Vol. 4 No. 2 JUNE 2010

FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

powder injection moulding INTERNATIONAL

in this issue

Company visits: Parmatech, AMT
Variants on the PIM process
PIM biomaterials for implants

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POWDER INJECTION MOULDING INTERNATIONAL

For the metal, ceramic and carbide injection moulding industries

A look back to the early days of our technology

PIM International's recent visit to Parmatech presented a good opportunity to look back to the early days of our technology (page 21). As the first commercial producer of MIM parts, the archives relating to Parmatech not only show us how challenging the early days were in terms of both technology and marketing, but also just how far the industry has come in nearly 40 years.

If you are not familiar with the early days of MIM, we hope that our report will give you an insight into just how the technology came into existence in California during the early 1970's. Our awards time-line, using Parmatech's award winning components from 1976 to the present day, effectively illustrates the steady expansion of the industry. From niche military and aerospace applications through to business machine parts and beyond, MIM has developed rapidly into the high growth global manufacturing technology that it is today.

As the industry has developed, however, various stages of the process have been adapted to suit specific applications and provide solutions to a wide variety of challenges. These 'derivatives' are explored in our feature 'Alternatives to PIM: variants on almost the same theme' (page 31).

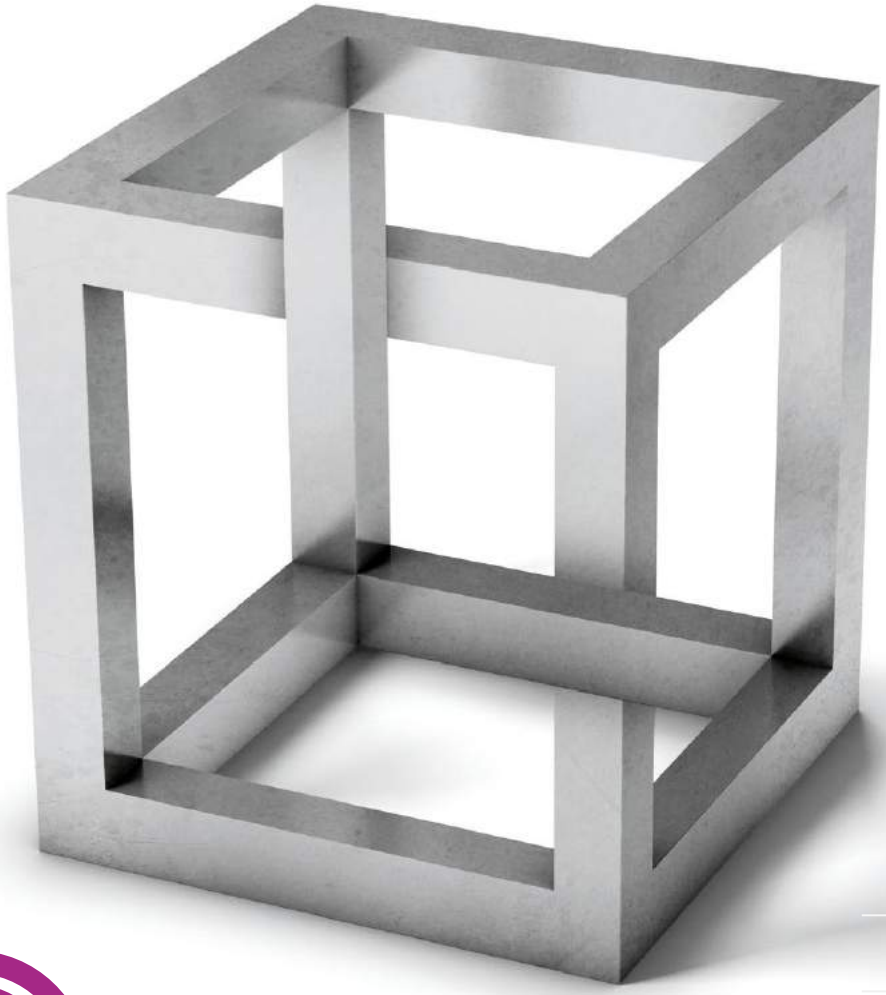
We also report on a visit to Singapore's Advanced Materials Technologies (AMT), one of Asia's longest established PIM producers (page 41). AMT has, like Parmatech, taken full advantage of international awards competitions to enhance its reputation with an impressive record of honours.

On a more general note, the PIM industry still appears to be in a positive position. A very successful MIM2010 conference in Long Beach, USA, attracted an international audience and many metal powder producers indicated that they were struggling to keep up with a surge in demand. Attention now turns to the industry's biggest event of 2010, the PM World Conference in Florence. We look forward to seeing you there.

Nick Williams
Managing Director and Editor

Cover image
Ceramic injection moulded parts for fuel cell applications. Courtesy Advanced Materials Technologies Pte Ltd, Singapore

Do you really know **the borders of possibility ?**



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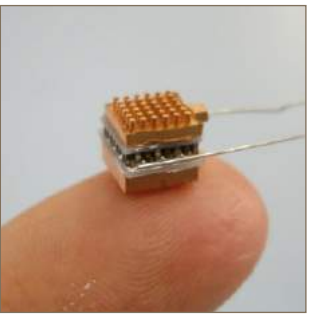

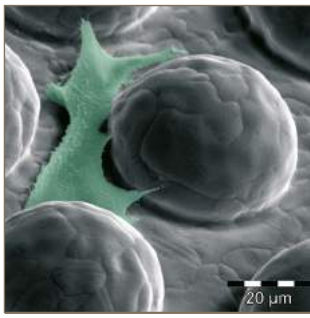

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In this issue

21 **Parmatech: The MIM industry's first commercial producer, and still going strong**
In the not too distant future Parmatech will be celebrating 40 years of powder injection moulding, a fact that may come as a surprise to many who believed that PIM was a far more recent technological innovation. We report on a recent visit to the company, which is widely accepted to be the first commercial producer of MIM parts.

31 **Alternatives to PIM: variants on almost the same theme**
Although there is much focus on the core technology of PIM, many variants have emerged over the years with shifts in binders, shaping, or other processing steps. Prof. Randall German introduces several variants to illustrate how core principles, similar powders and binders, and other parallel aspects spill over to a wide range of alternative approaches.

41 **Singapore's Advanced Materials Technologies celebrates 20 years of MIM excellence**
We report on a recent visit to Advanced Materials Technologies (AMT), one of Asia's longest established MIM producers which this year celebrates its 20th anniversary.

49 **The processing of biomaterials for implant applications by PIM**
PIM is an established process for the manufacture of medical products from biocompatible materials. Philipp Imgrund presents current work that includes degradable PIM implants, surface structured PIM products for implant applications, and implants with controlled porosity.

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Richard W. Bono from The Modal Shop Inc. explains how the 'Resonant Acoustic Method' of Non-Destructive Testing (RAM NDT) has been adapted to provide MIM producers with an effective in-line system to check 100% of parts for cracks and other flaws.

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Industry News

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

Adamou uses CIM route to produce valve seat

Adamou Sàrl of Etoy, Switzerland, is using ceramic injection moulding (CIM) to produce a sintered valve seat from 99.9% alumina powder. The company states the ceramic seat is used in conjunction with a ceramic ball to open and close a valve in order to achieve a high precision flow rate for homogeneous liquid mixing in a chemically aggressive environment. Different valve seat dimensions are produced depending on the flow rate required and customer specification.

A CIM valve seat produced by Adamou Sàrl

Key to the success of this application is producing the CIM valve seat to dimensional tolerances in the micron range, thus avoiding any leakage after the valve is closed by the use of the ceramic ball, states Adamou.

In addition to alumina CIM, the company also produces CIM parts from black or coloured zirconia, and recently launched a new website.

www.adamou.ch

Nissei sees growth in China and plans to target MIM

Nissei Plastic Industrial Co. Ltd., the injection moulding machine producer headquartered in Nagano, Japan, has reported that it expects to increase production to 900 machines a year at its Chinese manufacturing plant in Taicang, Jiangsu Province, by 2013.

Nissei has also indicated that it intends to allocate more resources to higher-tech areas such as MIM. The company states that it is increasing its research on new technologies to maintain global competitiveness, including developing the world's first system for moulding some grades of heat-resistant bioplastics.

www.nisseijushi.co.jp

New titanium powder plant to be on stream by year end

International Titanium Powder LLC (ITP) of Woodbridge, Illinois, USA, is expected to begin producing commercial quantities of low cost pure and alloyed Ti powders at its new plant in Ottawa, Illinois, later this year. The plant is expected to reach its capacity of 4 million lbs (2000 short tons) of Ti powder during 2011.

ITP has been commercialising its patented Armstrong Process for low cost titanium powder for a number of years at its R&D facility and pilot plant. The company became a wholly owned subsidiary of Cristal Global, headquartered in Jeddah, Saudi Arabia, in 2008. Since then Cristal Global has expanded the ITP team and is committed to bringing the new titanium powder on stream.

Cristal Global is said to be the world's second largest producer of titanium dioxide (TiO_2) with sales in the region of $2billion in 2008. "Ti and Ti alloy powders from the Armstrong process have been proven to have properties that will provide the foundation for an expanding powder-based titanium product market. We look forward to working with our suppliers, customers and titanium metal consumers in programmes to accelerate use in existing and new markets", said Robert Daniels, Vice President – Titanium Metals for Cristal US.

www.itponline.com

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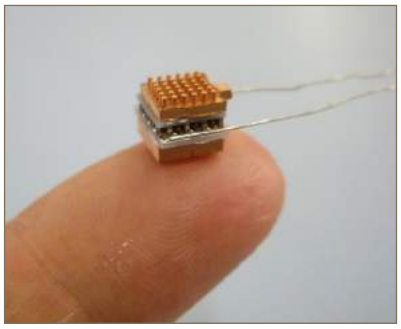
MicroMIM Copper Diamond composite heatsink promoted at CeBIT

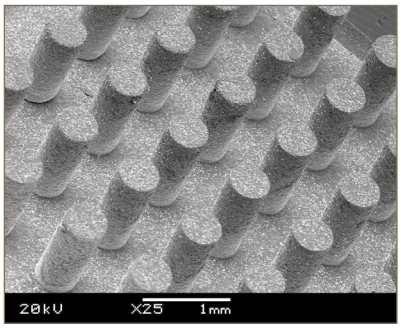
A copper and diamond composite 'Super Heat Sink' produced by micro metal injection moulding (μMIM) was promoted by a Japanese consortium at CeBIT, the world's foremost tradeshow for the digital industry in Hannover earlier this year. Developed and manufactured by Taisei Kogyo Co. Ltd. of Osaka, Japan, in collaboration with Osaka Prefectural College, the device is aimed at supercomputer applications, along with other demanding electronic devices. The heat sink is made of a copper diamond composite with a micro-structured surface to further improve thermal conductivity.

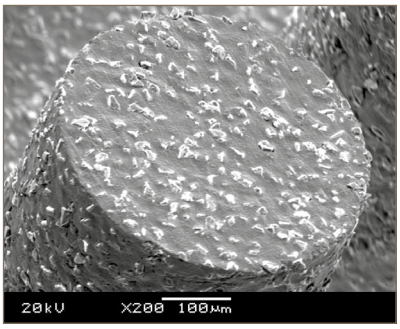
This technology, combining advanced materials with metal injection moulding processing, offers thermal conductivity of 580 W/mK, 1.4 times that of copper. Each heatsink pillar is 1500 μm high and 500 μm wide. "We can change the coefficient of thermal expansion (CTE) freely by adjusting the proportions of copper and diamond.", stated Dr Kazuaki Nishiyabu, formerly of Osaka Prefectural College.

Dr Nishiyabu recently took up a new position as Associate Professor in the Department of Mechanical Engineering at Kinki University, Osaka. His work on various aspects of MIM and μMIM in conjunction with Taisei Kogyo Co. Ltd. is expected to continue.

nishiyabu@mech.kindai.ac.jp







Left, the MIM Copper Diamond composite heatsink for supercomputer applications. Centre and right, SEM images showing details of the pillar designs and surface structure

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Spheric Technologies delivers continuous production microwave furnace

Spheric Technologies, Inc., the provider of industrial microwave equipment and technology, has completed the installation of a high-temperature (1600°C) continuous production microwave furnace at the NanoMaterials Innovation Center (NMIC), a wholly owned subsidiary of Alfred Technology Resources, Inc. (ATRI) in Alfred, New York, USA. ATRI also operates the Ceramics Corridor Innovation Centers hi-tech business incubation program.

Joseph Hines, Chairman and CEO of Spheric Technologies, stated, "Installation of this unit at the NanoMaterials Innovation Center provides valuable exposure for our industrial microwave furnace technology. This is the first large computer-controlled continuous high temperature production microwave furnace in the United States. It will be used for research and process development by commercial clients of the NanoMaterials Innovation Center, faculty and students of Alfred University and other educational institutions in New York State."

Alfred University has also established a Class 10,000 clean room environment, microwave sintering lab and manufacturing equipment for making multi-layer ceramics at the same location. The project was funded by a $1.8 million grant made to the Center for Advanced Ceramic Technology by the New York State Office of Science, Technology and Academic Research (NYSTAR).

www.SphericTech.com

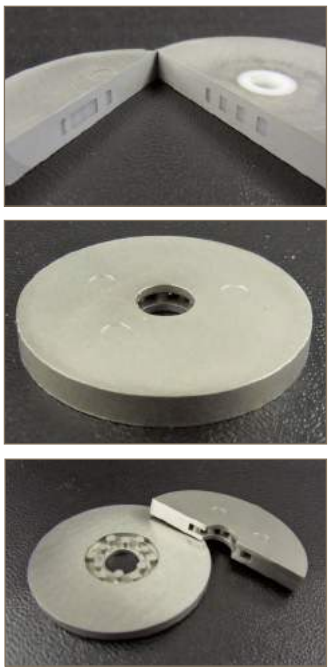
Megamet demonstrates hollow MIM components using 'Dissolving Core Technology'

Megamet Solid Metals, Inc., Earth City, Missouri, USA, presented their development of 'Dissolving Core Technology' for the processing of complex hollow MIM parts at the MIM2010 Conference, Long Beach, March 29-31. The process, the company claims, offers the ability to produce geometries that had previously been impossible using traditional MIM processing alone.

The process relies on the use of a dissolvable Acetal core to produce the required internal geometry. This core can then be removed during catalytic debinding, as Acetal is a primary binder ingredient in the MIM feedstock used.

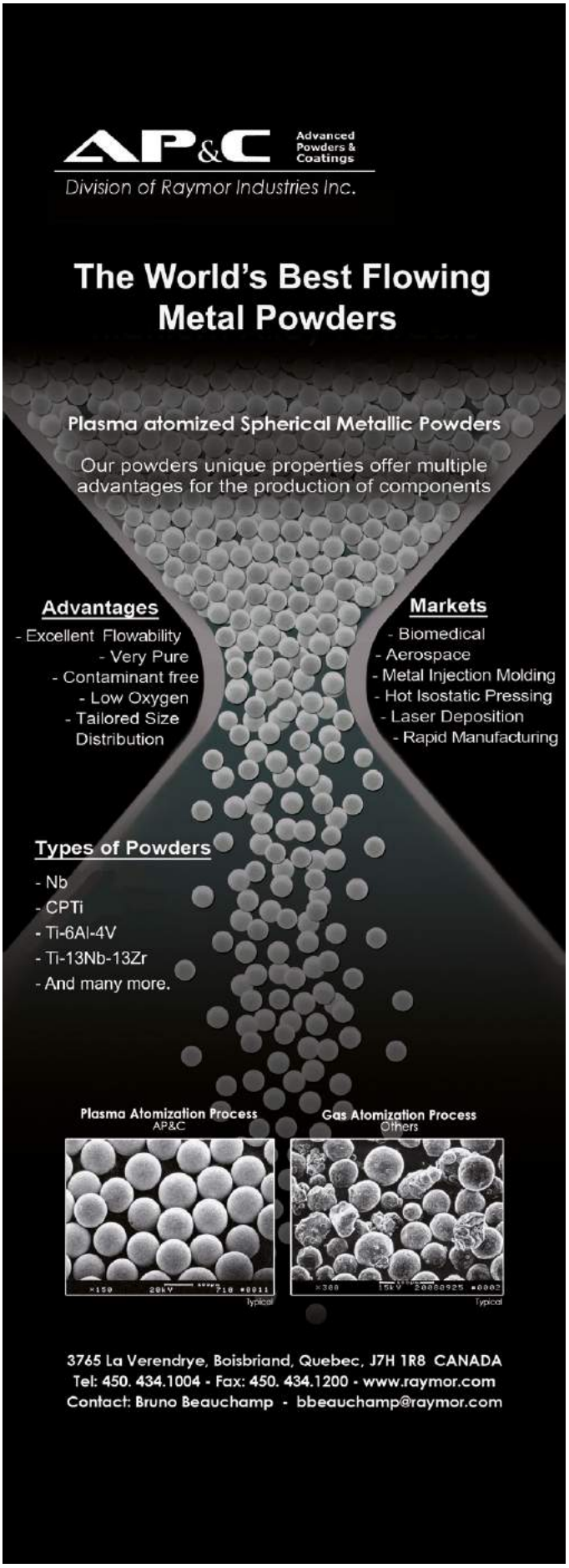
Megamet successfully produced demonstration parts using this process, with an internal geometry that would be impossible to produce using the slides or lifters available in standard MIM processing. Sintering was performed using standard furnace programming with no additional considerations for the internal cavity. The finished parts were then cross sectioned to inspect for the quality of the mould fill and for any distortion of the core, however no problems were observed.

It is expected that this process could be used with other feedstock and binder systems, however other systems have not been tested at this time. The company indicated that more research was needed into the size limitations of the dissolving core, thickness limitations during debinding, dimensional behaviour and consistency, and the condition of the surface of the as sintered MIM component adjacent to the acetal core.



Top, As moulded component cut open to show the Acetal core; Centre, A sintered component with core removed; Bottom, An exposed view showing the internal cavities (Courtesy Megamet Solid Metals, Inc.)

In an unrelated development, BASF SE, producer of the Catamold feedstock system, proposed the use of a similar process to enable the production of MIM turbine wheels for automotive turbocharger systems. In this instance, a dissolvable core was proposed as a route to reduce section thickness in MIM turbine wheels, reducing the possibility of cracks and distortion during debinding and sintering - see 'Turbocharger technology presents new opportunities for metal injection moulding' *PIM International, Vol. 3 No. 2 June 2009, pages 37-42* .



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Successful MIM2010 conference draws an international audience

The Metal Powder Industry Federation's MIM2010 Conference took place from 29-31 March, in Long Beach, California, USA. The three day event succeeded in attracting a diverse audience, bringing a distinctly international feel to what has traditionally been an event dominated by the North American PIM community.

The conference sessions were preceded by a well attended one-day "PIM Tutorial" presented by Prof. Randall German, San Diego State University. This provided a comprehensive foundation on all aspects of PIM technology, from materials through to processing, markets and financial considerations.

Within the main conference sessions, developments in the metal injection moulding of titanium (Ti-MIM) received much attention, with a number of high quality presentations from Asia and Europe as well as Canada and USA. There was general agreement that whilst the processing of Ti-MIM remains challenging, technical feasibility is no longer an issue and powder availability remains a greater challenge, along with the development of a greater market for Ti-MIM products. German estimated the current value of Ti-MIM part sales to be in the region of $8 million, shared between approximately 17 MIM operations.

The keynote luncheon featured a presentation by Lars Gehlback, VP Global Supply Chain, at Sybron Dental Specialties. Gehlback gave a thorough review of the impact of MIM in the orthodontics industry, citing its advantages over machining and investment casting, and highlighting current trends including the increased use of translucent ceramic injection moulded orthodontic components that offer significant aesthetic improvements for the consumer.

One of the most successful aspects of this conference series, thanks in part to its focused subject area and in part to its size and 'residential' type venues, is in the unique opportunity that it offers for networking with fellow industry professionals. Lively breakfasts, coffee breaks, lunches and receptions all offered delegates informal opportunities to meet with both colleagues and almost all the major international equipment and materials suppliers.

Exhibitors featured in the table-top networking reception included the following organisations:

Ametek, Inc.	Orton Ceramic Foundation
AT&M Powdered Metal	Plansee USA
Atect Corporation	Polymer-Chemie
Automated Cells & Equipment	Pressure Technology
AVS	Proton Energy Systems
BASF	Ryer
Carpenter Powder Products	Sandvik Osprey
Centorr Vacuum Industries	SCM Metal Products
CM Furnaces	Smart Metal Powders
Dynamic Group	Sunrock Ceramics
Elnik Systems	Yuelong Superfine Metal
Epson Atmix Corp.	Zircar Cermaics
Goceram	

The next conference in this series, MIM2011, will return to Orlando, Florida, 14-16 March 2011.

Top, the MIM2010 PIM Tutorial; lower left, MIM2010 succeeded in attracting an international audience of part producers, suppliers and researchers; lower right, Lars Gehlback, VP Global Supply Chain, Sybron Dental Specialties, presents the luncheon keynote

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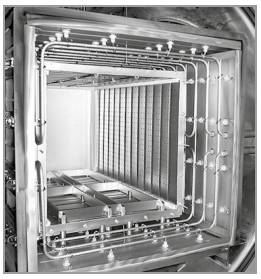

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Expansion at Yillik Precision Carbides reflects growth in demand for WC-MIM products

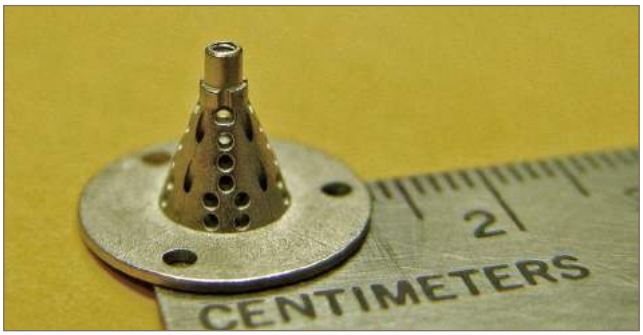
Yillik Precision Carbides announced earlier this year that it was moving into a larger 13,000 ft^2 new manufacturing facility in Ontario, California, USA. The company states that this move will allow for a more streamlined process workflow that is better able to support the growth in new products being developed by its 40 employees.

The company specialises in miniature carbide products with inside diameters as small as 0.010" (.25mm) and outside diameters of 0.040" (1 mm), with exacting tolerances for roundness and concentricity. The company reports that products of this type continue to fuel the recent growth into demanding applications for the oil field equipment, medical and sporting goods industries.

Yillik was acquired in 2007 by PSM Industries, "The synergy of adding a tungsten carbide processor to our family of PM technologies has fully expanded our manufacturing capabilities," stated Craig Paullin, President, PSM Industries. "The fact that Yillik presses and sinters their own carbide gives them exceptional control over the entire process".

An example of the synergy in the acquisition can be seen with the collaboration of Yillik with the PolyAlloys Injected Metals division of PSM Industries to introduce tungsten carbide net shapes with extreme shape complexity to the marketplace using the MIM process. "These two divisions are working together to produce geometries and tolerances previously unimaginable by conventional moulding techniques used in tungsten carbide production", stated Larry Totzke, Sales Manager at PSM Industries and PolyAlloys Injected Metals.

www.psmindustries.com



Top) A MIM tungsten carbide cone showing the complexity that can be achieved; Below) MIM tungsten carbide demonstration pieces. Both examples were moulded by PolyAlloys and sintered at Yillik Precision Carbides.

MIM furnace manufacturer Elino is acquired by PLC Holding

In March 2010, Elino Industrie Ofenbau, based in Düren, Germany was taken over by PLC Holding, a French industrial equipment company that also owns the Elmetherm and Wistra brands.

Elmetherm manufactures industrial kilns and furnaces in the low-temperature range from 0 to 500°C, whilst Wistra produces high temperature furnaces in the range from 1200 to 1850°C, commonly used to process ceramic materials. Elino fits into PLC Holding's portfolio well, covering the range between the two and specialising in the use of protective atmospheres such as nitrogen, argon and hydrogen. In the short term, Wistra will relocate from Cologne to Düren, where a 6000m² production area and around 2000m² of office space are available.

For Elino's PIM related activities this opens up new possibilities. Thanks to Wistra's 80 years of experience in sintering of technical ceramics (spark plugs, diesel particle filters, electronic components and CIM parts) a complete program for debinding and sintering equipment for both metal and ceramic injection moulding can now be offered. The first results of this new alliance will be shown during the PM World conference and exhibition later this year in Florence, Italy.

Philippe Blandinières, head of the holding company told *PIM International*, "We want to strengthen our presence on the German market , conversely Elino can use Elmetherm's and Wistra's sales channels and quickly build up a presence in the markets of Eastern Europe, India and especially the USA"

For over 70 years Elino has been manufacturing heat treatment and gas equipment. The company built it's first continuous sintering furnaces in the early 1950's and five years ago developed the world's largest continuous catalytic debinding furnace.

www.elino.de

Centorr improves its furnace line

Centorr Vacuum Industries, Nashua, USA, has reengineered its Sinterbar line of high pressure sintering furnaces. The redesign was overseen by a task force combining Centorr's own engineers and those from Fuad Barbar. The team not only expanded temperature and pressure capabilities, but also improved thermal performance, safety, durability and cost-effectiveness. The new Sinterbar line has temperature capabilities of 1650°C or 2200°C, with pressures of 60 to 100 bar (900 to 1500psig). Standard sizes of 1, 4, 10, and 12 cu ft (28, 113, 283, and 340 litres) are available with optional debinding capabilities.

CVI Sinterbar furnaces are used to densify hardmetals and ceramic powder parts. After a part has been debound and sintered in the furnace it is further densified by introducing an inert gas static back pressure of up to 100 bar (1500 psig) at high temperature, removing the majority of the remaining porosity.

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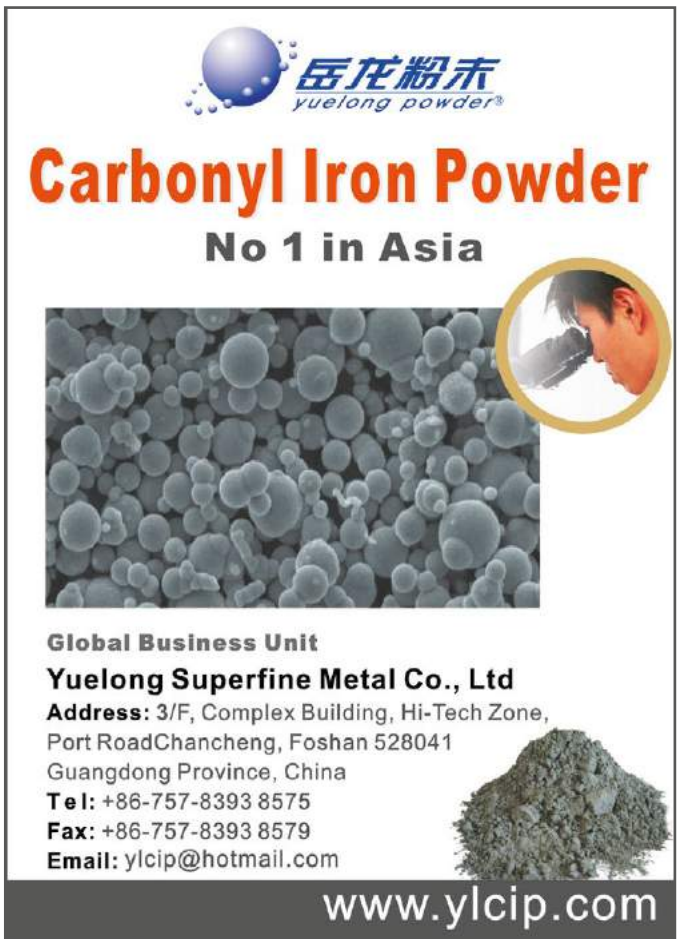
CIM Expert Group works to increase technology awareness

The Ceramic Injection Moulding Expert Group (Expertenkreis Keramikspritzguss) was founded during the 2008 Hanover Fair and brings together German and European companies, as well as research institutions, that use ceramic injection moulding and who have integrated the injection moulding, debinding and sintering process chain in their own production.

Like the German speaking MIM Expert Group, the CIM Expert Group aims to create a separate 'ceramic injection moulding brand' and offer increased benefits to end-users. This, it is claimed, will result in a strengthening of the global position of the CIM process in the areas of materials, technologies and markets. Other objectives of the group include the development of expertise and a combined approach to the resolution of complex technical and marketing-related challenges.

The CIM Expert Group has quickly grown since its launch and is now an important network of businesses and institutions with the aim of promoting innovative development throughout the CIM process chain. The group now has 17 members, including new members Oechsler AG, Ansbach, Germany and Philips Lighting B.V, Eindhoven, The Netherlands.

Members met at Arburg's headquarters in Lossburg, Germany, for their seventh meeting on 15 April 2010.



Members of the Ceramic Injection Moulding Expert Group met at Arburg in April

In addition to reports from the three working groups, Networking, Technology Development and Technology Marketing, participants also considered the exhibitions scheduled for 2010 and the integration of new members.

The Technology Development working group reported on the current status of filling studies, material characterisation, and process simulations for the development of test tools from which bars of varying geometries and bores can be produced. This sophisticated tool, it was reported, is an ideal solution for testing joint line and open jet patterns. Of particular interest in this context is the extent to which real conditions can be reproduced in a process simulation.

Practical trials were initially carried out and then compared with subsequent simulations. It was evident that there was still plenty of room for improvement in some aspects of the simulation. For example, the open jet formation in the simulation still cannot be replicated.

The Technology Development working group met again in May to further discuss the results of the trials with the test tool, as well as progress of the commissioned AIF ProCIM project, a non-destructive control system for early fault detection in ceramic injection moulded parts, which was scheduled to be approved this summer.

The Technology Marketing working group reported its work on the redevelopment of the group's website www.keramikspritzguss.eu and the initiation of a series of seminars at various universities. All of these activities, it is hoped, should contribute to making the injection moulding of ceramic materials more interesting to different target groups. The seminars are to begin at the College of Technology in Koblenz, Hörgrenzhausen, Germany, where the processes, options and opportunities offered by the processing technology will be presented.

The CIM Expert Group also indicated that its participation at Ceramitec 2009, held in Munich in September, was very positive. The group also had a showcase on the TASK (Technologie-Agentur Struktur-Keramik GmbH, Dresden) stand at this year's Hanover Fair.

The next members' meeting is scheduled for September 2010 and will be hosted by the Fraunhofer Institute for Ceramic Technology (IKTS) in Dresden.

www.keramikspritzguss.eu

Modelling properties of low viscosity feedstock for low pressure PIM

Producing ceramic and metal parts using low pressure powder injection moulding (LPIM) is said to offer an economic alternative to high pressure PIM for small series production. It is also said to allow the use of softer tooling helping to produce small complex shapes or prototypes as well as larger, higher mass parts with thicker cross sections.

Rupal Mehta, writing in the April 2010 issue of *Materials World Magazine*, reports on work being done at the Ecole Polytechnique de Montreal in Canada in partnership with a PIM producer, Maetta Sciences based in Boucherville, Quebec, to establish a model for the optimal rheology of feedstock for LPIM.

The research team is led by Francis Lapointe who stated that there has been no study of the rheology of LPIM feedstock for metal alloys. The research therefore focused on solid loading of feedstock using water and gas atomised stainless steel 17-4PH powders having a 10 micron particle size. Lapointe stated that further work is proposed using different particle sizes, adding the effect of granulometry as another sub-model. Multiplying the contributions of the individual sub-models will help to generate a global model to help manufacturers using LPIM to analyse the feedstock's rheological behaviour by identifying the optimum shear rate, solid loading and temperature.

Maetta Sciences recently received a capital injection of $1.6 million from its shareholders to help finance its metal injection moulding operation. The company was initially involved in the development of PIM for aerospace applications but has seen accelerated expansion in the medical sector especially in Europe and the USA.

Yvan Beaudoin, President and CEO of Maetta stated that interest in the company's PIM technology in the medical sector is phenomenal mainly because of the substantial cost reduction and new design possibilities it brings.

"We are working with leading European and US orthopaedic companies on key projects. The new financing will allow for the rapid growth required to meet our clients' rising demand, and the completion of products aimed at the medical and aerospace sectors. We also plan to hire personnel to meet the increase in sales", stated Beaudoin.

www.maetta.ca

Malaysian PM Symposium

The Advanced Materials Research Centre (AMREC), SIRIM Berhad in Kulin, Malaysia, is organising the 4th Malaysian Powder Metallurgy Symposium in conjunction with the Malaysian Metallurgical Conference to be held in Penang, November 22-23, 2010.

Powder injection moulding is expected to be included among the topics of the PM symposium and anyone interested in submitting a technical paper should contact Dr Mohd Asri Selamat at the Symposium Secretariat.

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Korean Powder Metallurgy Institute Spring Conference

The Korean Powder Metallurgy Institute's (KPMI) Spring Conference took place in Andong National University, Andong, Korea from April 8-9 2010. The event was attended by more than 230 participants and the conference programme included 132 scientific and technical papers.

Professor Randall German, San Diego State University, USA, gave the Conference Plenary Lecture, in which he emphasised the technical and economic barriers and opportunities for large scale metal injection moulding production.

As reported in the March 2010 issue of *Powder Injection Moulding International*, Korea has a thriving PIM industry that dates back to the mid-1980's and strong growth is anticipated in coming years, thanks to an increasing interest in PIM from Korean manufacturing giants such as Samsung Electronics, LG Electronics and the Hyundai-Kia Motor Company.

The KPMI was founded in 1993 and the association now has around 90 members, including industrial producers, research centres and academia. The association's journal is published six times per year and it also organises a number of seminars and meetings in each year, in addition to its annual conference.

German receives Honorary Membership of KPMI

Professor German was also awarded Honorary Membership of the Korean Powder Metallurgy Institute (KPMI) during a ceremony and dinner with former Presidents of the KPMI.

The KPMI further honoured Professor German by arranging a special book signing ceremony to coincide with the release of the Korean translation of his popular textbook *Powder Metallurgy and Particulate Materials Processing*.

Professor German also spoke at the Han Yang University Ansan campus, POSTECH (Pohang University of Science and Technology), Korean Institute of Materials Science in Changwon, and visited POSCO Steel in Pohang, an iron producer entering into powder production, PIM Korea in Gyoungasn, a producer of MIM components including turbocharger vanes, and ANC, a fabricator of molding machines used for microminiature metallic and ceramic devices.

President of the Korean Powder Metallurgy Institute Dr. Kim (left) and former President Dr. Lee (right)

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Measuring pressure peaks and temperature in PIM

Kistler AG of Winterthur, Switzerland, is producing a melt pressure measuring chain Type 4021A which can be used to measure melt pressure in injection moulding machines and hot runner systems. The Kistler measuring chain comprises a factory-calibrated sensor, cable and amplifier. The sensor is constructed of a stable, high-temperature piezoresistive silicon sensing element which can be mounted directly in the flow of material. This is said to have the advantage of being able to measure process parameters determining product quality.

The sensor is offered in multiple lengths and angular configurations, in pressure measurement ranges of 0 to 3000 bar and temperature ranges of 0 to 350°C. It is said to be ideal for use in short-cycle process monitoring environments such as those found in MIM, CIM or hot runner systems. Because the sensor does not contain any transmission medium it is said to be ideal for use in injection moulding of medical components.

www.kistler.com

New melt pressure measuring chain produced by Kistler

Chinese carbonyl iron powder producer Yuelong expands into atomised powders

Established a number of years as Asia's leading producer of carbonyl iron powders (CIP), Yuelong Superfine Metal Co., Ltd, located in Guangdong Province, China, is now offering a new range of atomised metal powders for the MIM and PM sectors. These include stainless steels, iron and pre-alloyed materials.

By the end of April this year the company had achieved the first stage of its ambitious expansion strategy, with the installation of both water and gas atomisation facilities with a reported 3000 t/a capacity. The company told *PIM International* that Stage II of its expansion plan is scheduled to be initiated by the end of 2010, increasing capacity up to 10,000 t/a.

Mr. Shangkui Li, President of Yuelong Superfine Metal Co., Ltd, explained the motivation behind this major investment, "This project has been planned for three years, and the advantage for Yuelong rests on the clear benefits of combining carbonyl powder production and atomised powder production. With our existing expertise, most of the recyclable materials can be collected and reused to lower the production cost and better protect the environment. A new level of reaction temperature and pressure can also be reached, improving the physical quality of the powders. We are very confident that these new powders will be very well received".

The availability of consistently good quality and cost-effective powders for MIM will have a positive effect on the industry in China, stated the company. It believes that despite a thriving MIM industry in the country, the materials supply situation has been chaotic in the past, with a few foreign suppliers offering high quality powders at a high price, whilst at the other end of the spectrum numerous local 'workshops' sold poor powders for a 'reasonable' rate. It is estimated that there are now around 60 MIM component makers in mainland China, with an annual growth over 30% in terms of turnover for the past three years.

With seven years of production experience in CIP and 3500 t/a capacity, Yuelong has already established itself in the MIM and PM industries, particularly in Asia. The company is, however, also looking to increase its presence on the international powder market, opening a sales office last year in St. Ingbert, Saarland, Germany.

www.yuelongpowder.com / bo.li@yuelongpowder.com

India's PM11 International Conference to include PIM

Westin hotel, Pune, venue for the the PMAI annual conference

The Powder Metallurgy Association of India (PMAI) has announced that its PM11 annual conference and exhibition will take place in Pune, 3-5 February 2011.

The event is expected to include sessions on metal and ceramic injection moulding for the first time, reflecting an increasing interest in the potential for PIM in India.

www.pmai.in

New consultancy specialising in MIM and PM established in Japan

Kato Yoshiyuki, a highly respected member of the Japanese and international Powder Metallurgy (PM) community, recently retired from his role as Director of Epson Atmix Corporation and has established a consultancy office in Yokahama, Japan. The new consultancy will serve all areas of Powder Metallurgy as well as the high growth Metal Injection Moulding (MIM) industry.

Kato Yoshiyuki has established 'Kato Professional Engineer Office', a new consultancy in Japan

After leaving Pacific Metals he was a central figure in the establishment of atomised metal powder producer and MIM parts maker Atmix, a venture supported by Seiko Epson.

"I have enjoyed 35 years in the PM industry", Kato Yoshiyuki told *PIM International*, "and look forward to keeping in contact with the many friends I have made. The Atmix business principles remain at the heart of my business philosophy. I believe that Atmix has made a positive contribution to the MIM industry's development, with its unique combination of powder manufacturing and MIM parts fabrication. I still, of course, enjoy a good relationship with Atmix and look forward to contributing to the industry."

Kato Yoshiyuki has been a valued supporter and a Consulting Editor of *Powder Injection Moulding International* since its launch in 2007 and we wish him every success for his new venture.

E-mail: pmkato@jd5.so-net.ne.jp

PM2010 World Congress programme now available

The European Powder Metallurgy Association (EPMA) has published the Technical Programme for the World PM2010 Congress & Exhibition, taking place in Florence, Italy, 10-14 October. Around 600 technical presentations are included in both oral and poster format, along with a number of special interest seminars.

Powder injection moulding features prominently, with seven sessions devoted to the processing of PIM parts. A special interest seminar will also present "A Global View on Metal Injection Moulding" through case studies from Asia, Europe and North America.

The EPMA's European Metal Injection Moulding Group (EuroMIM) will be meeting in Florence immediately prior to the World Congress, on Sunday 10 October, 15.00 - 16.30. This is an open meeting and anyone interested in attending should contact the EPMA.

www.epma.com/pm2010

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New solutions for analysing particle sizes

Fritsch GmbH of Idar Oberstein, Germany, is a long established supplier of instruments for sample preparation and particle sizing. The company recently introduced its fully automatic laser particle sizer, Analysette 22 MicroTec plus, capable of dry measurement of particle size distribution in the range 0.08 µm (80 nm) up to 2000 µm in sample volumes of less than 1 ccm to 100 ccm in an accelerated air flow.

The Analysette 22 MicroTec laser particle sizer from Fritsch GmbH

The company has also introduced the DynaSizer Analysette 12, for particle size measurements between 1 nm and 6 µm using dynamic light scattering. This enables fast and accurate particle size measurements down to the nano range and in very small sample quantities.

www.fritsch-laser.com

New Z-Sigma blade mixer for Powder Injection Moulding

Winkworth Group, based in Reading, UK, has introduced a new 7 litre Z-Sigma blade 'MZ7' mixer aimed at the MIM market. The MZ7 Sigma mixer is part of a wider range of mixers that vary in capacity from 100ml to 25 litres. The company states that its sigma mixers are used widely in research and production environments across the world for mixing metal powders.

The mixer is manufactured from 316 stainless steel and benefits from very easy cleaning, taking just 5 minutes, ideal for repeat small batches and helping to keep 'mix to new mix' times to a minimum. Mixing characteristics are fully monitored via temperature and torque sensing delivered straight from the mix via standard 4-20mA outputs. A touch-screen man machine interface (MMI) makes control and traceability simple and reliable.

Tim Simpson, Technical Sales Director, stated, "The ability of an unfamiliar technician or operator to strip down & remove the blades for thorough cleaning in just a few minutes is a key benefit of this Winkworth Z- Sigma blade mixer range. Combining this with a data logging facility makes this an essential and versatile addition to any R&D Laboratory or high value low volume production facility."

"The data logging function not only enables the recording of important parameters but also stores the changes in the actual mixing process. This data can then be easily downloaded for subsequent analysis in the form of graphs, trends and statistics and for data management/distribution purposes."

MZ7 Sigma mixer from Winkworth

www.mixer.co.uk

Wittmann Battenfeld back in black

Werner Wittmann, founder of auxiliary and robotics manufacturer Wittmann in Vienna, Austria, which in early 2008 acquired injection moulding machine producer Battenfeld of Kottingbrunn, Austria, stated that he expects both the auxiliary and moulding machine operations to be back into profit in 2010.

The company reported a loss for 2009. Wittmann Battenfeld recently organised a 2-day in-house Competence Day event at which it announced that the company will once again be marketing large tonnage machines (up to 1600 tons).

The company has also revamped its MicroPower micro-moulding machines available in 5 to 15 ton clamp force, and reports considerable interest in its EcoPower all-electric machine range introduced in the second half of 2009. The all-electric range currently includes 180 and 110 ton models with a 55 ton model being introduce in June 2010, and a 240 ton model to follow in the autumn.

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Arburg launches new micro-injection module

The demands on the injection side of the manufacture of miniature and micro parts have been clearly defined for some time. Previously the problem was simply how to achieve and implement these specifications technically. The new micro-injection module from Arburg now offers an efficient solution, combining an 8mm injection screw with a second screw for melting the material.

Precisely manufacturing products in series with shot weights that are often just a few milligrams, corresponding to the weight of a single grain of granulate, is only possible if the dwell time of the melts in the injection unit as well as the level of material shearing can be kept correspondingly low. Moreover, the prepared melts must be perfectly homogeneous, both thermally and mechanically. It must be possible to regulate the cylinder temperature precisely and the process must be easily reproducible.

One problem with extremely low shot weights of less than one gram is, in some cases, the very long material dwell time in the injection unit. Furthermore, the displacement distance of the screw is extremely short because of the small injection volume. An effective solution is the new micro-injection module from Arburg. Unlike other alternatives on the market which use a combination of screw plasticising and piston injection, this operates using two screws which 'share' the preparation, dosing and injection of the material.

Firstly, a servo-electrically driven screw pre-plasticising section, which is installed at 45 degrees to the horizontal injection unit, ensures standard granulates are prepared under ideal conditions. In terms of screw channel depths, the plasticising screw used is similar in design to a conventional three-zone screw. The molten material is then transported from the pre-plasticising stage to the injection unit. The injection screw is used purely for transport purposes. It has a diameter of only eight millimetres, is fitted with a non-return valve and operates according to the screw/piston principle. This permits the smallest shot weights to be achieved with great precision and the required travel distance. At the same time, the perfect interplay of screw pre-plasticising and injection ensures excellent processing that is gentle on the material. The melt is continuously fed from the material inlet to the tip of the injection screw. This ensures that the First-In-First-Out principle is fully observed.

Fig. 1 The injection screw has a diameter of only eight millimetres and is purely a conveyor screw (photo courtesy Arburg)

Constant injection conditions through dosage control

A prerequisite for continuous high part quality is a homogenous melt feed. In order to achieve this, the pressure at the transfer point between the pre-plasticising screw and the injection screw is monitored and regulated. For this purpose, appropriate nominal pressure and maximum permissible screw circumferential speed (limitation) values for the pre-plasticising screw are entered via the Selogica control system. If the actual pressure deviates from the nominal pressure, this deviation is compensated by adjusting the rotational speed within specified limits.

A homogeneously prepared, newly dosed melt is always available for every shot. This keeps the material dwell times correspondingly short, preventing thermal damage to the material. The result is high processing quality. An additional advantage of the design with two screws is the leak-tightness of the system.

Arburg claims that it has also

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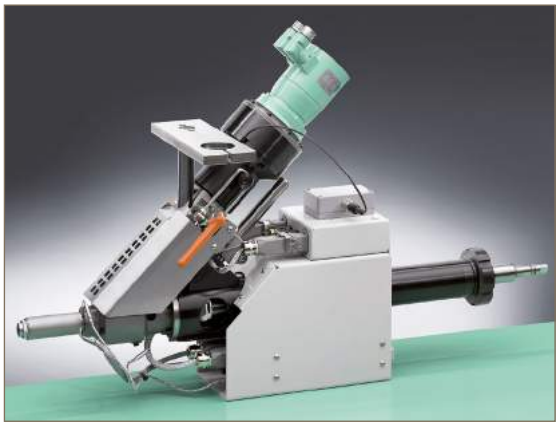

Fig. 2 The new micro-injection module from Arburg combines an 8 mm injection screw with a second screw for melting the material (photo courtesy Arburg)

Fig. 3 The new micro-injection module on an electric Allrounder 270 (photo courtesy Arburg)

succeeded in fully maintaining the modularity of its components with this new development. The micro-injection module is designed specifically for use on electric Allrounder A machines with a size 70 injection unit. Its enclosed construction means that it can be changed rapidly and then used on various machines, like any other Arburg cylinder module. This also means that the range of the machine's applications is not limited to micro-injection moulding. Consequently, other larger cylinder modules can also be used on this machine when a product change is required.

Fulfilling the crucial requirements for manufacturing micro components

With its new micro-injection module, Arburg has fulfilled all the crucial requirements for manufacturing miniature and micro components. It is particularly important, however, that the entire range of plastics can be used in production without forfeiting anything in terms of precision or part quality.

Compelling advantages of this technology are the homogeneous preparation of the melt with short dwell times of the plastics used, process reliability as well as working by the first-in first-out principle. These can be achieved through the "combined" use of two full-value screw systems for pre-plasticising and injection.

www.arburg.com

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Parmatech: The MIM industry's first commercial producer, and still going strong

In the not too distant future Parmatech will be celebrating 40 years of powder injection moulding, a fact that may come as a surprise to many who believed that PIM was a far more recent technological innovation. Nick Williams, Editor of *Powder Injection Moulding International*, recently visited the company which is widely accepted to be the first commercial producer of MIM parts, now located in the picturesque surrounds of California's Sonoma Valley.

Parmatech Corporation's story goes right back to the very first days of powder injection moulding (PIM). With its name created from the words PARticulate MAterials TECHnology, Parmatech was founded in San Rafael, California, in 1973 by four entrepreneurs, Karl Zueger, Peter Roth, Ray Millet and Ray Wiech, specifically to develop and commercialise PIM [1]. Nearly 40 years after the first trial parts were processed the company still remains a key member of North America's PIM community and is one of the most frequently honoured recipients of the Metal Powder Industry Federation's (MPIF) industry awards for product innovation.

The birth of a new technology

The story of Parmatech's early history is, in essence, the story of MIM. Prior to Parmatech's formation, Karl Zueger had established a small company in 1968 in San Rafael, California, called Small Precision Tools to produce tungsten carbide tools used to bond fine wires (0.001 inch in diameter) to printed circuit boards. It was during this time that Zueger met Ray Wiech. Wiech was investigating ceramic injection moulding at Cer Plas, a small company based in Huntington Beach, with his business partner Ray Millet [2]. Zueger saw an opportunity for Wiech's process to be used for the production of ceramic capillaries for the booming semiconductor industry and the decision to jointly develop the technology through a new company, Parmatech, was taken.

'The story of Parmatech's early history is, in essence, the story of MIM'

The efforts resulted in the development of a feedstock system that could be processed using a solvent based debind step followed by a second thermal debinding step. An initial patent application was first filed in June 1972, with a continuation of application filed in 1974. However, it was not until 1980 that the first MIM

Fig. 1 Parmatech's main manufacturing and development facility in Petaluma, Sonoma County, California

Fig. 2 View of a line of Arburg injection moulding machines with robotic handling systems

Fig. 3 Japanese Nissei injection moulding machines. In the foreground trays of green parts stacked prior to solvent debinding

patent was finally granted. The patent was awarded to Wiech, with Parmatech given exclusive manufacturing and licensing rights [1].

The final patent abstract in US Patent 4,197,118 describes the process as "a method of binder removal from a green body before sintering or the like wherein the green body is initially heated to a temperature above the flow point of the binder to liquify the binder and, at this elevated temperature, surrounded by a solvent for said binder in the vapor phase and at a temperature slightly above the melting point of the binder. The solvent vapor enters the green body slowly and dissolves the binder therein so that excessive stresses are not provided within the body due to binder expansion until binder-solvent substantially ceases to exude from the body. The body is then placed in a bath of the solvent, the solvent being maintained at a temperature above the flow point of the binder to remove remaining binder from the body." Pre-sintering in hydrogen to around 1150°C followed, then transfer to a vacuum furnace and sintering at above 1300°C.

'The turning point came in 1979 when Parmatech won two out of the five prizes awarded that year by the MPIF'

In 1981 the original founders of the company separated. Ray Millet took a license to produce gun parts and established Millet Sights in Huntington Beach, Peter Roth returned to Switzerland with a general license and established Moldinject, and Wiech, who died in May 1989, established Witec in San Diego under a cross-license agreement with Parmatech. Zueger remained as President and sole owner of Parmatech [1].

Progressing from ceramic to metal

The early days of Parmatech were characterised by an R&D oriented approach to business, with a "can we..?" attitude that resulted in a great deal of experimental work. Zueger stated in an interview in 1989 that, "the first seven years were largely spent in developing components by the injection moulding of ceramics followed by a gradual transition to metals" [1].

Whilst the initial driver for the development of the technology was for ceramic capillary tubes used to manufacture integrated circuits, some other noteworthy early applications for MIM were for IBM, where the technology found a niche in typewriter and dot matrix printer components. New markets soon followed, from disk drive components to parts for firearms.

One of the early challenges for Parmatech was the availability of suitable powder, with an article at the time stating that, "the process is at present limited by the ability to obtain metal powders of the right size, chemistry and shape. In some cases the company finds itself producing fine powers by unconventional methods" [3].

The turning point came in 1979 when Parmatech won two out of the five prizes awarded that year by the MPIF. The first of these award winning parts was a 2 inch diameter ring shaped screw seal used in the flap mechanism of Boeing 707 and 727 airliners, as well as the German VFW 614 transport aircraft. In addition to corrosion resistance, the part had outstanding properties as a result of its high density, which was over 96% of theoretical. The part was made of pure nickel with a complex configuration featuring a unique internal discontinuous thread (see awards inset).

The second 1979 award was for niobium-based alloy parts used in a

Rocketdyne rocket thrust system. The large 6 inch long chamber was moulded in one piece while the injector part, of an extremely complex geometry, was moulded in two pieces which were subsequently electron-beam welded together (see awards inset). Following these high profile successes, the world took note and, "a copious number of articles appeared in technical journals to announce the advent of this revolutionary technology" [4].

Fe-Ni steels were the main materials used in Parmatech's early MIM production, with a typical part being an Fe-2Ni typewriter head produced for IBM by Parmatech. "Parmatech never really had a marketing effort," Zueger stated in 1999, "The industry just became aware of us" [5].

Parmatech remained in San Rafael from 1973 to 1986, when the company relocated to its current premises in Petaluma.

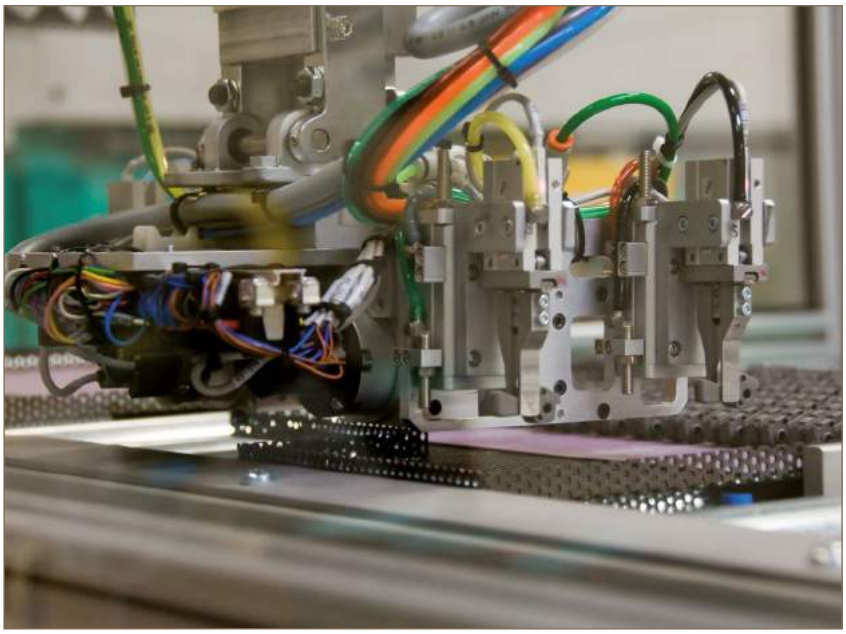


Fig. 4 Two green components being placed on trays in an automated production cell at Parmatech

MIM goes global: licensing Parmatech's technology

The technology patented by Parmatech is today used by many companies around the world thanks to the decision to license the process. Although from a business perspective some have questioned this strategy, the reality is that as a direct result of licensing, MIM was able to spread around the world far more quickly than otherwise may have been the case.

Licensees were granted to 12 companies, expanding the market for MIM products to ever more diverse areas. Although all agreements had come to an end by the late 1990's, the company still maintains contact with many of these licensees.

Carpenter invests in capacity

In 1995 Carpenter Technology purchased Parmatech. Carpenter had recently invested in a new structural ceramics business, although its core business related to the production of speciality metal powders, bars and billets. This transaction brought fundamental changes to the nature of Parmatech, with the new ownership coming at the same time as MIM technology worldwide was gradually transitioning from a niche technology to a fully fledged high volume component manufacturing industry.

The company benefited from a new 22,000 ft^2 facility built on a green field site when it relocated to Petaluma in 1986, and by 1994 it had reported annual sales of $6.5 million [6]. With production volumes increasing, Carpenter made substantial investments in the business, including a new continuous furnace from CM Furnaces, Inc., as well as investing in feedstock production capability.

Zueger retired at the time of the acquisition, though he remained as a consultant to the business.

Fig. 5 Solvent debinding tanks

Parmatech today

Parmatech was not a natural fit in the Carpenter portfolio and in 2003 the company was sold to the current owners, ATW Companies of Warwick, Rhode Island, a move which saw the company return to private ownership. ATW is a family business whose core activity is the production of metal components and products. The group includes metal stamping producer A. T. Wall, located in Warwick, Rhode Island and metal tube fabricator Judson A. Smith, located in Boyertown, Pennsylvania.

Brian McBride, General Manager of Parmatech, since 2006 told PIM International, "ATW is a natural fit for Parmatech. They are a family owned business, committed to precision metal products fabrication". McBride came to Parmatech from the plastic injection moulding sector, having worked previously at Honeywell and Pfizer, as well as a number of start-ups in both engineering and management roles. Commenting on his first years with

Fig. 6 Thermal debinding furnaces, with stacked parts ready for sintering in the foreground

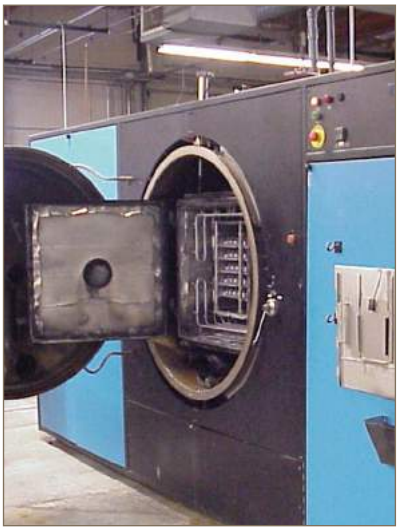


Fig. 8 An Elnik debinding and sintering furnace at Parmatech

Fig. 7 A pair of Abar Ipsen vacuum sintering furnaces (left), with additional thermal debinding furnaces (right)

Parmatech, McBride stated, "PIM is a blend of a wide range of technologies and a background in plastic injection moulding was certainly an advantage when I joined Parmatech".

Materials and feedstock

Parmatech's feedstock was for many years manufactured at an external location in the San Francisco area. In 2003, a custom mixing system was installed in a new private facility adjacent to Parmatech's manufacturing facility in Petaluma, bringing the total Petaluma site to around 30,000ft^2. The new mixer was developed and manufactured for Parmatech and one of its licensees in Switzerland (Parmaco AG) by a leading German producer of polymer processing equipment. Powered by a 100hp motor, the custom mixer gives Parmatech complete control over the quality and batch-to batch consistency of its feedstock and is able to produce 70kg batches.

McBride stated, "We aim to secure at least two powder suppliers for each material that we process. A good relationship with our powders suppliers is essential and we see value in providing feedback on the powders that we receive. This is essential to support the continued development of improved powders for MIM. The impact of powder costs can, of course, be significant, particularly on some of the larger components that we produce". Parmatech have recently moulded a cylinder shaped part weighing 200g as moulded, with final part weight around 180g. The largest part currently commercially produced at Parmatech is 52g.

Today, Parmatech typically processes low alloy steels (MIM-4140, MIM-4605, MIM-2200, MIM2700), stainless steels (MIM-304L, MIM-316L, MIM17-4, MIM-420), soft magnetic materials (MIM-Fe50Ni, MIM-Fe3Si, MIM-430L), controlled expansion alloys (MIM-F15) and specialty alloys such as Inconel™, Pryomet™ and F75 cobalt chrome.

Manufacturing facilities

Parmatech today operates 16 injection moulding machines with clamping forces ranging from 40 to 140 tons, a mix of German Arburg and Japanese Nissei machines. A significant number of machines feature automated production cells with robotic systems removing green parts from moulds and placing them on trays prior to solvent debinding. Computer vision systems are also used for the quality inspection of green parts prior to placement on trays.

The company has an experienced tooling department that is used for repairs and modifications, but uses specialist external toolmakers for all new tool production. "We prefer to put our trust in a small number of expert tool suppliers with whom we have a longstanding relationship", stated McBride.

The two stage debind process used at Parmatech initially involves removing the first stage binder in a solvent bath. Today this is done in the three large tanks shown in Fig. 5. The second stage sees the second stage binder thermally removed in debinding furnaces (Fig. 6). As a typical example, thermal debinding temperatures for a

Parmatech's industry awards mirror the growth of an industry

In 1979 Parmatech won its first two MPIF awards, bringing much needed awareness and credibility to MIM technology. These were the first of many awards that Parmatech has received.

Seen together, these parts illustrate well how MIM technology has, over time, been accepted by an ever-expanding range of end-user markets. Images courtesy MPIF.

1979

Aerospace / Defence

Airliner flap screw seal
MPIF Award of Distinction

This 2 inch diameter ring shaped part was used in the flap mechanisms of Boeing 707 and 727 airliners, as well as the German VFW 614 transport aircraft. In addition to corrosion resistance, the part was reported to have outstanding properties as a result of its high density, which was over 96% of theoretical. The part was made of pure nickel with a complex configuration featuring a unique internal discontinuous thread.

Aerospace / Defence

Rocket burner system
MPIF Award of Achievement

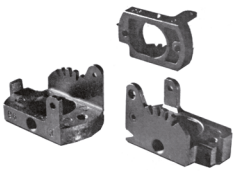
These niobium alloy components were used in a Rocketdyne rocket thrust system. The large 6 inch long thrust chamber was moulded in one piece while the injector part, of an extremely complex geometry, was moulded in two pieces which were subsequently electron-beam welded together.

1983

Business Machines

Tilt rings for IBM typewriters
MPIF Award of Achievement

These nickel steel tilt rings were produced by Parmatech for IBM's 'Selectric' electric typewriters. This was one of the very first high volume components for MIM and was central to securing the technology's future as a precision manufacturing process. IBM later established its own in-house MIM operation in Lexington, Kentucky, which operated the Parmatech process under licence. This part replaced a cast preform which had to undergo 14 machining operations. The MIM component required only minor grinding and coining operations.

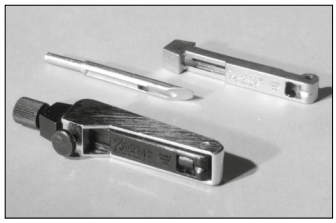


1989

Consumer Goods

Guitar pistol and saddle lever
MPIF Award of Distinction

These MIM 17-4 PH parts were used in the Kahler electric guitar tremolo. Formed to an average density of 7.8g/cm^3, the complex parts have a yield strength range of 20,000-25,000 psi(138-172 N/mm^2). The Kahler logo is also moulded into the lever. Secondary operations included sizing and a black oxide treatment.

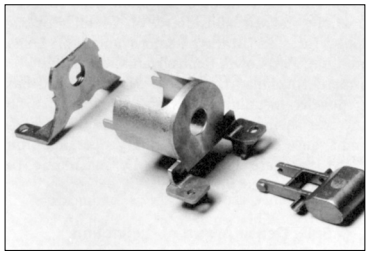


1990

Aerospace / Defence

Solenoid assembly
MPIF Grand Prize

This MIM-2700 nickel-iron tripping solenoid assembly consisted of a housing, rear plate and counterweight. Produced for SPD Technologies, the solenoid was used in a shock-hardened electronic moulded case circuit breaker, supplied primarily to the United States Navy. Density range is 7.78-7.84g/cm^3. Secondary operations included threading, straightening and zinc chromate plating.



1991

Automotive

Air bag sensor insert assembly
MPIF Grand Prize

Developed by Parmatech with Breed Automotive, this airbag assembly was a key early high volume application for MIM in the automotive industry. As airbags became commonplace in all cars, production volumes increased dramatically and inevitably production moved east. Such parts are still being produced by MIM today in China. *(see "MIM notches up nearly two decades of service in air bags", PIM International, Vol. 2 No. 2, June 2008, p 14-15).* Shown are the three parts that make up the assembly: D-shaft, tab insert, and firing pin. Made from MIM-17-4 PH, these MIM parts offered 'a marriage of rigidity, high strength, wear and corrosion resistance'. Tolerance control and repeatability in manufacturing were extremely important to the customer.

1992 Computer / IT

Disk drive pivot hub
MPIF Grand Prize

The electronics industry was one of the first industries to leverage the potential of MIM for the production of extremely high volume components. Hard drives quickly became established as a major market for the industry and this MIM 316L stainless steel pivot hub, developed by Parmatech for Hewlett Packard, was used in an integral support and positioning mechanism for a high density disc drive. The assembly is comprised of the MIM hub with a field coil and stator, plastic injection moulded directly onto the hub. The assembly functions as a non-magnetic, light-weight, structurally rigid positioning system for the drive. The part is produced to a density of 7.75 g/cm^3 and has a tensile strength of 73,000psi (503N/mm^2).

1993 Consumer Goods

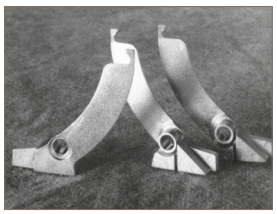
Wire crimper components
MPIF Award of Distinction

This collection of parts produced by Parmatech for Harris Corporation included three interchangeable blades and a cam/spring receptacle used in a crimping/cutting tool for telephone terminal wire connections. The interchangeable blades were made from 17-4PH stainless steel. The receptacle was made from nickel-iron to an average density of 7.8 g/cm^3. Coining assured the correct geometry and a grinding operation after heat treating produced a sharp knife edge.

1994 Computer / IT

Read/write latch arm for disk drives
MPIF Award of Distinction

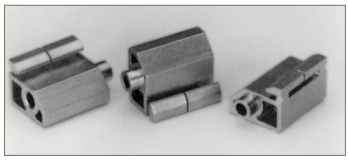
Developed by Parmatech for Seagate Technology, this MIM 316L miniature read/write latch arm was used in small high capacity hard disk drives. It is another good example of the adoption of MIM by computer hardware manufacturers. This complex part has a density of 7.65 g/cm^3 and a tensile strength of 70,000psi (482N/mm^2). The geometric form of the entire outside profile of the part was very critical relative to the pivot hole. Coining was the only secondary operation.



1995 Computer / IT

Disk drive magnetic latching post
MPIF Award of Distinction

This MIM 17-4 PH magnetic latch has a complex shape with variation in the cross-sectional area and a tapered hole that mates into a straight hole and unsupported bridge area with a thin slot. The part required only a light coining operation on the unsupported area to provide the desired air gap which establishes the magnetic field for the latching mechanism. The part has a density of 7.65g/cm^3, a typical hardness of 27HRC, a yield strength of 96,000psi (662MPa), an ultimate tensile strength of 127,000psi (875MPa) and an 11% elongation.



1999 Consumer Goods

Shower safety valve
MPIF Grand Prize

These MIM-17-4 PH valve parts work in a system that prevents scolding in showers during changes in water pressure. The parts were developed by Parmatech for Moen and replaced a screw machined spool containing a plastic insert. As this technology became mandatory in many countries around the world, volumes increased rapidly. The design is still in production, however it is now machined in China.

2002 Telecoms

Motorola cell phone hinge
MPIF Grand Prize

This MIM hinge assembly was developed by Parmatech for Motorola for a clamshell cell phone. Parmatech produced several hundred thousand parts monthly under very stringent process control conditions, however as production volumes increased beyond the capacity of any single MIM company at the time, multiple suppliers were contracted to produce these components.
The assembly is composed of four parts, a hinge, a right and left knuckle and a cam. Produced from 17-4 PH and 316L these parts had thin walls with highly complex geometries making them very difficult to manufacture economically by any process other than MIM. The length and slot diameter of the hinge barrel are coined and the knuckles were buffed to a Class A surface finish. The cam and hinge barrel were nickel Teflon plated for lubricity and wear resistance.

2004 Medical Devices

Laparoscopic jaw assembly
MPIF Grand Prize

This high compression jaw was used in a laparoscopic vessel fusion system manufactured by SurgX. The jaw design comprises top and bottom jaws, an anchor and an I-beam. The components were made from 17-4PH stainless steel metal powder and have as –sintered densities exceeding 7.6 g/cm^3. The parts have thin walls and highly complex geometries, making them difficult to manufacture economically by any other process. Secondary operations include coining, heat treating, wire EDM machining and bead blasting.

2008 Medical Devices

Articulation Gear
MPIF Grand Prize

This 17-4 PH stainless steel articulation gear is used in a surgical stapling unit. Challenging design features in this part included an interrupted gear tooth with window, overmoulding with plastic and assuring a seal in the over mould. The part is produced to a density of more than 7.65 g/cm^3 and has an ultimate tensile strength of 130,000 psi, a yield strength of 106,000 psi, and an HRC 25 hardness. The part is formed to a net shape.

2009 Firearms

Handgun Housing Block
MPIF Award of Distinction

The firearms industry has been one of North America's largest consumers of MIM parts. This 420 MIM stainless steel housing block is used in a 45-caliber handgun and contains the firearm's spring mechanism and provides sliding action with other mechanical parts. More than 100,000 units are produced annually. This complex MIM part features wings, undercuts, through-holes and blind holes, as well as thin and thick cross sections.

17-4 stainless steel range from 800-1000°C, with the parts then sintered in vacuum between 1300-1350°C.

The company uses a range of sintering furnaces, including Abar Ipsen vacuum furnaces, Elnik combined thermal debind and sintering furnaces and CM continuous furnaces. A new Centorr Vacuum Industries furnace is scheduled to come online in July 2010.

"There is a balance to be found between the convenience and speed of combined debinding/sintering versus the slower, cleaner and more 'maintenance friendly' separate thermal debind and vacuum sintering system. Either route has advantages and disadvantages in terms of carbon control and part quality", commented McBride.

Parmatech provides an increasing number of post-sintering services to its customers, including plastic overmoulding as was demonstrated in its 2008 award winning 17-4 PH stainless steel MIM articulation gear used in a surgical stapling unit (see awards inset). The company also performs laser marking on a number of components that it produces for the firearms industry. There are plans for some heat treatment processes to be available in-house.

ERP system helps maximise manufacturing efficiency

Parmatech operates an advanced Enterprise Resource Planning (ERP) system that it believes sets it apart from many other MIM parts production operations. "The benefits of using a 21st century business operations system are huge", states Dan 'DJ' Lauck, Parmatech's Sales Manager. "We can now accurately forecast part production times, calculate the impact of changes to existing orders, or new orders on overall production times, and manage inventory levels for maximum efficiency. The real-time nature of the system allows us to effectively control business processes that cross department boundaries. Ultimately we can shorten production lead times, delivery times and increase profitability".

Parmatech's approach to developing parts with customers

Parmatech operates a complex review system for all new products. Its New Product Introductions (NPI) process is a seven step gated process that ensures that all aspects of a new part are considered and planned for.

McBride told *PIM International*, "Such a rigorous NPI system enables the customer to extract the maximum benefit from MIM processing, but the earlier the discussions with us the better. We can leverage the potential design freedoms that MIM offers and incorporate changes to non-critical features to ease processing and reduce operating costs. After the design stage the NPI process addresses production requirements, tool design, engineering and finally details the necessary steps to agree with customers the appropriate inspection protocols, control plans and testing requirements".

"A crucial additional benefit", continued McBride, "is that such a system creates a team-oriented approach within the organisation, where all departments are aware of developments and have a input into the development of products at Parmatech".

For the last several years, Parmatech has introduced 20 to 25 new tools per year, and is looking to increase this rate of new business dramatically in the next few years. "Established customers now come to us from the start, enabling

Fig. 9 A MIM-420 stainless steel trigger component

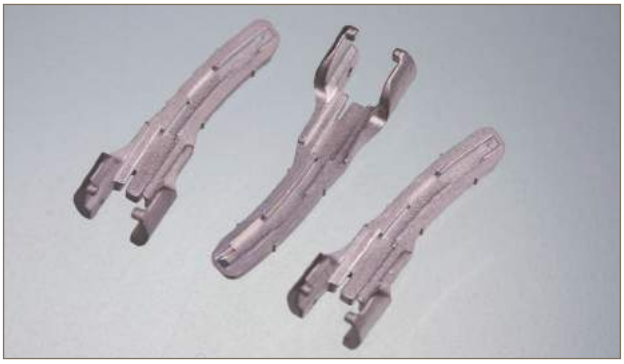


Fig. 10 MIM-17-4 surgical jaws for minimally invasive laparoscopic procedures, featuring multiple intersecting cores and shutoffs

Fig. 11 A helix shaped component for industrial magnetic application manufactured from HyMu80™ alloy

Fig. 12 A MIM-2700 alloy firearm component featuring an internally moulded thread

Fig. 13 A 304L component with external moulded thread for routing of communication fiber bundles

us to suggest tweaks to a design. They know that this can more often than not save on development time, processing costs and material usage".

"We don't claim to be the lowest priced MIM manufacturer out there, but in many instances we are seen as one of the 'lowest cost' manufacturers – when taking into consideration our reliability, consistency and ability to develop new parts. Medical and firearms are two of our biggest markets and both are markets in which reliability and accuracy are critical".

DFM – Design for Manufacturability

Design for Manufacturability is a key exercise for every project undertaken by Parmatech and is seen as crucial to maximise the benefits of MIM processing. The aim is to be able to recommend design adaptations to the customer with a view to improving a part's effectiveness for MIM processing.

"The earlier the engagement with us, the better. We can help designers maximise the potential design freedoms that MIM offers, and by incorporating changes to non-critical features ease processing and reduce operational costs", stated McBride.

"In a complex part, simulation software such as Moldflow can help us to quickly achieve 80% of the required dimensional accuracy; however the rest is done with iterations that can take weeks. Broadly speaking, we can have a part 'manufacturing capable' between 3-6 months, however on really complex components this could be up to a year."

'We can help designers maximise the potential design freedoms that MIM offers'

Another option for design validation is through the use of prototype moulds, which can produce functional parts within 4 to 6 weeks, allowing faster feedback to the design engineer on their development project. Overall project costs can be lower with this approach due to faster turnaround time for parts in hand, and discovering issues in design prior to large expense production tooling.

"The MIM process is particularly suited to products with a 3 year life cycle, enabling the customer to maximise their return on the investment in tooling and engineering development. MIM wins most often on components weighing 30g or less, especially on complex designs. Large MIM parts still struggle to be viable", stated McBride.

Markets

Parmatech's main customer base is in North America, with its main markets being medical, firearms, dental, electronics, industrial components (hand tools, lock components) and the textile industry (ceramics).

Whilst the company benefits from clients across a range of industries, it notes that the majority of its clients are at the higher end of the corporate scale. "The cost of tools is a big challenge for the industry. $25,000 - $75,000 for a tool for a newcomer to MIM is a daunting outlay, even for the biggest corporations. As a result, many of the companies that we supply parts to tend to be Fortune 500, if not Fortune 200", commented McBride.

Proform acquisition brings much needed capacity increase

Parmatech has enjoyed consistent year-on year growth for many years, including 2009. As a result, the company faced the challenge of increasing production capacity to keep up with demand. The high cost of real-estate in Petaluma, one of California's most desirable locations, made expansion of the current site unfeasible. The option of relocating a business that relies so heavily on the expertise of its staff to a new region was also seen as extremely difficult. The decision was therefore made to increase capacity through an acquisition.

In 2009 Parmatech announced the purchase of the Proform metal injection moulding operation of Morgan Advanced Ceramics (MAC). The operation, now renamed Parmatech-Proform, will soon be relocating from its existing premises in New Bedford, Massachusetts, to a new custom built 25,000ft^2 facility in East Providence, Rhode Island, and is due to be operational by September 2010.

Commenting on the acquisition, McBride stated, "Proform was a natural choice in many respects. It's location provides us with a presence on the East Coast closer to many of our customers, and is pretty much in the backyard of our parent company ATW."

"As well as investing in new premises, we are also investing in additional equipment for the facility, for example, in new larger injection moulding machines sourced from Battenfeld. These will supplement the five existing Battenfeld machines that the operators at Proform are currently using. Our intention is that Petaluma will remain as the sales and technical centre, with Parmatech-Proform operating as one of two manufacturing bases".

Commenting at the time of the acquisition, Peter C. Frost, President of ATW Companies, stated, "The combination of our businesses will prove beneficial by empowering us to serve our customers better. Proform will enhance Parmatech Corporation's current MIM business, expanding its MIM capabilities to the East Coast, as well as expanding production capabilities and improving product quality by leveraging ATW's expertise in precision metal components. This change in ownership will strengthen our relationships with customers and enhance the high level of service we provide."

Fig. 14 A MIM-17-4 part weighing 200g for an industrial application, shown next to a penny and a MIM orthodontic bracket weighing <1g. These parts illustrate well the size range capable with MIM

The binder system pioneered by Parmatech technology will eventually be rolled into the Proform operation.

Outlook and opportunities

Parmatech is optimistic about its future prospects. As well as continued organic growth, the company sees the acquisition of Parmatech-Proform as offering a number of important opportunities to build on relationships with the new operation's existing customers.

Parmatech also states that it is benefiting from synergies with its sister companies, all of whom operate in the metal forming sector. The three companies exhibited together at the Medical Device & Manufacturing (MD&M) West exhibition earlier this year and reported positive results from the experience. "From current sales of approximately $10-15 million, our aim is to double that within five years", stated McBride.

Plans are in place for a new in-house heat treatment facility, as well as additional injection moulding and sintering work cells. "We are confident that, particularly in the light of the acquisition of Parmatech Proform, we can continue to leverage the production expertise gained during more than 35 years of commercial MIM production. We are modernising our operations and plan to put the scientific knowledge gained from the influx of new personnel and equipment to good use."

Whilst the early decisions taken to license Parmatech technology overseas meant that the company could not look to become a global force, the company's track record of producing award winning MIM components decade after decade indicates that the spirit of innovation and ambition still exists as much today as it did in the pioneering days of the 1970's.

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Alternatives to Powder Injection Moulding: variants on almost the same theme

The initial demonstrations on powder injection moulding date from the 1930's while metal variants have been in production since 1975. The core process is well known, supported by a wide variety of powder, feedstock, and equipment firms. Although there is much focus on the core technology, based on high-pressure moulding and high-temperature sintering, many variants have emerged with shifts in binders, shaping, or other processing steps. In this article Prof. Randall German introduces several variants to illustrate how core principles, similar powders and binders, and other parallel aspects spill over to a wide range of alternative approaches that share many of the same concepts used in PIM.

Within the core PIM process we recognise many possible binders, powder types, moulding conditions, and even a wide range of debinding and sintering approaches. Still, in spite of such variation there seems to have emerged a fairly similar set of approaches, even though moulding might be at low pressures (few atmospheres), intermediate pressures, or high pressures.

However, in spite of the apparent diversity in the PIM industry, there are many other technologies out there that share some of the same platform, yet innovate around the same concepts for a variety of applications. Accordingly, this article is organised on the technical variants to the powder injection moulding process. Several variations in powder-binder combinations are reported. Many examples of different additive and reshaping routes are described, with a few comments on subtractive efforts that use green machining of PIM feedstock blocks to form green bodies. Several additive efforts are well known in the form of rapid prototyping techniques. Although binders might be tailored to the specific needs of the forming process, for most cases the debinding and sintering steps remain similar to that seen in PIM. The range of invention shows the possible new materials, products, features, and property ranges possible from the core PIM technology.

The central idea

In organising this article it became evident that humans are inventive. Thus, once an idea such as PIM takes hold, many exciting variations emerge as engineers put a twist on the central idea. Fig. 1 is a simple flow chart that is useful for discussing the many processing variants introduced in this article. As an example, the points specified in Table 1 provide one of many processes for producing 316L stainless steel by powder injection moulding. Although many variants are known, assume this reflects a baseline for discussing the several options.

The central point is that a small powder is mixed with a thermoplastic binder, granulated after mixing, those granules are then feed into a plastic moulding machine and shaped to a custom tool cavity, and upon ejection from the tool cavity the binder phase is removed and the powders sintered to near full density.

Obviously this fabrication approach is very adept at forming complex shapes in large quantities with excellent properties. However, often times the production quantities are low, the

Fig. 1 Simple process flow chart for powder injection moulding. Within each step there is a large possible variation. Many shaping approaches use similar feedstock, but avoid high pressure moulding

Outline of the PIM process for 316L stainless steels	
POWDER: spherical, gas atomised, prealloyed (18% Cr, 12% Ni, 2% Mo, balance Fe)	
particle size:	D_{10} = 5 µm, D_{50} = 11 µm, D_{90} = 22 µm
apparent density:	3.80 g cm^{-3}
tap density:	4.65 g cm^{-3}
pycnometer density:	7.96 g cm^{-3}
BINDER: 58% paraffin wax, 37% polypropylene-ethylene vinyl acetate, 5% stearic acid	
solids loading:	67 vol.%
MIXER: evacuated, oil jacket double-planetary mixer	
mixing temperature:	150°C
mixing time:	60 min
granule size:	2 mm (range from 0.7 to 8 mm)
MOULDING MACHINE: plastic reciprocating screw with low compression ratio	
metering back pressure:	3.5 MPa
gate diameter:	3 mm
die temperature:	30°C
barrel temperature:	116°C
nozzle temperature:	102°C
peak packing pressure:	5 MPa
peak injection speed:	100 mm s^{-1}
packing time:	2 to 4 s
cooling time:	20 s
DEBINDING: first stage solvent immersion to remove wax and stearic acid	
debinding fluid:	heptane
debinding time:	2 h for 6 mm thickness, 8 h for 12 mm thickness
debinding temperature:	60°C
SINTERING: batch hydrogen atmosphere furnace	
atmosphere dew point:	-40°C
atmosphere turn over:	5 min or 12 times per hour
support trays:	high purity alumina
sintering heating rate:	5°C min^{-1} to 600°C, hold 60 min, 10°C min^{-1}
maximum temperature:	1350°C
sintering hold time:	1 h
shrinkage:	11.9%
sintered density:	7.84 g cm^{-3}
sintered yield strength:	275 MPa
sintered tensile strength:	520 MPa
sintered ductility:	50%

Table 1. Outline of the PIM process for 316L stainless steels

structures are simple, or another aspect of the product or process leads to novel and innovative variants. Here are some examples.

Variations in powders and binders

Variants in powders and binders occur with most of the moulding through sintering steps remaining similar to traditional PIM. The use of wood particles was demonstrated in PIM of toys [1]. An example wood piece made into a keychain ring is shown in Fig. 2. One application was injection moulding alphabet blocks, but largely went into extruded products [2]. Other materials of note include titanium, ferrites, nickel-base superalloys, molybdenum, thermoelectric compounds, and even aluminium. Several other materials have been introduced into PIM, but after several years, materials such as aluminium have generated little interest.

Customised particle size distributions are known, and at times practised in PIM. Demonstrations of 82% solids loading were reported early in the technology [3]. Indeed, one company did a $65 million initial public offering based on a wide particle size distribution and a water-starch binder system. The high packing density powder was frozen in the mould, subsequently the water was sublimed to leave a starch bonded powder. The process works, but stumbled with metals since the starch tends to increases carbon content unless specific oxidation steps are added to the heating cycle.

The use of water as a binder has several variants. The most innovative was reported by King and Keswani [4], where the binder was simply water. The feedstock granules were frozen and pressure moulded into cold tooling. With sufficient pressure the water underwent an adiabatic melting (density of water is lower than the density of ice) so the water-powder mixture flowed into the cold tool. Once the pressure was relieved, the water froze again and the frozen powder ejected from the tooling. Sublimation of the water (vacuum freeze drying) enabled debinding and the powder was then heated to sinter the body.

Another variant of the water binder relied on ethyl silicate dissolved in the water [5]. The powder-water-silicate mixture was moulded into aluminium tooling at room temperature and then frozen. During freezing the silicate precipitated out of solution and became a permanent bond for the particles, delivering very impressive green strengths. Since the bond was strong, no care was required in heating to evaporate the water. This process is still used in production for alumina based ceramics, which contain small quantities of silica. Other water containing binder systems date back to the very beginning of PIM, including water-cellulose and water-agar binders. These systems generally passed the demonstration phases, and are used for example in extrusion, but largely have not been successful in moulding applications. Still the principles of mixing, shaping, debinding, and sintering are very similar.

Other variants include binders that decompose to form ceramic, and either polysiloxane or polycarbosilane polymers are employed to bond silicon carbide or other ceramics. Variations on this idea are seen in cemented carbides with thermosetting binders that become carbon sources during decomposition. This has seen its most

important application in repair kits for the space shuttle thermal protection system. Variations on this idea are seen in various gelation systems, including gelcasting [6,7].

Other binders abound, but one that is most interesting is the use of egg whites [8]. It was touted as an environmentally friendly binder that gave a high green strength.

Other options are in the area of filters, where sacrificial powders are added to the feedstock. A simple idea is to add salt particles. After moulding and debinding the salt is dissolved out of the body by water immersion, or if the binder is water soluble it is dissolved out in a water debinding immersion. The size and amount of salt added determines the final porosity and pore size. Filters are possible using this route, and various plastic particles such as poly methyl methacrylate serve similar roles [9].

Very small powders are known and several have been used in PIM. Usually a compromise is required since the normal polymers are now large compared to the particles, so a reduced solids loading results. With more sintering shrinkage and more surface area the issues of sintered density, purity, and defects shifts, so simply moving to a smaller powder is not a straightforward variant in PIM [10]. Agglomeration is common, so proper deagglomeration is important, and with smaller powders densification occurs at lower temperatures and that can inhibit oxide reduction prior to densification.

Fig. 2 This key chain includes an injection moulded wood component, showing evidence that PIM works even for organic particles

'The use of water as a binder has several variants. The most innovative where the binder was simply water'

Variants on moulding

In the shaping stage, there are three variants to high-pressure moulding. Often the powder, binder system, debinding, and sintering are similar to traditional PIM. Here the variants are categorised as follows:

- additive – the green body is built up by adding material,
- reshaping – the premeasured feedstock mass is shaped to the desired shape,
- subtractive – a preformed block has mass removed to generate the shape.

Additive processes

For ten years the idea of multiple nozzles working on a die cavity to form two-material bodies has been known. In recent times this has been modified using tape cast sheet that is cut and fit into the mould prior to filling with the second material [11]. From this base come several other ideas, even including sacrificial plastic moulds to enable creation of hollow and other novel bodies.

Most of the newer additive processes comes from developments in rapid prototyping. These ideas rely on computer files that hold the geometric specification and some means of adding or joining materials together to form the desired green body. By avoiding tooling there is an advantage when only a single component is desired. With respect to Fig. 1, these additive approaches only differ in how the moulding or shaping occurs. Some of the additive processes are variants such as the following:

Ink jet printing

A flat powder bed is laid out and bonded by binder that is jetted onto the top surface. This stereo-lithography route relies on a computer image of the component and at each slice through the part the ink jet printer coats particles where there is solid [12]. Where binder is missing the powder is loose and removed from bonded powder prior to sintering. What is formed is a green body, such as evident in Fig. 3, from layers of powders. Proper coordination of the area bonded due to jetted binder with the computer image of solid (dilated for shrinkage) on each slice is important, but even so there is a compound dimensional error over the several layers. One variant relies on hot feedstock for jetting while the more common variant jets just

Fig. 3 A scanning electron micrograph of a green body built using stereo-lithography, showing the laminated layers resulting from the progressive build process

Fig. 4 Porous torpedo noses formed by stereo-lithography with selective laser sintering

Fig. 5 An example turbine component generated by stereo-lithography techniques after infiltration

Fig. 6 A tool set for molding a camera body. This tooling was generated using rapid prototyping approaches adapted from PIM technologies

the binder onto a thin layer of powder which is built up to a three-dimensional object using an overlaid layer building routine.

To avoid distortion in sintering large objects, most of the variants rely on first attaining a presintered body that is then infiltrated with a copper alloy, often bronze. This approach minimises dimensional change. These rapid prototyping approaches are very similar to PIM with the key difference being in the shape building step. While a PIM part forms in seconds, a rapid prototype part requires hours, but is suitable for producing one of a kind.

Laser sintering

This is a related stereo-lithography approach where small binder particles are mixed with the metal or other build powder (metal, ceramic, or polymer). This polymer dusted powder is spread into a thin layer and everywhere the laser passes over the powder it melts the binder causing bonding between the powders. Where the laser is not passing over the powder no heating occurs so there is no bonding. After sequential building of many layers the green body is retrieved by brushing away the loose powders [13]. Fig. 4 shows an example of the laminated particle structure that makes up the green body. From this point the body is debound and sintered, and like other metal stereo-lithography approaches it is common to infiltrate the structure instead of heating to a high temperature where distortion might accompany sintering densification. Fig. 5 is a picture of a laser sintered and infiltrated demonstration shape.

Nozzle extrusion

This rapid prototyping approach relies on hot feedstock that is extruded through a small capillary tube attached to an x-y control system. The extruded feedstock forms a positive shape that follows from the computer image. The bead of extruded feedstock solidifies to form a three dimensional green body that is debound and sintered [14]. Like stereo-lithography, the computer image is realised by a progressive build process that generates the green body from the extruded bead. One difficulty is in forming cantilevers and overhangs, where there is no lower support. The feedstock is essentially the same as used in PIM.

Laminated objects
In this variant, a tape cast sheet of powder and binder is cut by laser or other means to match the two-dimensional image shape corresponding to the computer sliced cross-section of the target part. Tape casting spreads a powder, binder, and solvent mixture on a plastic sheet, and when the solvent evaporates the powder is glued together into a flexible sheet. The green sheets are stacked to form the three-dimensional green body. Subsequently the laminated object is debound and sintered [15]. Many of the demonstrations are in stainless steel or alumina, since the small powders were already available for PIM.

These are examples of the many rapid prototyping concepts proposed over the past few years. In the late 1990s there were over 20 similar commercial technologies all targeting limited production opportunities. Tooling such as shown in Fig. 6 is the largest area of interest. Unfortunately, as with PIM, the surface finish after sintering required polishing and dimensional precision is less than possible via machining. The rapid prototype approaches rely on similar powder-binder combinations and in some cases the feedstock is quite similar to that used in injection moulding, and the debinding and sintering steps are essentially the same as used in PIM.

Other additive approaches to forming a green body include dip coating, sprinkling, and electrophoritic deposition.

Dip coating
For dip coating, a powder-binder slurry is formulated with a solids loading slightly lower than typical for PIM feedstock (50 vol. %). A cold solid object is dipped into the slurry allowing the slurry to freeze on the surface to build up a feedstock layer. The added thickness can be small. Most interesting is the ability to change composition between layers, so functional gradient compositions are formed this way. For example, Miyake et al. [16] fabricated a nuclear tube material with titanium on the outside, stainless steel as the solid core, and molybdenum as the inner coating. This was performed using polyvinyl alcohol as the binder. Since the three layers differed significantly in thermal expansion coefficient, the composition was deposited in 25% composition steps.

Sprinkling
An additive coating is possible on a solid structure by first painting binder onto the substrate and then sprinkling powder to stick to the binder. Powder coating by shallow injection moulding or even sprinkling is a similar coating idea that has been taken up for tissue ingrowth in replacement orthopaedic surgery devices. Several competing ideas are reaching the market [17]. One approach is to infiltrate foamed polyurethane or polystyrene performs with feedstock [18]. Another is to sprinkle powder onto a polymer coated implant. The powder is then sintered to form the porous layer.

Electrorophoritic Deposition
Another way to build a structure in an additive manner is to electrically charge dielectric particles suspended in a solution. This is used to form a thin porous structure useful in microelectronics. For example, a small powder such as alumina or zirconia is mixed to form a suspension, such as acetone with added polyvinyl butyral. The fluid-powder suspension is deposited by applying an electrical voltage on a cavity placed inside the suspension. The voltage causes the particles to deposit as a green layer, similar in packing density to that formed in PIM.

Like other additive processes, the build rate is slow and the polymer is required to provide strength, but as with PIM, cracks will form if the fluid phase is removed quickly. Applications include making SiC deposits and then copper electroplating into the

'In the late 1990s there were over 20 similar commercial technologies all targeting limited production opportunities'

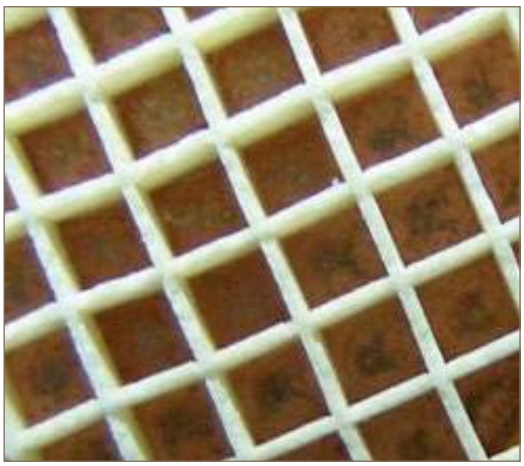

Fig. 7 This structure shows the electrophoritic deposition grid over a copper base used to generate a heat dissipation design out of silicon carbide with copper electrodeposited into the pores (photograph courtesy of Eugene Olevsky)

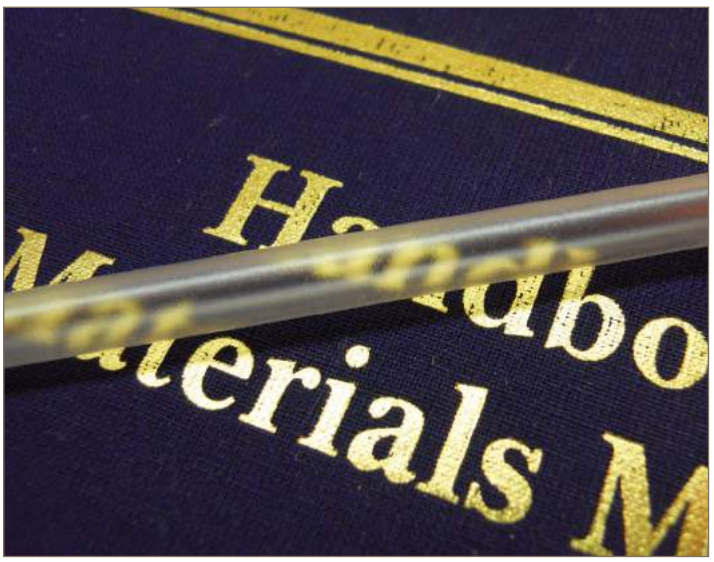

Fig. 8 Translucent alumina tube formed by extrusion of a powder-binder paste which is sintered to full density

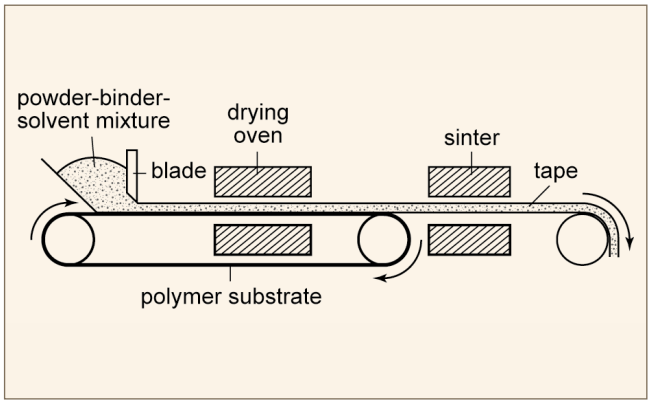

Fig. 9 Tape casting relies on powder and binder to form a constant thickness sheet, but unlike PIM where the rheology is adjusted with heat, tape casting relies on solvent to allow shaping

Fig. 11 This 2.5 kg bronze statue (150 mm high) was formed in rubber tooling using a slurry casting process with a feedstock designed for 82 vol. % solid loading. Shown here is the final product after debinding and sintering with a dark patina

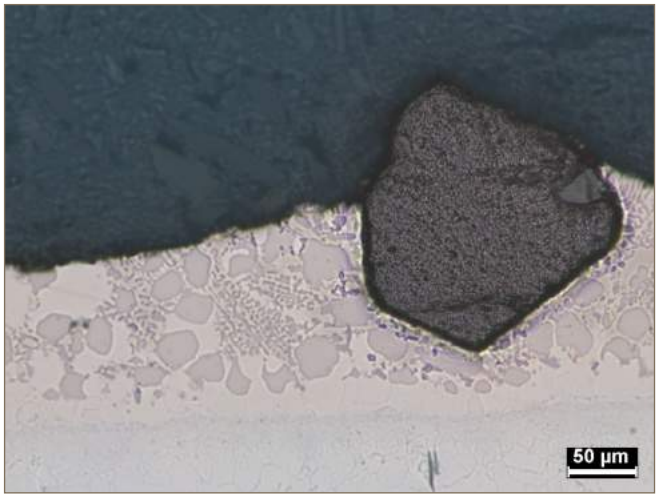

Fig. 10 Metallographic cross section through a diamond bonded to a stainless steel substrate. In this case the braze layer was formed using tape casting and the diamonds were glued to the braze layer prior to firing. The stainless structure is at the bottom and the cross-sectioned diamond is evident as the angular object

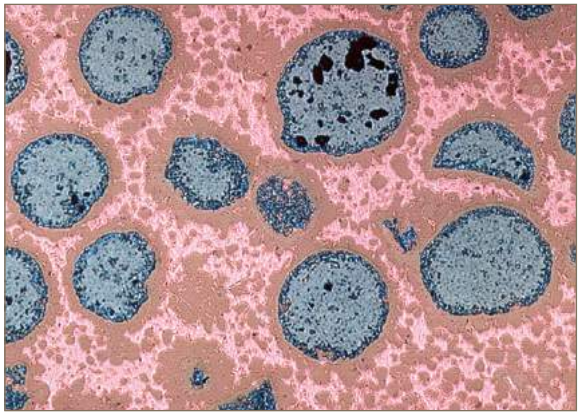

Fig. 12 A hard tool composite system designed for slurry casting, consisting of large cemented carbide (WC-10Co) grains in a matrix of 17-4 PH stainless steel with bronze infiltration. Wear tests determined tooling fabricated with this composition exhibited properties similar to tool steels (courtesy of Anthony Griffo)

structure to form a SiC-Cu composite such as shown in Fig. 7. The low thermal expansion and high thermal conductivity give excellent cooling for electronic circuits with thermal conductivities of 280 W/m/K and tailored thermal expansion [19]. Other systems include nickel with alumina, nickel with silicon carbide, alumina-zirconia, and such.

Reshaping processes

Several feedstock forming approaches rely on shaping feedstock without necessarily adding or subtracting material. Like casting, these reform a paste, putty, or slurry into a green body. One disadvantage is the need for hard tooling since often high pressures might be used to induce feedstock flow. Many variations exist as discussed below:

Extrusion

Powder extrusion with a binder is widely employed for the production of long and thin hard particle structures, mostly from ceramics or cemented carbides. An early application for metals was in the production of nickel gaseous diffusion tubes for uranium enrichment at Oak Ridge. The powder-binder feedstock is converted into uniform cross-section lengths by forcing the mixture through an orifice of desired cross-section.

The feedstock is heated to soften the polymer and relatively low pressures are used to force the paste through a die. Extrusion is used to form long, thin rods, tubes, honeycombs, and twist drills based on the die at the barrel exit. Usually the feedstock is evacuated during mixing to avoid bubbles in the extrudate. The binders are similar to those seen in PIM, and polyethylene and ethylene vinyl acetate are common backbones [20].

One example use is in forming lamp envelopes out of alumina, as seen by the translucent tube in Fig. 8. Other applications include forming micro-electronic substrates, porous tubes, welding rods, diesel exhaust particulate filters, and automobile exhaust catalytic converter substrates.

Tape casting

In tape casting the powder-polymer mixture has added solvent to lower the feedstock viscosity, versus PIM where heat is used to soften the polymer. It

is a means to fabricate thin, constant thickness sheets (wide with a smooth surface). The paste is placed on a moving plastic sheet as a thin layer as shown schematically in Fig. 9. The slurry thickness is controlled by the gap between the doctor blade and the moving plastic sheet. Subsequently, the solvent is evaporated, leaving residual binder behind to bond the particles and provide handling strength. Binders for tape casting are acrylics, waxes, polyvinyl alcohol, or polyvinyl butyryl and the solvents can even be water [21].

Once the solvent has evaporated, the polymer bonds between particles provide flexibility to the sheet. Depending on the application, the green sheet might be trimmed to size and draped over a solid for bonding, or stacked to form a multiple layer structure for rapid prototyping, or fired as a single layer to form an electronic substrate. Other uses of tape casting are to form battery electrodes, brazing layers, coatings, thin foils, bond tungsten carbide onto a steel body, or form diamond abrasive tools. Fig. 10 is a cross-section showing the integral bonding possible from use of a tape cast braze layer to affix diamond on a stainless steel substrate.

Slip casting

Another variant that relies on a solvent to lower the system viscosity is slip casting. It is a common ceramic forming process based on a paint-like mixture of powder in solvent with a dissolved polymer [22]. The key is to have a low solids loading to keep the viscosity low so the slurry can be poured into a porous mould. Pores in the mould extract the fluid and leave the particles behind. Usually the binder is a mixture of water and a gelation polymer, such as starch. The mixture is initially similar in viscosity to paint, but as water migrates into the porous mould the mixture viscosity increases rapidly. It is the absorption of water by the porous mould that thickens the remaining powder-fluid mixture, leading to a dense packing of particles on the surface of the mould.

To form a hollow body, most of the slip is poured out of the mould after a few minutes. Short times make for thin walls and long times give thick walls. After drying, the hollow body is removed from the mould and sintered. Slip casting is a favourite for forming ceramic statues and other complicated objects. Hausner [23] detailed stainless steel powder slip casting with an ammonium alginate solution in 1967, but it is not frequently employed with metal powders.

Slurry casting in soft tooling

A variant is termed slurry casting, where PIM feedstock is heated to form a slurry and simply cast into a warm mould, that mould often being rubber [24]. Upon cooling the binder freezes and the body is removed from the mould, debound, and sintered, so in all respect it is very similar to PIM except no pressure is applied to fill the mould. The concept is similar to casting, except with a binder that tends to be a wax-polymer mixture. High solids loadings require vibration to ensure filling out of the cavity. After filling, the tool is cooled so cycle times tend to be 30 min or more. Alternatives allow for freezing in the mould with a water-containing binder.

Two applications have arisen for slurry casting. The first is for the production of complex shapes, such as artistic pieces as shown in Fig. 11, fabricated from bronze powder. These are filled with an 82 vol. % solids loading, so only undergo a few percent shrinkage during sintering. The second is the fabrication of tool cavities similar to that shown earlier in Fig. 6. Special hard materials have been developed around this technology consisting of cemented carbide spheres, small stainless particles, and bronze infiltration. This composite, shown in cross-section in Fig. 12, has proven very durable with wear resistance comparable to tool steels [25].

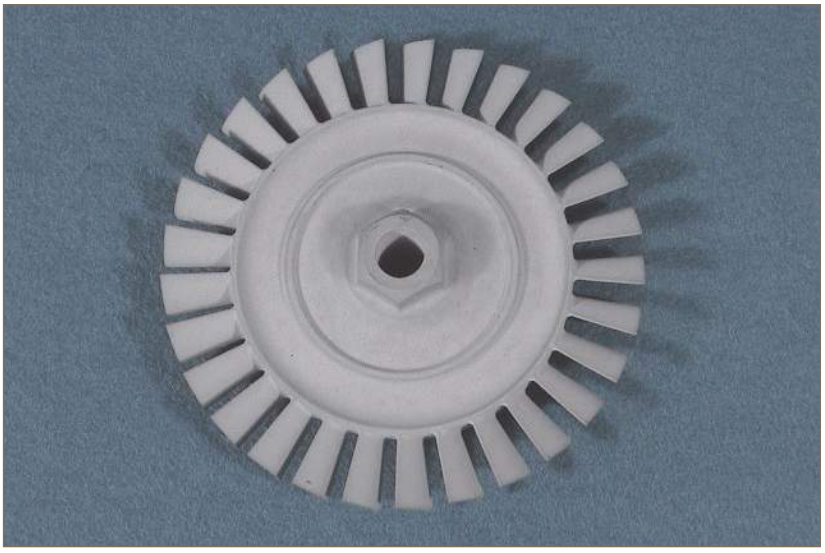

Fig. 13 Superalloy gel cast turbine component, with an outside diameter of approximately 200 mm (component courtesy of Mark Janney)

Fig. 14 Tungsten putty deformed to illustrate the pliable character of this 50 vol. % solids loading (sample courtesy of Travis Puzz)

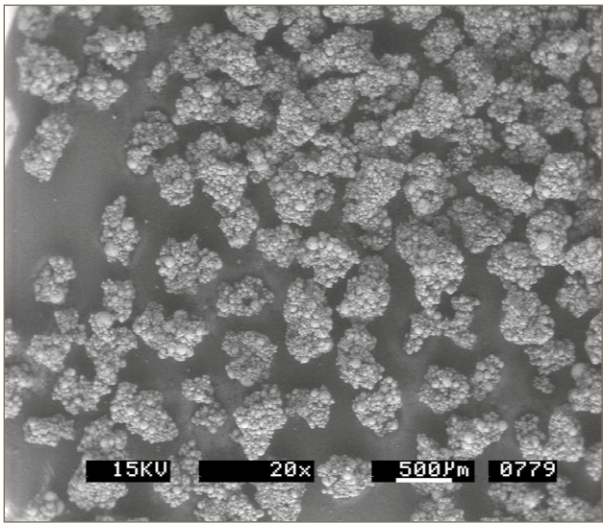

Fig. 15 Agglomerated stainless steel powder ready for die compaction. The small particles provide good sintering and the large agglomerates enable automated feed during uniaxial die compaction (photograph courtesy of Louis Campbell)

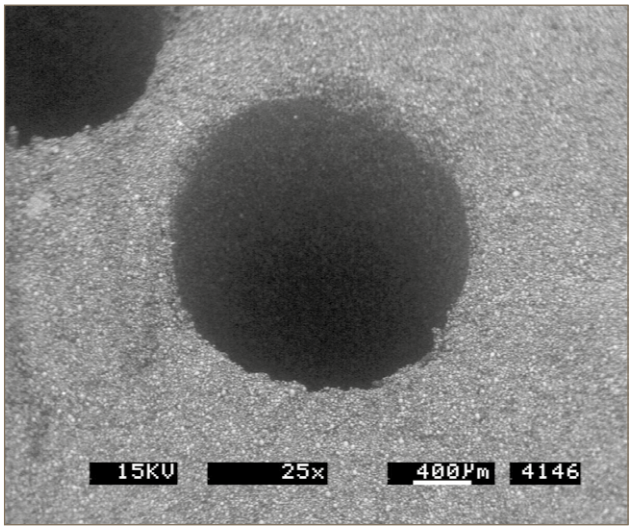

Fig. 17 A stainless steel green body imaged using scanning electron microscopy after green machining, showing only slight damage around the drilled hole

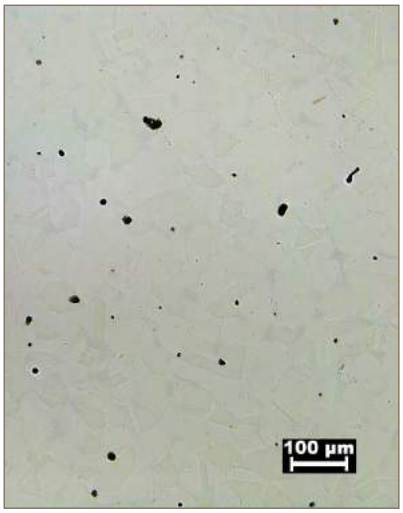

Fig. 16 Metallographic cross-section in a die compacted stainless steel formed by die compaction of an agglomerated MIM powder, the black spots are residual porosity (photograph courtesy of Louis Campbell)

A novel variant relies on filling the mould with a sacrificial open cell foam (urethane is common) that once burned out becomes the pore structure in the sintered body. Porous tantalum for tissue ingrowth is one application for this foamed structure.

Centrifugal compaction

Most PIM processing relies on a reciprocating screw to generate pressure on the feedstock to fill and pack a mould. Centrifugal compaction relies on a high rotation speed to generate pressure on the feedstock slurry, upwards of 10,000 times gravity [26]. Two variants have been demonstrated, one using room temperature shaping with a feedstock containing solvent and a second relying on heated feedstock. One advantage is that the tooling pressure is not severe and epoxy tools have been successful.

When applied to alumina the green density reaches 60%, which is above the range typical for a ceramic. This increase in packing density results in a lower sintering temperature. Further, for complicated shapes the resin mould is simply burned off during the sintering cycle. Demonstrations of the technology have included tool steels, alumina, stainless steels, silica, and copper.

Gel casting

As a variant to slurry casting, gel casting is a similar low or zero pressure moulding process that relies on a low viscosity slurry with a binder consisting of a monomer that polymerises in the die cavity [27]. Once the polymer forms, it gives a rigid body to hold the particles into the desired shape. A variant is to use wax as the tooling, where a wax mould is generated either by rapid prototyping techniques or machining [28]. Acceleration of the gelation step is possible by adding a catalyst just as the feedstock is moulded. Fig. 13 shows a turbine component from a nickel-base superalloy formed with this process. Other materials include stainless steel, silicon nitride, alumina, tool steel, and zirconia.

Putty and clay materials

A range of binders have been developed to enable forming of the feedstock at room temperature, using clay or putty type systems. The first of these was developed by Mitsubishi Materials, called Precious Metal Clay, and it has long been in use for artists as a means to form a metal object using clay forming [29]. The system relied on an old binder design consisting of cellulose and water, but firing in air avoided carbon retention that is often an issue with cellulose binders. Subsequent formulations have relied on wax and oils to form easily shaped materials. Fig. 14 is a picture of a putty containing nearly 50 vol.% tungsten formed with a simple oil-based binder system that includes a small amount of rubber. The powders and binders are similar to what is found in PIM, the shaping is often done by hand, and the firing cycles then burn out the polymer and sinter the powders.

Die compaction

Granulated PIM feedstock responds to die compaction [30]. At low compaction pressures large voids remain between the granules so the sintered body is not very dense. But when either a softer binder (rich in wax and oil) is used or when high pressures are employed, the green body has excellent integrity. Recently a polymer coated MIM powder with ethylene vinyl acetate has been promoted for this hybrid between PIM and traditional compaction. This approach enables simple shapes to be formed by uniaxial die pressing but

sintered to full density using standard PIM firing cycles.

Fig. 15 is a low magnification scanning electron micrograph of 316L MIM stainless steel powder agglomerated to provide larger granules for easy flow in die pressing, an idea similar to that employed in ceramic and cemented carbide compaction. The final microstructure after pressing, debinding, and sintering is given in Fig. 16.

Generally, if a simple wax-like binder is used the agglomerates are weak and easy to compact, have lower green strength, but do not contaminate the body. When higher green strength is required, then higher molecular weight polymers are used with more difficulty in avoiding carbon contamination in thick sections.

Fig. 18 Bronze block, consisting of powder and binder, and a final object carved from the block. The block is easily carved using hand tools, and then is debound and fired in a single firing cycle. A few chips have been lifted out of the block using the carving tool

Subtractive processes

Green machining

Here the concept is to form a large PIM block that is then machined. To avoid cracking, high speed, low cut machining is used to form the green shape. Trials with this approach found that several parameters in the binder, powder, solids loading, as well as tool geometry, cutting speed, and depth of cut had to be optimised to ensure a clean cut. The largest difficulty was crack propagation into the green body due to tool damage. An example hole drilled in a stainless steel green block is shown in Fig. 17. A slight amount of debris is evident around the hole.

One application is in forming prototype PIM parts without tooling, by machining the green body with standard debinding and sintering cycles. A more successful application has been to form artist blocks, in the same spirit as Precious Metal Clay, which can be carved using simple hand tools, debound, and sintered to form durable products. Fig. 18 shows one such block consisting of bronze powder and a mixture of waxes and the sintered product after carving.

Variants on sintering

Sintering is a heating process and it is hard to envision much innovation here. Even so, several process variants are under study, but none have reached far beyond demonstration levels. One of the variants is microwave heating, where a powder metallurgy product will couple to the microwave energy. Attempts to directly heat a MIM green compact resulted in a puddle, since binder softening means a loss of strength at a temperature far below the point of sintering. Thus, off-line debinding is required prior to sintering each compact individually. Several pilot facilities found that sintering could be performed in minutes, but only one compact could be controlled at a time. Thus, for a batch vacuum furnace load of 25,000 parts this means over 100 days to sinter with a microwave.

Likewise, a technology based on microwave driven plasma has the same limitation of heating one part at a time. Although relatively fast in heating each compact, the productivity is far below what exists with conventional technologies. Spark sintering is another approach being applied to complex shapes (without pressure), and it too is able to sinter each compact in about 10 min, but this is not competitive with batch and continuous furnaces installed to date.

One option of interest in vacuum sintering is to induce a discharge plasma using a voltage and slight vapour pressure of argon [31]. A key advantage of the glow discharge is any residual binder (backbone) is degraded so the product is free of carbon contamination, has excellent properties, while the furnace remains very clean. Data for a Nimonic 90 superalloy shows a tensile strength over 1100 MPa with 22% elongation. Besides superalloys, this option has been demonstrated with titanium, cemented carbide, and stainless steels.

Summary

Powder injection moulding has been practised in various forms for 35 years or more. The infrastructure of powders, binders, moulding and shaping equipment, debinding and sintering tools that has arisen around PIM is important to many variants as outlined here. Indeed the innovation continues. New companies in the microelectronics industry are now looking to use powders and binders to form low cost circuits. One approach is to print silicon powder as an alternative to traditional lithography. Another concept is to rely on screen printing of powders as a means to replace circuit board soldering. Thus, as PIM advances and further develops an infrastructure there are some exciting, and often more significant developments going on in parallel.

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Singapore's Advanced Materials Technologies celebrates 20 years of MIM excellence

Singapore, with its modern skyscraper skyline, epitomises the rapid ascendance of the Asian Tigers into highly developed economies. Metal injection moulding has benefited from this growth, particularly in relation to the dominant consumer electronics sector. In Singapore's case, the success of MIM is due in no small measure to the standards set by Advanced Materials Technologies (AMT). Bernard Williams reports on his recent visit to the company that was one of the first MIM producers in the region, and which this year celebrates its 20th anniversary.

The Republic of Singapore, an independent island state with nearly five million inhabitants, is situated on the southern tip of the Malay Peninsula. It is strategically located at the junctions of major sea trading routes in South East Asia and the region's huge economic expansion over the past three decades, combined with a well educated, skilled and industrious population, has combined to establish Singapore as an economic powerhouse of an importance that is disproportionate to its small geographical size.

The two dominant sectors which have helped to drive the country's growth since its independence from its neighbour Malaysia in 1965 are the service sector, which accounts for around 65% of GDP, and the manufacturing sector which accounts for a further 26%. Electronics related industries, for which the country is internationally renowned, make up nearly 30% of manufacturing output.

The global financial crisis which began in the final quarter of 2008 and led to the sharp and dramatic decline in demand worldwide for cars, consumer electronics and other products in 2009, also had a major impact on the Singapore economy. GDP is said to have fallen by between 4 to 6% in Singapore in 2009. The country has, nevertheless, managed to achieve

Fig. 1 The modern skyline of Singapore epitomises the country's desire to be one of the technology leaders in the rapidly growing region of South East Asia

Fig. 2 AMT's 44,000 ft² plant in Tuas Lane has the capacity to produce 500 tonnes of MIM and CIM components annually

Fig. 3 David Lau, General Manager (left) and CheeHoo Liang, Senior Manager, Technology (right), host PIM International on its visit in February 2010

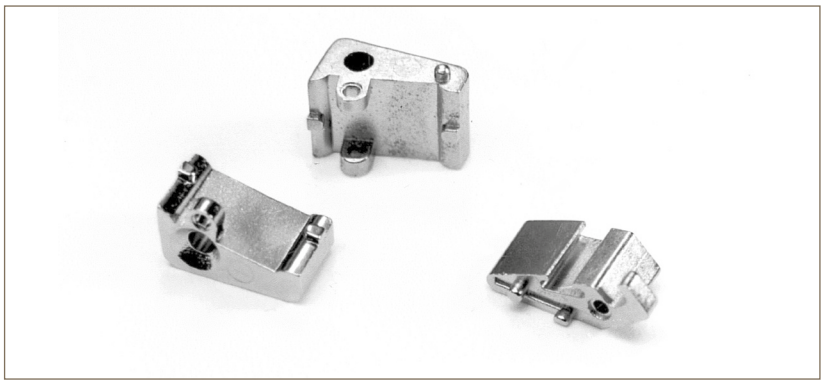

Fig. 4 Two of the tiny MIM parts produced by AMT and used in the first 1.8 inch miniaturised hard disk for computers. The parts won a MPIF Grand Prize in 1993

a very creditable growth, averaging over 5% in its manufacturing sectors over the nine year period from 2000.

'we are seeing the fruits of our efforts in diversifying into other sectors such as industrial, automotive, medical and energy'

Located in this small but vibrant country is Advanced Materials Technologies (AMT), the materials arm of Accuron Technologies which is fully owned by Temasek Holdings, an investment company headquartered in Singapore. Other subsidiaries of Accuron Technologies include Singapore Aerospace Manufacturing (SAM) a leading producer of engine case, engine mounts, airfoils, actuators, valves, flight/engine controls and hydraulic assemblies. Another subsidiary is Dornier MedTec, a manufacturer of medical devices such as lithotripters, orthopaedic shock wave systems, urology surgical tables and surgical lasers.

AMT - the evolution

AMT was incorporated in 1990 and the initial MIM operation was started with a small team, just one injection moulding machine, a solvent and thermal debinding system and a vacuum sintering furnace. The start up operation was housed in a 3,000ft^2 facility in the northern part of Singapore, and it had the capacity to produce around 1 tonne/month of MIM products. Early success came in 1993 when the company was given the National Technology Award by the Singapore National Science & Technology Foundation for its use of MIM technology to produce three miniature soft magnetic Fe-Ni parts weighing less than 1g each (Fig. 4).

These MIM parts were used in the world's first 1.8 inch hard disk drive. One of these MIM parts maintained the distance between the magnet housing plates, guides and spring locators for the other moving parts of the disk drive, whilst the other two MIM parts functioned inside the magnet housing.

The company also received the Overseas Grand Prize from the Metal Powder Industries Federation (MPIF) for these parts, an achievement which has been repeated four times since with further MPIF Grand Prizes for other innovative MIM components. In addition, AMT has received a number of Awards of Distinction from the MPIF as well as European Powder Metallurgy Association awards. In October 1994, the company delivered its millionth MIM part to a customer and just five years later AMT had increased MIM production tenfold to around 10 tonnes/month.

The company celebrated its 10th Anniversary at the end of 1999, having shipped over 25 million MIM parts. Sales for MIM parts continued to grow in the early years of the new Millennium and reached an all time high in 2006 thanks to success with the development and supply of new MIM parts specifically for mobile phones.

David Lau, who took over as General Manager of AMT in 2009, stated that sales were expected to be strong in 2010. "The combination of the global economic recession and the over reliance on the consumer electronics industry have taken their toll in recent years. However we are seeing the fruits of our efforts in diversifying into other sectors such as industrial, automotive, medical and energy", said Lau.

AMT has been at its Tuas Lane site on the western side of Singapore since 2004 where it has 44,000 ft^2 of production space and around 100 employees. Capacity for MIM and CIM production at Tuas Lane is put at around 500 tonnes/year. In addition AMT recently opened a new precision machining plant in Dongguan, China in December 2008. The machining

plant has a 78,000 ft^2 production area and is used to complement the MIM parts produced in Singapore as well as offering a complete machining service. The plant uses the latest high precision equipment for milling, turning, tapping, grinding and honing. Lau stated, "By relocating our finishing operations we were able to remain competitive as we capitalise on the low operating costs in China".

The range of MIM materials used by AMT has significantly increased over the past 20 years with the company processing Fe-Ni low alloy steels and iron-base soft magnetic materials, stainless steels, low thermal expansion alloys (Kovar, Invar, Fe-38%Ni, and AMT Alloys 42 and Alloy 50), non-ferrous materials including copper and tungsten, Inconel superalloys, and ceramics based on alumina and zirconia.

Feedstocks for MIM and CIM are prepared in batches of 50 to 100 kg in different production lines to avoid cross contamination. AMT uses heavy duty kneading mixers to mix the fine metal or ceramic powders and the polymer-wax binder at temperatures up to 150°C, followed by processing the mixture in a twin-screw extruder at 130-150°C. The extruded material is then pelletised and stored in drums ready for injection moulding. The ceramic or metal powders that are used have a typical particle size in the range 2 to 10 microns. Where possible AMT uses water atomised metal powders for MIM primarily because of their lower cost. Stearic acid is added as a surfactant to the powder/binder mixture to enhance mixing and typical solids loading is put at between 60 and 65%.

The normal binder systems used at AMT are wax and polymer based. Work has recently been undertaken to develop modified binder systems for μMIM parts which involves the use of low density polyethylene. This binder is said to produce feedstock having good viscosity and shape retention after moulding of micro and miniature PIM parts.

AMT uses an outside supplier to produce the injection moulding tools but undertakes tool design and maintenance in-house. Lau said that around 50 tools were currently being used for different MIM and CIM parts. The company operates an impressive range of automatic and semi-automatic hydraulic injection moulding machines, mostly Arburg Allrounders, ranging in clamping force from 30 to 100 tons. Operators check the weight of the green moulded parts on an hourly basis, feeding the data into an in-line quality assurance system. All the moulding machines are fully automatic with runners being removed after moulding and robots or mechanical pick and place devices feeding the green parts onto trays ready for debinding.

The company's debinding area houses a number of solvent extrac-

Fig. 5 Injection moulding lines at the AMT facility

Fig.6 Thermal debinding section at AMT

Fig. 7 Unloading one of the vacuum sintering furnaces

Fig. 8 Part of the quality control section at AMT

Fig. 9 Visual check being carried out on a MIM part

tion units operating at 60-80°C in air to remove the wax element of the binder from the 'green' parts. There is a further line of thermal debinding furnaces operating at temperatures up to 600°C for removing the remaining binder under controlled atmosphere (mainly nitrogen) with the waste gases burned off. AMT additionally uses a line of graphite batch furnaces each having 15 ft^3 usable volume for sintering, and Elnik furnaces with molybdenum heating elements capable of temperatures up to 1800°C. The latter combines debinding and sintering in one operation.

Sintering is mostly done in vacuum but some MIM parts are sintered using a controlled atmosphere such as nitrogen, hydrogen or argon. AMT uses an in-line PC system for controlling and monitoring the temperature of its furnaces with data being logged for each batch being debound or sintered. Dimensional tolerances of sintered MIM parts are generally held to ±0.5% or better and surface finish at 32 RMS; R_a 0.8.

The entire MIM and CIM manufacturing processes at AMT are supported by the Quality Assurance department which contains a range of equipment for metrology as well as metallurgical and chemical analysis (Figs. 8 and 9). The company was first registered for ISO 9002 in 1996, and has since received ISO 9001:2008 certification

Fig. 10 This single complex MIM part made from Fe50:Ni50 powder replaced 4 parts previously used in a control valve

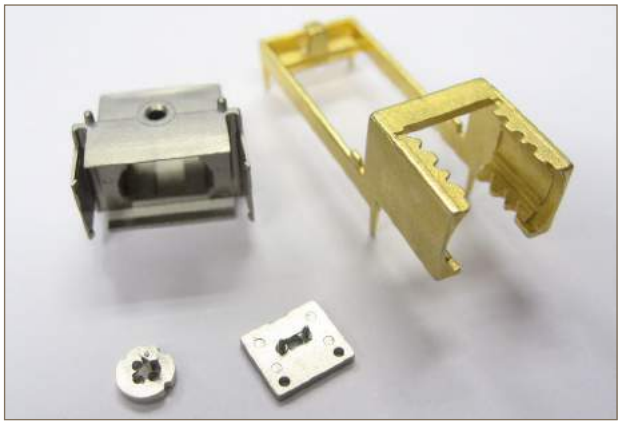

Fig. 12 Fibre optic transceiver housing with thin walls made from 17-4 PH stainless steel used in telecoms equipment

Fig.11 Stainless steel mechanical connector produced to incorporate the body base and centre shaft as an integral MIM component

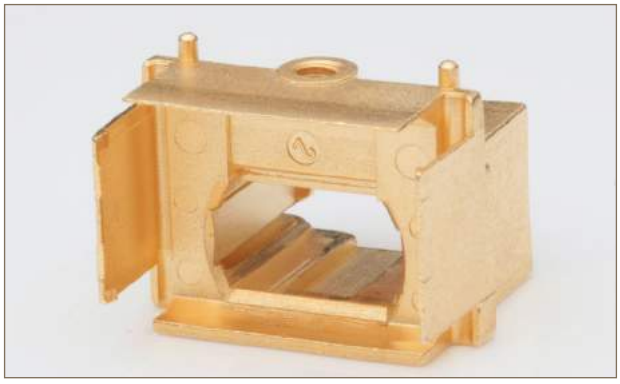

Fig. 13 17-4 PH stainless steel MIM connector for fibre-optic module. The overall size of the housing is 42 mm x 14 mm x 16 mm

in January 2010 for both the plants in Singapore and Dongguan, China. As the company is also serving the automotive industry, its systems are compliant with ISO/TS 16949 and maintain the usage of core tools such as APOP, PPAP, SPC, FMEA and MSA.

Expanding horizons through innovation

Underpinning the strong growth in sales at AMT over the past two decades has been an ongoing R&D effort in materials and process technology which the company believed would lead to new applications for MIM. In addition to R&D being carried out in-house, AMT also worked closely with the Singapore Institute of Manufacturing Technology and the Institute for Materials Research and Engineering on quality and production issues. The joint R&D efforts resulted in PIM innovations such as assembly integration, MIM parts with undercuts and internal hollow channels, parts with controlled porosity, and the development of anisotropic materials and two-material systems.

Liang CheeHoo, Senior Manager Technology, who has been with the company since the start of the millennium, told *PIM International* that over the past 20 years AMT has succeeded in developing numerous PIM innovations, with 30 patents filed in 16 countries.

In expanding its horizons, AMT sought to take advantage of MIM's potential for designing the cost out of complex components and assemblies. One project developed with a customer in the oil and gas sector resulted in a significant cost saving when AMT was able to replace a complex assembly of four parts (two conventional PM parts and two machined parts) with a single complex MIM part (Fig. 10) for a control valve. The part regulates the flow of highly flammable chemicals and gases in the control valve and is made from a 50%Ni-50%Fe powder to a final density of 8.05 g/cm^3. Magnetic flux is applied through the body assembly which is mounted inside the control valve, and because the whole assembly is integrated there is much less resistance to the magnetic flux compared to the previous design. AMT received a Grand Prize from the Metal Powder Industries Federation for this MIM application in 2000.

A further example of multi-component integration is a one-piece stainless steel mechanical connector, also developed for the same customer, where the previously separate body base and the centre shaft were produced as a single MIM part with significant cost savings. AMT received an innovation award from the European Powder Metallurgy Association (EPMA) for this application in 2003 (Fig. 11).

The company helped to overcome an engineering challenge when it designed a thin-wall (<1mm or 0.04 in.) intricate fibre optic transceiver housing made from 17-4 PH stainless steel used in a parallel optical module for ultra-high-speed transceivers in networking and telecom equipment. In addition to the very thin walls the MIM housing has four thin legs supporting two parallel strips (Fig. 12).

Another MIM part, manufactured from 17-4 PH stainless steel, is a connector which serves as an external connector to a high-performance fibre-optic module (Fig. 13). The company states that the one-piece MIM design would have been virtually impossible to produce by any other manufacturing process. Secondary operations on this part are limited to coining on the two latches and gold plating.

A further cost-saving electronics related application developed by AMT involved using an Fe-Co-Ni alloy to

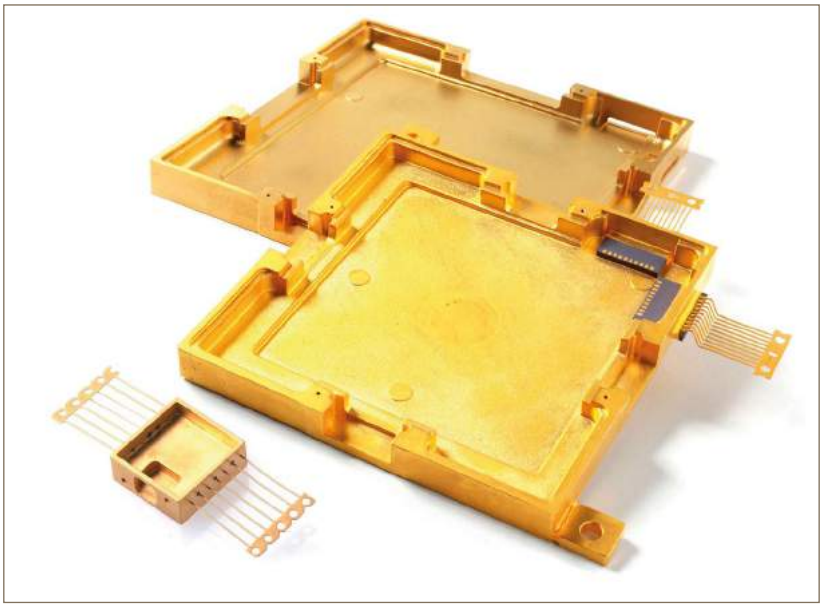

Fig. 14 AMT uses an Fe-Co-Ni alloy to produce this complex multi-port housing used in fibre-optic networks

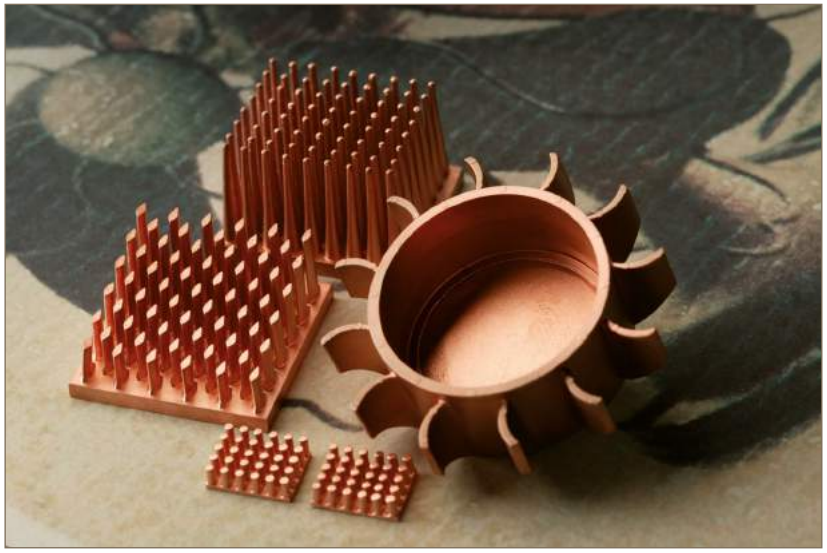

Fig. 15 Heat sinks produced by AMT's cuMIM™ technology have thermal conductivities comparable to copper barstock

Fig. 16 Complex shaped MIM electrical connectors used in a plug and adaptor for household appliances using cuMIM™ technology

Fig. 17 Hinge assembly of two MIM parts – a shaft and a plate – used on a mobile phone

produce a multi-port housing also used in fibre-optic networks (Fig. 14). The housing is required to give a 20-year minimum service life under extreme conditions.

In 2004 the company further expanded its horizons by developing MIM heat sinks for the rapidly growing thermal management market, and also introduced ceramic injection moulding for fuel cell applications. The need for high thermal conductivities led AMT to develop new tailored MIM materials based on pure copper (cuMIM) and copper-tungsten (wcuMIM). These materials, especially W-Cu containing 15wt% Cu, allowed the company to produce complex geometries with high thermal conductivity and thermal expansion coefficients suitable for many packaging and heat sink applications. Examples of cuMIM and wcuMIM parts are shown in Fig. 15.

AMT used the know-how developed with its cuMIM technology to produce three complex shape electrical connectors used in a plug and adaptor for the household appliance sector. The plug is designed make direct contact with the power track and can be engaged anywhere along the track. The cover of the plug is made from polycarbonate (Fig. 16). The company received a Grand Prize for this application from the MPIF in 2005.

A major surge in production at AMT came in 2005 and 2006 when the company succeeded in designing a number of intricate MIM parts for mobile phone handsets. One set of MIM parts involved a hinge assembly of two parts, a shaft and plate, which allowed the lid of the phone to be flipped open and then twisted by 180°, giving users a swivel screen on their mobile phones that facilitated the use of both the phone and its built-in camera (Fig. 17). The company received a 2005 EPMA award for this development.

A year later the company announced the production of MIM flip slider and hinge barrel parts for the Motorola PEBL phone. These parts made up the dual-hinge opening mechanism on the phone (Fig. 18). These complex thin wall 17-4 PH stainless steel parts have overhanging structures and a 3D design. Producing these by machining would have cost five times as much.

It was the fluctuating demand for mobile phone components and the phasing out of some phone designs which had a drastic impact on sales in 2007. This led to a decision by the company to focus on other end-user areas for future growth and to become less reliant on the consumer electronics sector. This led to the development of new proprietary MIM processes which allow the incorporation of features such as undercuts and

'new proprietary MIM processes allow the incorporation of features such as undercuts and internal channels into MIM and CIM components'

Fig. 18 MIM flip slider and hinge barrel parts which made up the dual-hinge opening mechanism on a Motorola mobile phone

internal channels into MIM and CIM components, and also the injection moulding of bi-material components. AMT has been successful in co-injection of two different materials to achieve different properties in a single component including magnetic/non-magnetic (Fig. 19), hard/soft, or high cost/low cost materials.

AMT developed and patented its In-Coring™ technology to produce MIM and CIM parts with undercuts features or internal channels (Fig. 20). This essentially involves a two-step process whereby a plastic part is injection moulded and this part is then placed as an insert into a second mould which receives another shot of MIM or CIM feed-stock. For PIM components with complex undercut features the process can be adopted using standard moulding machines and moulds. The plastic insert material onto which the PIM feedstock is moulded needs to stay rigid during the process, and should be easily removed from the green part in order to reveal the undercut or internal features. The plastic insert is removed either by solvent debinding or thermal debinding or a combination of both depending on the binder used.

Ceramic Injection Moulding and other materials

AMT has successfully applied its expertise in MIM technology to the production of alumina, zirconia and spinel ceramic components. One application is the manufacture of high quality, precision ceramics – both dense and porous – with customised thermal expansion properties for portable and stationary fuel cells used in the energy sector (Fig. 21). It has also developed a number of intricate shaped CIM parts from coloured ceramics.

Other high performance materials used by AMT include nickel-base superalloys for high temperature, high performance applications such as aerospace components, anti-corrosive components used in the oil and gas industry, and martensitic (high hardness) SUS 440C stainless steel for applications such as locking devices. The SUS 440C stainless steels produced by MIM are reported to have high strength (ultimate tensile strength of 1665 MPa) and hardness (55HRC) when sintered to a density of 7.54 g/cm^3 using water atomised powders of 10 micron particle size.

Outlook

AMT has notched up an impressive list of achievements in its first 20 years of PIM production with a growing portfolio of innovations and patents. David Lau, General Manager, is confident that ongoing projects will result in continuing growth in the years ahead. "With our track record of delivering what we promise to our customers as well as our capability of using MIM technology for innovative applications, we are well placed to benefit from the recovering economy. We remain focused on delivering cost-effective design and manufacturing solutions for all the sectors we serve", he said.

Fig. 19 AMT is capable of co-injection two different types of materials such as the non-magnetic disk and the magnetic shaft on this MIM part

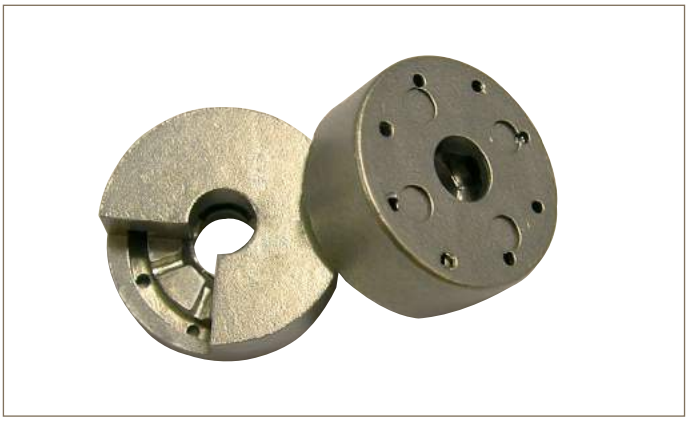

Fig. 20 AMT's 'In-Coring™' makes possible the production of MIM parts with undercuts and internal channels

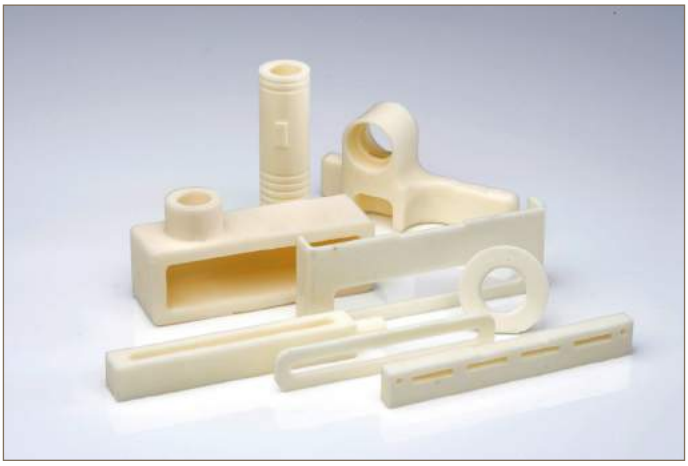

Fig. 21 Ceramic injection moulded parts for fuel cell applications. AMT is also producing complex coloured CIM parts

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USA

Innovation in different segments of powder injection molding (PIM) is responsible for the rapid growth of this field. The PIM industry (MIM—metal injection molding; CIM—ceramic injection molding; and CCIM—cemented carbide injection molding) has estimated sales of over $1 billion and could possibly double in a span of five years.

The objective of the conference is to explore the innovations and latest accomplishments in the areas of part design, tooling, molding, debinding, and sintering of PIM parts. The conference will also focus on the developments in PIM processing of different materials including metals and alloys, ceramics, and hardmaterials.

This specialized conference is sponsored by the Metal Injection Molding Association, a trade association of the Metal Powder Industries Federation, and its affiliate APMI International. The conference is targeted at product designers, engineers, consumers, manufacturers, researchers, educators, and students. All individuals with an interest in the application of powder injection molding will be encouraged to attend.

A "Call for Presentations" is being issued to solicit contributions for the technical program. The focus of the technical program is "**Manufacturing Best Practices.**" All submissions will be considered. All conference PowerPoint presentations will be distributed to conference registrants.

The following topics will be considered:

PROCESSING
- Computer aided engineering
- Consolidation technologies
- Debinding technology & equip.
- Micromolding
- Microstructural control
- Mixing science & machines
- Molding machines & technology
- Novel processing
- Part design considerations
- PIM tooling
- Powders/binders/feedstocks
- Secondary operations
- Setter technology
- Sintering furnaces

MATERIALS
- Alumina/AlN/alumina-silica
- Cermets
- Cobalt and Co-base alloys
- Copper and Cu-base alloys
- Ferrites
- Ferrous systems
- Magnetic materials
- Nanoscale materials
- Nickel and nickel-based alloys
- Novel materials & alloy systems
- Porcelains
- Precious metals
- Silicon carbides/silicon nitrides
- Stainless steels
- Titanium and Ti-based alloys
- Tool steels
- Tungsten carbide & alloys
- Zirconia

APPLICATIONS
- Aerospace
- Automotive
- Business machines
- Chemical processing equipment
- Computer hardware
- Consumer products/appliances
- Cutting tools
- Electronic packaging
- Heat exchangers
- High-temperature components
- Industrial applications
- Jewelry and watches
- Magnetics
- Medical and surgical instruments
- Novel applications
- Oil and gas
- Sporting equipment
- Thermal management

Abstract Submission Deadline: August 31, 2010

Comments or questions regarding abstract submission or conference should be directed to:

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The processing of biomaterials for implant applications by Powder Injection Moulding

Powder Injection Moulding is an established process for the manufacture of medical products from biocompatible materials. Processing innovations have, however, enabled a wider range of functional products to be considered than ever before. Philipp Imgrund and colleagues from IFAM, Bremen, Germany, present current work that includes degradable PIM implants, surface structured PIM products for implant applications, and implants with controlled porosity. The challenges of series production are also addressed.

The variety of materials and complexity of components makes powder injection moulding (PIM) attractive for the production of medical products and implants. For commercial processing of biocompatible materials (e.g. metals: 316L stainless steel, Co-Cr-based alloys, titanium alloys; or ceramics: alumina, zirconia) metal injection moulding (MIM) and ceramic injection moulding (CIM) are already successfully established.

In recent years the scope of powder injection moulding technology has expanded from standard powder based materials to a considerable range of functional materials and composites for medical use. Besides mechanical stability, they need to provide specific medical features such as bioactivity, degradability, or porosity and surface structure. This article presents current developments in the processing of biodegradable composites, the powder injection moulding of implant materials and the production of implants with defined surface structures while keeping tight processing tolerances.

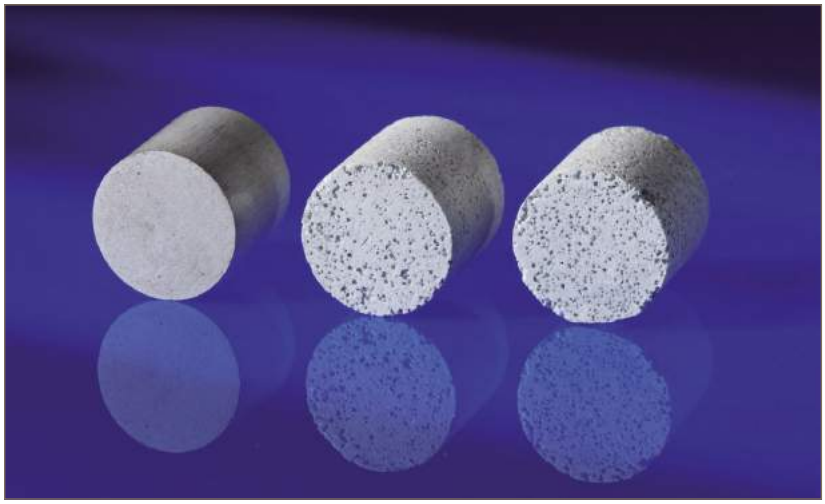

Fig. 1 PLA / HA composites with different porosity

Biodegradable composites

Following injuries such as bone fracture or damage, a replacement for the destroyed material might be necessary for the successful recovery of the patient. Currently, autografting and allografting methods are used to help repair these types of injuries. For the autografting procedure bone material of the wounded patient, mainly taken from the iliac crest, is placed into the injured region. Besides the limited amount of replacement material and the costs, this method causes pain for the patient due to at least two required operations.

By using the allografting method bone tissues from other natural sources can be used to fill the trauma. This approach, however, always carries the risk of disease transmission and incompatibility [1]. Therefore a bioresorbable material is preferred for integration into the human body that can be biodegraded and replaced by the body's own tissue after a certain time.

Through the combination of both manufacturing process and materials development, Fraunhofer IFAM has started the development of bioresorbable materials for the production of components with and without defined porosity, as illustrated in Fig. 1.

Bone consists of organic and inorganic components. The inorganic phase is a calcium phosphate based mineral and the organic phase is built by different proteins. Therefore in an initial step, a composite made of

Fig. 2 Interference screws made by injection moulding of PLA (left), hydroxyl apatite (centre), and 316L stainless steel (right)

hydroxylapatite powder (HA, a representative of the material class calcium phosphate) as the inorganic part and the biocompatible and bioresorbable polylactic acid (PLA), one of the most often used biodegradable synthetic polymers [2], assuming the task of the organic part, was prepared using a suitable mixing procedure. Both materials were compounded to obtain a powder of HA particles covered with a thin PLA layer.

The obtained composite granulate was then processed by pressing and then sintering the PLA phase. To improve the potential biological integration of the material into the bone, the described process was enhanced to manufacture components with defined porosity by integrating salt as a space holder during material preparation. The salt is washed out of the sample after pressing and the material is sintered to obtain a defined porosity, depending on the amount and size of the space holder.

Regarding dense composites, it was possible to produce materials with a HA content up to 70% by volume fraction of the solid phase. By applying the space holder method it was possible to produce components with porosity up to 52 % by volume (Fig. 1). Further work is envisaged to make the material suitable for injection moulding, targeting high HA shares of up to 65 vol% for enhanced bioactivity and osteointegration compared to today's commercial PLA / HA composites.

'Using a blend consisting of 33 vol% master alloy, 33 vol% carbonyl iron powder and 33 vol% nano iron powder, it was possible to replicate and sinter 5 µm structures'

Injection moulding of PLA, HA and metal implants

The versatility of injection moulding processes related to material choice is a well known advantage, especially considering its potential for the processing of metals and ceramics. For instance a range of medical devices made of 316L stainless steel, titanium, alumina or zirconia processed by PIM are commercially available.

One promising application for the technology related to implants is the manufacturing of bone screws. Commercial products are generally made of Titanium and sometimes stainless steel. The common manufacturing route is, similar to most metal implants, machining from bulk material, which remains a complex and material consuming process.

More recently, biodegradable polymers (PLA) and PLA based composites which can be processed by injection moulding are being chosen as alternatives to metal screws where appropriate. As the material is resorbed by the human body over time, the number of surgeries to extract screws after healing can be significantly reduced. For further enhanced bioactivity, commercial PLA / HA based composites (HA content up to max. 40%) are also on the market.

In recent developments IFAM set up a suitable MIM process for manufacturing 316L and titanium interference screws for ligament repair. Here MIM technology may help in decreasing material consumption and cost. All features of the screws including a hexagon bore can be moulded and sintered without post processing. In the next technology step, a feedstock of pure HA powder in combination with an IFAM binder system was prepared.

The material proved readily mouldable and bulk HA screws were obtained following sintering. Further steps will be powder mixture optimisation to obtain enhanced osteoconductivity and mechanical properties. However, in applications such as interference screws, compression forces mainly apply, so the ceramic based material would be highly appropriate for this application. Fig. 2 shows interference screws made of PLA, HA and 316L stainless steel as processed by IFAM.

Bioactive surfaces by Micro MIM

In addition to the chemical and physical characteristics, the biocompatibility of an implant is mainly influenced by the topography of its surface. Specific microstructuring such as defined roughness and/or precise structures can improve the biological integration and enhanced immediate post-operative implant fixation and long term biomechanical stability [3, 4].

Today, implant surfaces are functionalised by etching, blasting or coating technologies. However,

investigations prove that not only does an increase in surface area influence cell function and organisation, but also the type and arrangement of the structure. For example, hemispheres with a diameter between 5 and 50 µm which are hexagonally arranged show most promising cell migration and differentiation results [5]. However, in industrial manufacturing the post-processing of implant surfaces by etching or sandblasting leads to the desired rough surfaces but not to a regular micro-pattern.

Since the µ-MIM process is well-known for the production of near net shape, functionalised micro parts and microstructured surfaces with high accuracy [6, 7] the process was further developed for manufacturing implant surfaces made of biocompatible metals with defined micro- and sub-microstructures. 316L stainless steel, a biocompatible alloy, was used to replicate surfaces structured with hexagonally arranged hemispheres having a diameter of 50, 30, and 5 µm and 20 µm interspacing, see Fig. 3a. In Fig. 3b the structured surface carrying 50 µm hemispheres is documented by means of SEM.

For the generation of surfaces with regular micro pattern which are overlaid by sub-microstructures, the 316L was modified by preparing a blend of the master alloy powder CrNiMo 55-38-7 (d_{50} = 4.0 µm) with ultra fine iron particles (d_{50} = 1.4 µm) and nano iron particles (d_{50} = 17 nm), whereas the 316L composition was formed during the sintering procedure.

The development of the mixing process was a challenge due to the reduced powder particle size and the rising volume to surface ratio. These points lead to an increasing amount of binder needed to prepare a homogeneous and mouldable feedstock. Due to the increased risk of oxidation or even ignition of the nano iron powder, the blending process of the metal powder and the binder components was arranged under argon atmosphere in a planetary mixer installed in a glove box.

During optimisation of the manufacturing process it was established that higher feedstock and mould temperatures and higher injection pressures are required for processing of the material filled with nanoparticles. The structured stainless steel surfaces were produced with a varying amount of the different iron particles in the feedstock mixture. Using a blend consisting of 33 vol% master alloy, 33 vol% carbonyl iron powder and 33 vol% nano iron powder, it was possible to replicate and sinter 5 µm structures (Fig. 4). Such fine replication has not been reported for a µ-MIM process before.

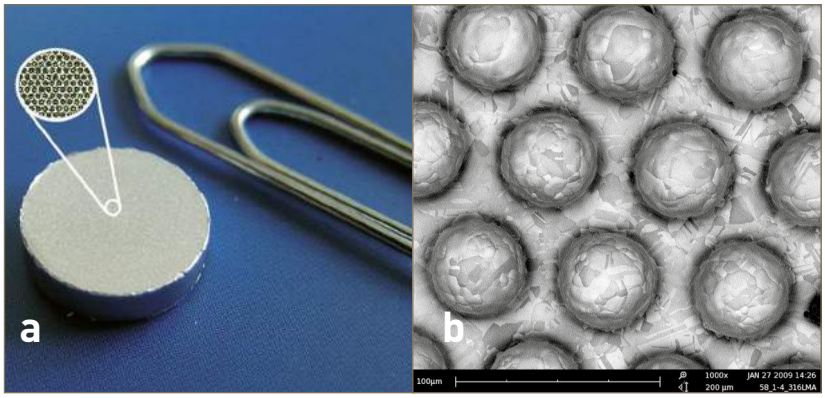

Fig. 3 a) Image of a sample with a hemispheric surface micro pattern; b) SEM documentation of surface with 50 µm hemispheres

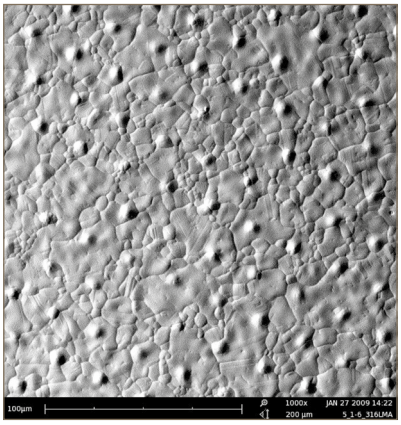

Fig. 4 Hemispheres produced via µ-MIM with a diameter of 5 µm and interspacing of 20 µm

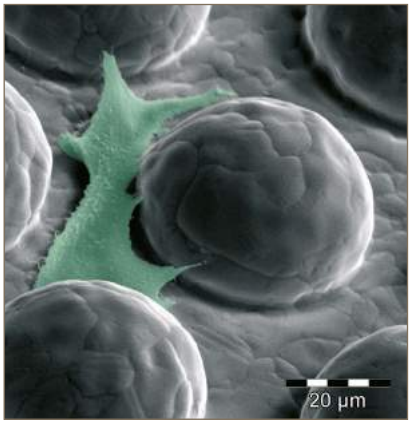

Fig. 5 SEM documentation of an osteoblast cell seeded on the surface patterned by µ-MIM

Furthermore, due to significant grain refinement induced by the incorporation of nano particles, the mechanical properties of the sintered parts were in the range of conventionally produced stainless steel (Yield strength > 200 MPa, Tensile strength > 500 MPa), whereby the finer grain microstructures lead to a significantly increased yield and ultimate tensile strength of 530 MPa and 730 MPa, respectively.

The project partner, EMPA Materials Science and Technology, St. Gallen, Switzerland, performed first tests of biocompatibility and cell culture tests. Biocompatibility of all 316L samples was confirmed independent of the nano powder content. Cell culture tests indicate that the hemispheres significantly enhanced osteoblast adhesion and mobility. Fig. 5 shows such a cell seeded on the micro structured sample.

Towards production of micro implants

In order to demonstrate the potential of µPIM regarding precision and reproducibility the smallest bone in the human ear, the stapes, was replicated under series production conditions. Design and manufacturing of the micro injection mould was performed by Kraemer Engineering, Rendsburg, Germany. Initial process setup was conducted using ultrafine stainless steel powder, as presented in Fig. 6.

Since biocompatible materials are required for medical implants the investigations were determined with titanium. A titanium grade 1 powder with d_{50} about 15 µm was used for the Titanium feedstock preparation. In comparison to conventional PIM the amount of backbone polymer inside the binder system was increased from approx. 25 % to 50 % to ensure the replication of the small component structures. Because of the higher polymer content, feedstock and mould temperatures had to be raised approximately 25°C during the injection moulding process, which

Fig. 6 Injection moulded stapes made of stainless steel

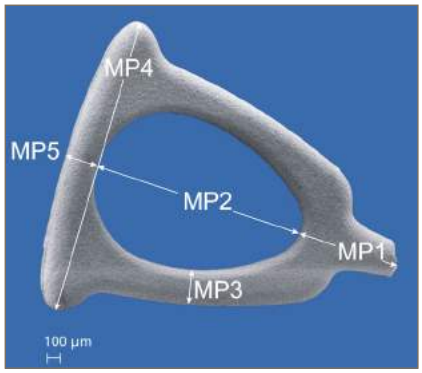

Fig. 7 Measurement positions for determining the tolerances of the titanium stapes

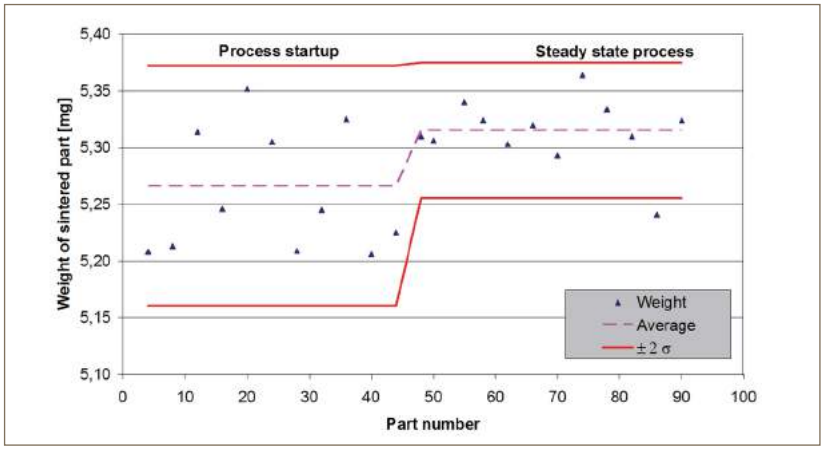

Fig. 8 Weight measurements of 90 titanium stapes

	MP1	MP2	MP3	MP4	MP5	Weight [mg]
Average length [µm]	714	1507	287	2284	263	5.31
Standard deviation [µm]	94	13	5	10	9	0,03
Standard deviation [%]	13.1	0.9	3.3	0.4	3.3	0.6

Table 1 Characteristics of the sintered titanium stapes at steady state conditions

was undertaken on a conventional micro injection moulding machine. Relevant data such as cycle time, mould pressure and volume per shot were recorded during series production.

In order to determine the tolerances of the titanium stapes 90 sintered samples were examined with respect to weight of the part and size variation at five different fixed distances (MP1-MP5, Fig. 7). The weight was measured with a precision scale from Sartorius, whereas the length measurements were performed with an optical measurement system produced by Gerwah Mikrotechnik.

As an example, the weight measurements of the 90 titanium stapes that were examined are shown in Fig. 8. It was observed that the variation of the first 50 shots was broader than the following 40 cycles. The length measurements show the same behaviour. A steady state condition is obtained after 50 cycles indicating that the system needs a starting phase before components with tight tolerances are produced.

The weight tolerances and the length tolerances of all measurement points under steady process conditions are summarised in Table 1.

Considering the thin sections of MP3 and MP5 especially, tight tolerances of maximum 3.3 % were obtained. The MIM Expertenkreis sectoral group, for instance, has suggested tolerances of ± 50 µm for MIM parts with wall thickness less than 3 mm [8]. Except for MP1 all determined tolerances are significantly better. The broader tolerance range of MP1 can be attributed to the different breaking points of the gates, leading to two different length scales and a bimodal distribution of the measurement points [9].

Conclusion

Powder injection moulding is a versatile manufacturing process for medical applications. Biocompatible materials such as ceramics, polymers, metals and composites can be processed regardless if they are modified with particles or filled with space holders. Furthermore, the development of µ-PIM allows moulding of bioactive surfaces with structures around 5 µm and manufacturing of implants with tight tolerances in a commercial series process.

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New methodology of resonant inspection applied to Metal Injection Moulded parts

Metal injection moulded parts are widely used in a number of safety-critical applications, from medical instruments through to automotive, firearms and aerospace products. Richard W. Bono and colleagues from The Modal Shop Inc. explain how the 'Resonant Acoustic Method' of Non-Destructive Testing (RAM NDT) has been adapted to provide MIM producers with an effective in-line system to check 100% of parts for cracks and other flaws.

Resonant Inspection (RI) is commonly used for quality assurance testing of powder metal components, providing a volumetric whole body inspection, sensitive to both external and internal structural flaws or anomalies. This technique measures a metal component's mechanical resonances and compares an individual part's signature to a template generated from a statistically significant sample space of 'good' parts.

Traditionally it has been limited to small to medium components with sizes ranging from about a 'dime to a dinner plate' given the requirements to fixture and excite the part effectively. However, using a new methodology that employs a drop testing fixture, very small components such as those commonly manufactured by powder metal MIM processes can be 100% inspected reliably, quickly and cost-effectively.

Resonant Acoustic Method (RAM NDT), a form of RI, is a non-destructive testing technique that evaluates structural integrity by striking a part with an impact and analysing its mechanical resonances from the acoustic ringing produced. This technique can be easily automated to eliminate human errors with fast throughput, providing cost effective 100% inspection with minimal disruption to production. With a large number of successes on the production lines of powder metal and cast parts, RAM NDT is the simple and effective solution to manufacturers' zero PPM challenge.

'very small components can be 100% inspected reliably, quickly and cost-effectively'

Traditional NDT techniques, for example magnetic particle or dye penetrant testing, focus on detecting and diagnosing defects. They use visual or imaging techniques that scan for indications of specific defects at specific locations. For production line quality inspection, identifying the type and/or location of defect is secondary to identifying the defective parts themselves. While diagnosing specific defects is applicable when evaluating and inspecting some systems, such as using ultrasonics to inspect gas

Powder Metal	Cast	Forged
Cracks	Cracks	Cracks
Chips	Cold shuts	Double strikes
Voids	Nodularity	Porosity
Hardness	Porosity	Hardness
Inclusions	Hardness	Inclusions
Heat treatment	Inclusions	Heat treatment
Decarb	Heat treatment	Quenching
Oxides	Stresses	Laps
Contaminants	Contaminants	Contaminants
Missed ops	Missed ops	Missed ops

Table 1 Typical structural defects commonly detectable by resonant inspection technique for powder metal, cast and forged processes

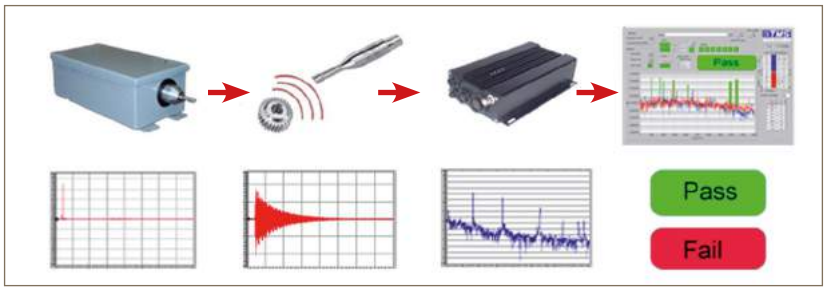


Fig. 1 NDT RAM process flow

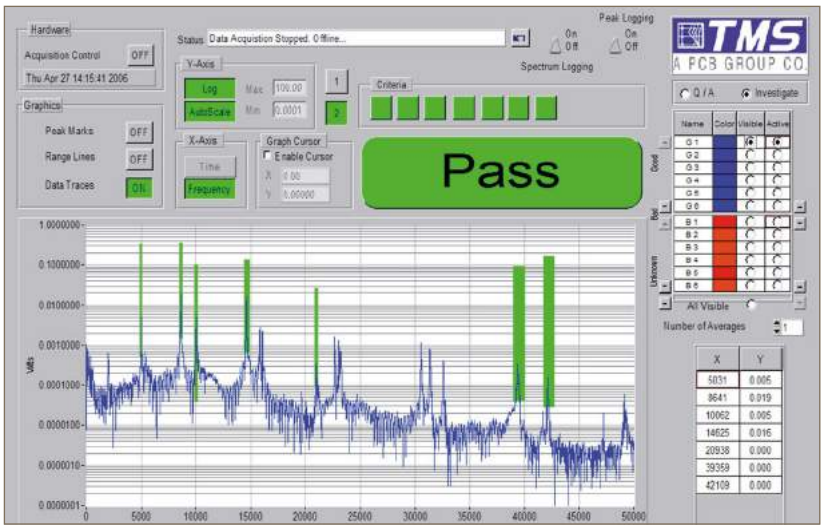


Fig. 2 Typical acoustic signature, power spectrum to 50 kHz, for a metal part, shown as processed by NDT RAM with criteria ranges (indicated with green "boxes") at seven natural resonant frequencies

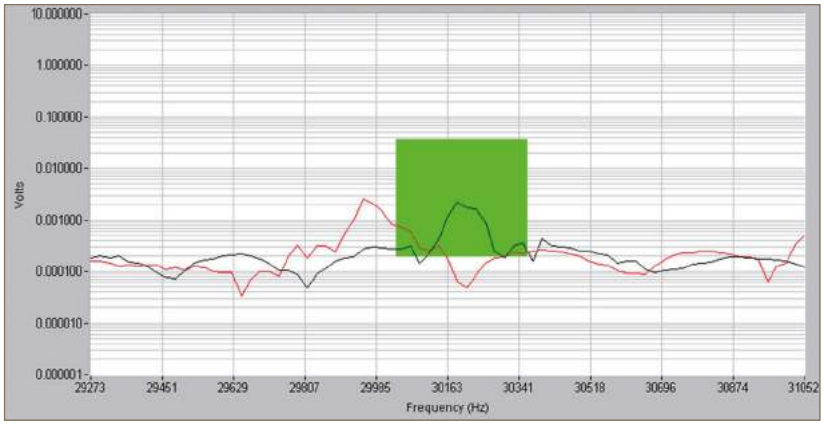


Fig. 3 Data showing a typical frequency shift detected by NDT-RAM

pipelines, it is not appropriate for high volume 100% inspection of manufactured metal parts. For these production lines it is of primary importance to detect if a part is non-conforming rather than why. Therefore, an end-of-line 'go/no go' objective inspection, such as by RAM NDT, which provides a reliable 100% sort, is preferred to a subjective diagnosis, perhaps useful for defect root cause analysis.

Resonant Inspection (RI) measures the structural response of a part and evaluates it against the statistical variation from a control set of good parts to screen defects. Its volumetric approach tests the whole part, sensitive to both external and internal structural flaws or deviations, providing objective and quantitative results. This structural response is a unique and measurable signature, defined by a component's mechanical resonances. These resonances are a function of part geometry and material properties and are the basis for RI techniques.

By measuring the resonances of a part, one determines the structural characteristics of that part in a single test. Typical flaws and defects that can adversely affect the structural characteristics of a part are given in Table 1 for powdered metal, cast and forged applications. Many of the traditional NDT techniques can detect these flaws as well, but often only RI can detect all in a single test, throughout the entire part (including deep sub-surface defects), in an automated and objective fashion.

After defective parts have been sorted with RI, complimentary traditional NDT techniques may provide a means for subjective diagnosis on the smaller subset of 'rejected' parts. This is useful for determining a defect's root cause and ultimately improving the production processes.

Science of resonant inspection

Modal analysis is defined as the study of the dynamic characteristics of a mechanical structure or system. All structures, even structures such as metal gears or similar parts that are apparently rigid to the human eye, undergo elastic deformation as a result of applied forces. The structure itself deforms in a distinct, specific pattern. This structural dynamic behaviour is defined by the mass, stiffness and damping of a given part's material properties and geometry. Specifically, all structures have mechanical resonances, where the structure itself amplifies any energy imparted to it at certain frequencies. For example, tuning forks or bells will vibrate at very specific frequencies, their natural frequencies, for relatively long periods of time with just a small tap. The sound that is generated is directly due to these natural frequencies. In fact, any noise made by a structure is done so by its vibration. RAM NDT utilises this structural dynamic behaviour to evaluate the integrity and consistency of parts.

The natural frequencies are global properties of a given structure and the presence of structural defects causes shifts in some or all of these resonances depending upon how the flaw interacts with the specific deformation pattern. For example, a crack will change the stiffness in the region near the crack and a variation

in density or the presence of porosity will change the mass. The resulting frequency shifts are measurable if the defect is structurally significant with respect to either the size or location of the flaw within a specific resonance mode shape. With some defects, a shift in resonant frequency can also be noticed audibly, such as a cracked bell that obviously does not ring true.

Practical application of resonant acoustic method

The Resonant Acoustic Method technique performs resonant inspection by impacting a part and 'listening' to its acoustic spectral signature with a microphone as shown in Fig. 1. The controlled impact provides broadband input energy to excite the part and the microphone allows for a non-contact measurement of the part's structural response. The part's mechanical resonances amplify the broadband input energy at its specific natural frequencies, indicated as peaks in the frequency spectrum (shown below the 'black box' signal processor in Fig. 1), measured by the microphone above the background noise in the test environment. 'Good' parts (structurally sound) have consistent spectral signatures (i.e. the mechanical resonances are the same among part samples) while 'bad' parts (structurally different) are different. Deviations in peak frequencies or amplitudes constitute a structurally significant difference that provides a quantitative and objective part rejection. NDT RAM processes the individual spectra, evaluating these changes compared to a baseline template for the given part. The results are displayed on the industrial PC workstation, with the pass/fail decision communicated to the system PLC. An enlarged display of a typical spectrum from 0 to 50 kHz is given in Fig. 2 and a zoomed display showing a typical frequency shift is given in Fig. 3.

Resonant acoustic method applied to small MIM parts

As shown previously in Fig. 1, RAM NDT has typically utilised a mechanical impactor to provide an impulsive force into the part specimen. However, this impactor can be ineffective for smaller parts, such as small MIM parts commonly used within medical devices as shown in Fig. 4. Given the obvious need for 100% inspection of components used within medical instruments to ensure quality, a new drop test fixture design allows for the application of RAM NDT with a reliable excitation technique and sorting mechanism for very small powder metal parts.

The drop test fixture design, shown in Fig. 5, uses gravity instead of an electromechanical impactor to generate the impulse force required for RAM NDT. Small MIM parts can be collected manually or with a bowl feeder and dropped through a tube directing the part into a chamber where it impacts the surface of a piezoelectric force transducer. This triggers the measurement of the part's acoustic resonant frequency signature by the microphone before the specimen comes to rest in a collector at the bottom of the chamber. Depending on the pass/fail result the collector sorts the "good" parts from the "bad" by rotating one direction or the other.

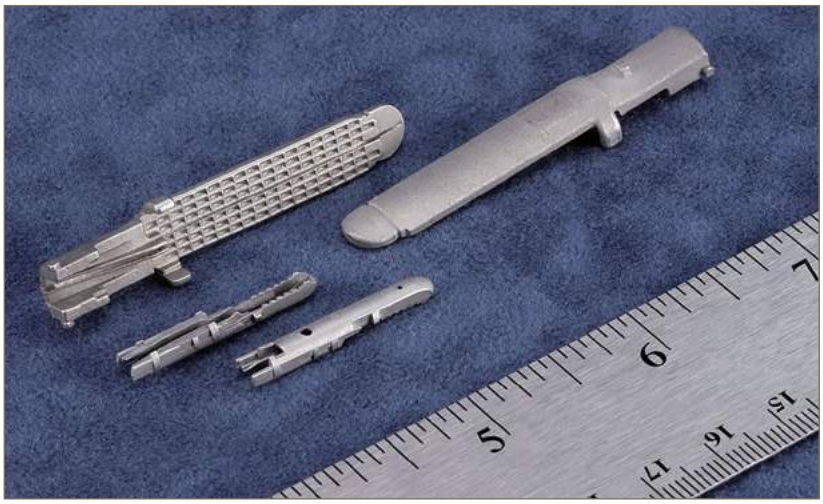


Fig. 4 Small MIM parts used in medical instruments

Fig. 5 NDT DTF Drop Test Fixture with LanSharc data processor and laptop controller

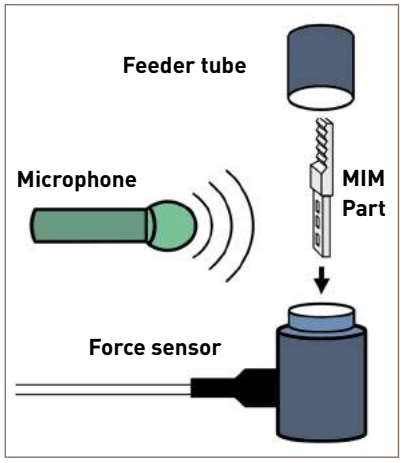


Fig. 6 Part dropping on force sensor

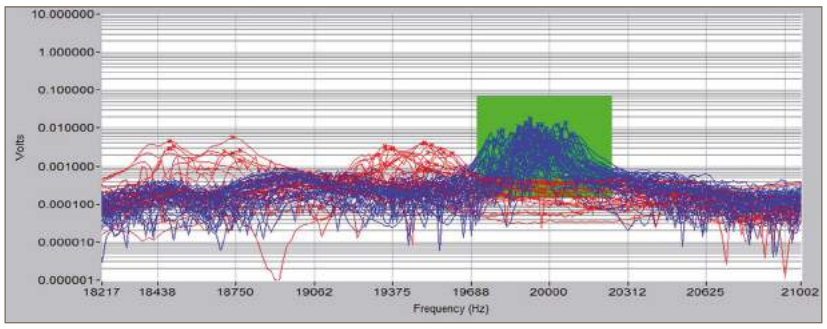
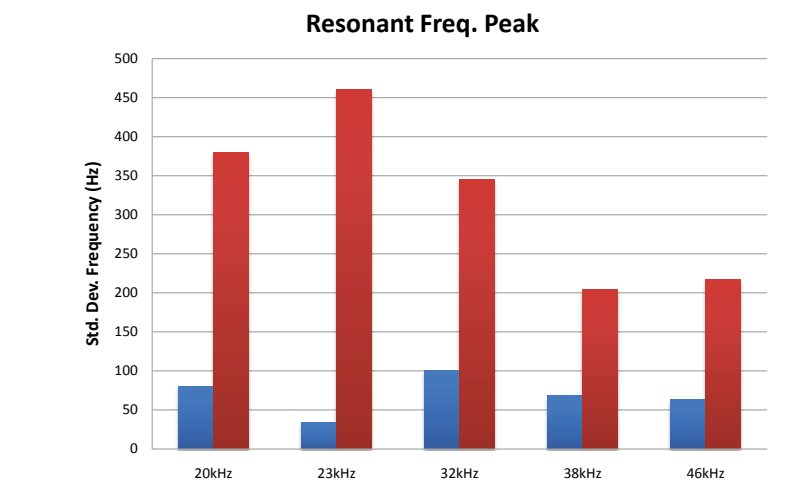


Fig. 7 Data from multiple MIM parts overlaid at approximately 20kHz



	20kHz	23kHz	32kHz	38kHz	46kHz
Good Part Standard Deviation	79.8	34.3	100.6	68	63.5
Bad Part Standard Deviation	379.5	460	345.6	203.9	216.5

Fig. 8 Standard Deviation of all parts at each of the five frequency ranges

Case study with experimental results

Fig. 6 shows an example of a MIM medical component used for clamping/grasping. The manufacturer was experiencing flaws typical to MIM parts: cracks at various locations, missing features, broken teeth, and internal porosity. Their current part inspection method was 100% subjective visual inspection, magnified via a microscope to check the part for cracks and other flaw indications. This method proved less than 100% effective on external cracks and had no means of inspecting internal voids or porosity.

With RAM NDT and the drop test fixture, the manufacturer is able to perform objective 100% automated inspection and sorting of the parts. Using a vibratory bowl-feeder to ensure that each part is in the correct orientation and proper part spacing is maintained, the part drops into a feeder tube and strikes into a force sensor. This triggers data acquisition; the microphone measures the response and the system processes the frequency spectrum. NDT RAM compares the specimen's resonant frequency peaks with statistically derived sorting criteria. These criteria contain frequency and amplitude limits for the given part type, indicated by green boxes as shown in Figs 2 and 3.

All criteria are established by experimentally testing a statistically significant number of good and flawed parts to determine which frequencies shift due to structural defects. These criteria represent a part's minimum and maximum acceptable values in frequency and amplitude, indicating significant changes in structural properties such as density, mass, stiffness and dimensions.

'The "good" parts have consistent resonant frequency characteristics clustered at the expected frequencies'

Fig. 7 shows the resonant frequency peaks measured from a large number of specimens at approximately 20 kHz. The blue traces are acceptable parts and the red traces are parts that have been proven defective. The "good" parts have consistent resonant frequency characteristics clustered at the expected frequencies. Parts with structural defects exhibit different resonant frequencies across samples that are inconsistent with expected values. These figures clearly show that structural defects cause a wide spread of resonant frequency shifts compared with the good parts. This wide range is caused by both external flaws (cracks, chips, missing features) and internal flaws (porosity, voids, inclusions, variations in densities) of varying degrees. The level or size of a flaw will directly reflect the amount of resonant frequency shift or movement.

Fig. 8 graphically displays the standard deviation of the all of the tested resonant frequencies for both the good parts and the defective parts. This illustrates the wide range of frequency shifts that accompany structural defects in parts compared with the very consistent resonant frequency peaks measured across good samples.

Conclusion

The Resonant Acoustic Method with the drop test fixture for small part inspection offers MIM manufacturers a fast, objective, whole body test that can be configured quickly to work on any number of small parts. 100% testing with RAM NDT removes the need for time consuming, subjective human visual inspection.

The benefit of removing doubt on whether shipped parts are defect free allows Plant Managers, Quality Managers, and Process Engineers more time to concentrate on high value projects without worrying about individual part quality contaminating production shipments. RAM NDT also saves substantial costs as compared with 100% visual inspection labour.

Additionally, fully inspected parts reduce liability and quality related expenses like external sorting, product recalls, and customer initiated penalties that can easily run in the tens of thousands of dollars per occurrence.

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Powder Metallurgy - A Global Market Review

Essential information for anyone with an interest in the current state of the global PM industry

'Powder Metallurgy - A Global Market Review' presents key statistical data highlighting the current state of the PM industry including both part and powder production.

The report includes detailed sector reviews for PM materials covering Ferrous PM, High Alloy PM Steels, Copper and Copper-base, Hardmetals/Cemented Carbides, Diamond Tools, Refractory Metals and Powder-Based Magnets.

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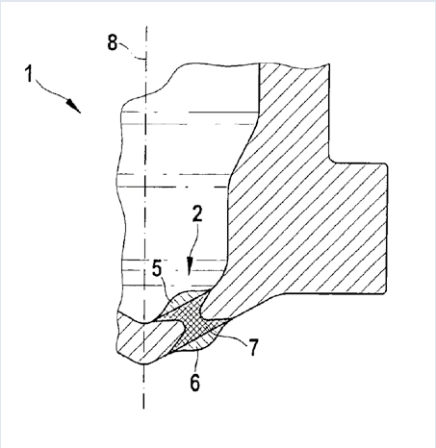
Global PIM Patents

The following abstracts of PIM-related patents have been derived from the European Patent Organisation databases of patents from throughout the world. Full information on individual patents (in the language of the country) is available through the *PIM International* editorial office.

CN101100378 (A)
MANUFACTURE OF ZIRCONIUM OXIDE CERAMIC PRODUCTS
Publication date: 2008-01-09
Inventor(s): L. Shunlu; et al, Hunan Hualian Special Ceramics Co., Ltd, China.
A method for making domestic porcelains such as cups, plates, bowls, dishes, forks, spoons and tea wares, by using zirconium oxide as the raw material. The production procedures involve high density zirconium oxide powder mixed with an organic binder (polypropylene, paraffin wax or the like). Then heated to 160-180°C for blending, fed into injection moulding machine (into the mould) under 150-200°C and 80-100mpa, finally cooled and demoulded to obtain the semi-finished products. The wax is removed followed by sintering to produce final products with high strength and high toughness.

CA2660484 (A1)
METHOD OF INJECTION MOULDING ALUMINIUM OR AN ALUMINIUM ALLOY
Publication date: 2008-02-14
Inventor(s): G. B. Schaffer, et al, University Of Queensland, Austrailia
The method comprises the steps of forming a mixture containing an aluminium powder or an aluminium alloy powder, or both, and optionally ceramic particles, a binder, and a sintering aid comprising of a low melting point metal. The mixture is injection moulded and the binder is removed to form a green body. The green body is sintered. The sintering is conducted in an atmosphere containing nitrogen and in the presence of an oxygen getter.

WO2008003660 (A1)
METAL POWDER INJECTION-MOULDING SECONDARY RESHAPING PROCESS
Publication date: 2008-01-10
Inventor: D. Holz , Robert Bosch GmbH, Germany
The invention relates to the production of a component by a metal powder injection-moulding process, wherein after sintering a reshaping step of a defined location of the component is performed to achieve local compression.



WO2008021508 (A2)
INJECTION MOULDING OF CERAMIC ELEMENTS
Publication date: 2008-02-21
Inventor(s): S. Annavarapu et al, Saint Gobain Ceramics, USA
New methods are provided for the manufacture of ceramic elements that include injection molding of two, three or more distinct ceramic layers or regions that form the element. Ceramic elements also are provided that are obtainable from fabrication methods of the invention.

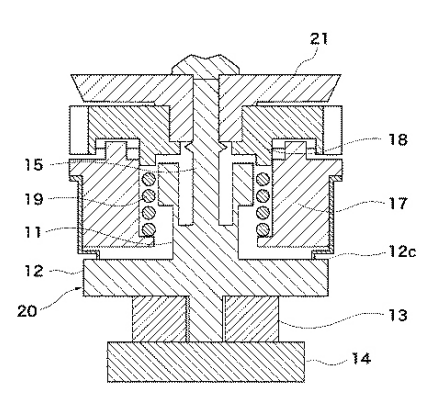
US2008075619 (A1)
METHOD FOR MAKING MOLYBDENUM PARTS USING METAL INJECTION MOULDING
Publication date: 2008-03-27
Inventor(s): L. Hosamani et al, USA
Molybdenum powder is mixed or blended with a binder to form a feedstock, which is injection moulded to form a green-state part. The green-state part is then sintered, such as by heating the part in a furnace for a predetermined period of time to effect consolidation and densification of the part. Desirably, the green-state part can be debound to remove at least a portion of the binder prior to sintering. In exemplary embodiments, sintering produces the final molybdenum article, and therefore the process does not require, but optionally may include, further processing of the sintered part, such as machining, cold-working, and/or hot-working

Kr20080032092 (a)
DEVICE AND METHOD FOR CONTINUOUSLY AND CATALYTICALLY REMOVING BINDER, WITH IMPROVED FLOW CONDITIONS
Publication date: 2008-04-14
Inventor(s): M. Bloemacher, et al, BASF SE, Germany
The invention relates to a device for continuously and catalytically removing binder from metallic and/or ceramic moulded bodies produced by pow-

der injection moulding. Comprising a binder-removing furnace, through which the moulded bodies pass in a direction of conveyance and in which they are brought to a suitable processing temperature; a conveying device, for introducing a process gas which is needed to remove the binder and contains a reaction partner; at least one device for introducing a protective gas into a reaction chamber of the binder-removing furnace, and a burner, for burning the gaseous reaction products that result from the binder removing process. One or more devices being included which lead to a targeted flow of the process gas in the device transversely to the direction of conveyance.

JP2008160989 (A)
ULTRASONIC MOTOR
Publication date: 2008-07-10
Inventor(s): H. Seki, Canon KK, Japan

Problem to be solved: to provide an ultrasonic motor that reduces cost, while reducing the vibration loss in each of the mating face of functional members. Solution: the ultrasonic motor is provided with a conversion device (13) for converting electrical energy into mechanical energy, and assembling members comprising a plurality of functional members (11, 12, 13, 14, and 15) incorporated into the conversion device (13). Three functional members (11, 12, and 13) of the assembly are respectively moulded by a metallic-powder injection moulding method.



JP2008133512 (A)
METHOD FOR PRODUCING HIGH DENSITY ALUMINIUM SINTERED MATERIAL BY METAL POWDER INJECTION MOLDING PROCESS
Publication date: 2008-06-12
Inventor(s): Kato Kiyotaka , Nat Inst Of Adv Ind & Technology, Japan

The moulding, which is formed in a prescribed shape by injection moulding of a compound composed of an aluminium powder and organic binder, is degreased and sintered to produce a sintered compact. Powder consisting mainly of aluminium powder with an average grain size of <35 [mu]m is used. A compound mixed with a silicon powder as a sintering assistant is used, improving the sinterability of the aluminium sintered compact without causing the reduction of its melting point by alloying. The high density aluminium sintered material has a relative density of at least 80%.

US2008147120 (A1)
METAL INJECTION MOULDING OF SPINAL FIXATION SYSTEMS COMPONENTS
Publication date: 2008-06-19
Inventor(s): F. Molz, H Trieu, USA

A method of making a component of a spinal fixation system. The method may comprise of providing a mixture of a powder of at least one metal or metal alloy and a polymeric binder. A metal-injection-moulding process may use the mixture to form a component for a spinal fixation system. The components may have varying flexibility across their cross-sections. Also, components are provided that are produced by this process, such as spinal fixation rods and plates.

KR20080055245 (A)
MANUFACTURING METHOD FOR PROSTHETICS
Publication date: 2008-06-19
Inventor(s): C. Hwang et al, Korea Ind Tech Inst, Korea

A prosthesis and a manufacturing method to manufacture a prosthesis by metal powder injection moulding to have good durability and good treatment effects. The manufacturing method of the prosthesis includes the steps of: (1) mixing metal powder, binder and polymer beads; (2) moulding the prosthesis by injecting the metal powder, binder and polymer beads into a mould which has a stem portion and a connection portion formed therein; (3) removing the binder from the moulded part; (4) removing the polymer by heating the part at a thermal decomposition temperature; and (5) sintering the part at a sintering temperature.

WO2008077776 (A2)
METHOD FOR THERMALLY DEBINDING A MOULDED METALLIC AND/OR CERAMIC BODY WHICH IS PRODUCED BY INJECTION MOULDING, EXTRUSION OR COMPRESSION USING A THERMOPLASTIC MATERIAL
Publication date: 2008-07-03
Inventor(s): J. MAAT et al, Germany

The invention relates to a method for thermally debinding a moulded metallic and/or ceramic body which is produced by injection moulding, extrusion or compression using a thermoplastic material, said material containing at least one polyoxymethylene homopolymer or copolymer as the binder. The method is characterized by heating the moulded body in a debinding furnace using at least a two-step temperature/time profile.

CN101235265 (A)
BINDER AND FEEDSTOCK MATERIAL PREPARED FOR METAL POWDER INJECTION MOULDING
Publication date: 2008-08-06
Inventor(s): W. JUNWEN et al, China

The invention relates to the metal powder injection forming field, in particular to a binding agent and feedstock which is used for metal powder injection forming. The binding agent comprises thermoplastic elastic compounded rubber, industrial paraffin, high density polythene HDPE, propene polymer PP and geoceric acid, and the feedstock is prepared through procedures of mixing according to material ratio, heating extrusion, cooling disintegration and vacuum packaging and the like. The effects of the invention are that the solid loadage of feedstock is over 60%, liquid and solid are not easily separated when in forming, the sticky point technology is simple, cost is low, sticky blank point height is high, sintering blank density is high and the product price is lower.

Sintering process of M2 HSS feedstock reinforced with carbides

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Metal matrix composites (MMCs) based on M2 HSS (High Speed Steel) are processed using a metal injection moulding route. Different types of reinforcement are added to the mixture and the effect in the sintering behaviour has been analysed. An optimised feedstock of M2 with carbides based on polyethylene and paraffin wax has been designed. The mixing procedure and the moulding conditions have been optimised to obtain parts without defects. A debinding schedule of thermal treatment has been established to partially eliminate the binder in order to promote the presence of some residual carbon into the parts. Finally, the sintering was studied under N_2-H_2 atmosphere. Density and microhardness measurements and SEM microstructures were used to determine the optimum sintering temperature and the sintering window of each system. This research has demonstrated that the addition of carbides produces an important reduction in the sintering temperature. Grain growth and coarsening of the grains are inhibited by the effect of the addition of the reinforcements. Although the addition of carbides reveals some general secondary effects in both cases, the addition of different reinforcement shows particular effects depending on which reinforcement is used. The mixture of carbides (WC+TiC+TaC+NbC) produces an important enlargement of the sintering window and the addition of VC produces an important reduction in the optimum sintering temperature.

Introduction

Metal injection moulding (MIM) is a cost-effective production method for small, complex and high performance components. This process allows the production of more uniform microstructures and improvement of mechanical properties. Moreover, it is possible to achieve higher densities, more intricate shapes and better surface finish [1]. Feedstock formulations are the area of greatest patent coverage in PIM. This factor serves to increase the cost of the final product. Basic exploration of innovative formulations increases knowledge and competitiveness, especially in high volume components. As can be seen in Table 1 [2] the part costs are dominated by feedstock price. This is the main justification for our research. The vendor A, who produces his own feedstock, is able to reduce his costs by 12% in the production of each part. For this reason, we focused our research on the development of innovative mixtures.

The production of HSS by MIM is considered to be better than other manufacturing techniques due to the inherent capacity of this technique to produce near net shape components, avoiding costly machining and obtaining more uniform shrinkage during sintering [3]. The principal difficulty presented by these steels is the complex densification process by SLPS (supersolidus liquid phase sintering) [4]. During the sintering process the high speed steel undergoes an unusual melting process; the liquid forms on the particle contact and grain boundaries and particle rearrangement causes rapid densification. A critical fractional coverage of liquid on the grain boundaries must be achieved for this process to occur [5]. The sintering temperatures and the carbon content are the most important variables in this process since these dictate the volume fraction of the liquid phase [6]. Accurate control of temperature and composition leads to accurate control of the volume fraction of liquid which in turn leads to full density, avoidance of shape distortion and minimisation of microstructural coarsening. The densification process and the microstructure development are the parameters for controlling the sintering window (temperature region in which optimum sintering takes place) because it is very narrow for M2 HSS [7].

The composition of HSS, especially the carbon content has a strong influence on the microstructure. The carbon content could be controlled by blending graphite with the HSS, leaving residual carbon during the debinding process or adding carbides to the HSS powder. The review of these possibilities leads to similar conclusions. In the studies developed in PM by Wright et al [8], blending M2 with graphite up to 1.2% of carbon content, a sintering window of 10ºC was determined. Also, it was demonstrated that

Factor	Vendor A	Vendor B	Notes
feedstock	$0.35	$0.67	vendor B purchases premixed feedstock
moulding	$0.17	$0.37	vendor A is in lower labor rate area
tooling	$0.13	$0.13	no difference
debinding	$0.11	$0.04	vendor A relies on batch process
sintering	$0.34	$0.07	vendor B relies on continuous process
piece cost	$1.10	$1.28	vendor A gets the job

Table 1 Contrast of component costs between two vendors on the same part, where vendor A uses self-mixing and vendor B relies on purchased premixed feedstock (values are US$ per part) [2]

the sinterability is determined by the competing effects from the addition of alloying elements. In the studies, developed with a thermosetting binder [9] in which 3% of residual carbon is left, a large reduction in the optimum sintering temperature (OST) of around 100ºC was observed. In the studies that we developed [10,11] with a thermoplastic binder, (Fig. 1) the maximum reduction of the OST was 20ºC and this reduction was achieved when 2% of residual carbon was left in the samples. Should the amount of residual carbon be increased the effect would not be more pronounced.

In other traditional research such as that made by Barkalow et al [12] the effect of the addition of other alloying elements such as Mo, V and W has been studied but none of these elements seems to produce a large gap between the solidus and liquidus temperatures.

We have focused our attention on M2 HSS because it has greater toughness at equivalent hardness and better wear resistance than other high speed steels. HSS's have traditionally been used as cutting materials and currently compete with cemented carbides in many applications [13]. It is possible to cover the gap of properties between both compounds through the development of composite materials with a HSS matrix and ceramic reinforcement. The addition of carbides to the feedstock formulation is expected to improve not only the mechanical properties, but also the uniformity of the liquid phase distribution thanks to the addition of a carbon source. A good understanding of how the sintering gate is affected by some variables, such as reinforcement particles, will enhance the production routes for HSS parts in order to make them cheaper and more flexible. Additionally, previous studies in PM have demonstrated that abrasive wear resistance can be increased [14,15] by adding NbC and TaC, although sometimes it is necessary to add some sintering activator that increases the joint with the reinforcement. Other common reinforcements added to produce composite based steel materials by PM are TiC and TiN [16], which look for some reactivity with the matrix so that a good bonding at the ceramic/matrix interface could be obtained. The full potential of MIM process for fabricating metal matrix composites is yet to be explored. Nowadays, although the attention is focused on refractory metal, titanium and intermetallics based MMCs [17], the main purpose of fabricating steel based MMCs is to improve the wear resistance of the steel, coupled with the unique shaping advantage of MIM. Previous research such as that developed by Liu et al [18] or Zhang et al [19] has demonstrated that reinforcement is able to inhibit the grain growth and microstructure coarsening, although the effect of inhibiting the densification with the addition of TiC [18] produces an important increase of the sintering temperature required for a near full density microstructure.

The main purpose of this research is to characterise and optimise the behaviour of the M2 HSS reinforced with different types of carbides, analysing the effect produced by each kind. It is our objective to widen the sintering window (SW) and to enhance the mechanical and wear properties of injection moulded M2 by the addition of carbides. It is expected that the carbides will provide additional amounts of carbon to the matrix that could promote the hardening of the steel.

Experimental Procedure

The metal powder used in our experiments is a prealloyed gas atomised M2 High Speed Steel sold by Osprey Metals Ltd. (U.K.). In this steel 90 % of the particles are smaller than 16 µm. The particles are spherical in shape, as is shown in the scanning electron micrograph, Fig. 2. The chemical composition is given in Table 2.

In this research two kinds of reinforcement were used to prepare different feedstock formulations. The first reinforcement is a commercial mixture of four different carbides, WC, TiC, TaC and

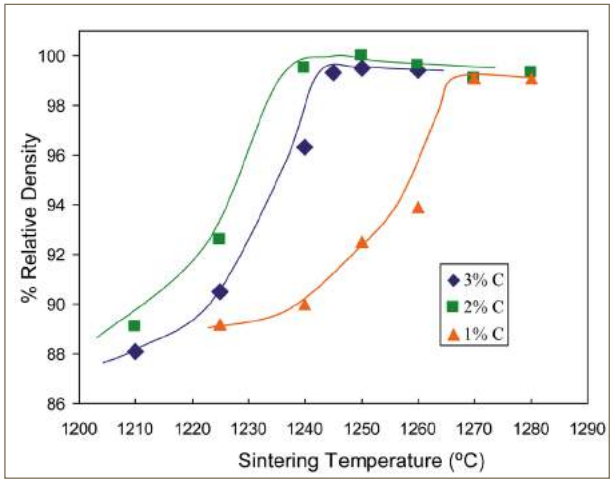

Fig. 1 Effect of residual carbon in the sinterability of M2 HSS processing by MIM [10]

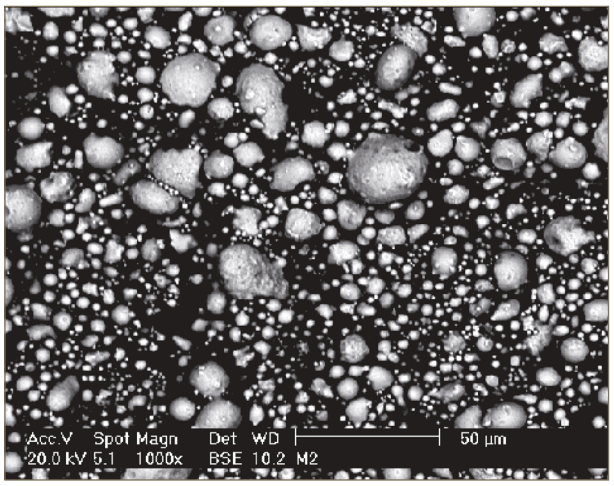

Fig. 2 M2 HSS powder as observed by SEM

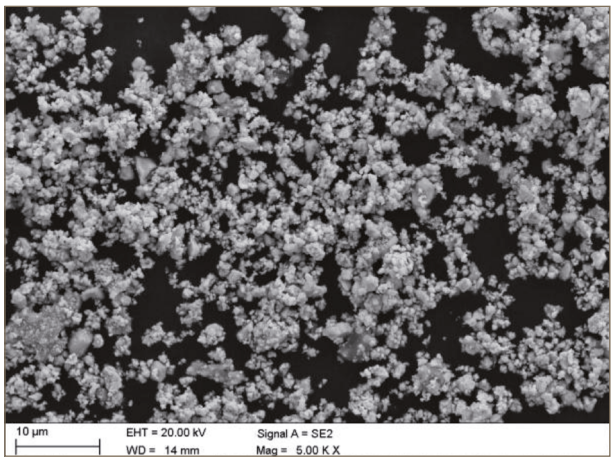

Fig. 3 Mixture of carbides as observed by SEM

	C	W	V	Mo	Cr	Mn
Present study	0.84	6.54	1.95	4.81	3.97	0.36
AISI specification	0.80-0.9	6.00-6.75	1.75-2.05	4.75-5.50	3.75-4.50	0.10

Table 2 Chemical composition (%wt) as received M2 powder together AISI specification

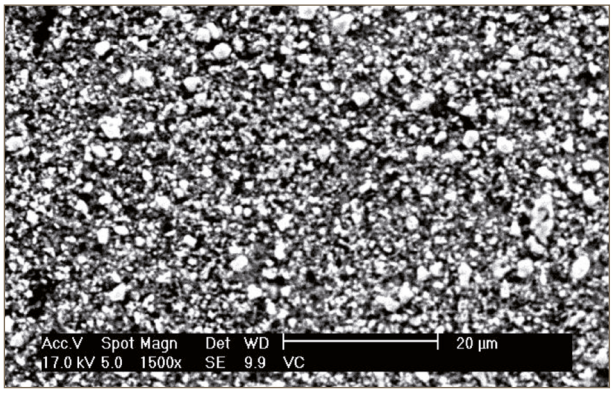

Fig. 4 Vanadium carbide as observed by SEM

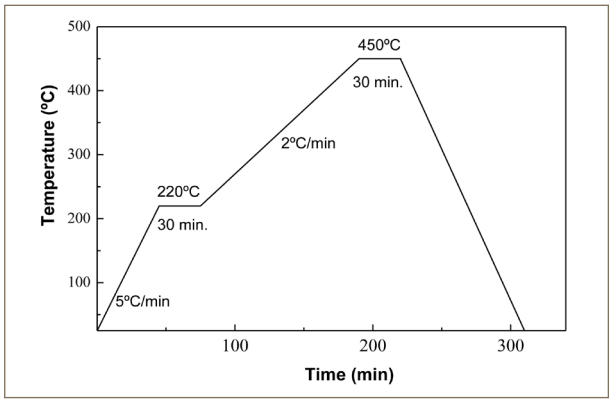

Fig. 5 Thermal debinding cycle applied

Feedstock	Reinforcement (% wt)
M2	-
M2 + Mixture of Carbides	13% (33.3 %vol WC + 33.3 % vol TiC + 26.8 % vol TaC + 6.8 %vol NbC)
M2 + VC	1 %
	3 %
	6 %
	10 %

Table 3 Feedstock formulations

NbC, Fig. 3. The second formulation is prepared with vanadium carbides, Fig. 4. All the particles of the added reinforcements are irregular in shape and smaller than the particles of the metal powder. The feedstock formulations prepared in this research have different amounts of reinforcements and the details are given in Table 3. The feedstock prepared with a mixture of carbides contains 13% wt (10% vol) of reinforcement. With formulations that contain VC the amount of reinforcement varies between 1 and 10% wt.

Previous rheological studies [20] have helped us to develop the binder used in this research. Complementary rheological measurements [10] have determined the optimal powder loading to be 68% volume of steel. In our current research the feedstock to which the reinforcements have been added have a constant percentage of powder loading, in order to maintain the rheological behaviour of the mixtures. This is obtained by substituting a proportion of the steel with varying amounts of the reinforcements. To corroborate this fact, preliminary rheological measurements have been made in some of the reinforced feedstock, and at this level of reinforcement the viscosity is mainly determined by the binder system.

Each component of the binder, besides the metal powder and the chosen amount of reinforcement carbides, was premixed in a turbula mixer at room temperature to homogenise the feeding of the twin screw extruder that was used to produce the feedstocks. All of them contain 68% by volume of powder loading and they were granulated and extruded twice to guarantee a complete homogeneity. Feedstock formulations were moulded into discs of 15 mm diameter using a low pressure system.

Thermal debinding in nitrogen was applied to eliminate the binder from the moulded samples, using the cycle that was designed using TGA studies made using Perkin Elmer equipment. The debinding schedule, Fig. 5, comprises a combination of suitable heating rate and moderate debinding temperature with two plateaus of 30min each to facilitate the degradation of the different components of the binder and to produce defect free parts. The maximum debinding temperature of 450ºC was set to leave a certain amount of residual carbon in the parts. The partially debound parts subsequently contain a minimum of 2.6% wt of carbon as was measured using a LECO CS-200 carbon analyser. Although the reinforced samples saturated the carbon analyser they contained 2.6% of carbon plus the carbon content provided by the reinforcement.

Sintering was performed at different temperatures under nitrogen-5% hydrogen (6 bar) in a tubular furnace. Densities of the sintered parts were measured using Archimedes's method. Microhardness was measured on polished surfaces with a Future Tech microdurometer. The powder and the microstructure evolution were observed using a Philips XL 30 scanning electron microscope equipped with BSE and EDS detectors. All the EDS spectra were collected at the same optimum conditions. The particles were identified using a Philips X'Pert automatic diffractometer (MPDPW3040) with Cu-Ka radiation.

Results and Discussion

To understand correctly the main results of this research it is useful to note the influence of the atmosphere in the M2 sintering, something reported previously by several researchers [7]. In accordance with these investigations and in the particular case of M2, which behaves differently from other HSS, the sintering in a N_2-H_2 atmosphere slightly increases the optimum sintering temperature (OST). The optimum sintering temperature (OST) is defined as the lowest sintering temperature leading to a density higher than 98% of the theoretical density. It can be said that the sintering window (SW) is the temperature interval comprised between the OST and the lowest temperature at which incipient melting of the M_6C carbides takes place [21]. We found that the optimum range of sintering temperatures is between 1260ºC and 1270ºC when the sintering process takes place in high vacuum conditions, while in a N_2-H_2 atmosphere the optimum range of sintering temperatures is 1280-1290ºC. This behaviour seems to be related to the low vanadium content in the M2 composition. At the optimum temperature the microstructure observation, Fig. 6, and the X-ray analysis leads to the identification of a ferrite matrix and a dispersion of W and Mo rich M_6C carbides, different kinds of carbonitrides M(C,N) and nitrides MN, which are very difficult to identify because they are very small. The absorption of nitrogen into the matrix from the atmosphere leads to the sequence of reactions described by Palma et al [22] MC+N → M(C,N) + C that justifies the dispersion of carbonitrides found.

Mixture of carbides

The comparison between the densification curves of M2 feedstock and the feedstock reinforced with the mixture of carbides shows the main consequence of the addition of the reinforcement. The OST (optimum sintering temperature) decreases by 50ºC. At only 1220ºC

the measured densities are higher than 98% as can be observed in Fig. 7. The added carbides serve as an additional carbon source, which decreases the solidus temperature. The carbides help the densification and prevent distortion possibly because the carbon source produces a modification of the phase diagram. These results offer the possibility to have better control of the complicated sintering process necessary for this kind of steel. The curves in Fig. 7 and the microstructure evolution of the feedstock reinforced with a mixture of carbides, Fig. 8, reveal a very stable grain size and a totally homogeneous distribution of carbides in a very wide range of temperatures. It can be said that the SW (sintering window) suffers a remarkable expansion by up to 40ºC. The hardness values of the materials are consistent with the relative density. The hardness is increased by around 70 HV, achieving 640 HV.

In this temperature range the grain size shows a minimum change, from 10 µm to 20 µm. This fact can be explained by the restrictive effect on the grain growth produced by the addition of carbides. This is in agreement with the results described by Bolton & Gant; [23] in conventional PM where M2 was reinforced with TiC and NbC, it was necessary to increase the temperature to 1300ºC to find carbide films in the grain boundaries. Bolton's results confirm that the sintering window of this feedstock is very wide.

Vanadium carbide reinforcement

The addition of VC has an important influence on the microstructural development of the M2 feedstock. The addition of only 1%wt of VC produces a decrease in the OST. The best microstructure is obtained at 1260ºC. During an already narrow SW (sintering window) a homogeneous distribution of bright carbides, gray nitrocarbides and black nitrides is detected, Fig. 9 (A). At higher temperatures the carbides migrate to the grain boundaries and coarsening of the grains begins. Analysing in more detail the optimum microstructure by X-ray diffraction, new types of carbides such as M_3C (Fe_2MoC) besides Fe_3W_3C can be identified. Numerous gray particles rich in iron and vanadium are observed and some of the original VC are found.

The addition of 3% wt of VC to the feedstock formulation has a more remarkable effect. As can be observed in Fig. 10, at 1225ºC complete densification is achieved. At 1250ºC, Fig. 9(B), the grain size and the homogeneous distribution of particles remain stable without the formation of any undesirable eutectic carbide. The SW is 30ºC wide. The temperature must reach 1260ºC in order to observe the coarsening of the carbides and the beginning of the grain growth. This enhanced sinterability is attributed to the expansion of the region of the phase field, the $L+M_6C+\gamma$ (L: liquid, γ: austenite) region [8], where the SLPS takes place.

This important effect has been reported before in conventional PM for vanadium rich high speed steels but it has never been

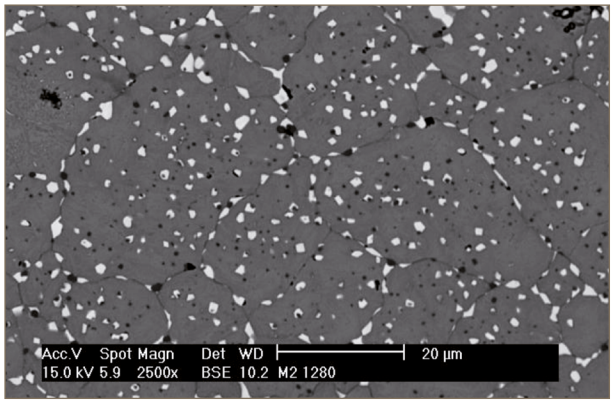

Fig. 6 SEM micrograph of M2 sintered under N2-H2, 1280ºC

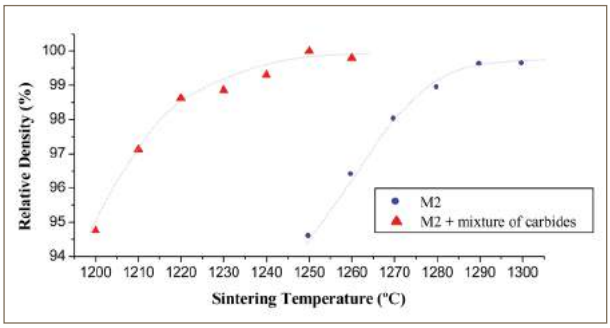

Fig. 7 Comparison of the densification curves M2 and M2+mixture of carbides

reported in M2 parts processed by MIM. In this research, we have several contributions that can together produce a synergic effect: the residual carbon from the partially debinding process, the presence of a vanadium rich reinforcement and the sequence of reactions produced by the adsorption of nitrogen into the ferrite matrix. This sequence of reactions is responsible for the formation of carbonitrides. If the pressure of nitrogen is high enough a progressive absorption of N by the material produces a transformation of the M(C,N) phase into MN [21]. So the complete sequence of reactions that takes place is:

$$MC+N \rightarrow M(C,N) + C \rightarrow MN + C \qquad (1)$$

Identification of the particles is very complex because if we look more closely, Fig. 11, a continuous evolution from VC to VN is taking place in several particles. The centre of the particles becomes more enriched in nitrogen as indicated by the black contrast when the microstructure is observed in backscattered electron mode (BSE). The outer rings of the particles are progressively clearer, indicating that nitrogen content gradually diminishes. The

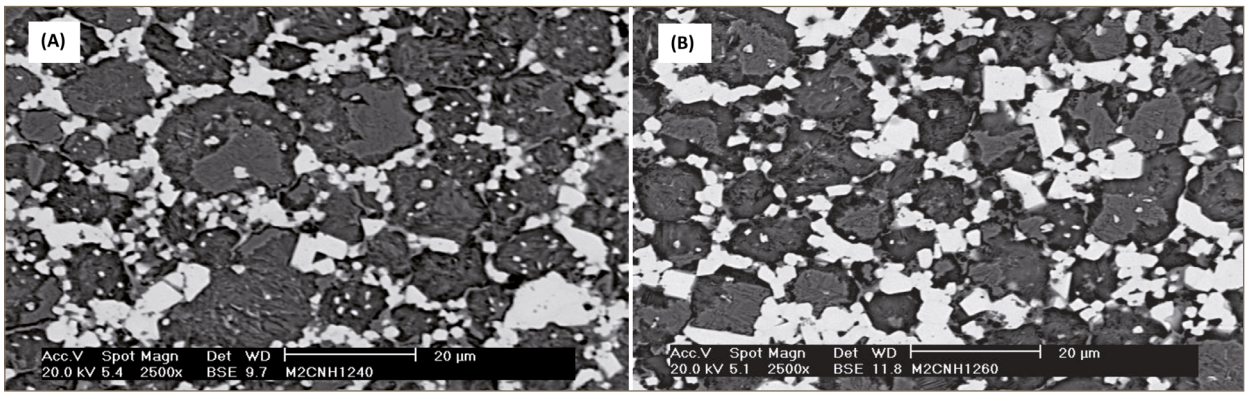

Fig. 8 Microstructures of M2 reinforced with a mixture of carbides sintered at (A) 1240ºC and (B) 1260ºC

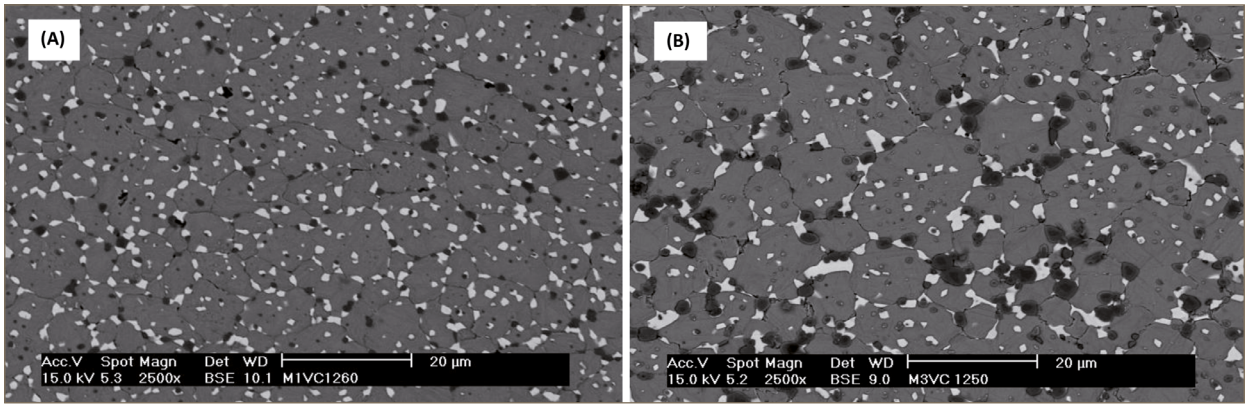

Fig. 9 Microstructure of M2 reinforced with (A) 1%wt of VC at 1260ºC and (B) 3%wt of VC at 1250ºC

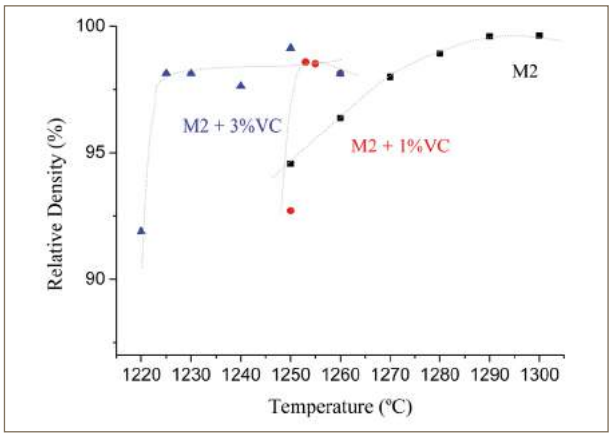

Fig. 10 Comparison of the densification curves of M2, M2+1%VC and M2+3%VC

outermost ring can be identified as VC, see Fig. 11. More research is needed to determine whether the carbon content of the nitrides is progressively increasing or if the carbides are being progressively enriched with nitrogen.

The microstructure of the samples reinforced with 6%wt of VC is completely different compared with previous descriptions, Fig. 12. The densification takes places at 1160ºC, Fig. 13, underlining the important influence of the addition of carbides. The progressive transformation of the iron and vanadium carbides into carbonitrides and nitrides promotes some modifications in the rest of the carbides in the sample. Bigger particles and rodlike carbides both rich in W and Mo and a small gray boundary film, rich in Fe and V, are detected. This gray film has a stoiochimetry M_3C that wasn't present before in such large quantities. It seems reasonable to suggest that M_3C is formed due to the enrichment of the matrix with carbon that is coming from the sequence of reactions described above. Although in this case the sintering window is 60ºC and the microhardness is 661HV, other properties such as toughness or wear resistance have to be measured to determine the positive or negative influence of these angular carbides.

Finally, the study of the feedstock reinforced with 10% wt of VC shows that at only 1125ºC, Fig. 13, a complete densification together with an optimal microstructure is found. The microstructure of the material sintered at the optimum sintering temperature, Fig. 14 consists mainly of a dispersion of equiaxed W and Mo rich carbides embedded in a ferrite matrix. Using X-ray diffraction analysis the evolution of the carbides can be observed. When 10% wt of VC and an increase in sintering temperature is applied, a gradual disappearance of M_6C is observed. At the same time other carbides such as Fe_2MoC and Mo_2C, appear. This transformation could be justified again by the progressive enrichment in carbon content when the reaction (1) takes place. Besides the formation of different types of carbonitrides, the presence of V_2N is detected at lower sintering temperatures, and at higher temperatures the presence of VN is more evident due to the higher absorption of nitrogen by the sample.

The maximum hardness achieved by the samples reinforced with 1, 3 and 6% of VC is quite similar, around 660HV. However the samples prepared with 10% of VC show more than 730HV within a range of 100ºC. To explain this effect, both the microstructure and the X-ray diffraction have been carefully analysed. The formation of VN particles may explain the high values of hardness of the samples prepared with this formulation of feedstock. Giménez et al [21] have described a similar effect in another vanadium rich high speed steel when sintering at a higher nitrogen pressure (8 bar).

As shown above, previous investigations developed in PM [21,24] demonstrated that in V rich HSS the nitrogen is easily absorbed by the matrix promoting a sequence of transformations. In the studies where the vanadium content of the alloy was modified, the most remarkable effect found was a reduction in sintering temperature, furthermore, the magnitude of the reduction increased with increasing vanadium content. In those studies where the increase of nitrogen content was analysed, a direct correlation with a decrease of the OST was observed. In our research the nitrogen pressure is kept constant during sintering but the VC quantity is varied. The great affinity between N and V produces a higher absorption of the nitrogen by the steel

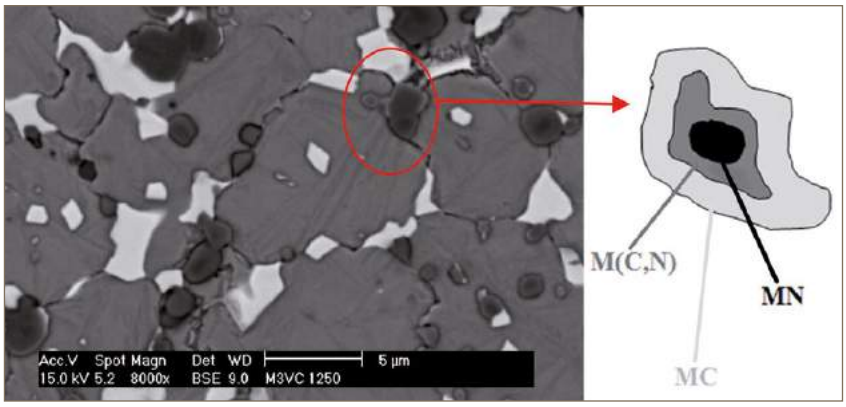

Fig. 11 Microstructure detail of M2 feedstock reinforced with 3%wt of VC at 1250ºC

matrix and then the same sequence of reactions described before is produced. Moreover, the large amount of carbon produced by these reactions promotes the formation of secondary carbides and carbon enriched carbides that increase the hardness of the parts. This improvement in the hardness anticipates an increase of the wear resistance properties.

Conclusions

Innovative feedstock formulations based on reinforced M2 have been successfully developed to be processed by powder injection moulding. The results presented in this paper offer the possibility of improving control over the complicated sintering process for this kind of steel and obtaining parts with complex geometries and higher hardness which is especially notable given that no thermal treatment is undergone. Consequently the development of these composite materials can cover the gap of properties between high speed steels and cemented carbides thus being of great interest for many tool applications.

The addition of a mixture of carbides (WC, TiC, TaC and NbC) enlarges the sintering window by up to 40ºC and reduces the optimum sintering temperature. The addition of carbides inhibits the grain growth and avoids the particle distortion for a wide range of temperatures.

Formulations which contain different amounts of VC show a clear reduction in the optimum sintering temperature although the effect is especially remarkable when 10% of VC is used. The hardness increases to 730HV. A better wear resistance is expected as well. The most important aspect in the microstructural evolution seems to be the relationship between the carbon content and the nitrogen absorption, and the amount of nitrogen absorbed seems to be directly related to the vanadium carbide content.

Acknowledgements

The authors thank Iberofon Plásticos for the financial support.

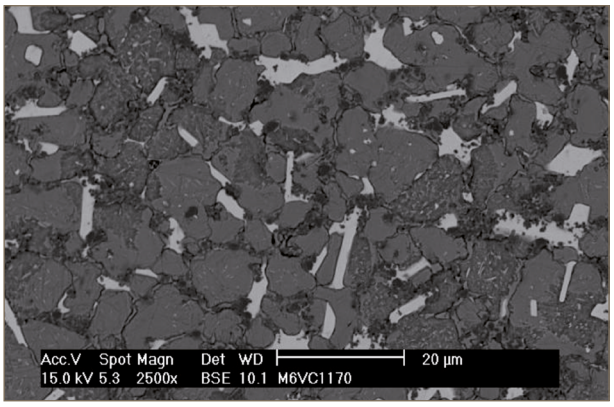

Fig. 12 Microstructure of M2 feedstock reinforced with 6%wt of VC at 1170ºC

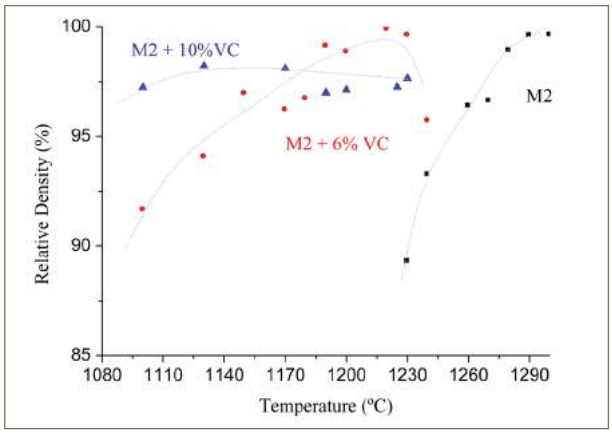

Fig. 13 Comparison of the densification curves of M2, M2+6%VC and M2+10%VC

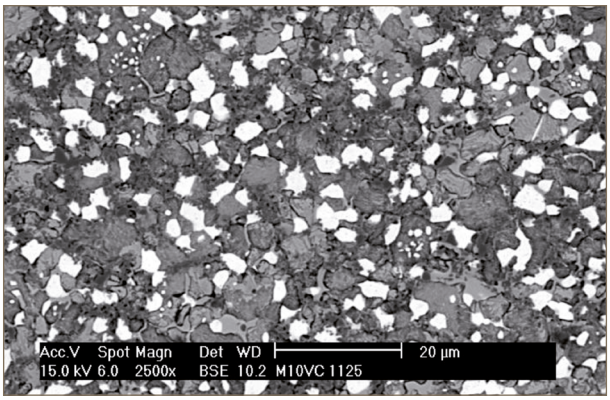

Fig. 14 Microstructure of M2 reinforced with 10%wt of VC sintered at 1125ºC

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Mechanical properties and corrosion resistance of vacuum sintered MIM 316L stainless steel containing delta ferrite

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A decrease in tensile ductility and axial fatigue strength was measured on increasing the content of delta ferrite in the metal injection moulding processed AISI 316L stainless steel. However, since delta ferrite activates densification, these effects can be compensated by an increase in the sintered density. No features attributable to delta ferrite were observed on the fracture surfaces in both tensile and fatigue loading. Only a slight increase in the passivation current density due to delta ferrite was observed in potentiodynamic tests in sulphuric acid solution, correlated to a selective corrosion of this secondary constituent. Despite these effects, all these properties remain comparable up to 8% of delta ferrite.

Introduction

AISI 316L stainless steel is one of the most widely used materials in high added value applications of Metal Injection Moulding [1] and is nowadays attracting a growing interest from new market sectors, such as that of the components for hardware [2].

AISI 316L is a typical austenitic stainless steel but, when processed by MIM, its microstructure may contain some delta ferrite [3] because of the extremely high sintering temperature (above 1300°C). The amount of delta ferrite increases with the sintering temperature and decreases with the carbon content which, as well known, stabilises the austenite phase. The effect of carbon, in particular, may be quite critical. The carbon content of the sintered austenitic steels depends on several parameters:

a) the carbon content of the starting powder;

b) the effectiveness of the debinding process, which may leave residual carbon in the brown parts that is subsequently dissolved in austenite on sintering; this effect has been investigated in the 17-4 PH stainless steel [4, 5];

c) the oxygen content of the powder, since carbon reduces the surface oxides, resulting in an activation of the sintering mechanisms but at the same time in decarburisation [6].

All these effects are active on sintering, irrespective of the sintering atmosphere: reductive (H_2), inert (N_2) or combined (N_2/H_2). Sintering of austenitic stainless steel at high temperature can be conveniently carried out in vacuum furnaces [7-10]. In these furnaces the graphite refractories, differently from the metallic ones, may produce a partial pressure of carbon which slightly increases the as sintered carbon content of the material. This effect, in turn, depends on the pressure of the backfilling gas which is added to prevent evaporation of Cr and Mn at high temperature.

The final content of delta ferrite is quite sensitive to all these parameters, and may vary during the industrial production.

The austenitic stainless steels are used in many applications where a high corrosion resistance is combined with a high ductility, toughness and impact strength, in particular when low temperatures are required [11]. Tensile and fatigue strength are a minor concern in such applications; nevertheless, minimum levels of both the properties have to be assured. Delta ferrite has an inherent lower ductility than austenite, because of its body centred cubic lattice, and a corresponding better strength. In addition, the coexistence of the two phases may negatively influence the corrosion resistance, because of the possibility of galvanic corrosion phenomena. Therefore, the knowledge of the effect of delta ferrite on properties of the austenitic stainless steel produced by MIM has a practical interest.

In this work, different amounts of delta ferrite were introduced in an AISI 316L steel produced by MIM using two vacuum sintering furnaces (having metallic and graphitic refractory), by varying the sintering furnace temperature and by a post-sintering heat treatment. This way, three significantly different amounts of delta phase were obtained, to investigate the effect on tensile and fatigue properties, as well as on the corrosion resistance of the material.

Experimental Procedure

A pre-alloyed gas atomised 316L powder was mixed with a proprietary binder and feedstock and was moulded to produce the specimens for tensile tests according to ASTM E 8M-03 – Standard Flat Unmachined Tension Test Specimen for Powder Metallurgy (P/M) Products. The same specimens were produced for fatigue tests, as well. Debinding was carried out in two steps: dissolution in water, to eliminate 80% of the binder, followed by thermal decomposition.

Material	Sintering Conditions
316L NF	Sintering at 1360°C in graphitic furnace
316L F	Sintering at 1380°C in metallic furnace

Table 1 Sintering conditions

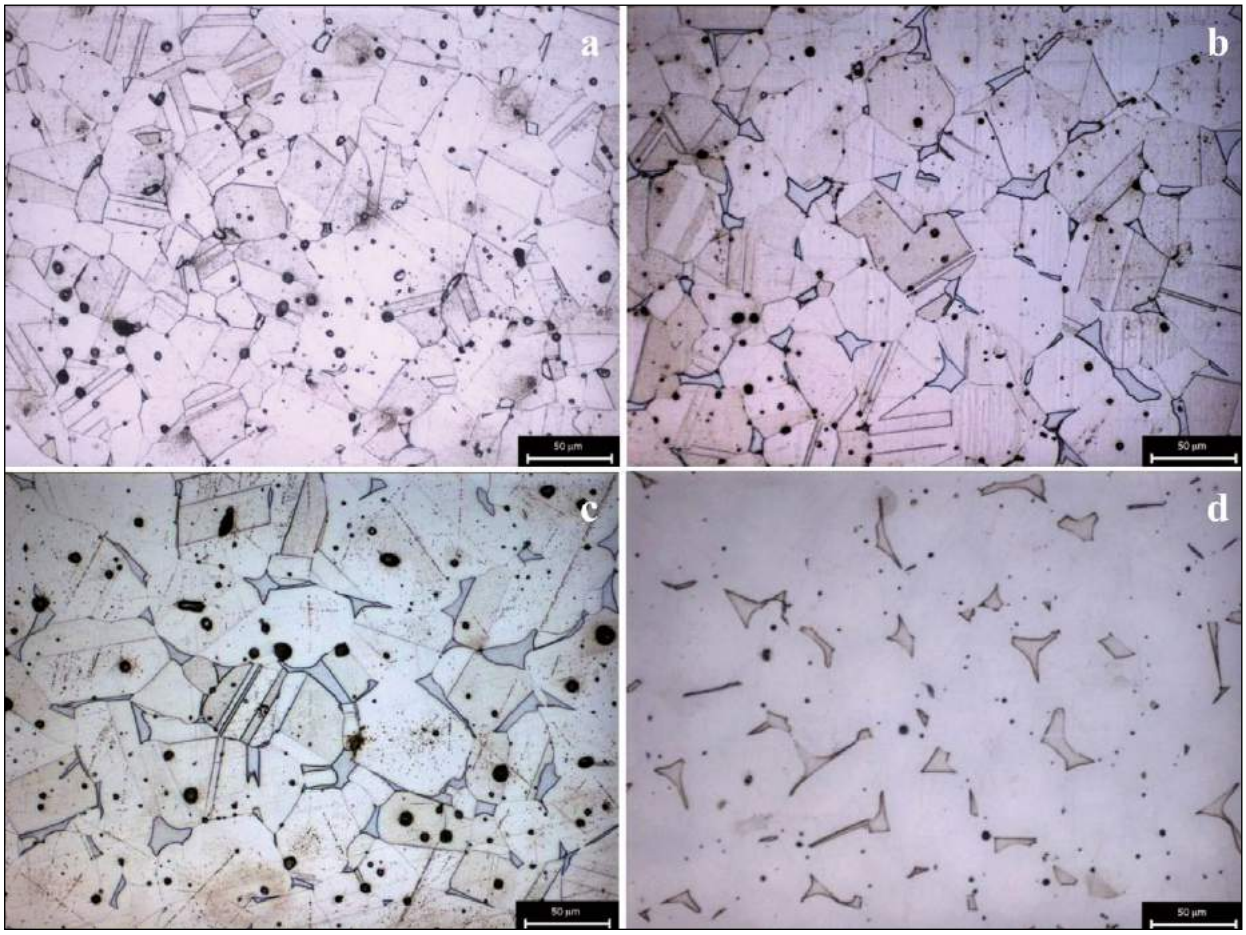

Fig. 1 Optical micrographs of 316L materials: NF (a), F (b), HF (c) and after specific etching for the quantitative determination of delta ferrite by Image Analysis (d)

The samples were sintered in two TAV (Caravaggio, Italy) vacuum furnaces with 1 hour isothermal holding in 100 mbar Ar backfilling at the temperatures reported in Table 1.

Cooling from the sintering temperature was carried out with 1 bar nitrogen flux. A third material was produced carrying out a heat treatment at 1390°C for one hour on 316L F to obtain a higher quantity of delta ferrite; it is called 316L HF in the following.

The carbon analysis was carried out by LECO CS125. Density was measured by the water displacement method. The microstructure was investigated by Light Optical Microscopy (LOM) after etching with a 25% distilled water, 50% HCl and 25% HNO_3 solution. For the quantitative determination of the amount of delta ferrite, specimens were etched with a 5ml distilled water, 2.5g potassium hydroxide and 2.5g potassium ferrocyanide solution, and characterised by Image Analysis.

Microhardness (HV0,02) and hardness (HV10) were measured.

Tensile tests were carried out on an Instron machine with a strain rate of 0.2 s^{-1} and by measuring strain with an axial extensometer with a gauge length of 12,5 mm.

High cycle fatigue tests were carried out on a Rumul Mikrotron 20kN machine with a frequency of 150Hz and a load ratio equal 0 (R=0). It was assumed $2x10^6$ cycles as run-out tests and the fatigue resistance was calculated by the Staircase Method. The fracture surfaces were investigated by SEM.

Corrosion potentiodynamic polarisation tests were carried out in a 0,5M H_2SO_4 solution. The samples were mechanically polished using SiC grinding paper and polished with a 1µm diamond solution to a mirror finish and then cleaned in alcohol and dried. The electrochemical cell was a three electrode cell using platinum as a counter and Ag/AgCl 3M as a reference. The scan rate applied was of 0.2 mV/s, starting from -1000 mV. Two measurements were performed for each sample and representative curves will be reported. The morphology of the corrosion attack was observed by SEM.

Results And Discussion

Microstructure

Fig. 1 shows examples of the microstructure of the three materials investigated: NF (1a), F (1b) and HF (1c). The residual porosity is very low, and is made of small, spherical and homogeneously distributed pores. Austenitic grains show several annealing twins, and the delta ferrite islands are isolated and distributed quite homogeneously. Fig. 1d shows the effect of the specific etchant used to prepare the specimens for the quantitative determination of the amount of delta ferrite. Etching is localised at the austenite-ferrite interface, and the contrast between the two constituents is very sharp.

Table 2 reports the volumetric percent of delta ferrite, along with density and carbon content. The main difference between NF and F is the carbon content. The smaller amount in F can be attributed to the metallic furnace and, to a lesser extent, the higher sintering temperature, which enhances the reaction between carbon and oxides. Because of the lower carbon content, F contains a larger amount of delta ferrite than NF. This enhances diffusion, which, in addition to the higher sintering temperature, results in a higher sintered density [3]. F and HF were sintered in the same conditions, hence the carbon content and density are the same. Heat treatment increases the content of delta ferrite as expected.

Material	delta ferrite, %	Density, (g/cm³)	C, %
316L NF	< 1	7.83±0.03 (98.5% of theor.)	0.013
316L F	4.5±0.9	7.94±0.01 (99.8% of theor.)	0.003
316L HF	8±1	7.94±0.01 (99.8% of theor.)	0.003

Table 2 Density, delta ferrite and carbon contents

Samples		Microhardness (HV0.02)	Hardness (HV10)
316L NF	ferrite	-	111.1±3.1
	austenite	170.3±12.9	
316L F	ferrite	223.5±5.8	120.4±1.7
	austenite	163.5±1.2	
316L HF	ferrite	223.0±29	126.0±3.1
	austenite	167.5±11	

Table 3 Microhardness and hardness of the investigated materials

Samples	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation %
316L NF	186±1.5	549.9±3.9	65.7±9.3
316L F	194.1±5	555.4±0.5	82.7±2.6
316L HF	211.2±2	571.8±7.5	77.7±3

Table 4 Tensile properties of the investigated materials

Samples	Fatigue Strength 50% (MPa)	FS / UTS
316L NF	248±15	0.45
316L F	293±1	0.53
316L HF	272±16	0.48

Table 5 Fatigue tests

Material	I_{pass} (Å/cm²)	Eb (mV)
316L NF	2,8 x 10^{-9}	900
316L F	4,6 x 10^{-9}	900
316L HF	7,5 x 10^{-9}	900

Table 6 Electrochemical parameters

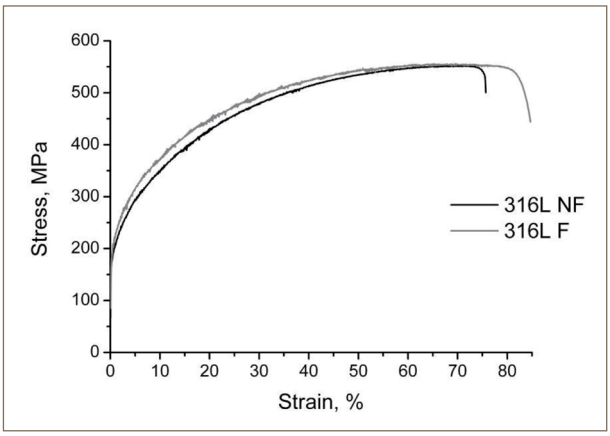

Fig. 2 Tensile test curves of NF and F materials

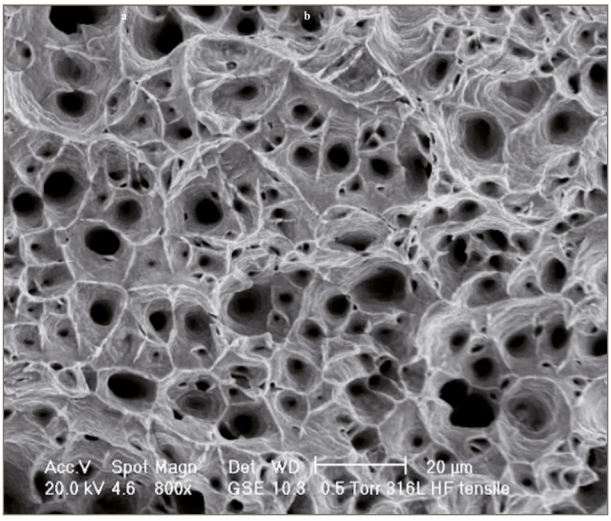

Fig. 3 Tensile fracture surface of 316L HF material

Microhardness and hardness

Microhardness and hardness data is reported in Table 3. Microhardness of delta ferrite is higher than that of austenite, but the effect on hardness is negligible (F versus HF). In comparing N and F, the contribution of density has to be taken into consideration, as well.

Mechanical properties

Fig. 2 shows examples of the tensile stress-strain curves of NF and F. The materials display a uniform plastic deformation up to the maximum stress, without any appreciable non uniform deformation, which is a typical feature of the austenitic stainless steel.

Table 4 reports the tensile properties of the three materials investigated. Results are comparable to those reported in the literature [12] and match the MPIF Standard 35 for MIM 316L stainless steel requirement [13].

The comparison between NF and F indicates that the latter has a slightly higher strength and a significantly higher ductility. In principle, delta ferrite is expected to increase strength and to decrease ductility. Differently, densification improves both the characteristics. Therefore, the difference between NF and F is attributable to the higher density of the latter. The effect of delta ferrite can be instead recognised in HF which actually has a higher strength and a slightly lower ductility than F.

The fracture is ductile, characterised by a dimpled morphology as shown in Fig. 3, relevant to HF and significant of the other

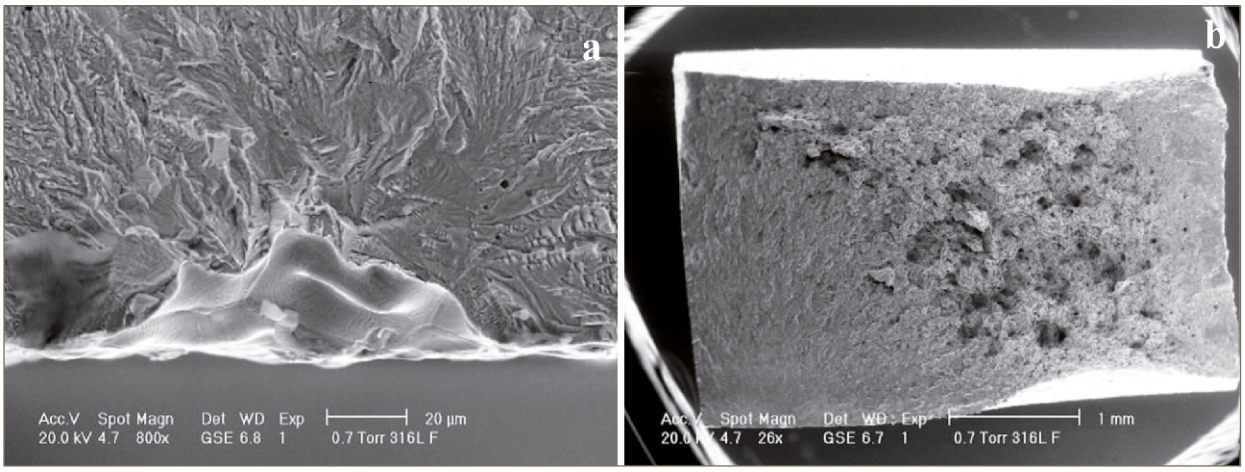

Fig. 4 Nucleation site of the fatigue crack (a) and fatigue fracture surface (b) of 316L NF material

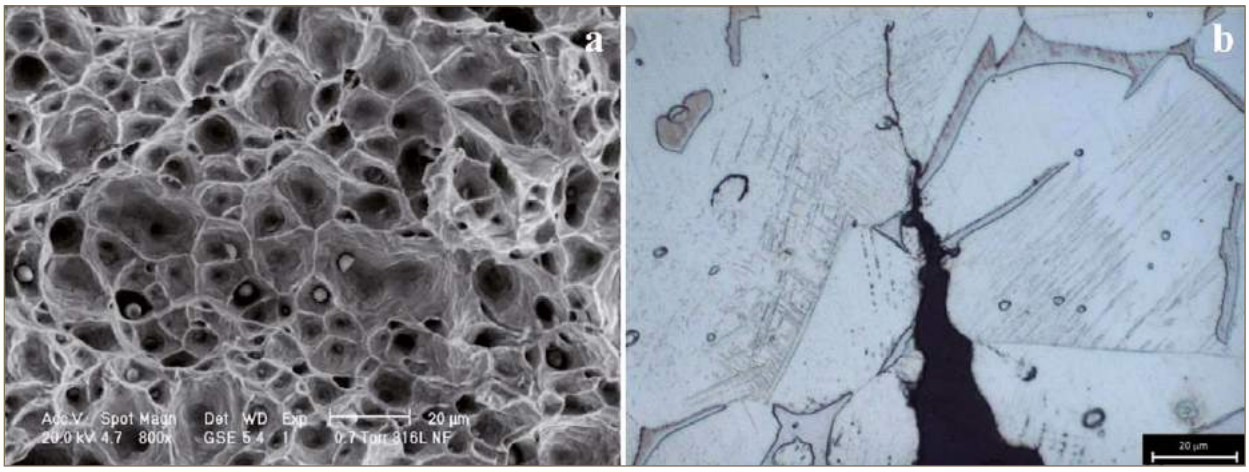

Fig. 5 Ductile morphology of the fast fatigue crack propagation (a) and path of crack propagation (b)

materials too. The fracture morphology does not show any feature attributable to delta ferrite, since this constituent also has a typical ductile behaviour.

Table 5 reports the results of fatigue tests. The fatigue strength at $2x10^6$ cycles is reported, along with the ratio between fatigue strength (FS) and UTS. Results are comparable to those reported in the literature [14]. F has a better fatigue strength than NF, but it can be recognised that the increase in fatigue resistance is higher than that in tensile strength. This is the well recognised effect of the enhanced densification. On the other side, HF has a lower fatigue strength than F, despite the increased tensile strength (Table 4). Therefore, the presence of delta ferrite tends to reduce fatigue strength. Its effect is overshadowed by densification between NF and F and is unambiguously displayed by HF in comparison to F.

The analysis of the fracture surface allows the nucleation of the fatigue crack to be individuated. It occurs on the surface, in correspondence of a pore, as Fig. 4a shows. There is an extensive slow propagation step of the fatigue crack (Fig. 4b), followed by fast propagation by overloading with the typical ductile morphology (Fig. 5).

As for tensile tests, there isn't any appreciable effect of the presence of ferrite on the fracture surface. The analysis of the propagating crack (Fig. 5b) confirms the absence of any preferential propagation path. The fatigue crack propagates through austenite grains as well as through delta ferrite grains.

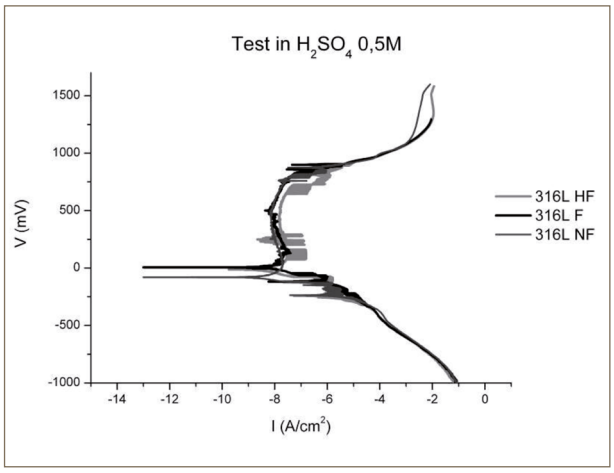

Fig. 6 Potentiodynamic curves

Corrosion resistance

The potentiodynamic curves of the three materials are shown in Fig. 6. All the materials present a passivity step followed by breakdown.

The main electrochemical parameters are summarised in Table 6. The passivation current density increases from NF to F and even more to HF confirming the effect of density on passivation and, on the other side, suggesting a negative effect of delta ferrite. However,

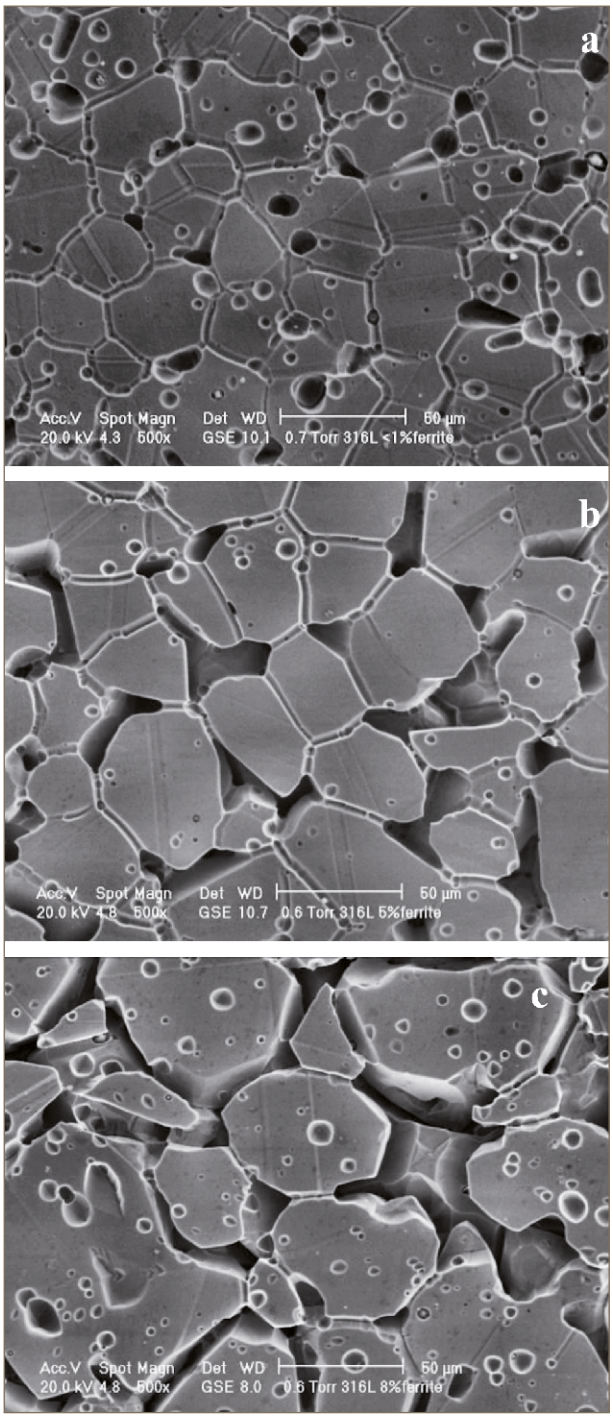

Fig. 7 Corrosion behaviour for NF (a), F (b) and HF (c) materials after breakdown.

the breakdown potential, which is a measure of the resistance of the passivation layer, is the same for the three materials.

The presence of delta ferrite slightly worsens the electrochemical behaviour of the investigated material. This is consistent with the corrosion morphology after breakdown, shown in Fig. 7. The delta ferrite grains are totally corroded, due to a strong localised corrosive attack. It is well known that austenite is nobler than ferrite, and the presence of the two constituents activates a galvanic corrosion phenomenon.

Conclusions

An AISI 316L austenitic stainless steel with different amounts of delta ferrite was produced by MIM, in order to investigate the effect of this constituent on mechanical and corrosion properties. Delta ferrite content increases with the sintering temperature, and with a post-sintering heat treatment at high temperature. Sintering temperature increases density from 98.5% to 99.8% of the theoretical one.

Delta ferrite increases hardness, yield strength and ultimate tensile strength, and reduces ductility. This effect can be overshadowed by that of density when the increase in delta ferrite is attained by increasing the sintering temperature. In this case, the effect of density on ductility prevails on that of delta ferrite. As far as fatigue strength is concerned, it is correlated to tensile ductility; it decreases with the increase in the delta ferrite content at a constant density. No features attributable to delta ferrite were observed on the fracture surfaces.

The corrosion resistance are almost the same for the three materials, with only a slight increase in the passivation current density due to delta ferrite. After breakdown the delta ferrite is totally corroded.

In conclusion, the effect of delta ferrite on tensile strength and ductility, fatigue and corrosion resistance is rather small since all these properties remain comparable up to 8% of delta ferrite.

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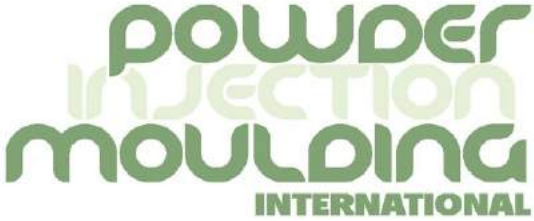

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www.titanium.org

2010 PM World Congress & Exhibition
October 10-14, Florence, Italy
www.epma.com/pm2010

55th Annual Conference on Magnetism and Magnetic Materials, MMM
November 14–18, Atlanta, Georgia, USA,
www.magnetism.org

2011

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www.mpif.org

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