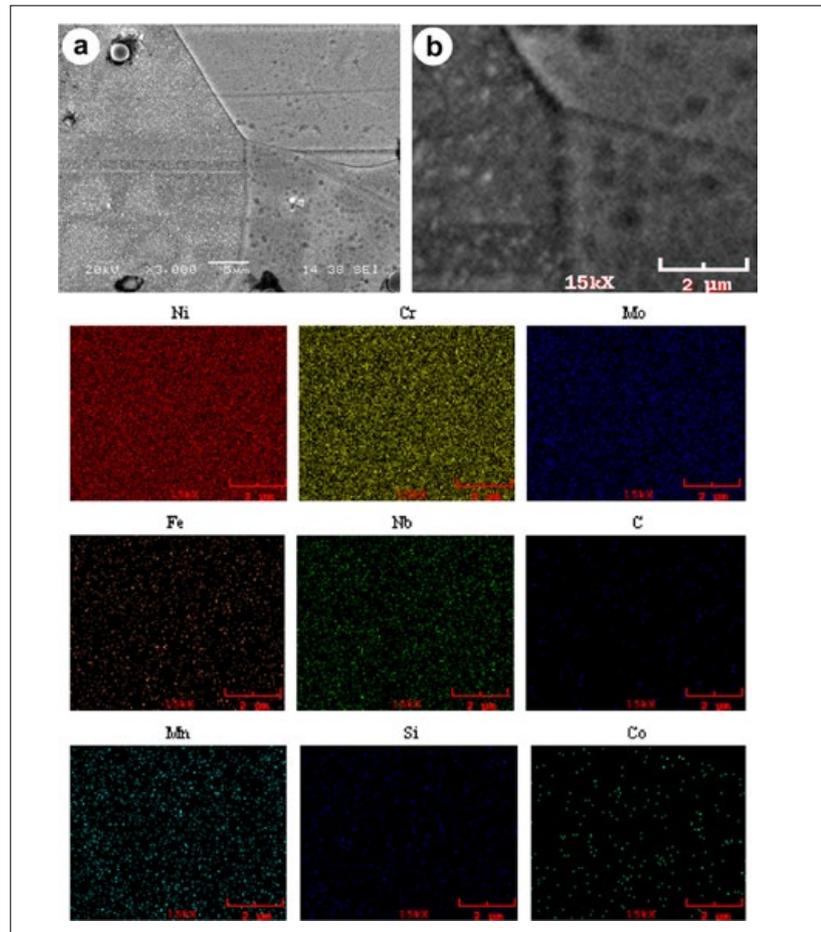


Fig. 3 The microstructure of MIM IN625 [27]

Run No	Temp (°C)	Hold time (min)	Density (%)	YS 0.2% (Mpa)	UTS (Mpa)	Elong (%)	Travel interm (min)	Travel incr. (cm)	Grain size <sup>a</sup> ( $\mu\text{m}$ )
1	1298	60	99.7	347.3	665.5	36.9	00	0.0	>175
2	1288	60	99.9	342.3	687.3	95.3	15	5.1	85
3	1288	60	100	344.3	805.8	48	10	10.2	85(42)
4	1289	30	99.3	400.6	751.9	80.7	5	10.2	46
5	1288	24	99.8	376.2	848	46.7	4	10.2	20(19)

<sup>a</sup> Grain size measured in transverse direction

Table 5 Sintering parameters, tensile properties and grain size of MIM processed IN 625 [10]

State	T (°C)	YS (MPa)	UTS (MPa)	Elong (%)	Ref.
Cast	20	350	710	48	[28]
Wrought	20	490	965	50	[29]
As-sintered	20	230	600	21	[19]
As-sintered	20	351	650	44.7	[27]
Sintered + heat-treated [SA]	20	305	605	46.2	[27]
Sintered + heat-treated [SA + AG]	20	385	674	40.6	[27]

Table 6 Mechanical properties of IN625

Powder	Solids part in feedstock (Vol%)			Binder	Debinding	Ref.
<b>IN 718</b>						
$d_{50} = 8.5 \mu\text{m}$	66			Wax-polymer		[10]
	$d_{10}$	$d_{50}$	$d_{90}$	Polyethylene glycol/acetal/ polyvinylbuteral	Solvent (60°C, 4 h) + Thermal (500°C, 1 h)	[11]
	3.6 $\mu\text{m}$	8.4 $\mu\text{m}$	13.4 $\mu\text{m}$			
Gas atomised	69			Paraffin wax/polypropylene/ zinc stearate		
	$d_{10}$	$d_{50}$	$d_{90}$	Wax-polymer	Solvent + thermal (900°C, H <sub>2</sub> )	[16]
	3.1 $\mu\text{m}$	7.4 $\mu\text{m}$	14.5 $\mu\text{m}$			
Gas atomised	65					
<22 $\mu\text{m}$ Gas atomised	62-65			Proprietary	Catalytic (HNO <sub>3</sub> )	[17]
Gas atomised, < 22 $\mu\text{m}$						[18]
	$d_{10}$	$d_{50}$	$d_{90}$	Wax-polymer	Solvent + thermal (900°C, H <sub>2</sub> )	[19]
1	3.0 $\mu\text{m}$	7.4 $\mu\text{m}$	15.8 $\mu\text{m}$			
2	3.1 $\mu\text{m}$	7.4 $\mu\text{m}$	14.5 $\mu\text{m}$			
Gas atomised	65					
Water atomised (<2 $\mu\text{m}$ , <10 $\mu\text{m}$ ) Gas atomised (<22 $\mu\text{m}$ )	62, 65			Wax-based	Solvent (heptane) + thermal (N <sub>2</sub> )	[20]
$d_{50} = 13.1 \mu\text{m}$	62			Paraffin wax, high density polyeth- ylene, ethylene-vinyl acetate	Solvent (CH <sub>2</sub> Cl <sub>2</sub> , 37°C) + thermal	[21]
	$d_{10}$	$d_{50}$	$d_{90}$	40% polyethylene 55% paraffin wax 5% stearic acid	Solvent (hexane, 60°C, 240 min) + thermal (different parameters)	[22]
1	3.6 $\mu\text{m}$	11.1 $\mu\text{m}$	22.8 $\mu\text{m}$			
2	18 $\mu\text{m}$	25.3 $\mu\text{m}$	34.9 $\mu\text{m}$			
3	57.3 $\mu\text{m}$	75.6 $\mu\text{m}$	102.9 $\mu\text{m}$			
4	68.5 $\mu\text{m}$	93.4 $\mu\text{m}$	156.7 $\mu\text{m}$			
Gas atomised	60 – 78					
<b>IN 625</b>						
$d_{50} = 8.5 \mu\text{m}$	66			Wax-polymer		[10]
	$d_{10}$	$d_{50}$	$d_{90}$	Paraffin wax (60%), polypropylene (35%), stearic acid (5%)	Solvent (60°C, 4 h) + Thermal (500°C, 1 h)	[11]
	3.0 $\mu\text{m}$	8.6 $\mu\text{m}$	12.9 $\mu\text{m}$			
Gas atomised	66					
	$d_{10}$	$d_{50}$	$d_{90}$	Wax-polymer	Solvent + thermal (900°C, H <sub>2</sub> )	[16]
	5.1 $\mu\text{m}$	9.7 $\mu\text{m}$	16.9 $\mu\text{m}$			
Gas atomised	65					
	$d_{10}$	$d_{50}$	$d_{90}$	Wax-polymer	Solvent + thermal (900°C, H <sub>2</sub> )	[19]
1	5.1 $\mu\text{m}$	9.7 $\mu\text{m}$	16.9 $\mu\text{m}$			
Gas atomised	65					
		$d_{50}$	$d_{90}$		Thermal (T <sub>max</sub> = 900°C, t <sub>max</sub> = 15h; with ramps and holding times)	[26]
1		12.5 $\mu\text{m}$	27.8 $\mu\text{m}$			
Gas atomised						
	$d_{10}$	$d_{50}$	$d_{90}$	Paraffin wax, polypropylene, carnauba wax, stearic acid	Solvent (heptane, 60°C, 6 h) + thermal (T <sub>max</sub> = 600°C, Ar; with ramps and holding times)	[27]
1	3.7 $\mu\text{m}$	11.1 $\mu\text{m}$	26.7 $\mu\text{m}$			
Gas atomised	60					
<b>IN HX</b>						
	$d_{10}$	$d_{50}$	$d_{90}$	Wax-polymer	Solvent + thermal (900°C, H <sub>2</sub> )	[16]
	3.2 $\mu\text{m}$	7.8 $\mu\text{m}$	15.1 $\mu\text{m}$			
Gas atomised	65					
<22 $\mu\text{m}$ Gas atomised	62-65			Proprietary (Catamold®)	Catalytic (HNO <sub>3</sub> )	[17]
	$d_{10}$	$d_{50}$	$d_{90}$	Wax-polymer	Solvent + thermal (900°C, H <sub>2</sub> )	[19]
1	4.5 $\mu\text{m}$	9.6 $\mu\text{m}$	18.4 $\mu\text{m}$			
2	3.2 $\mu\text{m}$	7.8 $\mu\text{m}$	15.1 $\mu\text{m}$			
Gas atomised	65					

Table 7 Powder and processing properties

of binders (which have been disclosed) are wax-polymer-based (for example polyethylene + paraffin wax). Debinding is usually a two-step process involving a solvent and thermal stage.

- In terms of heat treatment (Table 8) possible atmospheres are hydrogen, vacuum and an Ar-H<sub>2</sub> mixture. The minimum sintering temperatures, which are needed to achieve sufficient densities, are in the range of 1280°C (H<sub>2</sub>), 1260°C (vacuum) and 1235°C (Ar-H<sub>2</sub>).
- With respect to the tensile mechanical properties (Table 6) the strength values are generally comparable to cast material, but lower than those of wrought samples. A general tendency on how additional heat treatments change these values, cannot be concluded from the available data.

**Inconel HX**

IN HX is a solid solution-strengthened alloy. General properties are summarised in [30].

In [17] sufficient density values were achieved after sintering. The additional HIP treatment (1185°C, 100 MPa, 4 h) increased the density to 100%. Mechanical properties in the solution-annealed state (tensile test) were better than cast material, but the values were lower than those of a wrought sample.

In [19] the highest achieved density was 94.5%. No liquid phase formation was observed during the sintering process. It was noted that the starting powder may affect the liquid phase formation.

**Other properties**

In [16] the corrosion resistance of IN 718 was evaluated in different media and compared to pure Ni and stainless steel 316L. The superalloy performs well in nitric acid, bleach and sodium hydroxide, while severe corrosion occurs in hydrochloric and sulphuric acid.

In [19] the corrosion resistance of alloys IN 718, IN 625 and IN HX was evaluated in different media and compared to pure Ni and stainless steel 316L (Table 10). IN 625 and IN HX perform well under all tested conditions, while for IN 718 severe corrosion occurs in hydrochloric and sulphuric acid.

Atmosphere	T (°C)	T (min)	Density	Ref.
<b>IN 718</b>				
Hydrogen	1260	360	86% TD	[10]
vacuum	1250 - 1275	60 - 560	96.8 - 100% TD	[11]
Vacuum	1260	360		[14]
Vacuum	1260	120	98.3% TD	[16]
Ar	1300	180	97.2% TD	[17]
Vacuum	1265	60		[18]
Vacuum	1260	60 - 120		[19]
Vacuum	1260	120	94.9% TD	[21]
Vacuum	1275	120	98.9 % TD	
Vacuum	1290	120	96.7% TD	
Vacuum	1270 - 1300	120 - 360	94 - 98% TD	[22]
<b>IN 625</b>				
Hydrogen	1288 - 1298	24 - 60	> 99% TD for all parameters	[10]
Hydrogen	1280	30	100% TD	[16]
Hydrogen	1290	30	96.7 - 98.8 % TD	[19]
Ar-5% <sub>H</sub> 2	1235	120	> 96% TD	[26]
Vacuum	1260 - 1300	60 - 180	≈ 91 % TD (1260°C, 1 h) - 98.3% TD (1300°C, 3 h)	[27] <i>lower density value estimated from figure</i>
<b>IN HX</b>				
Vacuum	1260	120	93.8% TD	[16]
Hydrogen	1300	180	97.8% TD	[17]
Vacuum	1260 - 1300	60 - 120	94.5 % TD at 1275°C, 60 min	[19]
<b>U 700</b>				
Vacuum	1240	240	96% TD (< 150 μm), 99% TD (< 45 μm)	[6]

Table 8 Sintering parameters (theoretical densities: 8.2 g/cm<sup>3</sup> (IN 718), 8.44 g/cm<sup>3</sup> (IN 625), 8.2 g/cm<sup>3</sup> (IN HX))

C (%)	N (%)	O (%)	Ref.
0.08	0.03	0.05	[17]
0.06 - 0.09		0.05 - 0.055	[20]
≈ 0.04 (air) ≈ 0.08 - 0.6 (vacuum) ≈ 0.05 (Ar)		≈ 0.2 - 0.45 (air) ≈ 0.02 - 0.12 (vacuum) ≈ 0.04 - 0.09 (Ar)	[22] Values estimated from figure

Table 9 Impurity levels in the first generation of MIM superalloys

Corrosive media	316L mpy	HX mpy	718 mpy	625 mpy	270 mpy
Nitric acid	0.0	-	0.0	0.0	160
Hydraulic acid	8.8	3.8	16	0.3	5.5
Bleach	0.0	-	0.1	0.1	79
Sodium Hydroxide	0.8	-	0.0	0.0	0.0
Sulphuric acid	36	1.7	42	0.0	0.3

Table 10 Corrosion resistance of different MIM processed superalloys in comparison with stainless steel 316L and pure Ni 270 [19]

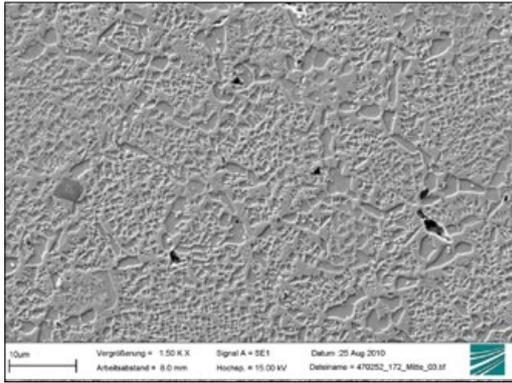


Fig. 4 Microstructure of MIM IN713 [37]

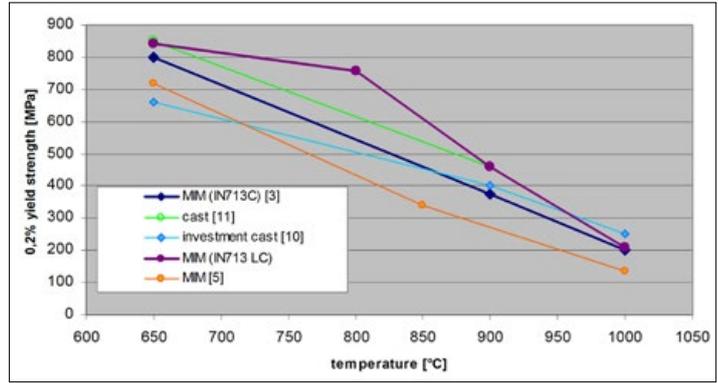


Fig. 5 High-temperature yield strength IN713, different processing routes [36]

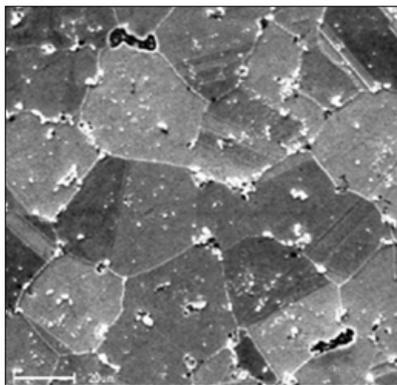


Fig. 6 Microstructure of MIM N90 [39]

### Second generation of MIM superalloys

The most commonly used superalloys (both single crystalline and polycrystalline) have a two-phase microstructure consisting of a fcc matrix ( $\gamma$  phase) and precipitates ( $\gamma'$  phase), mostly of type  $(Ni,Ti)_3Al$ . The precipitation-hardening effect leads to superior mechanical behaviour at elevated temperatures. In order to achieve these properties, the content of Al has to be increased compared to non- $\gamma'$  alloys.

The reasons why  $\gamma$ - $\gamma'$ -alloys have only recently found their way into MIM are not entirely clear, especially when taking into account that the alloy systems have been known for a long time. It is assumed that there was no market demand for this combination of alloy and technology until around ten years ago. Another possibility could be that MIM as a process had evolved to such an extent that other alloy systems apart from steels were given consideration.

Alloy systems will be described in

Powder	Solids part in feedstock (vol%)	Binder	Debinding	Ref.								
<b>IN 713</b>												
Gas atomised, < 22 $\mu$ m	62-65	Acetal-based (Catamold®)	Catalytic (HNO <sub>3</sub> )	[17]								
22 – 85 $\mu$ m Gas atomised			Solvent (water) + thermal (T <sub>max</sub> = 600°C)	[35]								
<b>Nimonic 90</b>												
Gas atomised, < 22 $\mu$ m	62-65	Acetal-based (Catamold®)	Catalytic (HNO <sub>3</sub> )	[17]								
<table border="1"> <tr> <td></td> <td>d<sub>10</sub></td> <td>d<sub>50</sub></td> <td>d<sub>90</sub></td> </tr> <tr> <td>1</td> <td>3.9 <math>\mu</math>m</td> <td>10.7 <math>\mu</math>m</td> <td>21.8 <math>\mu</math>m</td> </tr> </table> Gas atomised		d <sub>10</sub>	d <sub>50</sub>	d <sub>90</sub>	1	3.9 $\mu$ m	10.7 $\mu$ m	21.8 $\mu$ m			Thermal	[39]
	d <sub>10</sub>	d <sub>50</sub>	d <sub>90</sub>									
1	3.9 $\mu$ m	10.7 $\mu$ m	21.8 $\mu$ m									
<table border="1"> <tr> <td></td> <td>d<sub>10</sub></td> <td>d<sub>50</sub></td> <td>d<sub>90</sub></td> </tr> <tr> <td>1</td> <td>4.1 <math>\mu</math>m</td> <td>11 <math>\mu</math>m</td> <td>21.7 <math>\mu</math>m</td> </tr> </table> Gas atomised		d <sub>10</sub>	d <sub>50</sub>	d <sub>90</sub>	1	4.1 $\mu$ m	11 $\mu$ m	21.7 $\mu$ m	62.5	Paraffin wax, polypropylene, carnauba wax, stearic acid	Solvent (heptane, 60°C, 4 h) + thermal (T <sub>max</sub> = 600°C, Ar; 1 h)	[41]
	d <sub>10</sub>	d <sub>50</sub>	d <sub>90</sub>									
1	4.1 $\mu$ m	11 $\mu$ m	21.7 $\mu$ m									
<table border="1"> <tr> <td></td> <td>d<sub>10</sub></td> <td>d<sub>50</sub></td> <td>d<sub>90</sub></td> </tr> <tr> <td>1</td> <td>4.1 <math>\mu</math>m</td> <td>11 <math>\mu</math>m</td> <td>21.7 <math>\mu</math>m</td> </tr> </table> Gas atomised		d <sub>10</sub>	d <sub>50</sub>	d <sub>90</sub>	1	4.1 $\mu$ m	11 $\mu$ m	21.7 $\mu$ m	60	Paraffin wax, polypropylene, carnauba wax, stearic acid	Solvent (heptane, 60°C, 6 h) + thermal (T <sub>max</sub> = 900°C, Ar; 1 h)	[42]
	d <sub>10</sub>	d <sub>50</sub>	d <sub>90</sub>									
1	4.1 $\mu$ m	11 $\mu$ m	21.7 $\mu$ m									
<b>IN 100</b>												
Gas atomised, < 22 $\mu$ m	62-65	Acetal-based (Catamold®)	Catalytic (HNO <sub>3</sub> )	[17]								
<b>GMR-235</b>												
Gas atomised, < 22 $\mu$ m	62-65	Acetal-based (Catamold®)	Catalytic (HNO <sub>3</sub> )	[17]								
<b>N18</b>												
40-63 $\mu$ m Gas atomised		Polypropylene (60 wt%), paraffin wax (32.7 wt%), carnauba wax (7.4 wt%), stearic acid (0.03 wt%)	Solvent (heptane, 65°C) + thermal (T <sub>max</sub> = 650°C)	[38]								

Table 11 Powder and processing parameters

the following section and are summarised in the following tables:

- Table 11: Powder and processing
- Table 12: Sintering and density
- Table 13: Additional heat treatment
- Table 14: Tensile properties
- Table 15: Impurities

**Inconel 713**

IN 713 is a  $\gamma'$ -precipitation-hardened alloy with one of the highest Al contents of polycrystalline materials. It also has good corrosion resistance up to 1000°C. A summary of properties is given in [31].

The microstructure after sintering + HIP treatment is shown in Fig. 4. Some residual porosity is visible. Furthermore, the two phases are also clearly visible. Due to an optimised debinding regime, carbides could be reduced to a minimum.

In [17] samples in different states were analysed with respect to mechanical properties, impurities and surface roughness. The alloy had a density of 99.7% TD after sintering, which was increased to 100% TD after HIP treatment. In terms of tensile properties, values were compared to other processing routes. The values of the MIM samples are higher than those of cast material. In terms of impurities, values are compared with specification for carbon, which is fulfilled for this alloy.

In [32] an additional heat treatment was applied (parameters were not disclosed). Mechanical properties were also evaluated in different states. The additional heat treatment, which leads to a smaller size of the precipitates, helped to increase the tensile strength especially at elevated temperature.

In [33] samples in the sintered+HIP state were analysed with respect to tensile properties and oxidation resistance. MIM samples showed good oxidation resistance up to 1100°C, 100 h. The tensile strength at elevated temperature was, however, lower than that of cast material. This was attributed to a high degree of impurities, which was concluded to be due to incomplete debinding and impurities in the starting powder.

In [36] samples of IN 713C and IN713LC (lower carbon) were manufactured and analysed. By optimising the thermal debinding step through the use of Fourier Transform Infrared Spectrometry (FTIR) [34] the amounts of impurities were considerably lowered.

Atmosphere	T (°C)	t (Min)	Density	Ref.
<b>IN 713</b>				
Ar	1280	180	99.1% TD	[17]
Ar	1270	180	99.3% TD	[32]
Hydrogen	1310	60	99% TD	[35]
Vacuum	1265	180	95.2 – 96.7% TD	[37]
<b>N 90</b>				
Ar	1325	180	97.8% TD	[17]
Ar	1310	180	94.5% TD (GA)	[40]
	1280	120	97.7% TD (WA)	
Vacuum	1280	120	98.5% TD (WA)	[41]
	1330	180	97.3% TD	
<b>IN 100</b>				
Ar	1265	180	100% TD	[17] [32]
<b>GMR-235</b>				
Ar	1270	180	98.6% TD	[17]
<b>U 720</b>				
Vacuum	1230	120	96.7 – 98.6% TD	[37]
<b>MAR-M 247</b>				
Ar	1335	180	98.4% TD	[32]
<b>N 18</b>				
Vacuum	1265	60		[38]

Table 12 Sintering parameters and density (GA = gas atomised, WA = water atomised)

HIP	Solution anneal	Aging	Ref.
<b>IN 713</b>			
1200°C, 103 MPa, 4 h			[17]
1200°C, 100 MPa, 4 h			[33]
			[36]
<b>N 90</b>			
1185°C, 100 MPa, 4 h	1080°C, 8 h, Ar	705°C, 16 h, Ar	[17]
1160 – 1185°C, 100 MPa, 4 h, Ar	1080°C, 8 h, Ar	700°C, 16 h, Ar	[40]
	1080°C, 1 h	700°C, 16 h	[41]
	(no parameters given)	750°C, 16 h	[42]
<b>IN 100</b>			
1220°C, 103 MPa, 4 h	1080°C, 8 h, Ar	870°C, 12 h, Ar	[17]
<b>GMR-235</b>			
1185°C, 100 MPa, 4 h	1150°C, 2 h, Ar		[17]
<b>U 720</b>			
1130°C, 140 MPa, 4 h	1100°C, 1 h	650°C, 24 h +	[33]
		760°C, 16 h	[36]

Table 13 Additional heat treatments

Furthermore, the tensile strength was increased and is competitive with other MIM samples and processing routes, respectively (Fig. 5).

- The following conclusions are drawn:
- In terms of powder and process parameters (Table 11) no general

trend can be concluded. Based on the obtained data it is likely that this alloy can be processed with similar parameters to, for example, IN 718. In terms of heat treatment (Table 12) there is a variety of atmospheres that lead

State	T (°C)	YS (MPa)	UTS (MPa)	Elong (%)	Ref.
<b>IN 713</b>					
Sintered	20	916	1082	12	[17]
	20	890	1319	16	[35]
	650	720	997		[35]
	850	340	493		[35]
	1000	135	170		[35]
Sintered + HIP	20	959	1375	25	[17]
	650	800	1043		[33]
	900	373	455		[33]
	20	768	1210		[36] LC variant
	650	843	1085		[36] LC variant
	800	756	839		[36] LC variant
	900	459	537		[36] LC variant
<b>N 90</b>					
Sintered	20	785	1144	22	[40]
	20	690	858	11.4	[42]
Sintered + HIP	20	723	1161	21	[40]
Sintered + heat-treated (SA + AG)	20	732	1222	25	[17]
	20		1112	15	[41]
	20	906	1249	22	[40]
	20	760	920	10.6	[42]
Sintered + heat-treated (HIP + SA + AG)	20	791	1271	27	[17]
<b>IN 100</b>					
Sintered + heat-treated (SA + AG)	20	785	1184	5	[17]
Sintered + heat-treated (HIP + SA + AG)	20	780	1353	21	[17]
<b>U 720</b>					
Sintered + heat-treated (HIP + SA + AG)	650	677	1000		[33]
	800	595	656		[33]
	900	343	390		[33]
	20	1127	1534		[36], Li variant
	650	1026	1373		[36], Li variant
	800	931	955		[36], Li variant
	900	592	598		[36], Li variant

Table 14 Tensile properties

to sufficient densities. However, there are differences in temperature and time depending on the sintering atmosphere.

- With respect to the tensile mechanical properties (Table 14) as-sintered samples yield the lowest values in terms of strength. Applying the HIP treatment generally increases tensile strength to values, which are comparable to

cast material. Material purification (lower impurity contents in the powder) also seems to be beneficial to tensile properties; however, the percentage of tensile strength increase due to this cannot yet be estimated. Furthermore, some results on high-temperature mechanical properties were obtained (Fig. 5), which show the same general tendency.

C (%)	N (%)	O (%)	Ref.
<b>IN 713</b>			
0.17	0.02	0.04	[17]
0.32	0.02	0.25	[33]
0.178	0.023	0.102	[36]
0.063	0.017	0.102	[36], LC variant
<b>N90</b>			
0.08	0.01 – 0.02	0.05	[17]
<b>IN 100</b>			
0.14		0.07	[17]
<b>GMR 235</b>			
0.06		0.06	[17]
<b>U 720</b>			
0.09	0.002	0.26	[33]
0.038	0.006	0.093	[36]
0.032	0.002	0.107	[36], Li variant

Table 15 Impurities

**Nimonic 90**

N90 is another precipitation-hardened superalloy and the microstructure after sintering is shown in Fig. 6.

In [17] samples in different states were analysed with respect to mechanical properties, impurities and surface roughness. The alloy had a density of 97.8% TD after sintering, which was increased to 100% TD after HIP treatment. In terms of tensile properties, values were compared to other processing routes. The values of the MIM samples are comparable to those of cast/wrought material. In terms of impurities, values are compared with specification for carbon, which is fulfilled for this alloy.

In [40] a special process (plasma assisted debinding and sintering) was applied. Both water and gas atomised powders with slightly different compositions were used to manufacture samples. All samples had densities <94% TD after sintering, which was increased close to 100% TD after HIP treatment. The water atomised powder showed better mechanical properties. This was attributed to the higher content of strengthening elements (Cr, Co, Al).

Özgül *et al.* [42] analysed samples in the sintered and aged state. The optimum sintering range was determined by thermal analysis. It was observed that the density increases with sintering temperature and time

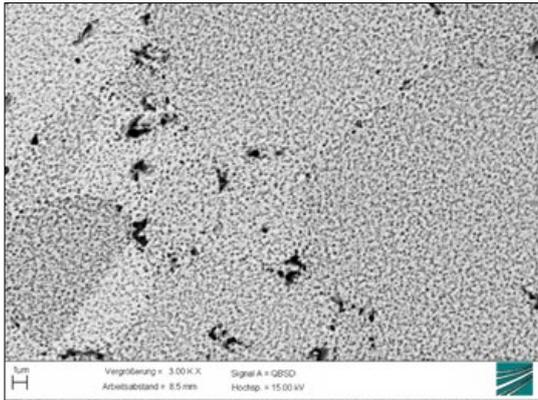


Fig. 7 Microstructure U720 [37]

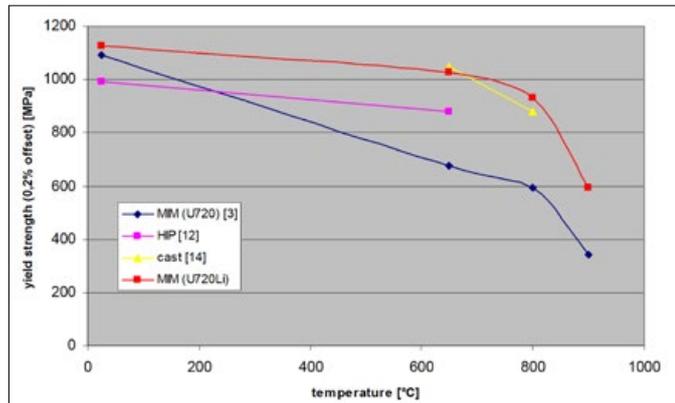


Fig. 8 High-temperature yield strength of U720 [36]

and the lower threshold is 1310°C, 1 h. Furthermore, an analysis of possible carbide precipitates was undertaken and for the samples in this study TiC was detected. Hardness was evaluated with different aging treatments with the optimum parameters being 750°C, 16 h. The measured tensile properties were comparable to cast material, but inferior to wrought and PM. This was attributed to the lower density.

In terms of powder and process parameters (Table 11) no general trends can be concluded. Based on the obtained data it is likely that this alloy can be processed with similar parameters to IN 718. In terms of heat treatment (Table 12) both Ar and vacuum lead to sufficient densities. However, there are differences in temperature and time depending on the sintering atmosphere. Independent of atmosphere, the minimum sintering parameters seem to be 1280°C, 2 h.

With respect to the tensile mechanical properties (Table 14) as-sintered samples yield the lowest values in terms of strength. Applying additional heat treatments generally increases tensile strength to values, which are comparable to cast material.

### Other alloys

In [17] samples of IN 100 and GMR-235 were analysed in different states with respect to mechanical properties, impurities and surface roughness. All alloys had densities > 97% TD after sintering, which was increased to 100% TD after HIP treatment. In terms of the surface roughness, the values of reference materials (316L and FN02) were reached. In terms of tensile properties, values were compared to other processing routes. Most values of the MIM samples are comparable

or in some cases higher than those of cast/wrought material. In terms of impurities, values are compared with specifications for carbon, which is fulfilled for all alloys.

In [38] samples of alloy N18 were manufactured. A compression-moulding procedure was applied in order to simulate the MIM process. Focus was put on the sintering mechanism, which was systematically analysed in terms of density, fraction of liquid and microstructure.

In [33] samples of Udimet 720 in the sintered + heat-treated (HIP+SA+AG) state were analysed with respect to tensile properties. The tensile strength at elevated temperature was lower than that of other processing routes. This was attributed to a high degree of impurities, which was concluded to be due to incomplete debinding and impurities in the starting powder.

In [36] samples of U720 and U720Li were manufactured and analysed. By optimising the thermal debinding step through the use of Fourier Transform Infrared Spectrometry (FTIR) [34] the amounts of impurities were considerably lowered. Furthermore, the tensile strength was increased and is competitive with other processing routes (Fig. 8). Fig. 7 shows the microstructure after sintering + heat treatment. The two-phase structure with finely dispersed precipitates is visible.

### Applications for MIM superalloys in industry

Almost all the data in this paper originates from publications which primarily deal with basic materials development. Advancement of this know-how into applications is significantly less documented. In [43] and [44] information is disclosed with respect

to aero-engine parts. It is indicated that superalloys processed by MIM are an attractive alternative to forged or electrochemically milled material for stator vanes, an example of which is shown in Fig. 9. The main motivation is the net-shape manufacturing process, which is seen as a key success factor with regard to costs. Furthermore,



Fig. 9 A single MIM compressor vane (top) and MIM vane cluster prototype (bottom) [43]

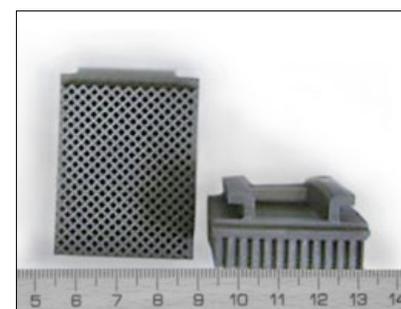


Fig. 10 A MIM honeycomb seal [43]

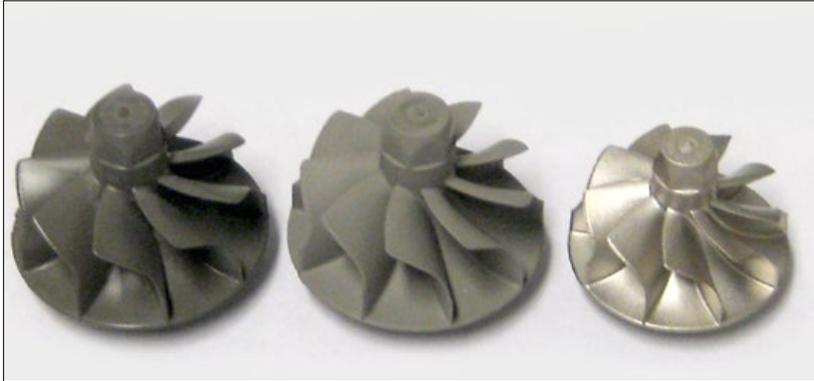


Fig. 11 Green (left), brown (middle) and sintered (right) turbocharger wheel made of IN713LC (Courtesy Schunk Sintermetalltechnik GmbH)

minimising material waste is a driver. It is stated that similar strength and ductility as with forged material were achieved. Furthermore, a high dimensional quality and sufficiently low surface roughness was achieved. Single vanes were brazed to a vane cluster (Fig. 9) and qualified for engine testing. A further mentioned applica-

aerospace sector. Further material and process prerequisites are mentioned:

- Assure property and microstructure repeatability
- Inspection and quality assurance
- Control of chemistry and processing effects
- Robust supply chain.

***'According to industry sources, testing and production in the aerospace sector has progressed significantly, particularly in the US, where all major aero-engine companies are believed to be involved in the process'***

tion is a honeycomb seal (Fig. 10). Further possible jet engine parts include adjusting levers, locking nuts and retaining plates.

In [45] a brief summary is given on the development of stator vanes made of IN 718 at Rolls Royce. Powders with particle sizes of 5 – 40  $\mu\text{m}$  were mixed with a binder containing PMMA and water soluble polymer. The debinding process consisted of solvent extraction (water) and a thermal step. After sintering, a single forging step and the finishing process were applied. The main advantages of MIM over forging are seen in a reduction of component manufacturing times, material waste and energy consumption.

The experimental findings in [23] were summarised earlier. Besides this, some more insight is given on what is necessary for MIM superalloys to receive better acceptance in the

Additionally, the issue of industry specifications is discussed. Based on the observed lack of information a document (AMS 5917) was developed and approved for IN 718. One of the conclusions was that MIM IN 718 has strength levels near 95% of wrought material and ductility levels between wrought and cast IN 718. However no further details on aero-engine parts are disclosed.

According to [46] prototype samples for the automotive sector have been produced recently (Fig. 11).

The available information suggests that MIM superalloys have not progressed very far in terms of application maturity. However, this is unlikely to be entirely true. In fact, the development is more advanced compared to what the available data suggests. According to industry sources, testing and production in

the aerospace sector has progressed significantly, particularly in the US, where all major aero-engine companies are believed to be involved in the process. However, the processing or test data remains confidential. This is because each individual company paid for many years of research in order to compile reliable data that allows a part to be in full compliance with Federal Aviation Administration (FAA) testing, and sharing these investments with competitors by making the data publicly available is not an option. It is therefore very likely that data from actual MIM parts in aero-engines is not going to be in the public domain for some time.

### **A review of published processing and properties data, and challenges ahead**

Looking at the information available, either from publications or other non-confidential sources, it becomes apparent that MIM processing of superalloys has been investigated for some time. For all alloys, processing parameters have been found which ensure sufficient density values even after sintering alone.

Sintering aside, the processing of MIM superalloys is generally comparable to other classes of MIM processed alloys such as steels. No significant changes were made to powders, binders, feedstock compositions and the respective debinding processes.

For the majority of alloys additional heat treatments such as HIP, solution annealing and aging are known from cast/wrought materials and were directly applied to MIM samples. Furthermore, the microstructure has been characterised and there exists data on hardness (not compiled in this paper) and tensile properties. Based on these values, most alloys can compete with cast and/or wrought counterparts.

In terms of commercial powders and feedstocks respectively, MIM grade superalloy powders are available from suppliers such as Sandvik Osprey, while BASF [17] and PolyMIM [35] have superalloy feedstocks available.

However, as mentioned previously, MIM parts made of superalloys are a niche product and there are a number of reasons for this. Firstly, processing and properties data is spread very unevenly with only a few alloys contrib-

uting more than just one reference. In terms of the number of references, Inconel 718 has been studied the most by far, followed by Inconel 625 and Inconel 713.

In order to assess the application potential more thoroughly, significantly more data would be needed. This refers on one hand to testing already done (for example hardness and tensile testing), but on the other hand and more significantly to test methods such as creep, fatigue and fracture analysis.

Secondly, the data on binder systems and impurities is rather scarce and binders are one of the main sources of impurities, especially carbon, besides the powder. Furthermore, control of impurities is generally difficult due to the nature of the superalloys, as most of these alloys contain a significant percentage of reactive elements, which tend to form either carbides (Mo, Nb, Ti) and/or oxides (Al, Cr, Ti). Taking all this into account, the following main challenge ahead exists:

- Control of impurities in the process. This actually starts with process optimisation of powder atomisation and continues with the choice of binders and especially

with parameter control during debinding.

Thirdly, in terms of processing, there are two main challenges ahead:

- Achievement of impurity threshold levels, their control and reproducibility in order for the process to be accepted by customers.
- Sintering can only be done in semi-continuous furnaces (walking beam) or in a batch process if high vacuum is needed. This significantly influences the process time and thus costs.

### Summary and outlook

MIM superalloys have been investigated for around 30 years. In terms of processing, there are several alloys available from which parts can successfully be produced. Concluding from the literature, there are a number of challenges ahead, with the main ones being:

- Compilation of more reliable data
- Overcome processing issues such as impurity control and sintering
- Establishment of material standards.

Some of these challenges may have

been overcome already on an industrial scale, but as stated above concrete data is not available. Generally, information on applications is rather scarce, but from what is available there are indications that the most promising parts in aero-engines are stator vanes, while in the automotive industry there is great potential for parts with high thermo-mechanical loads such as those used in turbochargers.

Another aspect, which has not been discussed yet, is the expansion of the MIM process into other classes of superalloys such as Fe- or Co-based ones. This has not been covered in this review as there is nearly no data available. In fact only one example is cited, [36], where the potential to manufacture oxide-dispersion strengthened (ODS) alloys by MIM is evaluated. A manufacturing route is suggested, which differs from the usual MIM routine insofar as that the powder is mechanically alloyed (MA). It is shown that the MA powder can be processed by MIM with no major modifications other than from binder content. Sintering is challenging and sufficient densities are yet to be reached. ODS alloys are not in use today, however with MIM as the technology to manufacture parts this may change.



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## List of abbreviations

TD	Theoretical Density
WA	Water Atomised
GA	Gas Atomised
MIM	Metal Injection Moulding
HIP	Hot Isostatic Pressing
SA	Solution Annealing
AG	Aging
HT	Heat Treatment
ODS	Oxide Dispersion Strengthened
MA	Mechanical Alloying



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# Global PIM Patents

The following abstracts of PIM related patents have been derived from the European Patent Office's database of patents from throughout the world.

## CN201659577 (U)

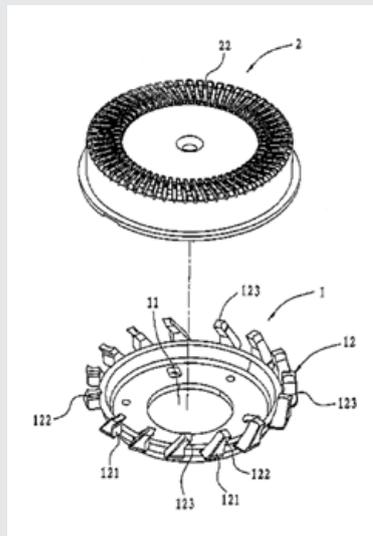
### SHAVER CUTTER STRUCTURE

**Publication date:** 2010-12-01

**Inventor(s):** Jingru Yan, China

This patent describes components used in a shaver cutter assembly. In this instance the shaver cutter structure comprises of a movable cutter and a fixed cutter. The movable cutter is assembled on a rotating shaft of the shaver body motor, with the fixed cutter covering the movable cutter. Rotating blades are arranged on the movable cutter driven by the shaver's motor.

Each blade is made of ceramic powder or metal powder via injection moulding and comprises a blade body and a cutting portion extending from the blade body. The fixed cutter has cutting edges arranged on the top surface. As the movable cutter and the fixed cutter are both made of



ceramic powder or metal powder via injection moulding, the sharpness of the cutting bevels can be increased greatly.

## WO2010104652 (A1)

### GOLF CLUB FACE HAVING ENCAPSULATED TUNED STRUCTURE

**Publication date:** 2010-09-16

**Inventor(s):** Richard Steven Wahlin et al, NIKE International, USA

The patent describes a golf club head as having a face portion and a body portion. The face portion has a support frame, a matrix structure within the support frame, and a face material surrounding the matrix structure.

In the example discussed, a face portion is prepared with a metal support frame having wires whose ends are attached to the support frame, forming a 'racquet'. The wires are then encapsulated with a metal/polymer composite material to form a face material. The face material is applied over the matrix structure by Metal Injection Moulding.

## US2010044929 (A1)

### METHOD OF FORMING A SPARK PLUG INSULATOR

**Publication date:** 2010-02-25

**Inventor(s):** Jeffrey Boehler, USA

The method for forming spark plug insulators, particularly asymmetrical spark plug insulators, without required machining after an initial heating or firing are described in this patent. The method for forming an asymmetrical spark plug insulator includes forming a first mixture comprising a binder made from water and agar, then forming a second mixture comprising a ceramic material. The first mixture and the second mixture are combined to form a moulding compound. The moulding compound is injection moulded to form an asymmetrical spark plug insulator. The asymmetrical spark plug insulator is then sintered, resulting in a final net shape and size.

## DE102009000463 (A1)

### METHOD FOR MANUFACTURING FITTING MODULE OF GAS SENSOR

**Publication date:** 2010-08-05

**Inventor(s):** Thomas Loibl et al, Robert Bosch GmbH, Germany

This patent describes a method for manufacturing a fitting module of a gas sensor. The method involves injection moulding and sintering a ceramic body, where the ceramic body is formed to fit in a housing.

A sensor element for the detection of the physical characteristics for measuring gas is surrounded by the ceramic body. The injection moulding of the ceramic body is made directly on to the sensor element. The sensor element is inserted as an unsintered green body in an injection moulding tool. The ceramic body is formed from a ceramic material using injection moulding.

## CN101857433 (A)

### COLOURED ZIRCONIUM OXIDE CERAMIC COMPONENT

**Publication date:** 2010-10-13

**Inventor(s):** Guanwei Liu et al, Tsinghua University, China

Described is a process for preparing a coloured zirconium oxide ceramic component. The method comprises of mixing the injection moulding ingredients by mass, 85-90% of zirconium oxide powder, 8-12% of low melting point binder and 2-4% of non-soluble skeleton binder.

The resulting feedstock is injection moulded. The debound blank is soaked in a colouring ion-containing solution and sintered to obtain coloured zirconium oxide ceramic. The degree of colour can be controlled by varying the soaking time. The method offers a simple, low cost process, suitable for industrial production.

# Moulding equipment for the processing of coarse powders by Metal Injection Moulding

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Metal Injection Moulding (MIM) is a mature technology for the net shape production of complex metallic components. Typically the MIM process utilises small spherical powders, having particle sizes below 30 µm. Today there are tendencies to use coarser powder particles for MIM processing. There are different reasons for these tendencies: on one hand there is a growing demand for titanium MIM parts, where coarser powder particles are used because of the high reactivity of titanium powder. On the other hand there are economical considerations to use coarser powders also for the production of traditional MIM materials such as steels. The processing of coarser powders generates additional demands on the design of the plasticising unit of injection moulding machines because there is a significantly higher wear compared to the processing of standard powders. This paper presents methods to minimise wear and discusses tests for the processing of low alloyed steel powders with particle sizes < 45 µm and < 63 µm as well as Ti6Al4V powder < 45 µm.

For the production of Metal Injection Moulded parts spherical powders with particle sizes below 30 µm are typically used. In the case of pre-alloyed powders the size ranges include powders with particle sizes of D80= 22 µm, D90=16 µm and D90= 22 µm [1, 2]. In other publications the use of coarser powders with particles sizes D90= 31 µm [3] up to particle sizes of D90 of 157 µm have been discussed [4, 5].

For the processing of titanium and titanium alloys the use of powders with median particle sizes of 30 µm to 60 µm is reported [6] but typically particle sizes below 45 µm are used [7, 8].

The use of coarser powders during Metal Injection Moulding is generating additional demand on the design of the plasticising unit of the injection moulding machine because there is a significantly higher wear during the processing of powders having particle sizes of less than 20 µm.

The plasticising unit of the moulding machine comprises a barrel, a screw and a non-return valve. The function of the non-return valve (Fig. 1) is two fold. During dosing it allows the molten feedstock to be dosed in front of the screw tip. When the screw is acting as a piston during injection the non-return valve needs to close in order to move the melt into the mould. Furthermore, it prevents the melt from flowing back into the barrel by floating across the outer diameter of the bushing. To ensure this, the clearance between the inner diameter of the barrel and the outer diameter of the bushing needs to be as small as possible. Typically this is in the range of 10 µm to 24 µm. With this it can be expected that wear will occur at the bushing of the non-return valve and/or the barrel, when coarse MIM powders are processed.

Depending on the powder characteristics (hardness/ductility) different wear mechanisms can be present during MIM processing, including abrasive wear and adhesion. For the system screw/barrel there is a different situation. The clearance between the screw and

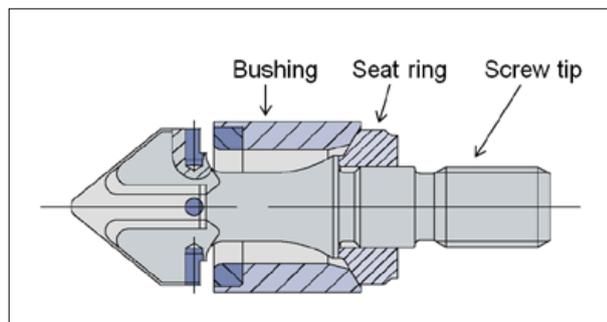


Fig. 1 Schematic view of the non-return valve. The non-return valve is made out of a high wear and corrosion resistant steel. The bushing and screw tip is equipped with cemented carbide inserts (Courtesy Arburg)

the barrel is larger and therefore the risk of wear being caused by the particle size of the powder is not present, as long as the powder particles are smaller than approximately 100 µm.

## Experimental procedure

### Powders and feedstock

For the experimental work carried out in this investigation, two grades of a low alloyed steel Micro-Melt 100Cr6 with particle sizes <45 µm and <63 µm supplied by Carpenter Powder Products GmbH and a Ti6Al4V < 45 µm supplied by AP&C, A Division of Raymor Industries Inc., were selected (Table 1).

The particle sizes of the powders were characterised by Beckman Coulter LS and the morphology was analysed by SEM (Fig. 2).

The steel powders were subsequently processed into feedstocks on a Bellaform Shear Roller Extruder BSW 100 without pre-mixing in a kneader using a binder system from eMBe Products and Service

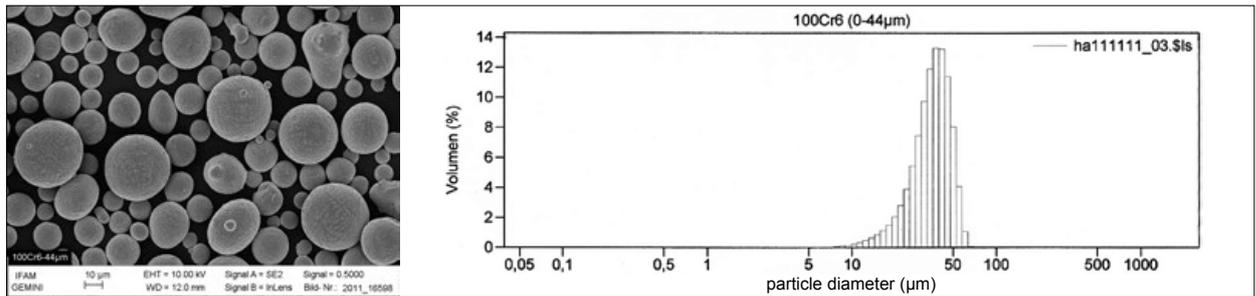


Fig. 2a SEM; 100Cr6, <45 μm and particle size distribution (Courtesy Fraunhofer IFAM)

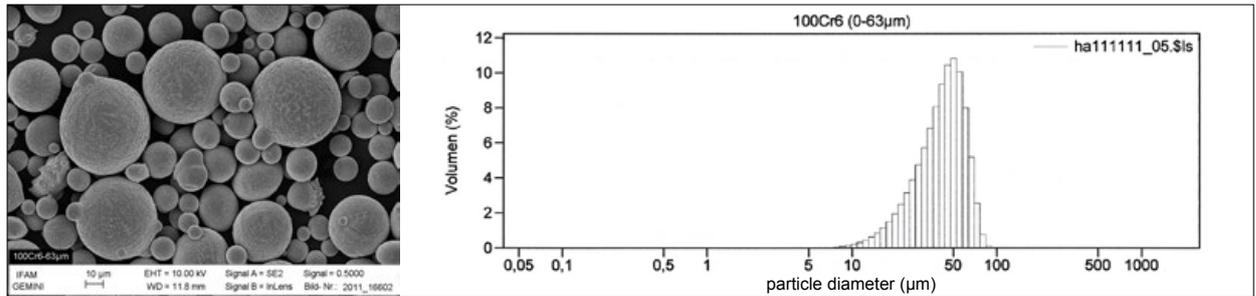


Fig. 2b SEM; 100Cr6, <63 μm and particle size distribution (Courtesy Fraunhofer IFAM)

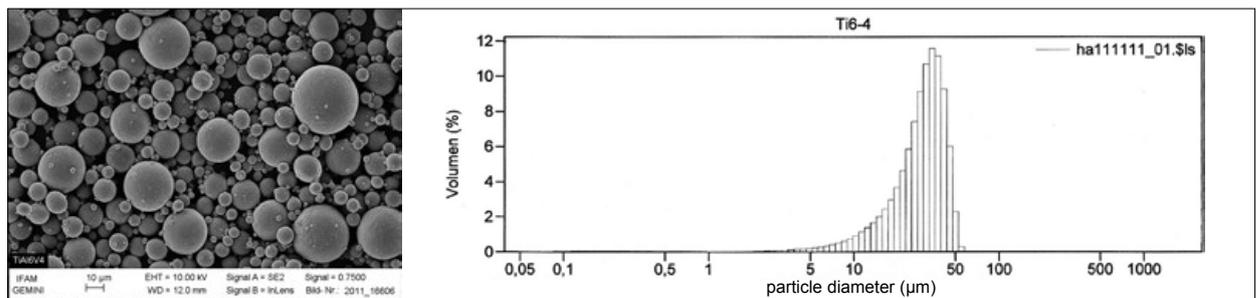


Fig. 2c SEM; Ti6Al4V, <45 μm and particle size distribution (Courtesy Fraunhofer IFAM)

Powder	Particle size in μm				
	Dv10	Dv25	Dv50	Dv75	Dv90
100Cr6, < 45 μm	22.41	29.26	36.53	43.56	49.82
100Cr6, < 63 μm	22.60	31.63	42.84	53.50	62.46
Ti6Al4V, <45 μm	13.87	22.20	30.44	37.53	43.10

Table 1 Particle sizes of the test powders



Fig. 3 The standard machine Allrounder 270 S can be equipped for PIM (Courtesy Arburg)

GmbH. The feedstocks contained 6.7 wt% binder, which resulted in a solids loading of 65 vol%.

The feedstock formulation for the Ti6Al4V powder was performed by eMBe Products and Service GmbH using their binder system on a Shear Roller Extruder.

Additionally, commercially available Catamold titanium feedstock from BASF SE was chosen for testing.

**Injection moulding**

Moulding trials were performed on an Arburg injection moulding machine, Allrounder 270 S (Fig. 3), with a clamping force of 400 tons and a size 70 injection unit, which is equipped for MIM processing with a position regulated screw and an 18 mm MIM cylinder assembly.

For all tests a single cavity test mould with a cylindrical geometry was selected. Using this mould, different screw strokes during injection could be achieved by a special ejector function, which allowed a reduction of the mould cavity height (Fig. 4).

Two different screw strokes and thus two different shot volumes were realised:

- A short stroke of less than the length of the bushing of the non- return valve,  $L_{Stroke} < L_{Bushing}$  and
- A long stroke of approximately double the length of the bushing of the non-return valve,  $L_{Stroke} \leq 2L_{Bushing}$

The machine settings for dosage stroke, injection speed and switch over position were kept constant during the tests and are summarised in Table 2.

The barrel and mould temperatures, as well as holding pressure profile, were adjusted to the actually processed feedstock and kept constant for the tested feedstock. Non-return valve bushings with different diameters were used to allow for a clearance between the barrel and the bushing of 20 µm, 70 µm and 115 µm. After each test, consisting of 100 injection cycles, the plasticising unit was cleaned and the non-return valve was inspected.

**Results**

**Wear depending on type of powder**

The trials revealed that already after 100 moulding cycles wear was observed for each material when the standard clearance of 20 µm for the non return valve was used. The wear itself was different for the different types of material. During moulding of the low alloyed steel feedstocks only abrasive wear could be observed at the bushing, leading to grooves in the axial direction over the complete length of the bushing. When moulding the titanium feedstocks fewer and different scratches and grooves could be observed. The grooves were not only oriented in the axial direction. Additionally, a minimal build up of titanium on the bushing was already visible after 100 cycles (Fig. 5).

**Wear after elongated test time**

After the initial testing of the steel feedstock 100Cr6 - 45 µm it was observed, that the wear starts only at one side of the lateral area of the bushing while at the opposite side no wear was observed. When the test time was extended to 200 and 300 cycles, grooves were visible on the complete lateral area of the bushing.

For titanium feedstocks this could not be observed. Here the complete lateral area of the bushing showed scratches, grooves and material build up already after the first 100 cycles. After additional testing these scratches/grooves intensified and more material build up was visible (Fig. 6).

**Wear depending on the clearance between bushing and barrel**

Tests were carried out with different clearances between the barrel and the bushing of the non-return valve. These tests showed that



Fig. 4 Test part produced with test mould at different cavity heights (Courtesy Arburg)

Dosage stroke	21 mm / 39 mm
Switch over position	6 mm
Injection speed	40 mm/s

Table 2 Injection moulding parameters

increasing the clearance from 20 µm to 70 µm and then 115 µm leads to a decrease of wear on the bushing. Again the appearance is different when comparing the tested low alloyed steel feedstocks and the titanium feedstocks. For the low alloyed steels the length of the grooves created by the feedstock decreases to approximately 40 - 45% and 15 - 20% of the length of the bushing (Fig. 7).

With the Ti6Al4V feedstock, fewer scratches occurred on the lateral area but more Ti6Al4V was welded to the surface of the bushing when the clearance was increased (Fig. 8). At higher magnifications it can be seen that after the Ti6Al4V has been welded to the surface of the bushing it creates deep grooves in the bushing during the axial movement of the bushing during injection (Fig. 9).

**Wear depending on screw stroke**

No significant differences on the wear of the bushing could be observed for the trials carried out at different screw stroke of  $L_{Stroke} < L_{Bushing}$  and  $L_{Stroke} \approx 2L_{Bushing}$ .

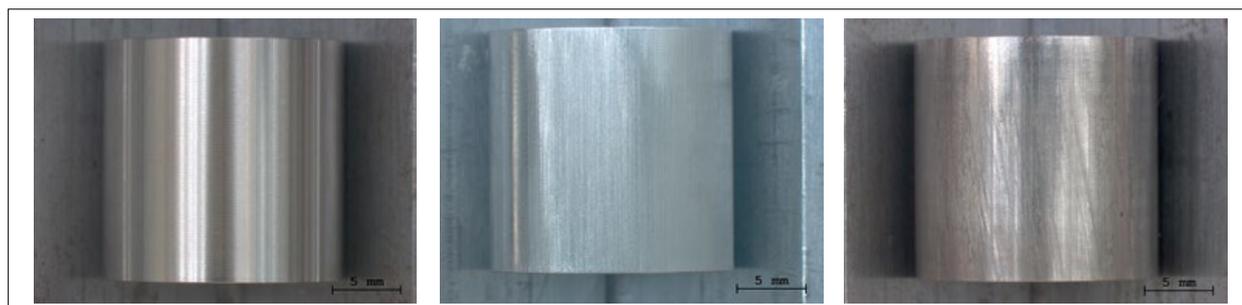


Fig. 5 Bushing before testing (left); after 100 injection cycles with 100Cr6 - 45 µm (centre) and after 100 injection cycles with titanium (right),  $L_{Stroke} < L_{Bushing}$  (Courtesy Arburg)

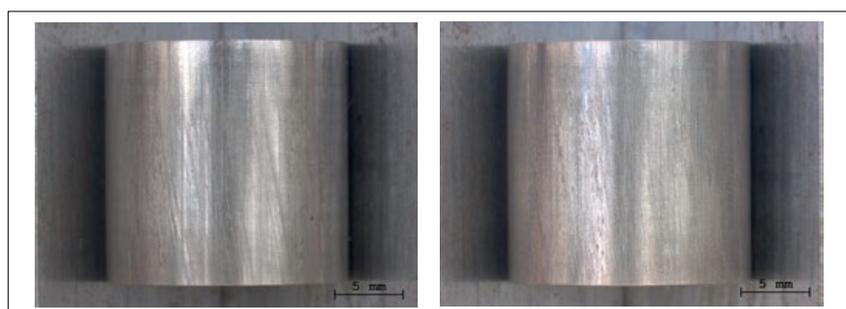


Fig. 6 Bushing after 100 and 200 moulding cycles, Ti6Al4V Feedstock (Courtesy Arburg)

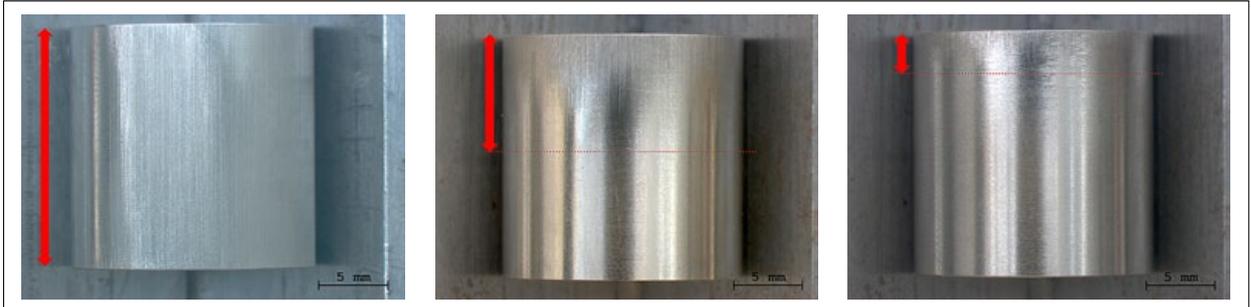


Fig. 7 100Cr6 - 45 μm feedstock, wear depending on clearance. Clearance increases from left to right from 20 μm to 70 μm to 115 μm (Courtesy Arburg)

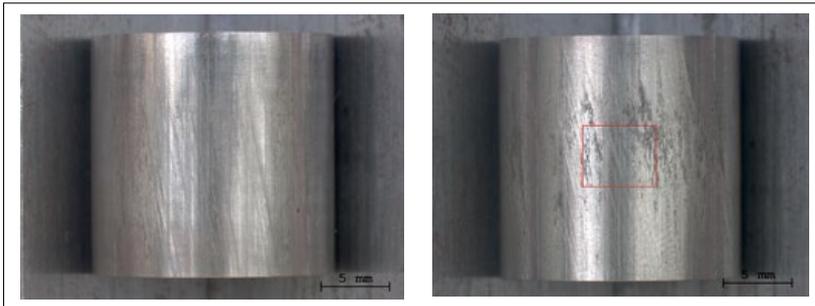


Fig. 8 Ti6Al4V feedstock, wear depending on clearance. Clearance increases from left to right from 20 μm to 70 μm (Courtesy Arburg)

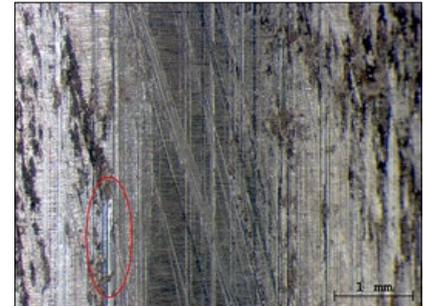


Fig. 9 Detail of Fig. 8, showing start of deep groove of cold welded Ti6Al4V (Courtesy Arburg)

**Discussion**

As could be demonstrated by the results, the wear of the bushing can be influenced by the clearance between the plasticising barrel and the bushing of the non-return valve.

Differences in the wear depending on the type of material could clearly be demonstrated.

This includes abrasive wear for the steel powder and a combination of cold welding and subsequent abrasive wear for titanium materials.

In the case of low alloyed steel, an increase in the clearance will lead to a decrease in wear of the bushing.

For titanium there is a similar tendency regarding the number of scratches. It was however observed that with titanium cold welding occurs. This in turn will create new wear in the form of grooves when the cold welded material is removed from the surface during the injection movement.

It can be concluded that an increase of the clearance between bushing and barrel is a good method to minimise the wear of the non-return valve. Although the test results for the titanium materials could not clearly show this, it is known that increasing the clearance between barrel and bushing leads to an extended life time of the non-return valve in high volume commercial production.

A second approach to minimise the wear of the bushing could be to use a more wear resistant material such as cemented carbide or ceramic. This will work if the wear is purely abrasive.

In case of a combination of cold welding and abrasion, as could be seen with titanium, using a harder material could lead to even more problems due to the risk of brittle fracture of the bushing.

Within the plasticising unit the wear will not only occur at the non-return valve but also at the barrel. Since it is known that a blocking of the non-return valve as a result of cold welded material can lead to severe damage at the screw tip, and since it is more economic to change the non-return valve, the investigation did not focus on wear of the barrel.

Influences arising from potential wear at the screw tip and the seat ring have not been discussed in this paper.

**Outlook**

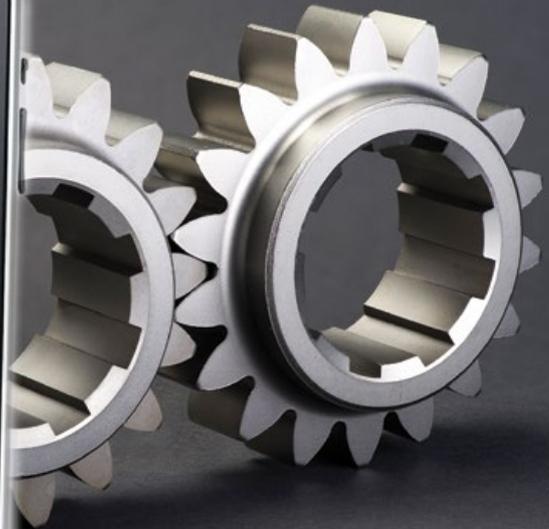
Depending on the viscosity of different feedstock the material cushion consistency needs to be investigated for the different clearances between the barrel and the bushing of the non-return valve.

Furthermore, the wear occurring at the screw tip and seat ring should be considered since the conducted trials already indicate an influence on the wear of the bushing.

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# POWDER METALLURGY REVIEW



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# Development of the Ultrasonic Spray Pyrolysis (USP) process for nanopowder production (Part 1)

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Nanostructured materials and their applications have been one of the key research topics for industry in recent decades. Various nanomaterials and numerous applications for them are known today, and much research has been undertaken on the use of nanopowders in the Metal Injection Moulding (MIM) process. However, the industrial application of nanomaterials is still challenged by the limited number of methods suitable for large scale nanomaterials production, especially when it comes to nanomaterials with target morphology and complex composition. This paper reports on the scale-up of the Ultrasonic Spray Pyrolysis (USP) process. The USP process as a nanoparticle production method is a relatively inexpensive and versatile technology based on an aerosol process to produce fine metallic, oxidic, composite nanoparticles of precisely controlled morphology and defined chemical compositions from water solution using different metal salts and their mixtures [1-4].

This paper is the first of a two-part report on the results of research into the Ultrasonic Spray Pyrolysis (USP) process. This first paper features a description of the construction of a prototype device to be launched in the production phase in the first quarter of 2013. The trial phase began in December 2012 and preliminary results show the suitability of the design, as well as the success of the scientific and engineering work of the IME Process Metallurgy and Metal Recycling team at RWTH Aachen University in collaboration with engineers from EliNo GmbH. The second part of the report will be devoted to the control system, the technological process itself and the results of final tests of the equipment presented.

## Introduction

Over the last ten years ultrasonic spray pyrolysis synthesis has been the subject of research at the IME Process Metallurgy and Metal Recycling department at RWTH Aachen University. Different organic and inorganic salts were used as precursor materials for the preparation of metallic, oxidic and composite nanopowders by ultrasonic spray pyrolysis using the equipment shown in Fig. 1a.

Synthesised powders can be divided in three groups:

1. Metals (Au, Ag, Co, Cu, Zn, Ni, Fe) [1-2]
2. Oxide (TiO<sub>2</sub>, ZnO, Al<sub>2</sub>O<sub>3</sub>, RuO<sub>2</sub>) [3-4]
3. Composite materials with partially core-shell structure (CuNi, FeCo, NiCo, RuO<sub>2</sub>/TiO<sub>2</sub>, La<sub>0.6</sub>Sr<sub>0.4</sub>CoO<sub>3</sub>, C/LiFePO<sub>4</sub>, Au/TiO<sub>2</sub>, Ag/TiO<sub>2</sub>) [4]

Next to variations in chemical composition, it is possible also to produce nanopowders with various morphologies (spherical, cylindrical, triangular, dense, porous, hollow, core/shell nanoparticle). Some of the nanopowders produced by USP are presented in Figs. 1b and 1c.

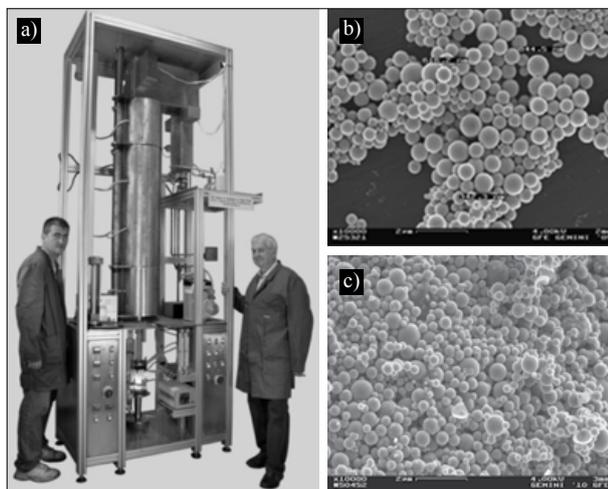


Fig. 1 a) USP Equipment at IME Process Metallurgy and Metal Recycling, b) NanoAg [2] and c) TiO<sub>2</sub> [4]

## Design of USP equipment

A decade of experience in nanoparticle synthesis by USP was the basis to develop a system for industrial scale production. The main parts of the demonstration scale Ultrasonic Spray Pyrolysis equipment are, A: Aerosol ultrasonic generator, B: High-temperature furnace with five wall heated reactors, C: Electrostatic filter, and D: Vacuum system. The concept draft and device photo are shown in Fig. 2.

In cooperation with EliNo GmbH engineers, and based on a previously built small scale prototype by the IME Institute (Fig. 1), after nearly six months of intensive work by two teams, EliNo

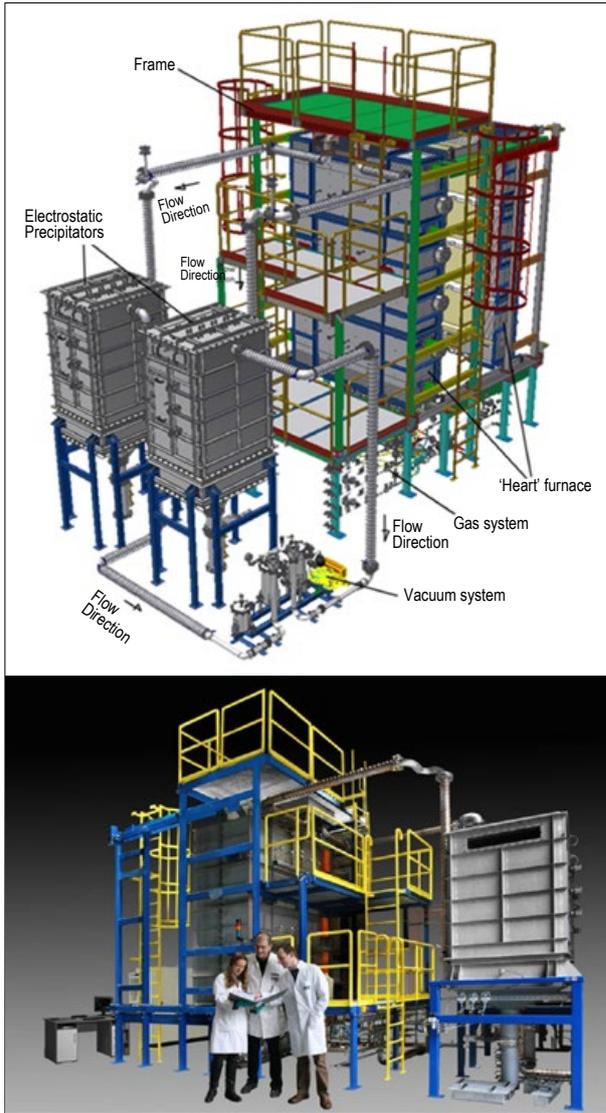


Fig. 2 The concept of Demo scale USP and photo of the equipment

GmbH and the IME Institute, the first design of the demonstration scale version of the device with the working name “MIRANDA” was developed.

The main engineering challenge was to transfer scientific achievements and maintain process specifics on a macro scale. This involved many complex calculations relating to gas flow, temperature uniformity and the impact of these factors on the process.

The “hearth” furnace consists of four separately regulated heating zones (Fig. 3). An additional unique aspect of the device is its full tightness. A specific challenge related to the thermal decomposition in an inert atmosphere ((Ar, N<sub>2</sub>), reduction (H<sub>2</sub>) or oxidation (O<sub>2</sub>) gases) of reaction pipes to flange connections, further affected by the negative influence of high temperatures and thermal expansion. Engineers from Elino GmbH established solutions to eliminate the above mentioned problems.

Each heating zone in the furnace is controlled separately, and each has separate temperature monitoring. Temperature uniformity is min. +/- 10°C, max. +/- 15°C based on data collected after the first trials. The proper construction of heating elements and their heating power used in each of the four zones enables the setting of much higher temperatures, which additionally make this device very flexible in the full temperature range necessary for nanotechnology.

Additional advantages of this construction, following the first trials, have been identified as very good thermal insulation. At a working temperature in heating zone 2 and 3 of 1000°C, the temperature of the shell does not exceed 30°C, which ensures safe usage of delicate ultrasonic aerosol generators, which as electrical units are very sensitive to high temperatures.

Further important components are ultrasonic aerosol generators and the gas system. In order to understand the essence of the gas system, one should understand the basis of the process. A gas flow is fed to the control valve where it is adjusted depending on the type of gas or mixture of gases. Successively the gas is sucked by vacuum pump to the aerosol generators and then to the reaction pipes. This gas flow has two main functions: reaction gas and carrier gas. A vacuum pump provides the pressure below atmospheric pressure in the whole system, which has positive influence on the process itself. The gas and aerosol generation system are shown in Fig. 4.

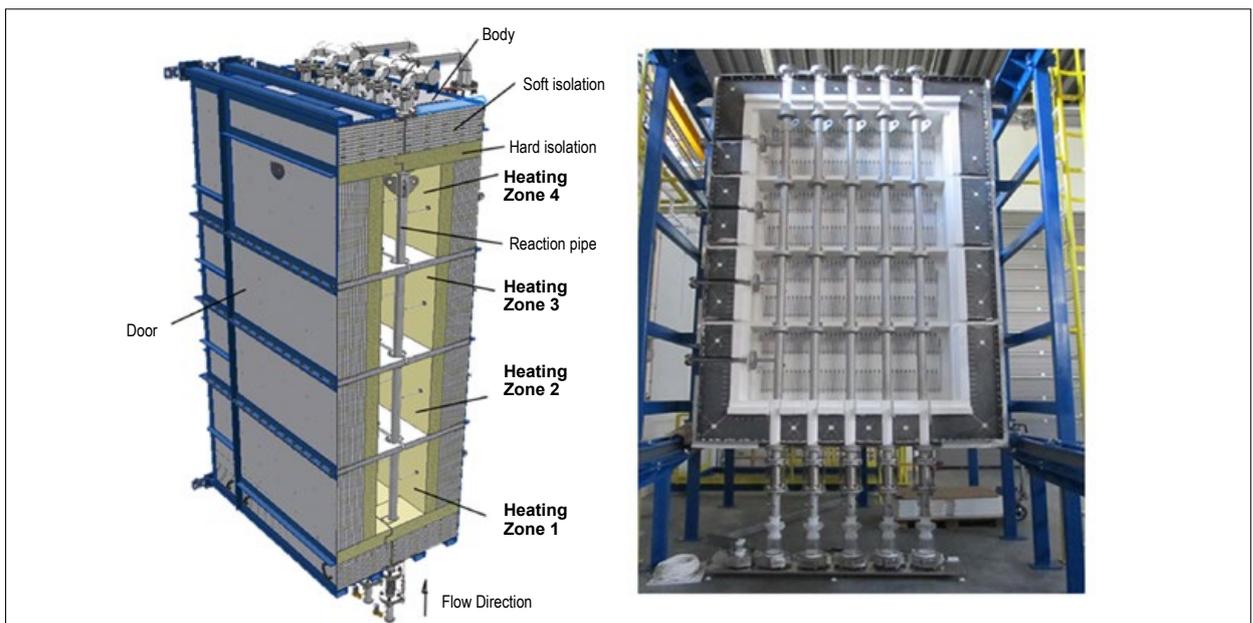


Fig. 3 Furnace with four heating zones and wall heated reactors

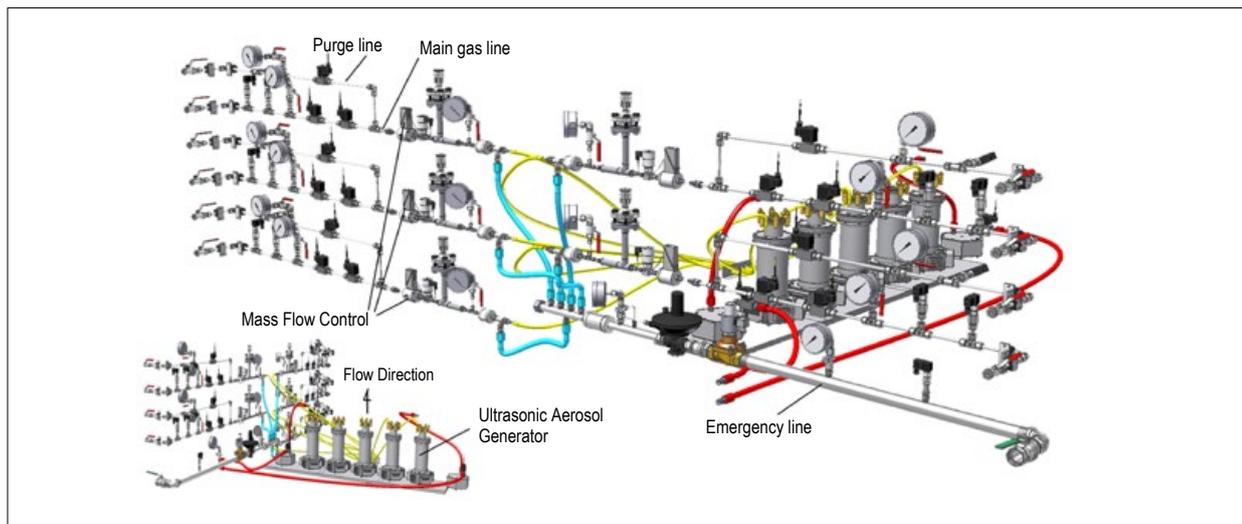


Fig. 4 The gas and aerosol generation system

There are five aerosol ultrasonic generators, called “Priznano”. Their design is the effect of the joint work between IME researchers, engineers from Elinio GmbH and engineers from PRIZMA Company, Kragujevac, Serbia. The main advantages of this system compared to other systems for aerosol production are the small droplet size, established industrial design, continual processability, high corrosion resistivity and the ability to operate with H<sub>2</sub>.

Nanopowder collection occurs in specially designed Electrostatic Precipitators (Electro Filter). Based on the mini version and results of previous research, the IME and Elinio GmbH team developed, on paper, a concept that was mathematically an extension of the laboratory design previously developed by IME and Schnick Industrieberatung. It should be mentioned that there are no exact rules on how to design or build such devices as electrostatic precipitators with regard to this particular technology. Professional involvement of members of the “MIRANDA” project showed that there is no easy transfer from the micro to macro version. Additional factors, such as the type of gasses, acids, gas flow, influence of temperature on certain materials and their nonlinear behaviour forced the engineers to reject the original version and develop an entirely new design shown in Fig. 5.

An additional factor that had an influence on the electrostatic precipitator design was making it such that it could operate with harmful gasses (which requires a gas-tight design) and acids while maintaining proper temperature inside the EGR, and simultaneously obtaining the final properties of the nano product in the required industrial quantities. It must first be clarified that this unit uses electrostatic precipitators connected to the reaction pipes coming from the furnace in such a way that redundant operation and flexible adjustment of throughput is possible. The electrostatic precipitators will always be the part of the device that requires the most maintenance and this is made possible due to the specific design.

As previously mentioned, gas and nanopowder is collected by vacuum pump from the reaction pipes placed in the furnace. The temperature of this stream is measured on the inlet to the electrostatic precipitators and is maintained automatically. This is necessary for the protection and resistance of the seal to corrosion and acids during the process.

The inner design of the electrostatic precipitator consists of a few emitting/collecting electrodes made from adequately selected stainless and temperature-proof steels.

The emitting electrode is connected to a high voltage generator

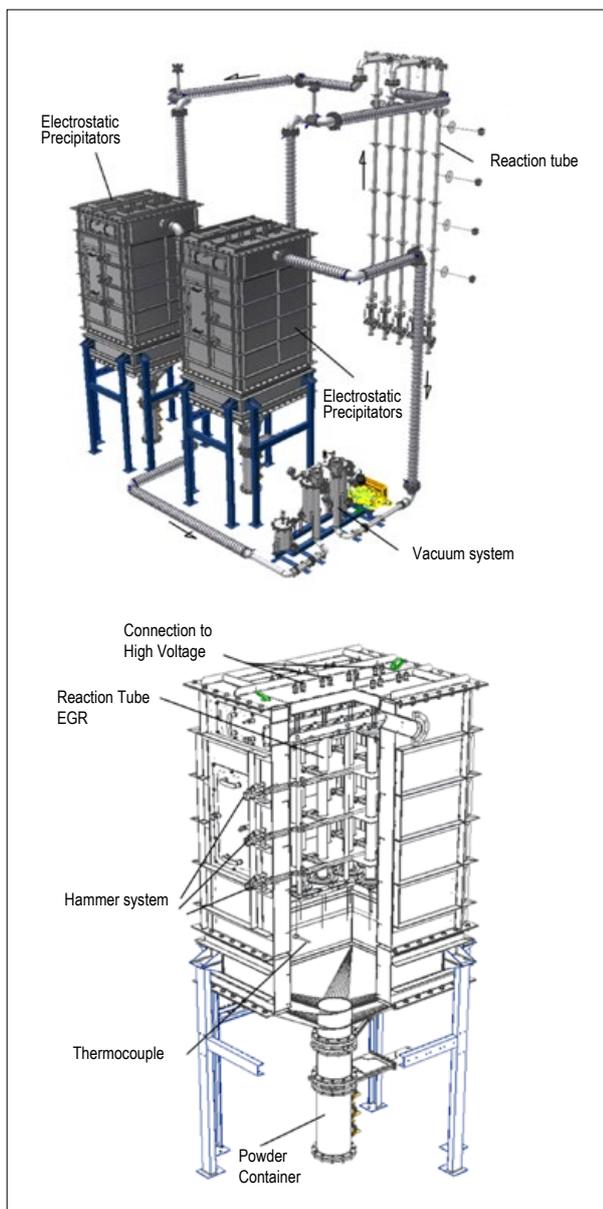


Fig. 5 Design of electrostatic precipitator

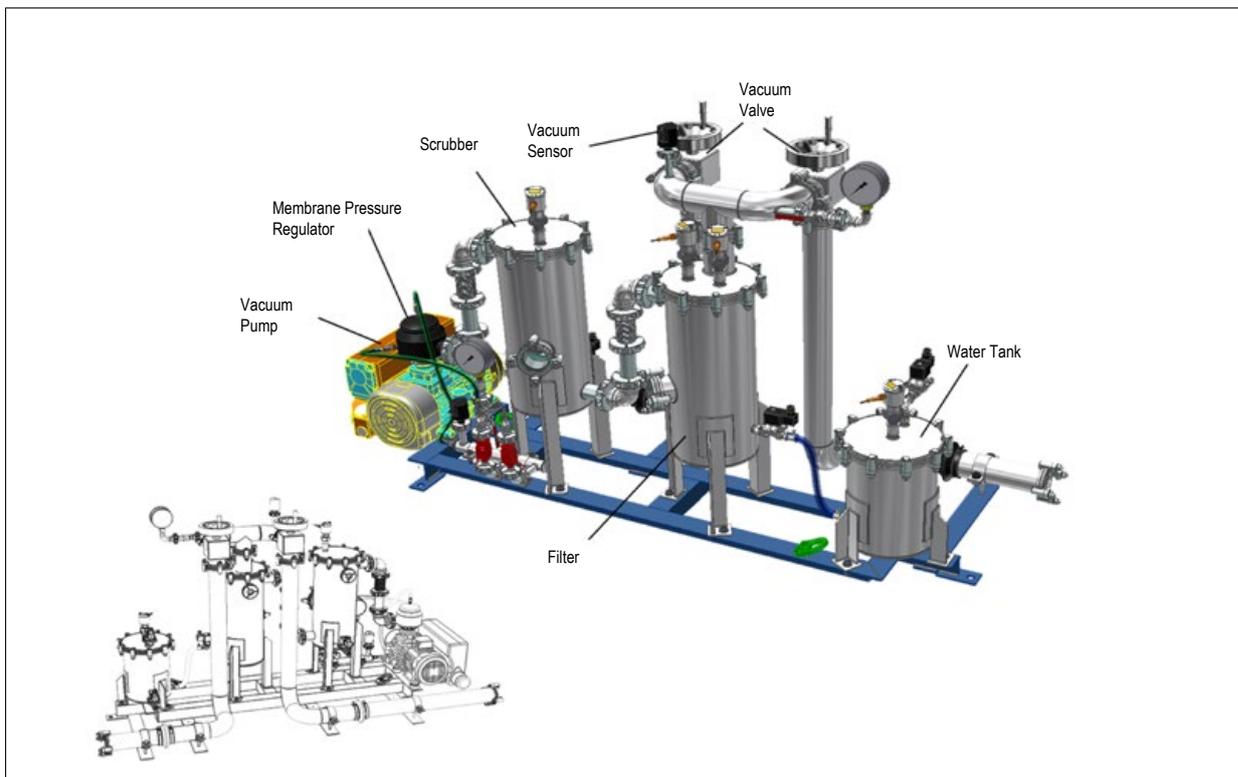


Fig. 6 The vacuum system

that creates a powerful electrostatic field. As a result, nanoparticle is kept in collection pipes and the “carrier atmosphere” is discharged to the vacuum system. It should be mentioned that pressure, flow and temperature of the gas, otherwise called the “carrier atmosphere”, have a major impact on the process. However, when the “carrier atmosphere” enters the electrostatic precipitators, there are two more additional factors decisive for obtaining the nanoparticle - the voltage that creates the electrostatic field and the geometry of the reaction pipes. In order to collect nanoparticle during production, each of the electrostatic precipitators is equipped with a special hammer system, which enables the “pouring” of the nanoparticle into containers under each EGR.

The last element of the equipment is the vacuum system, shown in Fig. 6. The vacuum system, in addition to the pump and vacuum valves, consists of two filters with a design that ensures the completely safe operation of the vacuum pump without the need for frequent oil replacement, as well as ensuring that the pump itself is not damaged by the remnants of nanoparticle in the “carrier atmosphere”.

Using water as a natural filter, the system operates automatically and pollutants are removed thanks to installed sensors and electromagnetic valves coupled into the automatic control system.

**Conclusions and outlook**

Initial tests conducted in the last quarter of 2012 have proven the suitability of this design in terms of safety, control systems operation and such parameters as heating rate, maximum temperature, vacuum level in the system and gas flow. Previous conclusions clearly show full readiness for technological trials, which will be described along with the theoretical background in the second part of this paper.

**Acknowledgements**

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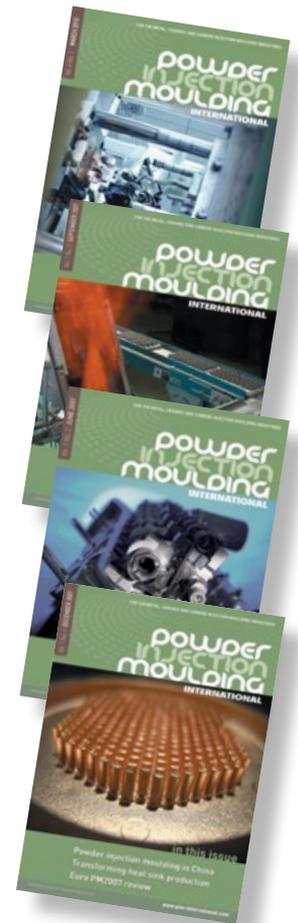
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