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FOR THE METAL, CERAMIC AND CARBIDE INJECTION MOULDING INDUSTRIES

POWDER INJECTION MOULDING INTERNATIONAL

in this issue

PIM in the aerospace industry

Company reviews: OBE, Carpenter

Water soluble binder systems

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POWDER INJECTION MOULDING INTERNATIONAL

For the metal, ceramic and carbide injection moulding industries

PIM set to fly high in the aerospace industry

The PIM industry is now starting to see the rewards of several decades of investment in process control, materials and quality management with increased interest from the lucrative aerospace industry. Although this sector has explored the potential of PIM since the late 1970's, it is only now that there is a sense that aerospace engineers are willing to move forward with confidence towards the increased use of PIM for critical aero engine components. In a major report for *PIM International*, Prof. Randall German reviews the history and opportunities for PIM in this high growth area (page 28).

In this issue we also report on a visit to German MIM producer OBE Ohnmacht & Baumgärtner GmbH, a long established precision machining company that has successfully combined its knowledge of automation with a diverse range of in-house finishing facilities to create a successful niche in high volume MIM parts production (page 39).

The continued growth of MIM parts production is having the effect of transforming the fine metal powder industry from what was originally a "sideline" activity into a lucrative business area. One of the early participants was UltraFine Powder Technology Inc., now part of Carpenter Powder Products. We report on the company's plans to expand their MIM powder supply capabilities (page 45).

We also present work being done in France to evaluate a selection of water soluble binder systems (page 51), and an innovation from the Orton Ceramic Foundation in the USA that provides part producers with a physical record of furnace performance for individual sintering runs (page 55).

Finally, our technical papers from Japan and Germany demonstrate the effect of the addition of Zr on the sintering behaviour of water-atomised 316L stainless steel (page 60) and progress of Two-Component MicroPIM technology (page 68).

Nick Williams
Managing Director and Editor

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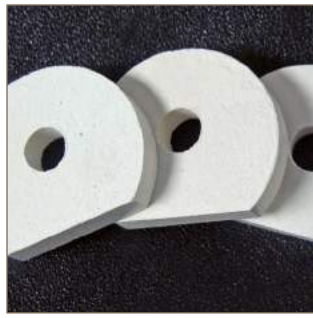

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To submit news for inclusion in *Powder Injection Moulding International* please contact Nick Williams nick@inovar-communications.com

Chromalloy breaks ground at a new $5 million ceramic core production facility

A new 40,000 square foot ceramic core facility is being built next to Chromalloy Castings's newly operational $30 million aerospace components foundry in Tampa, Florida, USA.

The ceramic core facility will be built in 2011 and be online by the first quarter of 2012. The company's new 150,000 square foot investment casting foundry in Tampa, Chromalloy Castings, was unveiled in December 2010 and is fully online. The foundry expanded the company's casting capability to pour up to one million pounds of superalloy turbine components and parts for aerospace, aero-derivative and industrial gas turbine engines.

Ceramic cores are utilised in the investment casting process to form complex cooling passages within the components, which are necessary to operate effectively in the hot and highly stressed sections of gas turbine engines.

"The new facility will supply the critical ceramic cores used to cast superalloy turbine engine vanes and blades," said Armand F. Lauzon, Jr., President. "Being co-located with the foundry, it will help us to serve our customers with even stronger production times."

Florida Governor Rick Scott joined Chromalloy officials and employees as the company broke ground on the new production facility. "This is an important day for Chromalloy, Tampa Bay and the State of Florida," said Lauzon. "We are pleased to be a vital member of the community with a new world-class foundry, and now a new companion core production facility that will help us better serve our customers worldwide."

Chromalloy's parent company, Sequa Corporation, has corporate headquarters in Tampa. The company also operates a joint venture company, BELAC LLC, in nearby Oldsmar, Florida, also in the Tampa Bay area. BELAC is a producer of turbine engine components for aerospace turbines.

The company, with 52 locations worldwide, casts components for the "hot section" or critical gas path of the engine, for the entire range of jet aircraft engines as well as marine, aero-derivative and heavy frame industrial turbines, including the largest and most complex turbine blades and vanes for power generation engines (IGT).

www.chromalloy.com

EPMA Powder Metallurgy Summer School, Dresden, Germany, 27 June – 1 July 2011

The European Powder Metallurgy Association (EPMA) is planning a Powder Metallurgy Summer School, which will take place in Dresden, Germany. The course, which consists of a five-day training programme, takes place from Monday, 27th June to Friday, 1st July 2011.

The EPMA Summer School is particularly designed for young graduate designers, engineers and scientists drawn from a wide range of disciplines such as materials science, design, engineering, manufacturing or metallurgy. The course will offer them an advanced teaching of Powder Metallurgy's advantages and limitations by some of the leading academic and industrial figures in Europe.

Powder injection moulding will also be covered during the course, and all participants will receive a copy of the June 2011 issue of *Powder Injection Moulding International*.

The participation fee for the whole event is a very reasonable €425 per person. For this non-refundable fee participants will receive all relevant course documents plus refreshments, meals and accommodation. The course is open to graduates under the age of 35 who are citizens of a European state. The deadline for applications is 31st March 2011, however spaces are allocated on a first come first served basis.

www.epma.com/summerschool

Moldex3D Interfaces with Siemens NX Software

CoreTech System Co Ltd based in Hsinchu, Taiwan, has been providing CAE simulation programs for injection moulding since the mid-1990s. The company's Moldex3D simulation software has not only found applications by plastic moulders, but also in the processing of MIM and CIM components. The company states that as long as the rheological properties of the MIM/CIM feedstock can be fully characterised then Moldex3D can simulate its filling, packing and cooling behaviours successfully.

The company has announced that an interface has been developed with Siemens' NX software, the flagship digital product development solution from Siemens PLM Software. The interface is called 'eDesignSYNC for NX' and will serve as the bridge allowing NX users to access Moldex3D/eDesign from within the NX environment. With the new interface, NX users are able to synchronise their design changes with Moldex3D simulations. Specifically, within the NX environment, designers are able to directly launch Moldex3D/eDesign analyses and acquire simulation as they make changes to a part's design, helping speed modification and optimisation. "Seamless integration between CAD and Moldex3D has been one of the strongest demands of our customers. The release of 'eDesign-SYNC for NX' is expected to benefit the mutual users of NX and Moldex3D with a flexible simulation-driven design platform," said Venny Yang, CoreTech's President.

Joan Hirsch, VP, product design solutions, Siemens PLM Software added, "NX is widely used throughout the plastic mould industry for CAD part design, tool design and computer-aided manufacturing (CAM), and the integration of Moldex3D/eDesign will create additional value for our mutual customers for part design validation and mould optimisation."

Moldex3D R10.0 offers a new dimension of injection moulding simulation

The latest version of Moldex3D R10.0 includes a number of new modules, for example Injection Compression Moulding (ICM) and Fluid-Assisted Injected Moulding (FAIM), to accurately simulate the 3D moulding phenomena in these special injection moulding processes. Key process parameters can be investigated and major moulding blemishes can be predicted.

"This is a completely new dimension of a 3D CAE simulation software," stated Cristoph Hinse, General Manager of SimpaTec GmbH, reseller of Moldex3D, about the new Release R10.0. "More than 120 new features further assist part and mould designers to optimise their product design with fast computation and accurate 3D simulation results at every stage of the production."

"Moldex3D R10.0 combines an interactive platform, powerful automatic meshing, parallel computing, and fast feedback of the design for all designers and CAE specialists at every stage of the product development. It integrates your innovation, efficiency and productivity to reach your design expectation and product optimisation," said Dr. Venny Yang, President of CoreTech System. "The ability to have better simulation results and accurate predictions encourages more effective product cycle management and increases profitability at the same time."

www.moldex3d.com
www.simpatec.com

Laser sintering speeds up mould production

Direct metal laser sintering (DMLS) of metal powders has been gaining industrial acceptance both as a process to produce prototype parts (Rapid Prototyping) and prototype tools (Rapid Tooling), as well as Rapid Manufacturing (eManufacturing) for the optimised production of laser-sintered customised products for a growing number of sectors.

EOS Electro Optical Systems GmbH based in Krailling, near Munich, Germany, was founded in 1989 to exploit the new technology of stereolithography and later developed DMLS technology, particularly for metals. Today EOS employs around 300 people worldwide, and claims to be the world's leading manufacturer of laser sintering systems.

At the recent EuroMold exhibition in Frankfurt, Germany (December 1-4, 2010) EOS exhibited its new EOSINT M 280 machine, which is the updated and further improved version of the EOSINT M 270, said to be the leading system on the market for laser sintering of metal components. The EOSINT M 280 system is equipped with a solid state laser of 200 watt, or an optional 400 watt, and an optimised Gas Management System operating in both protective nitrogen and argon atmospheres to guarantee consistent processing conditions.

Peter Klink, Executive Vice President Global Sales at EOS, states, "The system sets new standards in terms of part quality and reproducibility, at the same time improving cost-effectiveness and user-friendliness. The EOSINT M 280 adopts all features and advantages of the well-established EOSINT M 270 system, and makes it even more attractive for a wide range of demanding applications including injection moulding tooling and series production."

www.eos.info

The EOSINT M 280

Submitting News

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

MIM and CIM parts used in Cochlear hearing implant devices

Cochlear is one of the world leaders in advanced hearing technologies with headquarters in Macquarie Park, Sydney, Australia, and additional manufacturing facilities in Gothenburg, Sweden. Cochlear implants, first introduced in the early 1980s, have the ability to restore hearing for individuals who are severely hard of hearing and for whom conventional hearing aids are of little help.

The company states that the implant device bypasses damaged hair cells in the inner ear, or cochlea, which stimulate the hearing nerve directly. For example, the Baha system developed by Cochlear combines a sound processor with an abutment and a small titanium implant with the implant being placed behind the non-functioning ear. It takes around 3 months for the implant to osseointegrate with the bone. The Baha sound processor may then be attached to the implant abutment via a snap coupling. The implant ensures the optimal transfer of sound vibrations, via the skull, directly to the functioning cochlea.

The company recently introduced its Baha BP100 sound processor which it states sets a new benchmark in hearing aid performance. Key to the design of the Baha BP100 is the use of MIM technology which allows Cochlear to produce the unique shape and tolerances required for the two outer shell casings used in the chassis of the sound processor. Titanium was selected because of its biocompatibility and light weight, yet it is a strong and stable material. The MIM titanium parts are said to have stood up well in all the 18 reliability tests, even in challenging environments. The design of the Baha BP100 combines the use of the Europlug and the Cochlear Baha fitting software to allow direct audio input from MP3 players, phones and other audio sources.

Cochlear also reported the use of ceramic injection moulded Al_2O_3 in one of the most critical components in its hearing implants, the feedthrough, at the PM2010 Powder Metallurgy World Congress in Florence. This is the mechanical structure that provides the electrical connection for the platinum pins in and out of the device housing. Biocompatibility and long term implantation stability as well as mechanical and electrical functionality are some of the requirements that had to be met with the CIM Al_2O_3 feedthrough.

www.cochlear.com

The snap coupling and abutment of the of the Cochlear Baha BP100

Sunrock Ceramics to more than double production capacity

Sunrock Ceramics, based in Broadview, Illinois, USA, has announced that it has raised equity capital from Stantine Limited Partnership. The proceeds will be used to increase press and kiln capacity to meet growing demand for its industrial ceramics, allowing Sunrock to more than double the production capacity of its 20,000 square foot facility, as well as putting more resources into the development of new products and markets.

Sunrock President and CEO, Doug Thurman, stated, "We are grateful for the confidence that Stantine has shown us. In this period of tight financial markets, we are pleased to be able to take full advantage of the robust order pipeline in our highly specialised markets. Business has been very strong for some time now, with many great opportunities for further expansion, and now we can take Sunrock to the next level."

Sunrock Ceramics produce a wide range of alumina consumables

Steve Dehmlow, Stantine's Managing Director, also added "We are very pleased to be involved with Sunrock. They are participating in some exciting markets related to cutting-edge energy, transportation and medical applications, and their ability to deliver the highest quality products in a fraction of the time of others impressed us. We believe Sunrock also provides us with a platform to co-invest in related niche markets down the road."

Sunrock Ceramics is a manufacturer of high performance alumina consumables used in the production of MIM and PM parts.

www.sunrockceramics.com

Sintering 2011 conference to be held in South Korea

Jointly organised by the Korean Powder Metallurgy Institute and the Korean Ceramic Society, the Sintering 2011 conference will be held at the Shilla Jeju Hotel, Jeju Island, Korea, from 28th August to 1st September 2011.

Sintering 2011 will address the latest advances in sintering science and technology of powder-based materials. Topics range from sintering fundamentals to industrial applications.

All classes of materials will be covered. The topics will include: Fundamental aspects of sintering, Modeling of sintering at multiple scales. Sintering of multi-material and multi-layered systems, Microstructural evolution in sintering processes, Novel Sintering Processes (field-assisted, microwave, etc), Sintering of nano-structured materials, plus a range of other topics.

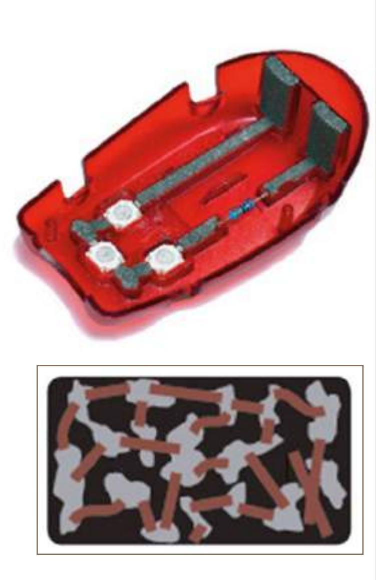
www.sintering2011.org

Moulding electrical circuits into plastic components

Plastics compounder A. Schulman Inc., based in Akron, Ohio, USA, together with Siemens AG, IKV Aachen, and HEK GmbH, Germany, have successfully developed a new group of plastic-metal compounds with excellent electrical conductivity.

The electrically conductive plastic compound designated SCHULATEC® TinCo can be used to injection mould electric circuits into plastic housings.

The material is based on copper fibres and tin loaded at very high levels into polyamide 6. The tin acts like a solder connecting the copper fibres. This allows the direct production of complex injection moulded, electrical conducting structures with completely new design possibilities, states Schulman.



An example of a plastic injection moulded housing with integrated moulded circuit. Inset is a schematic of the function of the conductive network

The specific electrical conductivity of the moulded compound is said to be in the range of 5 x 10^5 S/m. The electro-magnetic damping is 80 dB in the frequency range between 30 kHz and 1.2 GHz (sample thickness = 1.5 mm). The thermal conductivity is > 7 W/(mK). The new material is expected to find applications in automotive housings and lighting applications.

www.aschulman.com

EPMA's 'Stiffness Moduli of Hardmetals' project to be launched

The European Powder Metallurgy Association (EPMA) will soon launch a club project on 'Stiffness Moduli of Hardmetals' in partnership with the UK's National Physical Laboratory (NPL), London, as the required number of industrial partners has now been reached. The project will have two objectives:

1) To conduct research using the standard impact excitation method on a wide range of hardmetal grades, resulting in a report describing the results of testing and the levels of discrimination that impact excitation can achieve between hardmetal grades.

2) To develop a simple table-top method using similar excitation principles, which can be delivered to project members for subsequent use with in-house measurements.

The research will be jointly funded by NPL and the industry project partners.

www.epma.com | www.npl.co.uk

Arburg launches new Selogica "Set-up Assistant" module

Arburg has launched a new Selogica "Set-up Assistant" module that enables installation technicians to perform simple, quick and reliable set-up of Allrounder injection moulding machines without detailed knowledge of the control system. The set-up assistant accompanies the installation technician throughout the set-up process, from installation of the mould through to automatic initial calculation of the parameters and the finished sequence.

In the first step of five steps, the installation technician selects the necessary machine functions with which the Allrounder is to operate, such as ejector, core pull or sorter unit. The available selection options depend on the machine equipment.

In the second step, the set-up assistant actively supports mould installation. It specifies the optimum sequence of operating steps. These only need to be consecutively performed and confirmed. The installation technician selects the necessary sub-sequences, such as referencing (zeroing) of individual machine axes in a central screen page and starts them with the single push of a button. The control system then performs the relevant task automatically. A schematic display acts as an additional guide and no parameter entries are required.

The third step prompts the installation technician to enter the key data for the injection moulding process. This includes the material, screw diameter, mould type, projected area of the moulded part, shot weight, wall thickness and flow paths. Very little product data is thus necessary in order to have all the processing parameters such as temperatures, pressures and speeds calculated automatically by the Selogica control system. An extensive embedded database is used for this purpose.

In step four, the installation technician then determines which parameters the control system is to calculate automatically. Modular selection options make it possible to, for example, redefine only the injection unit temperatures when changing materials. All the other parameters can remain unchanged. Furthermore, on this set-up assistant screen, all the available monitoring and log functions can be automatically initialised "at the click of a button". The usual entry of parameters in a variety of screens and the selection of various monitoring and log functions "in the depths of the control system" is thus completely dispensed with.

The fifth and last step finally serves to "teach-in" the machine sequence by setting it up following the menu guidance sequence. In other words, all the installation technician has to do is move consecutively to the required positions and confirm them. All the parameter entries, as well as completion of the machine sequence, is performed automatically by the control system. The Selogica system ensures that all the machine functions selected in step one are taught-in. During the accompanying set-up procedure, protection mechanisms, such as the position for mould protection are determined. Once all five steps have been completed, the Allrounder has been fundamentally prepared for operation with very little effort.

The New Selogica "Set-up Assistant" module enables menu-guided, accompanied set-up, or so-called "teaching", of the entire injection moulding process in only five steps

Two main advantages of the Selogica "Set-up Assistant" module are of prime importance. Firstly, the installation technician no longer needs expertise in control systems in order to set up the entire injection moulding process. Moreover, preparation of the Allrounder for production is much faster than previously thanks to the high flexibility of the set-up assistant and the automatic functions which run in the background. The new Selogica module is, state Arburg, therefore a further, milestone towards the simple set-up of injection moulding machines.

www.arburg.com

High precision moulds for microfabrication

Microworks GmbH, founded in 2007 as a spin-off from the Institute of Microstructure Technology of the Forschungszentrum Karlsruhe (IMT/FZK), Germany, offers a specialised service for high precision tools used to produce metal micro devices by LIGA technology.

LIGA technology uses x-ray lithography to produce polymer structures with vertical and extremely smooth sidewalls of 100 to 2000µm thickness. These lithographic templates are then filled by electroplating to obtain micro- to millimeter-sized metal parts of the highest precision.

Properties of the electroplated material can be customised with respect to hardness, magnetic properties, wear resistance, etc. Inverse metal structures can be used as cavities for hot embossing or metal or ceramic injection moulding for µPIM components.

Microworks fabricates LIGA micro structures from Ni, NiCo, Au and hardened Au in cooperation with IMT/FZK. The smallest details can be less than 1µm, with heights between 100 and 2000µm. Dr. Joachim Schulz is the Managing Director, while Dr. Pascal Meyer is responsible for process development.

www.micro-works.de

Malvern Instruments to run short courses focused on particle and molecular characterisation

Malvern Instruments will run two one-day short courses as part of the established program at Pittcon 2011, taking place from March 13 -18, in Atlanta, Georgia, USA.

The first of these covers the fundamentals of particle sizing with an emphasis on light scattering techniques, and the second examines molecular and particle characterisation by dynamic light scattering and zeta potential.

Fundamentals of Particle Size Analysis with an Emphasis on Light Scattering Techniques

This course will take place on March 14 and will bring newcomers to the particle sizing field up to speed on the basics of particle size analysis.

The main techniques (sieves, sedimentation, electrozone sensing) will be covered with an emphasis on dynamic light scattering and laser diffraction.

Molecular and Particle Characterization by Dynamic Light Scattering and Zeta Potential

The second short course takes place on March 16 and will discuss, review and provide useful tips for dynamic light scattering (DLS, PCS, QELS), molecular weight and electrophoretic light scattering (zeta potential) measurements.

Registration is open for both via the Pittcon website.
www.pittcon.org

Expansion-matched heat sinks made by microMIM

Sintered Cu-W heat sink with a mounted laser bar and a copper n-contact sheet (Source SPIE Newsroom)

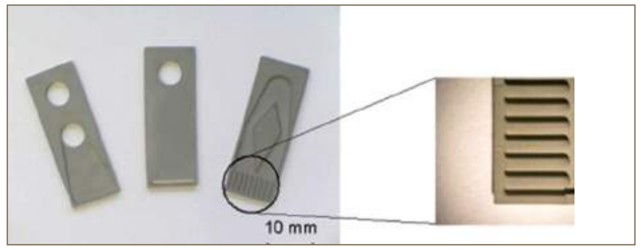
According to a recent report issued by SPIE, the international society for optics and photonics, laser bars (arrays of multiple parallel emitters on a semiconductor substrate) are on the verge of becoming a consumer product. It is estimated that more than a million laser bars will be needed within two to three years to meet the demands in the materials processing and medical sectors. This expected surge in demand should lead to a significant drop in the price of high-power diode lasers.

Interest has also been growing in using thermal expansion matched microchannel heat sinks with very high cooling performance to increase the reliability of high power diode laser bars. The report outlines research done at the Fraunhofer Institute for Laser Technology (ILT) Aachen, Germany, in cooperation with the Fraunhofer Institute for Manufacturing and Advanced Materials (IFAM), Bremen, Germany, to develop manufacturing technology to produce heat sinks made from fine (5µ) copper-tungsten (80:20) powder using µMIM. The research at ILT and IFAM was funded by AiF (German Federation of Industrial Research Associations).

The report states that combining tungsten, a material with a very low coefficient of thermal expansion (CTE) and moderate thermal conductivity, with copper, which has moderate CTE and high thermal conductivity (>400W/mK), allows to mass produce the complex flow-channel structures in the µMIM heat sinks economically and with the desired mechanical and thermal properties.

Densities of more than 98% are achieved during sintering. The sprue material can be recycled after moulding and reused. The sintered µMIM copper-tungsten heat sinks have a gallium arsenide (GaAs)-matched CTE, combined with high thermal conductivity. The report states that another advantage of µ-MIM is that the needed green injection moulded components can be joined together in a co-sintering process. In co-sintering, individual green bodies are positioned one on top of each other and are joined together at temperatures >800°C, with no need for additional joining steps. The joining material is said to be copper. As an alternative to µ-MIM, the researchers used silver diffusion soldering. Here, pre-sintered heat sinks are silver coated (which constitutes one additional processing step), braced together, and then also joined together at temperatures >500°C.

www.spie.org



Three 'green body' components of µMIM 80/20 Cu-W heat sink (Source SPIE Newsroom)

New MIM pilot plant to be established in Spain

Work on a new MIM pilot plant has started at the University of Castilla La Mancha's Energy Research Institute and Industrial Applications (INEI), in Central Spain. It is hoped that the plant, which is under the directorship of Dr Gemma Herranz and Dr Gloria Rodriguez, will contribute to the growth of MIM technology both in Spain and further afield.

The plant will consist of a laboratory with the capability to cover the complete MIM process. This includes equipment for the manufacture, design and assessment of MIM feedstock, an injection moulding machine to prepare parts for complete mechanical and magnetic analysis, a thermal debinding furnace, and a tubular sintering furnace able to work under different atmospheres and high vacuum conditions. The purchase of an additional high temperature furnace to cover studies related to ceramics is also scheduled.

"We are in the process of purchasing equipment and preparing students to collaborate in the implementation. Although we intend to work with some equipment this summer, the new pilot plant will be fully operational from January 2012," Dr Herranz told *PIM International*.

"This new facility will be a test centre for initiatives that have industrial potential. We therefore plan to cover a range of fields according to the needs of the companies with which we will work, as well as covering the essential scientific and technological needs that will allow an expansion of the use of MIM. Our ambition is to help transfer knowledge about MIM technology to domestic and European markets," stated Herranz.

"We would like to increase the awareness of MIM technology within Spanish industry. As you can imagine we are only at the very start of the journey compared with other countries. We cannot yet compare our situation with Europe's most advanced MIM centres in Germany, Austria or France, for example, but now at least we will have the facilities to start to contribute," stated Herranz. In addition to cooperative research, the plant will be used by students studying and researching for masters degrees and doctoral theses.

The laboratory's facilities will be supplemented by the mechanical characterisation equipment of the University's Metallic Materials Research Group, which includes durometers, tensile testing equipment, tribometry equipment, and microstructural characterisation facilities including optical microscopy with image analysis, SEM and XRD.

The plant is funded by the University of Castilla La Mancha, with the support of the European Union. The management team states that it has the support of six research groups within the University, ten major Spanish businesses and two important pan-European companies, as well as numerous high profile industry professionals and related associations.

"It is also our intention that the plant is open to exchanges by researchers from other research institutes interested in collaborating with our group. We will look for all possible opportunities to support the needs of national, European or international projects," stated Herranz.

www.uclm.es

Polymer Technologies looks to the international market, appoints new President, adds gas farm at MIM plant

Polymer Technologies Inc. (PTI), a custom plastic and metal injection moulding company based in Clifton, New Jersey, USA, has named Neal Goldenberg as its new president. Goldenberg previously served as Chief Financial Officer of PTI, where he implemented fundamental changes to the cost accounting system enabling a keener focus on profitable markets

Earlier in his role of Executive Vice President, Neal implemented a company wide ERP system leading to comprehensive efficiency gains. In this role he also initiated PTI's attainment of its ISO9001:2000 designation and AS9100B Certification in 2007.

In his new role, Goldenberg will be responsible for identifying new opportunities, both domestic and international, within the aerospace, medical and surgical markets. This includes aligning domestic and international reps as well as implementing strategies to increase PTI's presence in the Asian market.

"I am pleased to take on my new role as President and look forward to meeting the many challenges I will face within this position," said Goldenberg. "I have a very clear, strategic vision for this company that includes the identification and execution of opportunities that add value to our clients' businesses and I am confident that between my experience and the knowledge of the seasoned team here at PTI, this vision will be brought to fruition."


The company has installed Linde-hydrogen and AGL-nitrogen tanks

The company recently completed a significant equipment upgrade to expand its metal injection moulding capabilities by installing a Linde-hydrogen and AGL-nitrogen tank farm on company grounds. The gases are used during the sintering of MIM parts. PTI state that product quality is enhanced by tank farms, providing a cleaner gas supply versus bottled gas. In addition, an on-site tank farm allows the team at PTI to support uninterrupted sintering runs while avoiding frequent gas deliveries and bottle switchovers. The assurance of an interrupted gas supply was key in PTI's recent decision to hire weekend staff to run the sintering furnaces around the clock. PTI states that it joins the top 20% of toll sinterers industry wide by adding this capability.

www.polymertechnologies.com

Hunter Chemical supplies nickel powder

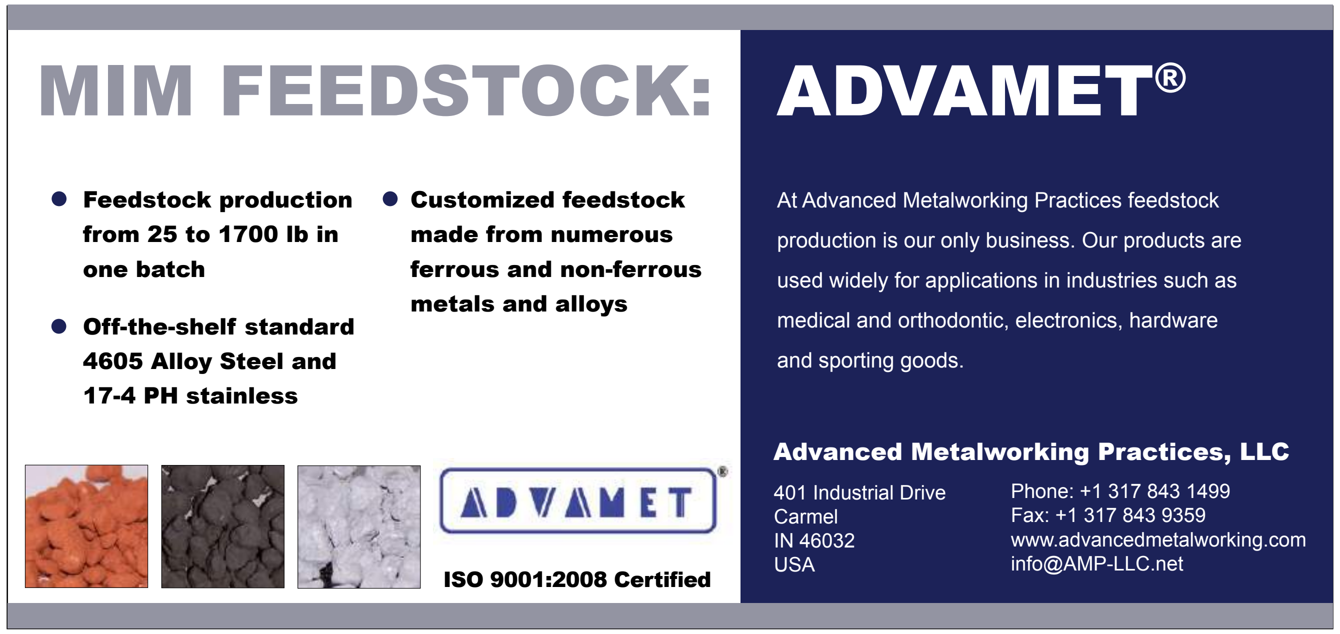
Hunter Chemical LLC, headquartered in Pennsylvania, USA, recently announced the availability of its nickel powder Grade AH50. AH50 is processed with the specific purity, morphology and particle size distribution needs of the sintered parts industry in mind.

Hunter Chemical LLC is an ISO9001:2008 certified supplier of nickel, cobalt and chrome raw materials. The company states that it maintains direct relationships with the world's primary producers of raw materials enabling it to offer competitive prices for finished products that meet PM and other application requirements.

Ben Brock, President of Hunter Chemical said, "We are pleased to provide another option for part producers. Many companies still have in-house powder blending operations or need to supplement with pure nickel from time to time. Our customers have learned from last year not to rely on one supply stream."

Grade AH50 is produced through a carbonyl process and is spherically shaped with a spiky morphology. Raw material is processed and tightly monitored to achieve a particle size distribution of 3-6 microns (FSSS). The company also offers free particle size analysis for metal powders to its customers.

www.hunterchem.com



US MIM producer Phillips Plastics acquired by Kohlberg

Phillips Plastics Corporation has been acquired by Kohlberg & Company L.L.C., a private equity firm based in Mount Kisco, New York, USA.

Brad Wucherpfennig, Phillips' current Chief Executive Officer will continue to lead the company and the entire management team will remain in place. "Today we go forward under new ownership, but our commitment remains the same – to provide our customers with a resource for state-of-the-art technology and engineering expertise that will give their products a competitive edge in the marketplace. Working with Kohlberg, we will be able to strategically grow our business to meet the needs of our customers worldwide," said Wucherpfennig.

Backed by the financial strength of Kohlberg's private equity funds, it is expected that the acquisition will accelerate Phillips' ability to serve its customers on a global basis. "We are excited to be working with the people of Phillips," commented Sam Frieder, Managing Partner of Kohlberg. "The company already possesses a strong market presence and recognised brand, we look forward to helping Phillips enhance and expand its service offering, which will directly benefit customers across all market segments."

Kohlberg plans to maintain current operations. "Partnering with Kohlberg represents an enormous opportunity for our company," said Wucherpfennig. "Together we will focus on expanding our business to provide increased value for our customers, shareholders, employees and the communities in which we live and work."

Phillips Plastics Corporation is a leading manufacturer of highly engineered injection-moulded plastic and metal products with annual sales of over $250 million. The Company employs 1,300 people in 14 locations throughout the United States, including design centers in Wisconsin and California, and a medical campus with 180,000 square feet of FDA registered facilities dedicated to high volume medical and clean room manufacturing. The company's medical operations are cGMP compliant and registered to 21 CFR parts 820, 210, and 211.

www.phillipsplastics.com

Höganäs reports best ever Q4

Höganäs AB, Sweden, has reported its best ever fourth quarter results. In a statement issued by the Swedish metal powder manufacturer, CEO Alrik Danielsson commented, "We are benefiting from our strong position on emerging markets, as volume growth in Europe has not been as high. Sales volumes in China and India reached new record levels in the quarter."

"The year as a whole exceeded our expectations by a good margin. The downturn in Europe late in the year was expected, and although uncertainty regarding demand conditions for the short term persists, we are looking to the future with confidence. Höganäs advanced its position in 2010," added Danielsson.

Fourth quarter 2010 results showed net sales of MSEK 1,612 (1,364), up 18% year on year, with sales volumes 10% higher.

www.hoganas.com

Rare earth industry develops outside of China

China's recent reduction in its 2011 export quota of rare earth minerals has caused great concern in many manufacturing industries around the world. China currently provides around 97% of the world's supply of these minerals and recent reports state that the government will reduce exports by up to 35% in the first half of 2011.

Rare earth minerals are used in many applications, from smart phones to wind turbines, with around 26% (35,000 t) used in the production of magnets. The use of neodymium-iron-boron (NdFeB) permanent magnets, produced via the powder metallurgy (PM) process, has seen spectacular growth since the technology was developed in the early 1980's. NdFeB magnets can also be produced via the powder injection moulding (PIM) route, allowing for small, complex shapes to be produced.

In an attempt to decrease the global reliance on China, both America and Australia plan to begin mining deposits of rare earth minerals.

In the USA, Molycorp has announced an expansion plan that is expected to provide an annual rate of up to approximately 40,000 metric tons of rare earth oxide (REO) equivalent per year at its Mountain Pass facility by the end of 2013. In Australia, Arafura Resources Ltd state that annual production of 20,000 tonnes of rare earth oxides from the Whyalla Rare Earths Complex, equivalent to about 10% of the world's supply, is on schedule to commence in 2013. Also in Australia, Lynas Corporation estimates the capacity of its Mount Weld mine will be 22,000 tonnes per year by 2014.

Molycorp and Hitachi to join forces manufacturing rare earth magnets in the USA

Hitachi Metals, Ltd., based in Japan and Molycorp announced in late 2010 that they have entered into an agreement regarding the planned formation of joint ventures for the production of rare earth alloys and magnets in the USA. The ventures are to be focused on the manufacture of neodymium-iron-boron (NdFeB) alloys and magnets.

"As the world's top manufacturer of NdFeB magnets, we are well positioned to satisfy the growing demand from global customers for these rare earth magnets, and to contribute to an energy efficient society," stated Nobuhiko Shima, President of NEOMAX Company of Hitachi Metals, Ltd.

Signing of definitive agreements, subject to the satisfactory conclusion of the feasibility study and other conditions, for the joint venture to produce rare earth magnets will take place in late 2011.

"We look forward to launching these joint ventures for production in the USA together with Molycorp, which has long experience and deep expertise in the rare earth industry," added Shima.

"These joint ventures are an integral part of Molycorp's 'mine-to-magnets' business plan, and they move our Company and the United States one step closer to realising the strategic goal of re-establishing a complete rare earth manufacturing supply chain in the USA," stated Mark Smith, Molycorp's Chief Executive Officer.

Sinter joining produces complex micro MIM parts

Miniaturisation has required the development of new manufacturing methods for complex micro components, particularly those which have integrated functions. One example is a ferrous-base micro check valve developed using metal injection moulding (MIM) which contains a freely moveable ceramic ball. The operation of the valve is based on the movement of the valve ball which is pressed against the internal valve body at the valve outlet when the fluid passing through flows in a reverse or forward direction, thus closing the valve by blocking either of the outlets (Fig.1).

The geometry of such a micro check valve would be difficult to manufacture using conventional manufacturing methods, and impossible if the ceramic valve ball is incorporated in the production process. Researchers at the Institute of Production Science (wbk) at the Karlsruhe Institute of Technology, Germany, have found a unique solution to this problem which involves combining two separate MIM parts joined together during sintering with the ceramic valve ball in situ. C. Munzinger and his colleagues at the wbk reported on their work in a paper presented at the PM2010 Powder Metallurgy World Congress . The research was supported by the German Research Foundation (DFG) within the Collaborate Research Center 499 program.

The authors stated that because PM (and MIM) components are subject to shrinkage during sintering, the parting plane of the valve body was chosen as parallel to the flow direction and vertical to the longitudinal axis. The rotationally symmetrical valve halves were fitted with tapered joining surfaces in the form of internal and external tapers that fit into each other (Fig. 2). Tests were carried out in order to identify the optimum geometry and roughness parameters of the joining surfaces required for sinter joining in order to achieve maximum strength.

Some 47 valve-like pairs of experimental samples were made from carbonyl iron powder (d_{50} < 4 µm) and 8 wt% of a polyolefin wax-based binder that can be thermally debound. The injection moulded samples were all manufactured as hollow cylinders with an external diameter of $d_{a,green}$ = 3 mm and an internal diameter of $d_{i,green}$ = 2 mm, produced by turning. The joining surfaces were created having taper angles which varied between 11° and 15° in order to determine its impact on the quality of the join (Fig. 3).

Surface roughness of the samples was deliberately varied by using different machining methods to manufacture the joining surfaces.

After sintering the properties of the resulting joined surfaces in the 47 samples were tested by means of internal pressure tests and 20 samples were subjected to tensile testing. The tests showed that sinter joined connections can withstand internal pressures of ≥ 700 bar and can achieve a tensile strength of ≥ 216.37 N/mm² if built to a suitable design. This tensile strength is equivalent to about 60% of that of full density sintered carbonyl iron, and results suggest that improvements of angular accuracy of the taper could further increase the tensile strength achieved. Also key in the results obtained are the individual parameters required for successful sinter joined connections so that a specific strength can be given in the MIM part specification.

www.kit.edu

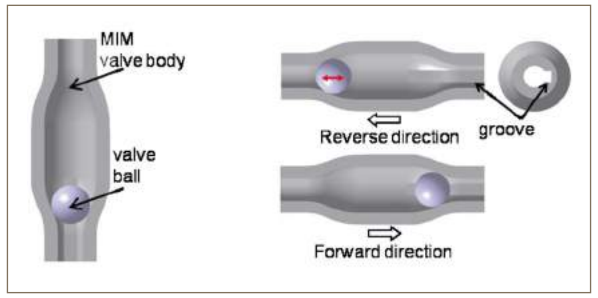


Fig. 1 Left, design of the micro check valve; Right, principle of operation of the micro check valve. (From paper by C. Munzinger, etal (Karlsruhe Institute of Technology) presented at PM2010 World Congress, Florence, October 2010, and published in Congress Proceedings available from EPMA Shrewsbury, UK)

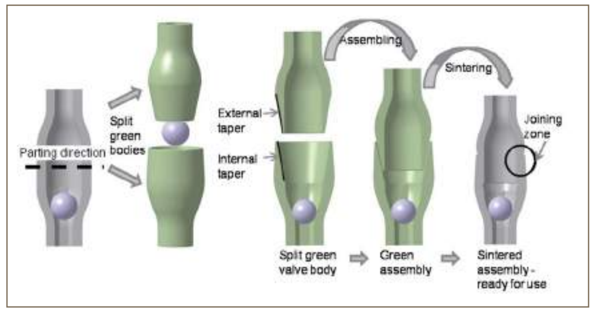


Fig. 2 Procedure for producing the micro check valve based on sinter joining. (From paper by C. Munzinger, etal (Karlsruhe Institute of Technology) presented at PM2010 World Congress, Florence, October 2010, and published in Congress Proceedings available from EPMA Shrewsbury, UK)

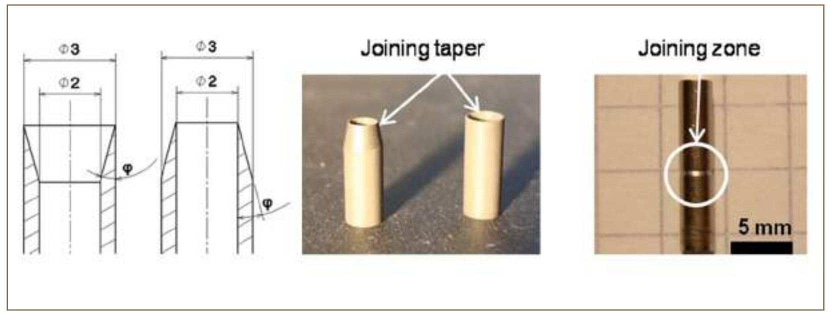


Fig. 3 Geometry of the two sample halves of the micro check valve, valve parts in the green state and sinter joined assembly. (From paper by C. Munzinger, etal (Karlsruhe Institute of Technology) presented at PM2010 World Congress, Florence, October 2010, and published in Congress Proceedings available from EPMA Shrewsbury, UK)

Ecka Granules/ SCM to place greater focus on aluminium powders

Ecka Granules / SCM Metal Products, headquartered in Research Triangle Park, North Carolina, USA has announced that it will put a greater emphasis on its aluminium PM business.

At its inaugural global R&D meeting between SCM and Ecka Granules, CEO Barton White introduced Dr. Shuhai Huo as the Group's new Vice President of Research and Development. Dr. Huo comes to the Group from the University of Queensland in Australia where, along with Professor Graham Schaffer, he has been instrumental in developing innovative materials for the aluminium PM industry.

Dr. Huo and his team from the US, Germany and Austria will look to expand its product portfolio, develop the next generation of aluminium PM powders and build on the technical strengths already in place at Ecka Granules.

"Along with our strategic customers, the powders we develop will target new aluminium PM applications that previously have not been feasible due to technically demanding properties," stated White.

www.ecka-granules.com
www.scmmetals.com

Boron addition improves fatigue properties of MIM titanium

It is well known that a small addition of 0.1wt% boron to Ti-6Al-4V alloys during melting and casting decreases grain size in the cast product by an order of magnitude thereby enhancing mechanical properties. The Institute of Materials Research at GKSS Research Centre in Geesthact, Germany, has been actively researching into MIM of titanium alloys for some time, and whilst it has been possible to produce Ti-6Al-4V alloys by MIM with excellent tensile properties (UTS >800 MPa) and elongation values of >14% the fatigue endurance limit is often not sufficient for demanding applications. For example, GKSS has already published data which shows that fatigue strength of MIM Ti-6Al-4V alloy at 10^{-7} cycles is around 350-380 MPa, which is significantly lower than the typical value of around 600 MPa, for annealed, wrought material. This could preclude lightweight Ti-6Al-4V MIM from many high performance components for permanent medical implants, automotive and aerospace parts.

A paper by O.M. Ferri, T. Ebel, and R. Bormann, presented at the 2010 PM World Congress in Florence and published in *Advanced Engineering Materials* (January 17, 2011) outlines work done at the GKSS Research Centre to improve the fatigue properties of MIM titanium alloys by using small additions of boron to improve the microstructure, as has been previously done in casting. Boron powder (< 2μm) was added to spherical gas atomised Ti-6Al-4V powder (<45μm) in quantities of 0.1 and 0.5 wt% during MIM feedstock preparation. The feedstock contained 35 vol% binder. Subsequent to injection moulding the paraffin wax portion of the binder was removed from the samples by chemical debinding. This was followed by thermal debinding, to remove the remainder of the binder, and vacuum sintering with both processes carried out in the same furnace. MIM Ti-6Al-4V alloy samples with 0, 0.1 and 0.5 wt.% boron added were sintered at 1250°C with further samples containing 0 and 0.5 wt.% of boron also sintered at 1400°C.

In order to minimise any possible influence of surface quality difference on the fatigue behaviour, some samples were exposed to shot peening after sintering. Shot peening was done with an air-blast machine using zirconia particles with a diameter of 500 μm. Four-point bending high cycle fatigue testing was done with the fatigue endurance limit defined as 10^{-7} cycles. The fatigue results of Ti-6Al-4V-0.5B samples with and without surface treatment were compared to the MIM Ti-6Al-4V obtained from a previous work published by the authors*.

The authors reported that TiB particles formed during the sintering process (in situ reaction) as indicated by the white arrows in Fig. 1b and Fig. 1c. The addition of boron apparently promoted a refinement of the microstructure; however, at the sintering temperature of 1250°C, an increase of porosity was observed with 0.1 wt.% boron reaching approximately 96% of theoretical density. The addition of 0.5 wt.% boron decreased sintered density even further to approx 91%. Therefore, in order to obtain samples with high density (>95%) and at the same time a finer microstructure, samples with 0.5 wt.% of boron addition were sintered at the higher temperature of 1400°C for 2 hr. This resulted in a significantly finer microstructure than was observed for the standard MIM Ti-6Al-4V alloy. The authors conclude that the finer microstructure is obtained mainly due to the fact that TiB particles act as new nucleation sites for the phase (heterogeneous nucleation), and as pinning sites to restrict grain growth. Such a pinning effect promotes a delay in the starting point of the sintering process. On the other hand, they state that this effect is beneficial for densification in the later stages of sintering due to the delayed separation of pores and grain boundaries.

A significant increase of yield strength and UTS is observed for the Ti alloy containing boron sintered at 1400°C compared with the standard MIM Ti-6Al-4V alloy as shown in Table 1. However, the ductility decreases by approximately 3 %. The significant improvement in the fatigue behaviour to an endurance limit of approximately 640 MPa is most probably related to the finer grain size. The results presented in this investigation demonstrate that it is possible to produce MIM titanium alloy parts with sound mechanical properties even in terms of high cycle fatigue behaviour.

www.hzg.de

* Published in Materials Science and Engineering A 504 (2009) 107-113

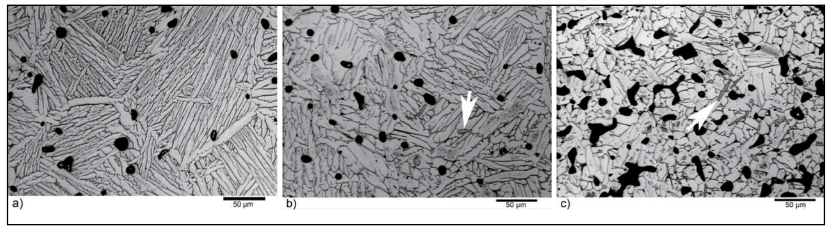
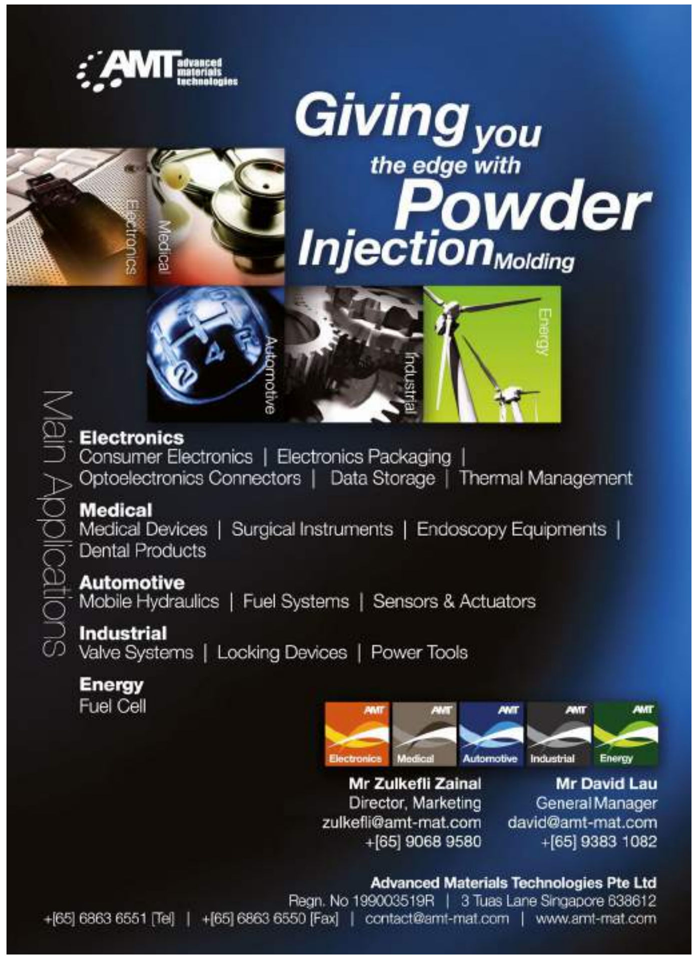


Fig. 1. Microstructure of Ti-6Al-4V sintered at 1250 °C with different levels of boron addition: a) 0 wt.%, b) 0.1 wt.% and c) 0.5 wt.%. (From paper by O.M. Ferri, T. Ebel, and R. Bormann (GKSS Geesthacht) presented at PM2010 World Congress, Florence, October 2010, and published in Congress Proceedings available from EPMA Shrewsbury, UK)

Microstructural features and tensile properties	Ti-6Al-4V	Ti-6Al-4V-0.5B
Relative density [g/cm³]	4.26	4.31
Porosity [%]	3.3	2.3
O [μg/g]	1917 ± 30	1960 ± 21
C [μg/g]	577 ± 13	390 ± 33
N [μg/g]	147 ± 9	164 ± 6
$\sigma_{0.2}$ [MPa]	744 ± 5	787 ± 1
UTS [MPa]	852 ± 2	902 ± 3
ε_f [%]	14.7 ± 0.7	11.8 ± 1
Grain size [μm]	247 ± 22	18 ± 5

Table 1. Microstructure and tensile properties of Ti-6Al-4V and Ti-6Al-4V-0.5B sintered at 1400°C. (From paper by O.M. Ferri, T. Ebel, and R. Bormann (GKSS Geesthacht) presented at PM2010 World Congress, Florence, October 2010, and published in Congress Proceedings available from EPMA Shrewsbury, UK)



Neodymium enhances microstructure/properties of MIM HDH titanium

In a further report on the influence of alloying additions on the microstructure and mechanical properties of MIM titanium, Hao He and colleagues from the State Key Laboratory of Powder Metallurgy, Central South University in Changsa, China, describe the impact of adding Nd to hydride-dehydride (HDH) titanium powder. The authors state that although HDH titanium powders are cheaper than gas atomised powders, the large surface area of the fine HDH powders used in MIM makes reduction of surface oxides (around 0.35%) 'very difficult, if not impossible'. Furthermore, the MIM processing steps of binder addition and later removal helps to aggravate the oxide problem. After debinding and sintering, the final Ti parts generally have a total oxygen content substantially higher than the amount typically allowed in commercially pure titanium (ASTM Grade 4).

In order to remove oxygen from HDH titanium the authors undertook research into the use of alloying additions which have a high affinity to oxygen, and which could be used as an 'oxygen scavenger' during the sintering process to keep oxygen values at acceptable levels. They state that Ca, Al, Nd, Gd, and Y could be added to fine HDH titanium powders used in MIM to prevent their reaction with oxygen. Earlier work on a TiNdAl alloy system used to produce conventional PM parts indicated that Nd reduced oxygen content and significantly improved tensile strength and ductility values. However, it remained unclear as to what the reaction mechanism of oxygen and Nd was, or the amount of Nd needed for successful scavenging of the oxide. In the current study the authors undertook to define the effect of Nd addition on the microstructure and mechanical properties of HDH titanium powders using MIM processing .

HDH Ti powder (O_2 content 0.33 wt%, D_{50}=75μm) was mixed with 0 – 20wt% Nd powder (D_{50}=45μm) and a multi-component binder containing HDPE, paraffin wax and stearic acid with powder loadings of 55 vol.%. The granulated feedstock was injection moulded, and the binder was removed through a combination of solvent (n-heptane) followed by thermal debinding at 800°C (vacuum, 10^{-1} Pa). The specimens were then sintered at 1200°C (vacuum, 10^{-3} Pa) for 2 hrs. Table 1 shows the relative densities and mechanical properties of the various sintered TiNd specimens. It was found that relative density, tensile strength and elongation increase with increasing Nd content with an optimum value of Nd put at 15 vol%.

The authors reported that the Ti15Nd specimens appeared to consist of three distinct phases, titanium matrix (white), Nd clusters (grey) and pores (black), see Fig. 1. The residual porosity is reduced both in size and amount when Nd was added, with small and spherical pores distributed inside the grain while a few large pores are trapped in the Nd clusters. Although there are differences in the porosity and pore distribution, the grain size of TiNd specimens is said to be similar to pure titanium.

In addition, a liquid phase can be found along the grain boundaries. The melting temperature of Nd is around 1024°C, so when sintering at 1200°C, Nd powder melts to form a liquid between the particles and wets the titanium phase, thereby improving the density. The small Nd particles, which are uniformly distributed in the titanium matrix effectively absorb the oxygen atoms during sintering and have been identified as Nd_2O_3

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Nd nominal content (%)	Relative density (%)	Tensile Stress (MPa)	Elongation (%)	Total oxygen Content (%)
0	94.9	386	0	0.70
5	97.2	661	2.4	0.65
10	97.8	644	4.5	0.72
15	98.2	634	6.5	0.69
20	96.5	630	1.0	0.70

Table 1 Relative densities and tensile properties of the sintered MIM TiNd specimens. (From paper by Hao He, Yimin Li, Liang Xiong, Jung Zeng (State Key Laboratory, Central South University, China) presented at PM2010 World Congress, Florence, October 2010, and published in Congress Proceedings available from EPMA, UK)

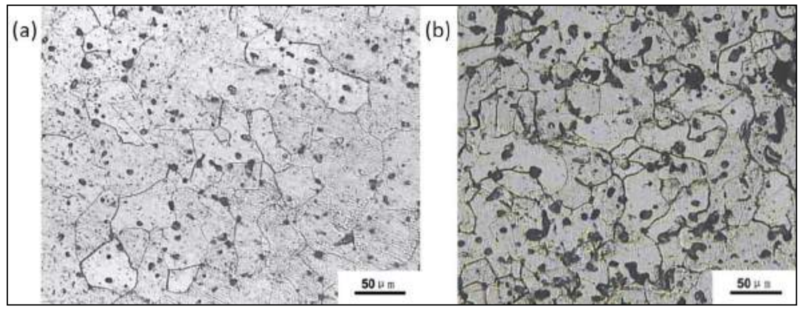

Fig. 1 Micrographs of (a) pure Ti and (b) Ti-15%Nd specimens liquid phase sintered at 1200°C (From paper by Hao He, Yimin Li, Liang Xiong, Jung Zeng (State Key Laboratory, Central South University, China) presented at PM2010 World Congress, Florence, October 2010, and published in Congress Proceedings available from EPMA , UK)

PowderMet2011 conference programme now available

PowderMet2011, the latest in the series of annual PM conferences sponsored by the Metal Powder Industries Federation (MPIF) and APMI International, will highlight the latest developments and trends in metal powders, as well as powder metallurgy and PIM processes, products, and applications. The event takes place from May 18–21 at the San Francisco Marriott Marquis Hotel in San Francisco, California. The 2011 International Conference on Tungsten, Refractory & Hardmaterials VIII will run concurrently with PowderMet2011 at the same location.

The conference technical program, now available on-line and in print, will feature over 225 technical presentations by authors from over 30 countries. A trade exhibition will showcase leading suppliers of metal powders and particulate materials, processing equipment, and PM products.

www.mpif.org

San Francisco Marriott Marquis Hotel, venue for PowderMet2011

Back to basics: Metal Injection Moulding of copper feedstock

Complex shaped sintered copper components are increasingly being produced by metal injection moulding (MIM) for applications such as heat sinks used in high thermal conductivity devices for electronics systems and modules used to cool high-power Insulated Gate Bipolar Transistors (IGTBs). However, whilst a considerable amount of literature exists on the processing and characteristics of ferrous and stainless steel materials produced by MIM, there are few detailed reports on the MIM processing of copper powders.

This is to some extent being rectified through research being undertaken at the Dept. of Mechanical Engineering at the University of Technology Petronas, in Perak, Malaysia, in cooperation with the Metal Injection Molding Laboratory at the Advanced Materials Research Centre (AMREC) based in Kulim, also in Malaysia.

In a recent paper published in the Journal of Applied Sciences (Vol.10, No 24, 2010, 3295-3300) G. Goudah and colleagues at the Petronas University report on their investigation into the preparation and characterisation of three formulations of MIM feedstock based on high purity (99.95%) spherical (gas atomised) copper powder with a particle size distribution of 4 to 22 μm and using a polymeric binder system (Table 1). This binder comprises paraffin wax as a major component (65%) plus high density polyethylene (HDPE – 30%) and stearic acid (5%). The degradation temperature of this binder system already starts at 170°C so the processing temperatures during mixing and injection moulding should be lower.

Four feedstocks were prepared to give powder loadings ranging from 55 vol% to 61 vol%. After low pressure injection moulding (4 bar) the copper parts were first solvent debound in heptane to remove the lower stability paraffin wax and stearic acid components of the binder from the moulded pieces. The HDPE has the effect of holding the remaining powder particles together whilst the lower stability parts of the binder are being removed and thereby helps to maintain component shape during debinding. The interconnected pore channels generated inside the compacts during solvent debinding in turn allows the gaseous products produced during thermal debinding under argon atmosphere to diffuse harmlessly out of the structure. Sintering was done at 900°C in the same furnace, also using argon to produce defect free parts.

The authors describe the heating rates used during thermal debinding and sintering for the four different copper powder feedstocks, and also the results on sintered density and shrinkage. It was found that increasing the powder loading in the feedstock increased density to 7.87 g/cm^3 or 88% of theoretical. They state that optimising the processing conditions should allow >97% of theoretical density to be achieved with good mechanical properties of the sintered Cu MIM parts. The dimensional shrinkage of the sintered Cu MIM samples was found to be nearly the same in all three dimensions (Table 2).

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Binder component	Weight %		
	Formula 1	Formula 2	Formula 3
Paraffin wax	55	65	70
HDPE	40	30	25
Stearic acid	5	5	5

Table 1 Binder system formulation used in Cu metal injection moulding feedstock. (From paper by G. Goudah, etal published in Journal of Applied Sciences No 24, 2010, pp 3295-3300)

Feedstock (Vol.%Cu)	Shrinkage %		
	Length	Width	Thickness
55	11.89	11.21	11.29
57	10.52	10.54	10.56
59	10.13	10.02	10.07
61	9.62	9.21	9.94

Table 2 Dimensional shrinkage of Cu metal injection moulded test parts. (From paper by G. Goudah, etal published in Journal of Applied Sciences No 24, 2010, pp 3295-3300)

CIM clevis sensor for online substance concentration measurements

Robust and acid-resistant clevis sensors with a capacitive sensor effect are used in the online measurement of substance concentrations in pipeline systems. With this method even low substance concentrations down to 1/10th of 1% can be identified. Zirconium oxide (tetragonal zirconia polycrystal TZP) was found to give the required properties for the clevis sensor and ceramic injection moulding was found to be the only cost effective manufacturing method for series production of the complex part.

Researchers from Technology Platform for Product Miniaturisation in Saxony-Anhalt at the Otto-von-Guericke University of Magdeburg, Germany, recently reported at the "Electronic System-Integration Technology Conference" (ESTC) held in Berlin, September 2010, that they used a 3D CAD model to design the sensor element into which was incorporated a flanged joint for problem-free connection to pipelines. The TZP material behaviour in the injection moulding process was simulated with a FE-tool, and the simulation results were used for the optimisation of the IM tool. The developed zirconia clevis sensor was successfully tested in multiple fluidic systems.

www.uni-magdeburg.de

Submitting News

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

Powder Injection Moulding in the aerospace industry: Opportunities and challenges

After several decades of cautious and discreet interest in Powder Injection Moulding, there now appears to be a growing level of enthusiasm for the technology from the commercial and military aerospace industries. In the following exclusive report for *PIM International*, Prof. Randall German explores the history of MIM and CIM in aerospace component fabrication, and outlines the challenges that are faced by producers to succeed in the market. A number of noteworthy aerospace components are presented.

Aerospace component fabrication using metal powder injection moulding (MIM) traces back to the late 1970s. This application area was one of the first to employ MIM. However, after a burst of early demonstration successes, the penetration of MIM into commercial and defence markets has been relatively slow. That may change soon as evidenced by increased research and development activities, increased awareness, improved quality systems, and a need for new application areas that differentiate from commodity MIM products.

This article traces the history of powder injection moulded aerospace materials and applications to provide a status report on prospects. Several opportunities are evident in the next few years. The downside is that significant investment is required to take on some of the large, longer-term projects, largely because qualification testing requires about three years of effort. A balance between technical developments and market management is envisioned to bring about significant gains in aerospace applications.

'aerospace casting cores constitute about $100 million in annual PIM sales, or about 8% of global PIM production'

Introduction

Aerospace applications for powder injection moulding are not new. The first two MPIF awards for metal powder injection moulding (MIM) were a screw seal used on a Boeing aircraft and a niobium alloy thrust-chamber and injector for a liquid-propellant rocket engine developed under US Air Force contract for Rocketdyne [1]. Indeed, for several years Rocketdyne maintained its own MIM facility. The early successes, by actors such as Ray Wiech, Ron Rivers, and Stan Zalkind, led to a proliferation of larger-scale developments, many of which are evident in the more than 360 firms that practice PIM today.

By the early 2000s, alumina and silica ceramic casting cores used in the fabrication of jet engine turbine blades became a dominant application for PIM. The alumina cores were highly valued since they enabled the directional growth of elongated or single crystal superalloy blades with precise internal cooling passages. Even today casting cores remains the single largest commercial use of PIM and two new facilities have opened in recent years; Chromalloy in Tampa, Florida and ENGIMICS in Ticino, Switzerland.

To properly appreciate the importance of the aerospace casting core field (and the related applications in industrial gas turbines and marine turbines), consider that a typical casting core ranges in cost from $10 to $100. The higher cost is associated with the more precise cores used for the highest pressure regions in the turbine. As a perspective on this application, note that about 400 cores are used in the fabrication of each jet engine and about 4,000 engines are built per year (new plus replacement engines). This means that aerospace casting cores constitute about $100 million in annual PIM sales, or about 8% of global PIM production.

Part of the justification for the component cost comes from the tight tolerances. Generally, casting cores are produced with critical dimensions held to a coefficient of variation (allowed standard deviation in size divided by mean size) of 0.02% to 0.05%. This is much more precise than seen elsewhere in PIM. Indeed, user audits of typical MIM parts show production is often only holding a coefficient of variation ten times larger, in the 0.3% to 0.5% range. Firms servicing the casting core market are some of the largest PIM producers; for example, one firm reported annual sales of $91 million a few years ago when the aerospace industry was at record levels.

Although casting cores are not directly used in the jet engine, still the application and its tight specifications require aerospace quality levels in terms of testing, process control, certification, record keeping, and first article qualification protocols. These quality criteria are more rigid than typically encountered in other PIM products.

Yet in spite of several early efforts in aerospace, today PIM aerospace applications are considered a new frontier. In a survey of the MIM industry it is surprising to learn how many firms have activities in this market. However, few custom MIM firms focus on the aerospace market. Most likely this is because product development and qualification times are long compared with many other applications. So a MIM firm might participate in a few longer term aerospace efforts, but overall their business is dominated by nearer term projects in medical, dental, industrial, automotive, electronic, firearm, or other areas that are faster to develop.

Why might aerospace be the next growth engine for MIM? Several indicators come to mind that include the following:

- Improved awareness about MIM in both the military and commercial aerospace sectors, coupled with a push to lower manufacturing costs (the US has already spent $50 billion developing the F-35 fighter and this sort of cost escalation is not sustainable).
- A significant increase in studies, publications, and presentations. Today 25% of the MIM literature deals with aerospace components, materials, or designs, and this is a significant step up from 10 years ago.
- MIM has historically undergone sales growth in a sector on the heels of user education via publications and presentations, and prior studies show in general the peak in publications leads the significant sales gains by about seven years.

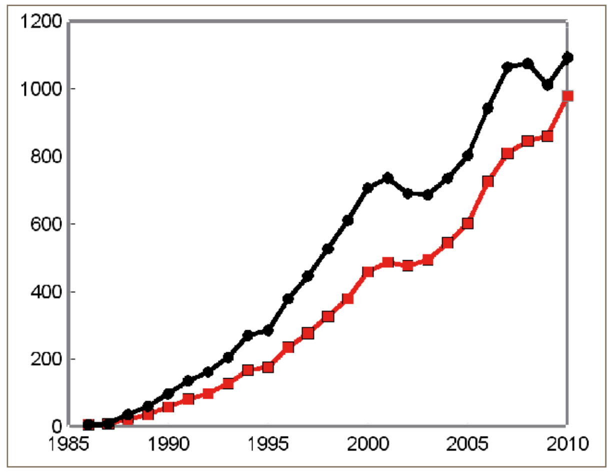

Fig. 1 The global annual sales history in millions of dollars for powder injection moulding (upper black curve) and for metal powder injection moulding (lower red curve) since statistics were first collected in 1986. For 2010 the total PIM sales were just short of $1.1 billion

Primary MIM aerospace materials
Stainless steels:
316L
410
420
17-4 PH
13-8 PH
Superalloys:
Hastelloy X
Inconel 625
Inconel 713C
Inconel 718
Nimonic 90
Udimet 700 and variants
Titanium alloys:
TiAl
Ti-6Al-4V

Table 1 The primary MIM aerospace materials processed today

- Concern over loss of market as evident by the migration of high volume MIM component production to lower cost regions; North American and European MIM firms are seeking out markets where there is no Asian presence - aerospace and medical jump to the top of the list with strong local markets and excellent prospects for profits.
- Quality systems have significantly improved from the early MIM days when Allan McMillan, head of General Motors manufacturing research, said, "... not in my cars" with respect to MIM; today the bulk of the MIM industry is much better and several firms have moved to meet aerospace quality concerns.
- Technical issues associated with meeting property goals are largely settled, although most of that literature is carefully guarded, still the data on specific components show MIM can satisfy aerospace needs.

History

Early PIM efforts in aerospace and the closely aligned military/defence sectors were impressive. I remember serving as judge on the MPIF "Part of the Year" panel and seeing one of the early MIM entries. The shapes were complex, the surface finish was outstanding, and the mechanical properties were unrivalled in PM outside hot isostatic pressing or powder forging. A hinge for a US Air Force jet fighter fabricated by MIM using 17-4 PH stainless steel caused much discussion by the judges. It had an impressive tensile yield strength of 1070 MPa (155 ksi) and far outstripped any of the press-sinter entries. Indeed, the combination of features, shape complexity, cost, and performance led to several early MIM aerospace victories.

Generally aerospace applications are associated with a few materials - high strength stainless steels, superalloys, titanium alloys, and some cobalt-chromium. By the late 1980s all three materials were demonstrated for aerospace applications. Diehl *et al.* [2-5] reported impressive properties for MIM Udimet 700 superalloy subjected to a final hot isostatic pressing densification step. Subsequent reports added IN 625 and IN 718 superalloy compositions, and the testing expanded to include hot tensile testing, creep, and toughness [6-9]. Honeywell moved MIM into larger superalloy components, up to 2 kg, and provided an impressive array of MIM alloys and properties [10-13]. By 2003 the MIM process was demonstrated on a range of aerospace compositions and components. Table 1 lists today's primary MIM aerospace materials.

Various reports detailed the powder-processing-property details, investigated different types of powders (water atomised, master alloy, versus gas atomised), and added to the test data [14-17]. One byproduct of the early work emerged in the form of automotive turbocharger components, where lower cost superalloy MIM components were adopted [15,18,19]. Like aerospace, the early MIM turbocharger parts were subjected to 100% radiological inspection. Thus, non-aerospace applications were enabled by the early aerospace developments.

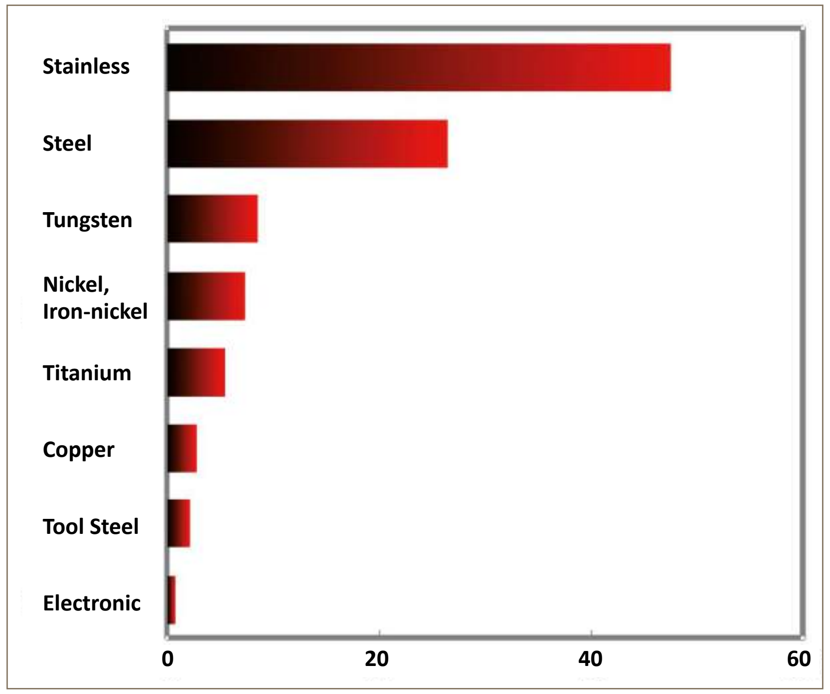

Fig. 2 Sales partition by metallic materials based on percent value of product sold (not tonnage), showing how stainless steels continue to dominate MIM applications

Current activities

First a few comments on the MIM and PIM markets by the end of 2010. Metal powder injection moulding is a subset of PIM, and is often designated as MIM. There are about 365 PIM operations globally, and about 77% of those firms produce metallic components. In 2010, as plotted in Fig. 1, PIM reached nearly $1.1 billion in global sales, and the MIM subset exceeded $920 million in sales. The global sales for 2010 were enhanced by increased production of components for automotive, medical, firearm, and consumer products, but fewer industrial, hardware, cutting tool, watch, computer, dental, and electronic devices (except for the higher end

cellular telephones). A large gain came from two Apple products, the iPhone and iPad, each of which became very popular with consumers and put some interesting stress on the MIM supply chains. Today the MIM sales distribution is roughly 21% in North America, 27% in Europe, 49% in Asia, with a small amount distributed across the rest of the world.

Production is heavily skewed toward stainless steels, amounting to 53% of all MIM sales as illustrated in Fig. 2. This is also true in aerospace MIM [20]. After stainless steels, MIM metals include 27% steels, 10% tungsten alloys, 7% iron-nickel compositions (mostly for magnetic applications), 4% titanium and titanium alloys, 3% copper, and other materials such as cobalt-chromium, tool steel, and special electronic alloys. The nickel alloys, primarily superalloys, account for about 2% of global MIM sales. The lower cost stainless steels are used extensively in aerospace applications.

Applications for MIM in aerospace components, including military and defense aircraft parts, have been demonstrated for more than 30 years. Today, the aerospace MIM sector amounts to a small portion of the global market. The current activity is in the range of $18 to $25 million in sales, so aerospace component production is a small fraction of the overall activity. But a new wave of effort is starting, driven by envisioned savings. About five firms are leading in this area, but more firms are participating.

Example Mechanical Properties for MIM Superalloys [2,11,14,28-30]				
Alloy	Yield strength, MPa	Tensile strength, MPa	Elongation, %	Test
Nimonic 90*	906	1249	22	20
IN 625	230	600	21	20
IN 718	1056	1380	29	20
IN 718	900	2065	4	20
IN 713	800	1043	---	650
IN 713	400	455	---	900
Udimet 700	910	1340	14	20
Udimet 700*	1100	1400	12	20
Udimet 720	677	1000	---	650
Udimet 720	595	656	---	900
** hot isostatically pressed*				

Table 2 Selected superalloy tensile property reports

Evidence of MIM in aerospace can be gathered from several areas, especially from conversations with metallurgists at aircraft and jet engine firms. In 1992 Chong [17,21] of McDonnell Douglas Missile Systems Company provided early test results on MIM stainless steels. She found internal void defects, unhealed weld lines, low tensile properties, large grains, and out of specification carbon. Since those days much progress has been made. Efforts in MIM aerospace components are evident by the prior programs involving Boeing, US Navy, Pratt and Whitney, Moog, McDonnell Douglas,

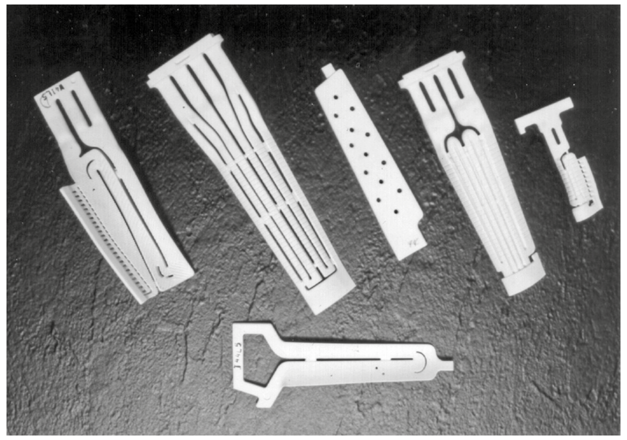

Fig. 3 Examples of alumina casting cores, which meet the rigors of PIM manufacturing for aerospace and represent a large market segment

Fig. 4 One example of a large MIM superalloy component, known as the flow body, it was widely touted as a success, but failed to reach production

Rolls Royce, General Electric, Thiokol, Rocketdyne, Eaton, GKN Aerospace, MTU, Parker Aerospace, Elliott, and Honeywell. Parallel efforts are evident in land-based turbines, for example at Solar Turbine. Now MIM is identified as a cost saving process for the aerospace industry [22,23]. For example, in a rare disclosure of mechanical property data Voice of Rolls Royce [24] reports excellent MIM properties for one of their 316L applications; namely 170 MPa yield strength, 515 MPa ultimate strength, and 50% elongation.

Technology status

The materials sought for aerospace/defence aircraft components are straightforward reflections of long established compositions. These include stainless steels, nickel superalloys, titanium alloys, and possibly some cobalt-chromium. Several aerospace counterbalance and inertial devices rely on tungsten heavy alloys. The titanium most commonly in the form of the Ti-6Al-4V alloy. The nickel superalloys include several compositions as listed earlier. Reportedly IN 725 is nearing qualification. Cobalt-chromium alloys are already in the MIM arsenal, and might be applied to some of the lower temperature components, such as in fuel pumps. Even titanium aluminides are under consideration [25-27]. This follows on from several successful material science projects involving injection moulding of intermetallics and intermetallic matrix composites.

These alloys are offered by several MIM firms, including Polymer Technologies, Maetta Sciences, PCC Advanced Forming Technology, Parmatech, Advanced Materials Technology, Lupatech Steelinject, and others. One of the spin-off growth areas from nickel-base superalloy work is evident in the use of superalloys for automotive turbochargers. In the area of ceramics, there is a large aerospace use of PIM for casting cores, with probably a half dozen large actors relying on lower moulding pressures to form alumina or silica airfoil shapes. Fig. 3 shows examples of these geometries.

'One of the spin-off growth areas from nickel-base superalloy work is evident in the use of superalloys for automotive turbochargers'

With respect to mechanical properties, generally the choice is to use gas atomised powder. After sintering, the components are subjected to hot isostatic pressing to ensure best properties. Selected superalloy tensile property reports can be seen in Table 2.

Test data at room temperature are not necessarily good predictors of high temperature or fatigue behaviour. Accordingly, for critical aerospace applications stringent test data are required under relevant conditions. This means more than just a few tensile tests. Advanced testing has been performed on several MIM superalloys and includes high temperature tensile testing, as well as room temperature fatigue, high temperature fatigue, creep and stress rupture testing, fracture toughness, oxidation, and corrosion testing [14,28-39]. Most of these properties are measured during the component and process qualification effort and the results are not in the public domain for a variety of reasons. A few examples that have been disclosed include the following:

- IN 718 processed by MIM has a fatigue endurance strength at room temperature of 385 MPa.
- IN 718 subjected to MIM and HIP with a final heat treatment is reported to give a fatigue endurance strength at room temperature of 452 MPa.
- IN 713 fabricated by MIM and HIP has a 373 MPa yield strength at 900°C.
- IN 625 by MIM has excellent corrosion resistance in a range of environments.

In a parallel manner, titanium MIM compositions have evolved with property combinations attractive for aerospace applications. Indeed, the technical literature is about four-fold larger on titanium versus superalloys. Yet, titanium MIM in aerospace is not evident. Generally prealloyed spherical powder can be moulded, sintered, and hot isostatically pressed to full density with minimal contamination [40,41]. The most popular alloy, Ti-6Al-4V, delivers a yield strength near 800 MPa and tensile strength between 850 to 900 MPa with 12% elongation to fracture [42,43]. These properties are competitive with other titanium production routes.

Manufacturing quality systems

Customers for MIM want post-HIP machining combined with the MIM operation, so integrated partnerships with machining facilities are common. In the overall MIM industry, about 90% of the firms are ISO 9000/9001/9002 certified, about 25% are ISO 14000 certified, but only a few are certified as complying with quality systems required for aerospace component production. The customer's final engineering definition must specify that a MIM material is acceptable. Extensive testing is required for this to happen, such as oxidation, corrosion, mechanical properties, low cycle fatigue, high temperature creep, stress rupture, or other attributes that vary with the application.

A small fraction of the MIM firms are moving with vigour into the aerospace MIM field, as evident by efforts to qualify components, materials, and processes. For aerospace component production, AS 9100 or a similar quality system is required. Chemistry, radiography, and mechanical testing are expected in addition to dimensional checks. Note that if coining is employed it too is subject to validation including the tooling. Thus, every step in the production cycle needs to be controlled and validated. Accordingly, in a production operation probably at least two people must be devoted to quality issues.

Engineering source approval is performed using production equipment, which interferes with other production opportunities. All parameters need to be documented for sensitivity and robustness. Smaller MIM firms find the cost of quality to be particularly high since the quality system must be sustained (people, calibration, equipment) independent of production levels. Companies operating in MIM aerospace component production estimate the added cost of inspection probably runs from 15 to 40% of production cost, averaging 30%.

Fig. 5 A superalloy turbine wheel demonstration component for small jet engines

Business development

There are several aerospace components fabricated by MIM that were taken to at least a pilot production stage. Globally over 30 firms show activity supporting the fabrication of MIM aerospace components. Some of these are government affiliated facilities, often associated with military contractors. Table 3 lists some examples of aerospace MIM applications ranging from routine components to life-critical components in roughly increasing sophistication.

A few examples are shown in Figs. 4 to 7. Of these devices, about 24 specific components were identified in sufficient detail to sense the current applications in terms of material, mass, and production quantity. Although the majority of the identified components were from the USA, it was surprising that MIM aerospace efforts were also found in Brazil, Canada, China, Korea, Singapore, Switzerland, Turkey, and the United Kingdom. The distribution in MIM firm size tended to range from MIM sales from $4 million per year to $30 million per year. In no case was the MIM production of aerospace components a significant part of the business, implying that most of the current production is acquired without a focused marketing effort.

The median component design had a mass of 27 g, but the mean design was 137 g. Since there were a few large part designs, they skewed these statistics. The smaller components tended to be in higher production rates, and the smallest was 1.2 g. Curiously, tungsten inertial components showed up frequently in the industry survey, even though the research and development efforts have tended to emphasise

MIM Aerospace Applications
Seat belt latches for passenger seats
Luggage compartment latches
Stabiliser fins
Arming inertial components on rockets and munitions
Anti-missile, anti-aircraft, and fragmentation projectiles
Fibre optic connectors
Structural fasteners
Timing fuses, rotational sensors, trigger switches
Hermetic electronic packages
Heat sinks for electronic components, including phase array radar systems
Fuel-air adjustment vane levers
De-icing spray nozzles
Fuel pump components, butterfly valves, valve plates
Flow bodies, swirlers, inlet valves
Turbine blades, turbine wheels, compressor components.

Table 3 Examples of aerospace MIM applications ranging from routine to life-critical components in roughly increasing sophistication

Fig. 6 A superalloy valve plate fabricated using MIM

titanium, nickel superalloys, and some of the stronger stainless steels. Annual production quantities ranged from 1,000 per year to one example reaching over a million per year. One company reported a project of 280 million parts. For 18 of the components sufficient information was disclosed to give a surprising estimate of a median production quantity of 100,000 parts per year. Note that in some cases these statistics are misleading since variants in size or orientation (for example left and right) were reported together as a single component. In one case the design involved 12 variations at 20,000 per year each, giving 240,000 as the annual production quantity. A crude estimate of the sales price gives a typical value near $20 per part for the average design.

The general sense is that aerospace MIM components over about 150 g tend to be economically unfavourable [44,45]. This was illustrated by one firm using titanium alloys. They claimed titanium bar stock is priced at 10 to 25% of the powder price ($20 to $40 per kg for bar stock versus $125 to $250 per kg for powder). This ratio between wrought material and MIM powder means that MIM starts with a high cost disadvantage. Cost of powder is one of the first barriers to large aerospace components. In general, conversion to MIM requires at least a 30% cost savings to attract attention from the customer.

Aerospace MIM development cycles are long compared to other application areas. One company reported it took about three years to validate the material and process for a jet engine application. Another report said four years. Several firms claimed that after extensive work the project never went to production. These appear to be typical frustrations. As noted earlier, development work must be performed in production equipment. However, once in production the aerospace components tend to sustain for about 20 years and customers are reluctant to make technology or supplier changes. Success depends on picking the right partner, right material, right part, and right features (where tolerances are realistic for MIM).

Obviously, during a three year qualification period financial stability is required and funds might be needed for new equipment beyond the engineering, quality, and production staff. In short, the financial rewards in aerospace MIM are attractive, but significant financial resources are required to be a player in this field.

Future opportunities

Several individuals working in aerospace, especially jet engine materials, have now been exposed to MIM. This familiarity is a first step towards acceptance.

Research is needed to help mature aerospace materials and the use of MIM. For the smaller batch sizes it is important to quickly qualify the component and process, since a large number of opportunities appear to exist in the 10,000 parts per year range. Larger components continue to be desired from the users, but typically MIM has been drifting toward smaller components. Hence, focused work on shortening the process time for larger components and thicker cross-sections is appropriate. This might best happen in the context of a new MIM operation dedicated to aerospace, larger and thicker components, in lower production quantities, with high quality levels.

Based on anticipated production, a dedicated MIM aerospace facility would be expected to attract a substantial portion of the future business. In such situations MIM operations have been able to command excellent profit margins. Note the competition is not other MIM operations but other production technologies.

The time to reach production in MIM aerospace components is not a trivial barrier. In the past, several MIM projects were used to create competition with other production technologies. The projects were successful, but failed to move into production. A few MIM firms complained that they could ill afford to play this role and for that reason downplay aerospace.

Growth strategy

The cost of the special alloy powders used in many aerospace applications, namely titanium alloys and nickel-base superalloys, remains a barrier to growth. This is often termed the chicken-egg situation. Current powder use quantities are low, so powder prices are high. To increase the number of applications and overall powder use requires a lower powder price, but a lower price only comes with increased consumption to keep production capabilities running near capacity. Research on novel atomisation technologies geared to MIM grade powders at lower costs with the required quality would enable more aerospace component production.

Much interest is in applying MIM to thicker and larger parts. However, debinding and sintering times increase as the section thickness increases, leading to higher relative costs. Larger parts in batch vacuum sintering furnaces, have a low furnace loading due to the use of conformal setters that reduce furnace loading. New means for binder extraction are needed, possibly via binder modifications. Some ideas under discussion include supercritical, solvent, and plasma treatments. A key goal is to increase the rate of binder extraction especially for thicker components. A subsequent goal would be to marry debinding and sintering into a single cycle.

Sintering technology is another area needing attention. Limitations in current practice include poor heat transfer in vacuum furnaces at low temperatures. Faster cycles are needed to improve sintering. This might include options such as microwave-assisted heating at lower temperatures. Another option might be to increase the heating element area per unit of furnace volume. Most of the batch vacuum furnaces used for aerospace MIM components are cylindrical and have a low heating surface area per unit volume, most likely to reduce construction costs. On the other hand, faster heating would suggest just the opposite.

Additionally, tight tolerances add considerably to manufacturing costs, in part because of the added instrumentation and testing required in the manufacturing environment to ensure a stable process. Would it be best to use MIM only as a near-net shape technology and to machine most surfaces? For some components this is probably a cost effective option.

Summary

The use of MIM for aerospace applications, largely stainless steels, is roughly a $20 million market for 2010 split over several firms, but the use of CIM for aerospace casting cores is roughly a $100 million market. The injection moulding of aerospace components has been in incubation for many years. A variety of components are qualified and in production. In the past MIM was used to create competition for established technologies, but did not reach production. These competitions are guided by cost-reduction teams that are divorced from the final buying decisions. However, that situation is changing and a few technical developments can significantly accelerate the penetration of MIM into aerospace applications. Probably more than anything the largest need is for efficiency changes, since technical credibility is now established. With changes to the powder cost and debinding-sintering times, aerospace MIM components could become a significant growth field.

Fig. 7 A demonstration turbine component, about 200 mm in outside diameter

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Automation and a focus on value-added services helps Metal Injection Moulding thrive at OBE

Located in Ispringen, close to Stuttgart and the Northern area of Germany's Black Forest, OBE is a manufacturer of high volume, precision metal parts. More than 100 years of machining experience has enabled the company to become the market leader in hinges and related components for the eyewear industry. In 1996 the company diversified into MIM and has now combined its expertise in automation with its broad range of in-house finishing operations to set itself apart from many other MIM operations in Europe. Nick Williams reports on his recent visit.

OBE Ohnmacht & Baumgärtner GmbH & Co. KG (OBE) was founded in 1904 as a machine shop for jewellery and watch parts in Pforzheim, close to the company's current site in Ispringen, Germany. Over the following decades expertise was developed in the production of ever more precise components thanks to investments in the latest production technology and the company diversified to supply components to a wide range of markets. In 1947 the company first came into contact with spectacle manufacturers, an introduction that ultimately led to the successful development of a range of innovative hinges for spectacle frames. By the 1960's the company employed nearly 1000 people.

The company went through a major restructuring in the late 1980's and early to mid-1990's, during which time the company's two factories in Isprignen were consolidated onto one site. The new factory was opened in 1995.

OBE's machining division, called Optic, is dedicated to the production of spectacle frame parts from titanium and stainless steel profiles. The machining facility is located in the same building as the MIM operation. The components and hinge assemblies that the company makes for the optical industry range from simple but high precision hinge mechanisms to highly complex systems with internal springs. OBE sells the assembled hinge mechanisms to eyewear manufacturers worldwide and owns more than 150 international patents.

All the equipment used for machining is manufactured and maintained in-house by OBE, except for a bank of CNC milling machines. The machining area benefits from a high level of automation. After machining, parts are processed in an automated cleaning area before receiving additional processing such as precision welding, nickel and gold plating, galvanising, tumbling, laser engraving and polishing.

In the early 1990's two important decisions were taken. The first was to diversify into MIM production and the second was to develop expertise in the production of machined titanium parts.

Success came in 1996, when the

Fig. 1 OBE's factory and headquarters in Ispringen, Germany

Fig. 2 Automation machining of stainless steel and titanium strip to produce spectacle frame parts for OBE's Optic division

Fig. 3 The machine building and maintenance area at OBE

Fig. 4 OBE currently operates eight Arburg Allrounder injection moulding machines, all with automated part handling systems for the efficient processing of high volume parts

Fig. 5 The company's debinding and sintering area operates a range of furnaces from Elnik Systems and Gero GERO Hochtemperaturöfen GmbH & Co.KG

company introduced the world's first titanium spring hinge for spectacles after intensive development work. In the same year, the company opened subsidiaries in the two most important markets for spectacle manufactures, Italy and Hong Kong. Further regional sales offices followed, and in 2002 the company founded a joint venture, called Globe Precision, in Shenzhen, China together with a Taiwanese company to machine optical components.

The company has today evolved into an international business with three distinct divisions; Optic, producing machined components for spectacles; MIMplus, manufacturing MIM components for a range of industries, and Trevista, a division that markets the company's own automated surface inspection system.

OBE's Ispringen facility now covers an area of 15,000m², including both the Optic and the MIM operations. The company today employs 220 people and has an 80% export rate. It has representative offices covering many locations worldwide and remains a family owned enterprise. Total turnover for 2009 was reported as €30 million.

'OBE's Ispringen facility now covers an area of 15,000m², including both the Optic and the MIM operations'

The development of MIM at OBE

OBE started commercial MIM production in 1996. This was in part a strategic decision to protect the company against what was regarded as a growing dependency on the spectacle components division. "The eyewear market is strongly fashion driven, and as such liable to rapid changes in design and component use. This can make life difficult for a supplier, so our board took the decision to diversify into a technology that offered opportunities in a much broader range of markets", stated Josef Heckert, Technical Director of Production at OBE.

When discussing the early days of the MIM facility, the team at OBE said that they had to overcome many challenges in order to reach commercially acceptable standards.

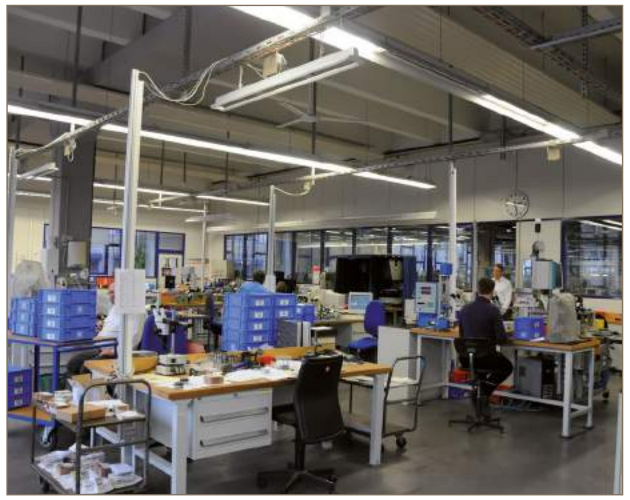

Fig. 6 The inspection and testing laboratory at OBE

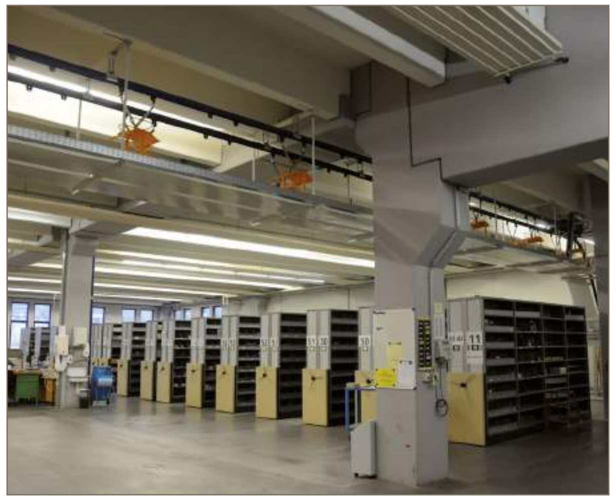

Fig. 7 A conveyor system that connects all areas of the factory can be seen passing along the ceiling of this dispatch area

"If anyone thinks that starting a MIM operation is easy, they are mistaken. It's far more than the simple matter of buying the equipment and finding a source of feedstock. Thankfully we had the resources of a well established and successful business behind us, able to support the transition to commercial production and achieve a constant and stable process", continued Heckert.

The company today operates eight Arburg Allrounder injection moulding machines with automatic handling systems (Fig. 4). The debinding and sintering area uses four debinding and five sintering furnaces (Fig. 5). These thermal processing facilities at OBE operate on a 24 hour basis, whilst the injection moulding operation operates two shifts a day.

The MIM operation has significantly expanded since its opening in 1996 and OBE states that there is still space reserved for further expansion in the coming years.

Materials

OBE has used BASF's Catamold feedstock since the start of production. The core materials processed are 316L and 17-4-PH stainless steels and titanium. "We have been processing MIM titanium parts since 1996 and MIM-Ti remains an important area for growth. The biggest challenge was to achieve the ductility needed, through careful control of oxygen, carbon and nitrogen content", stated Heckert. The company also has experience of other titanium feedstock systems for the development of MIM medical components, and is also able to process copper and copper base alloys.

Tooling

OBE initially started out using external tooling suppliers, however in 2007 an in-house tooling department was established, building on the experience gained by the company during more than 10 years of MIM development and production. The company now produces around 50% of all new tooling, as well as maintaining existing tooling. Approximately 50 new tools are added each year.

A facility with automation and traceability at its core

OBE sees itself as distinct from many MIM producers in that its factory as a whole benefits from an extremely high level of automation, making it well suited to high volume production. To leverage this advantage, OBE targets potential customers whose requirements are in the multi-million part volume.

Each injection moulding machine is fitted with advanced custom built robotic part handling systems that have been adapted by OBE to the high volume production of very small parts.

All areas of the OBE plant are connected by an advanced computer controlled conveyor system. This system automates the movement of standardised boxes of components, either in a semi-finished or finished state, between 'stations' located at various manufacturing points, as well as the quality department and the dispatch area. A complete circuit of the entire factory takes 45 minutes (Fig. 7).

The boxes can be automatically identified by machine readable barcodes as they move around the system, as well as containing a printed report of the contents, including full order details and the manufacturing history to-date.

Fig. 8 Josef Heckert, OBE's Technical Director of Production, stands in front of a vast automated component storage area that links directly to the factory's conveyor system

Fig. 9 One section of the electroplating facilities at OBE. The area in view is dedicated to small volume processing, whilst a second significantly larger electroplating facility is out of view to the right

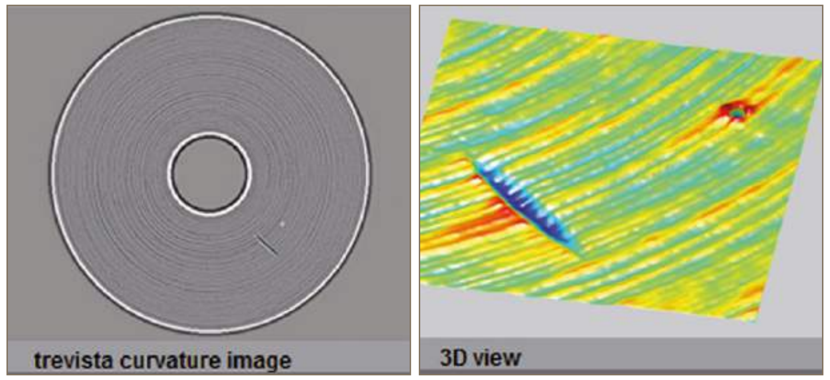

Fig. 10 Images generated by theTrevista automated surface inspection system developed by OBE

In the case of an operator identifying defect parts during production, or parts that appear out of tolerance, sample parts and a report detailing the complete production history are immediately placed in a box and conveyed to the quality laboratory for analysis.

The conveyor system also links to a large automated store that can house up to 20,000 boxes of part-finished or finished products (Fig. 8). "When needed, a specific box of parts can be immediately recalled and sent to the required location for processing or shipping", explained Marcus Mayer, Project Management Director at OBE.

'For many, the convenience of receiving highly processed parts or completed sub-assemblies is a major benefit'

All stages of the MIM production cycle are carefully managed and recorded, from the receiving and testing of incoming materials to the various processing stages and final inspection. "With regards to feedstock, we test each batch on arrival as part of our release process. This involves injection moulding and sintering a standard test specimen and then analysing the test piece for density, hardness, carbon content etc.", continued Mayer.

Each operation or action in the factory is recorded, with machine operators using a swipecard and PIN number to log functions such as setting or pausing a machine. This system tracks and records every step so that any quality problems can be traced quickly and efficiently. "With our full checking plan, each worker is responsible for the parts that he or she produces, with identity verification via the PIN and swipe card."

MIM parts are quality checked throughout the production cycle, with frequent weight, dimensional and visual checks.

The concept of a full service supplier

In addition to the efficiencies brought by automation, OBE's MIM operation benefits from the wide range of machining and finishing operations that are available in-house.

"Everything we need in terms of post-sintering and finishing operations is available here. We have large scale advanced automated machining and welding areas that were specifically designed around the processing of very small precision components. In addition, we are able to offer numerous surface treatment facilities including high capacity deburring, polishing, galvanising and electroplating systems (Fig. 9). It is for this reason that we branded our MIM operation 'MIMplus', to highlight our ability to efficiently process MIM parts after sintering, be it for subsequent machining, surface treatment or sub-assembly".

"For most of the MIM parts that we produce our customers require some additional post-sintering steps, even if it is just a tumbling or calibration process. Our customers can benefit in terms of efficiency from the additional services that we can offer. For many, the convenience of receiving highly processed parts or completed sub-assemblies is a major benefit compared to simply receiving the 'raw' MIM parts. Thanks to our machining facilities and experienced operators, we are also able to supply our MIM customers with machined sample parts within 1-2 weeks of receiving drawings", continued Mayer.

In addition to machined eyewear parts and MIM production, OBE's third and youngest division develops and markets the Trevista automated optical inspection system. Developed in-house by OBE, the Trevista system is able to automatically and continuously perform a visual surface inspection of metal parts, rejecting any that have scratches or similar visual defects. Interest is growing from a range of industries, and the Trevista system now accounts for 5% of OBE's turnover.

Trevista was developed for the 100% inspection of a range of surfaces, from shiny components to diffusely scattering surfaces. Topographical defects which are relevant for the function of a part can be reliably distinguished from irrelevant stains caused by lubricants, for example. A structured diffuse illumination of the part and a special calculation algorithm guarantee high-quality images for the subsequent automatic evaluation (Fig. 10).

MIM markets

OBE currently supplies MIM parts to a broad range of industries including the eyewear, medical, automotive, aerospace and general engineering sectors. "In recent years MIM turnover has doubled each year and we have strong expectations for the future. 2009 was another good year for our MIM business and there are now around 100 people involved in our MIM operation, including MIM related finishing, sales and administrative operations", stated Frank Schroeder, Marketing Director at OBE.

MIM now accounts for 25% of OBE's turnover and strong growth is expected over the coming years. MIM eyewear components for the company's Optic division account for approximately 10-15% of the company's MIM production (Figs. 13-16).

"We believe that our real advantage is with high volume MIM parts in the smaller size range because we have a lot of experience in the production, handing and finishing of extremely small machined parts for the optical industry. We are also well accustomed to dealing with extremely small parts with very tight tolerances. MIM technology is also at its most competitive with smaller parts thanks to the relatively low quantities of powders need. This widens the advantage over competing processes", continued Schroeder.

Fig. 11 A MIM titanium dental fixation plate produced by OBE

Fig. 13 An innovative spectacle frame hinge system produced by MIM for TAG Heuer

Fig. 12 A 316L MIM clock casing for use in a high-end automotive dashboard. A unique feature of this component is the brushed surface effect which is integrated into the tool

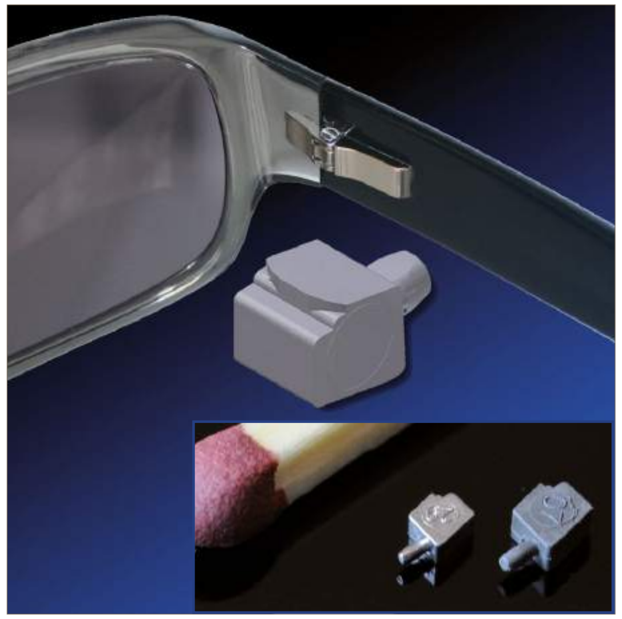

Fig. 14 OBE won a 2010 EPMA Award for this extremely small MIM 316L part for a spectacle frame locking device. The part weighs 0.028g and more than 4 million parts are produced each year. The part is manufactured in a 32 cavity mould

Fig. 15 A MIM spectacle frame hinge that is both decorative and functional

Fig. 16 A Laguiole spectacle frame featuring MIM hinge parts

In October 2010 OBE received an award from the European Powder Metallurgy Association (EPMA) for a MIM locking component for spring hinges in spectacles. This small 316L part weighs just 0.028g, however despite its small size, production volumes are high, with more than four million parts being produced each year.

Reflecting on the biggest challenge of all, making designers and engineers aware of MIM and its possibilities, OBE express the importance of promoting the technology. "We are present at major trade fairs, and support trade and networking groups such as the EPMA and the MIM Expert Group (MIM Expertenkreis), but still see a lot of possibilities for the dissemination of information about MIM. Of most concern is that so few engineering and design students graduate from University with any knowledge of MIM. Without this being achieved, it will be an even harder task to spread the message about MIM's potential to end-user markets" stated Schroeder.

'few engineering and design students graduate from University with any knowledge of MIM. Without this being achieved, it will be an even harder task to spread the message about MIM'

OBE is exhibiting at a number of European trade fairs in 2011 including Medtec in Stuttgart, the Hannover Fair, Affidabilità & Tecnologie, a technology and innovation event in Turin, Italy, EPHJ, a watchmaking and jewellery exhibition in Lausanne, Switzerland, and GIFA, located in Dusseldorf.

Environmental awareness

OBE places a great emphasis on its environmental responsibilities and was one of the first companies to have passed an EMAS 'Ecological Audit' in 1996, thanks to the installation of a state-of-the-art water reprocessing system. Today the company is certified according to the DIN EN ISO 14001 Environment management system.

Additionally, the company believes that the environmental benefits of MIM processing, when compared to conventional manufacturing technologies, need to be highlighted. "More needs to be made of the environment advantages that MIM offers, in terms of efficiency of material usage. The ability to produce net shape parts, and to recycle any green defect parts and runners etc, is a huge plus for the technology," added Schroeder.

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Carpenter Powder Products: Stepping up the production of gas atomised powder for the MIM industry

Carpenter Powder Products (CPP), based in Rhode Island, USA, is one of the world's largest and most diversified producers of pre-alloyed gas atomised metal powders. In the following article, the company outlines its plans to increase its UltraFine® MIM powder producing capacity by around 75% by the end of 2011, positioning itself to meet the just-in-time needs of the MIM industry. The company also shares its view of the MIM industry's regional development and major markets.

Carpenter Powder Products (CPP), a business unit of Carpenter Technology Corp., has for many decades been an important force in the metal powders industry. The business unit produces fine, medium and coarse spherical metal powders in a variety of ferrous and non-ferrous alloys using different melting processes to meet customers' requirements. This includes fine powders for metal injection moulding.

The story of fine powder production at Carpenter

CPP's roots date back to the 1960s, well before the development of MIM technology. The earliest incarnation of what is now CPP's metal powder production story can be traced to the R&D laboratory of Universal-Cyclops's specialty steel division in Western Pennsylvania, USA. Universal-Cyclops's powder operation was acquired by Dynamet Inc. in 1991, which itself was subsequently brought into the fold of Carpenter Technology Corp. in 1997.

Carpenter's first toehold into the MIM powder market came with its 1999 acquisition of the Anval Group, based in Torshalla, Sweden. However CPP's real push into fine powder technology came with its acquisition of UltraFine Powder Technology Inc. (UFP) in 2008. UFP had been formed in 1986 to commercialise production of gas atomised metal powder. The company's team of materials and process experts had made significant advances in the production of fine spherical powders suitable for MIM based on technology first developed at the Massachusetts Institute of Technology, USA.

Fig. 1 This inert gas atomisation unit is used by CPP in the production of spherical metal powders

Fig. 2 A powder capture station at CPP Rhode Island

'CPP has recently invested more than $7 million in equipment and added to its workforce to improve efficiency and increase production'

"The merger not only allowed each entity to do what they do best but gave UFP, now CPP Rhode Island, the added benefit of direct access to the expertise of Carpenter's metallurgical team. It is good to know that when anything needs to be handled from a metallurgical point of view, whether for new products and materials or existing ones, that Carpenter Technology is there to support us and our customers," said Dr. Philip E. Jones, Director of Business Development for CPP and former President and CEO of UFP.

Carpenter states that UFP's powder atomisation process is the only one that was focused on fine powder production from the start, making it ideally suited to MIM, and adds that the process has been constantly improved over the years to give maximum yield and consistency. "Our reactors, some of the largest in the world for this application, allow CPP to supply powders with exceptional consistency in size and chemistry while maintaining competitive cost," stated Jones.

Carpenter Technology Corp's fiscal year 2010 sales (year ended June 30 2010) were $1.2 billion; net income $2.1 million, covering all divisions including CPP. For the first half of the 2011 fiscal year, sales were $727.3 million and net income, $16.9 million. Carpenter Technology is listed on the New York Stock Exchange (NYSE:CRS).

Powder production today

CPP today produces metal powders in three production facilities on two continents. Carpenter Technology's central R&D laboratory in Reading, Pennsylvania, USA, develops coarse and MIM grade powders prior to scale up at the company's main powder facilities. The primary location for the production of MIM powder is CPP Rhode Island, where the majority of the powder has an average particle size of 10-12 microns, compared to 70, 80 or even 100 microns for the higher volume powders produced elsewhere.

Investing to meet customer demands

Whilst suppliers of atomised fine powder for MIM are struggling to meet demand, CPP states that it is 'pulling out all the stops' to ensure that the facility can continue to meet customers' needs in a timely fashion. As part of this move, CPP plans to significantly increase its MIM powder producing capacity by the end of 2011, thereby further improving lead times. CPP has recently invested more than $7 million in equipment and added to its workforce to improve efficiency and increase production. Additional melting capacity is anticipated in the near future that would double current capacity levels.

"Customer expectation today is that their materials will arrive just in time. You need to be able to produce it quickly, test it quickly, turn it around and get it right into the marketplace. Our present investment primarily has been directed toward improved efficiency and yield, additional support equipment and additional production time utilisation. We have committed to add additional melting capacity as needed," stated Jones.

"We are also seeing the differences between water and gas atomised powder narrowing with the properties merging and becoming more interchangeable and gas atomised powder getting cheaper than it was and water atomised powder getting more expensive," Jones added. "It is getting to the point that some people are looking at water as an alternative to gas and gas as an alternative to water."

The company believes that gas atomised powders offer some convincing benefits compared to water atomised powders. "Gas atomised powders are cleaner, more spherical, and more consistent lot to lot. This leads to a more predictable and controllable customer process yielding parts that are closer to net shape and improved properties. All this reduces the need for additional secondary processing steps and gives the customer a higher yield and thus lower cost," commented Jones.

Considered as a whole, CPP has a melt capacity of more than 14,000 metric tons per year, one of the largest concentrations of gas atomised powder production in the world. The company states that it is increasing capacity at its Rhode Island facility by more than 300% through increased efficiency, manpower and equipment investments. The initiative launched in late 2010 and is expected to conclude by summer 2012.

CPP produces any particle size range to match the needs of the customer. Initially this meant d90 -22 µm, however as the market grew and matured other sizes were included from very small d90 -3 µm to -44 µm and above. Today, indicates CPP, the MIM market is focused on the 15 to 32 µm range of powders with d90-22 µm still the most popular.

Materials processed

A wide variety of materials are atomised by CPP for the MIM market, including iron-base alloys such as stainless steels, duplex stainless and tool steels, nickel-base alloys including superalloys, cobalt-base alloys, copper-base alloys and chrome alloys.

"We work with the customer to aid them with our extensive metallurgical expertise and help them select whatever alloy best fits their needs. With our experience in melting metals both at CPP's Rhode Island plant, and in our other divisions, we are uniquely positioned in the market to supply almost any metal needed," stated Jones.

The company also offers commercially pure (CP) and alloy (Ti 6-4) spherical titanium powder for a variety of applications including MIM. R&D efforts focus on producing powder from special titanium grades and on reducing the overall cost of titanium powder.

A focus on R&D

The MIM industry has historically put significant resources into research and development efforts. "In particular there are significant R&D programmes under way in Europe, and Carpenter is an active participant in similar efforts in the United States. Our R&D centre in Reading, PA, acts as the driving force for much of that development work, coordinating the R&D activities of not just Carpenter Powder Products but the entire company," commented Jones.

This collaborative environment allows CPP to use the advanced testing and analysis capabilities and metallurgical technical support already in place at other Carpenter facilities. Customer development work, in collaboration with Carpenter, has included addressing the challenge of developing powders that work effectively in the production of the complex, relatively small parts that operate in certain high temperature, high cycle, high wear applications, such as automotive engine valves, which must open and close millions of times over the life of the engine.

The company is also collaborating with universities and R&D facilities

Fig. 3 Powder V blenders at CPP Rhode Island

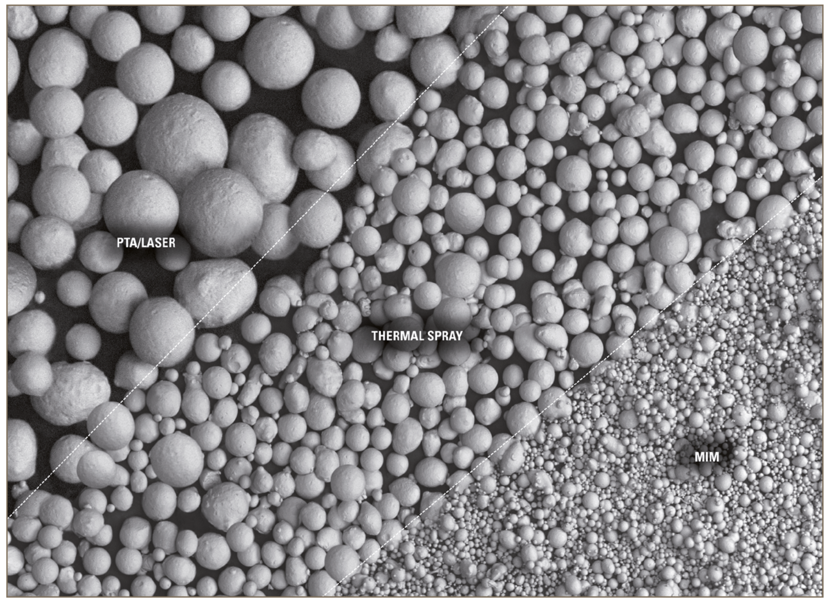

Fig. 4 CPP produces a variety of powder sizes for various markets, including PTA/laser, thermal spray and MIM

Fig. 5 Various small, complex MIM parts can be cost effectively produced in large volumes using metal injection moulding

on various technology, product and materials innovations. Goals include studying the size and chemistry of powders and their effects on the properties of the final product. Whether automotive, medical, aerospace or consumer applications, CPP states that it is looking to push the limits of traditional cast and wrought alloy properties.

Carpenter's view of the MIM industry

While 2010 industry statistics have not been released yet, CPP states that MIM sales have certainly risen from 2009 levels, after falling from the previous year. "The MIM industry, much like other industries, has been generally conservative with cash flow and inventory strategies. In the fourth quarter of 2010, it's been noted that demand for MIM powders in the industry in general was beginning to grow," Jones said.

Asia

MIM demand, however, has and will likely continue to vary by region. As a whole, demand has been following economic trends, growing with the economy. One exception is Asia, where the MIM industry is growing even faster than the economy, pushed primarily by the 3C (Computing, Communication and Consumer) markets.

"Partly due to lower production costs, MIM has become well established in China, Taiwan, Japan, Singapore, India, Malaysia and Thailand with these MIM houses ranging from high volume producers to very small start-ups. Recognising the potential of the Asian region, for both MIM and non-MIM products, Carpenter Technology has increased the company's presence there to better market its products, including MIM and other metal powders, in that region. MIM demand is expected to grow more than 15% in Asia over the next several years according to industry sources," stated Jones.

'MIM still needs to gain further acceptance, embrace standardisation, and reduce the cost of both the powder and processing'

North America

Despite the tremendous growth potential in Asia, Carpenter believes that the United States remains the world's leading user of MIM parts, as well as having a number of major MIM producers.

"The U.S. metal injection moulding market is highly diverse, and demand comes not only from the automotive market, which is dominant in Europe, but also from the medical, dental and aerospace industries. These industrial sectors, on the whole, remained profitable even during the economic downturn. The medical industry is a major market for MIM. Any operation that is performed through endoscopy is probably made possible by MIM technology," Jones commented, noting that the complex end piece on the endoscopic surgical device, or gun, is generally made by MIM.

"Another growing MIM end use is dental braces, which require very small, intricate parts. You can make these advanced metal dental adjusters, or braces, very cost effectively with MIM." According to industry sources, the United States is expected to see about a 5% increase per year in MIM usage for the next few years.

Europe

As previously stated, CPP recognises that the European MIM industry is dominated by the supply of automotive components. There are however a number of other significant segments including the aerospace sector and a thriving market for 'luxury' parts for watch components and jewellery. CPP also notes that a significant number of research initiatives in Europe are aimed at promoting the use of microMIM, as well as the use of certain non-conventional materials.

Developing the market for MIM

"Many manufacturers within traditional MIM markets, such as the automotive, medical, aerospace and 3C industries, will continue to explore new applications for the MIM process. They are comfortable with the MIM technology and see the advantages of using it versus other manufacturing processes," Jones said.

But to really grow, CPP believes that MIM still needs to gain further acceptance, embrace standardisation, and reduce the cost of both the powder and processing. This includes reducing secondary operations by truly making a 'net shape' part, as well as reducing the cost of making MIM tooling. "The use of MIM is an educational process," stated Jones. "When people adopt MIM for their parts production, they realise it is a viable technology that, once in place, offers distinct advantages with the right applications."

CPP recognises that there are some practical limitations to MIM, including those involving the size of parts that could be produced by this technology, both from a manufacturing capability and cost perspective. Larger parts have a larger powder cost on a percentage basis, so casting may often be more competitive for these applications, especially since larger parts also tend to be less complex.

CPP also sees high performance superalloys as a significant growth area and is presently supplying these materials in standard and special grades to a variety of customers.

"There are definite advantages to using MIM, as is well known, particularly when the parts maker can combine several individually manufactured parts into one intricate MIM part. A parts maker can make net shape or near net shape, high-volume, intricate, small parts in one step with MIM. MIM is typically an attractive manufacturing process when annual demand is in the hundreds of thousands of parts per year. A MIM mould can incorporate several parts from different manufacturing technologies into one part," John D. Hunter, Sales and Marketing Manager for CPP stated.

There are also some producers around the world that offer more than one technology, including MIM. "Offering MIM alongside more traditional manufacturing methods is an advantage to CPP because it raises MIM up there with the big boys," Jones concluded, "MIM is being offered as a viable production alternative, the beginning of a paradigm shift."

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Investigations into water soluble binder systems for Powder Injection Moulding

Water soluble binder systems for metal and ceramic injection moulding offer several distinct benefits. Compared with thermal debinding, water solvent debinding is regarded as more environmentally friendly, with a faster debinding rate, simpler processing, and a wide range of feedstocks are available. Delphine Auzène and colleagues from CRITT-MDTS, Charleville-Mézières, France, present a comparative study of some commercially available feedstock systems that can be processed using water solvent debinding.

CRITT-MDTS, a centre for innovation and technology transfer specialising in materials, coatings and surface treatment, was founded in 1984 and is an important research and inspection laboratory in France. A team of technicians and engineers offer technical assistance for the characterisation and analysis of materials, as well as the development of new products. In particular, the manufacturing of complex shaped parts by metal and ceramic injection moulding has been studied since 2006.

In 2007 an injection moulding machine, debinding equipment and a sintering furnace was purchased and installed. The mission of CRITT-MDTS is to promote powder injection moulding primarily in the Champagne-Ardennes region of France, but also in Wallonia, Belgium, via the INTERREG IV PRISTIMAT program with SIRRIS and in wider France via collaboration with French PIM networks.

Introduction

Metal injection moulding has evolved to become a versatile mass production method for a wide range of complex-shaped metal components [1]. The powder is first incorporated into a polymer by high shear mixing at a volume loading typically between 50 and 70 vol %. The melted suspension is injected into a cavity where the polymer solidifies. The task that remains is to remove the organic components before sintering the metal powder assembly. Binder removal is a critical processing step and plays a central role in PIM part production. The main reason for this is that the risk of introducing defects into the components is particularly high during this step [1]. In the past binder removal involved reheating the moulded part to cause thermal, degradative or evaporative loss of the organic phase [2]. This processing step has to be very long in order to avoid sample distortion and defects. Therefore the development of feedstocks for MIM is the area of the technology with the greatest improvement potential. In recent decades considerable progress has been made in feedstock development and binder removal.

The feedstock or binder systems are classified by their debinding techniques. The conventional thermal debinding and vacuum debinding processes involve one step only: controlled heating in an inert or reducing gas atmosphere. The more advanced debinding techniques require a two-stage process. During the first stage a major binder fraction is removed in order to create an open pore structure within the metal powder assembly. Solvent extraction, water debinding, and chemical degradation techniques

Fig. 1 Stéphane Roberjot and Delphine Auzène in the CRITT laboratory

are most common. A certain binder fraction remains rigid during the first step in order to provide mechanical strength during the chemical and physical removal of the main binder content. In a second processing step the remaining binder fraction is removed thermally. The advantage of two-step binder systems is that the thermal binder fraction is greatly reduced, thus minimising the risk of defects such as cracking, blistering or viscose- and gravity-driven part deformation [1].

This study compares different water soluble feedstock systems:

- PolyMIM® 316L commercially available from POLYMIM (Germany)
- AquaMIM® 316L commercially available from RYER (USA)
- Inmafeed® K1010 Al2O3 commercially available from INMATEC (Germany)

Experimental details

Step 1: Injection moulding
Rectangular parts of 3 mm thickness made from the three different feedstock systems were injected using an ARBURG 320C 600-100 moulding machine. The composition of the screw, plasticising cylinder and mould were specifically chosen to resist abrasion and corrosion. Analysis of the feedstock binder was undertaken by SEM, TGA-TDA and infrared analysis

Step 2: Water debinding
Samples were plunged into demineralised water; different temperatures were tested: 30°C, 50°C and 70°C. Each hour, a sample was removed from the bath. After water debinding treatment, the parts were dried for 2 hours at 100°C. For each sample the mass loss was measured. A corrosion inhibitor (2% inhibitor 4000 commercialised by Zschimmer and Schwarz) specified by POLYMIM was added even if the metal tested was a stainless steel.

Step 3: Thermal debinding & sintering
The second debinding and sintering cycle consisted of the following steps for the 316L feedstocks, under pure hydrogen at 400mBar:

- Heating at 5K/min to 600°C, holding at 180 minutes
- Heating at 5K/min to 1350°C, holding for 120 minutes
- Furnace cooling.

For the alumina feedstock, the second debinding and sintering cycle consisted of the following steps, under air, at atmospheric pressure:

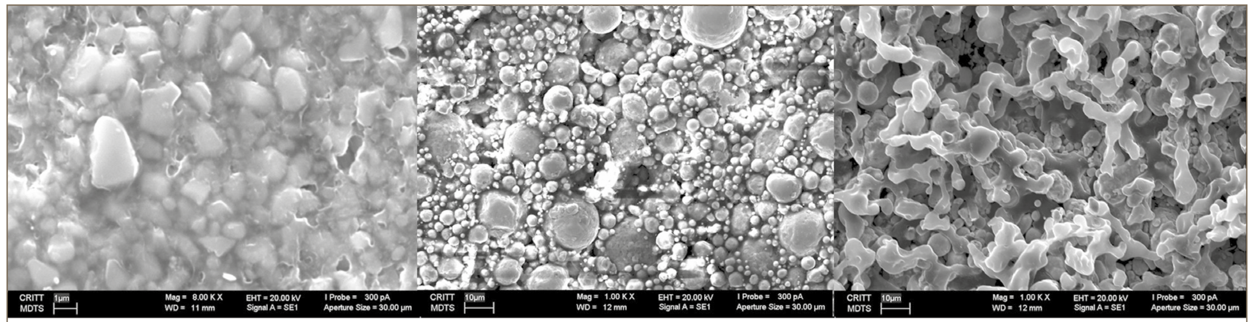
- Heating at 5K/min to 600°C, holding for 180 minutes
- Heating at 5K/min to 1680°C, holding for 120 minutes
- Furnace cooling.

Results and discussions

The characterisation of feedstock systems shows differences in the polymeric binders owing to the feedstock producers (Fig. 2):

- PolyMIM® 316L has a similar infrared spectrum to polypropylene
- AquaMIM® 316L has a similar infrared spectrum to plastic-coated polyethylene associated with saccharide polyhydric alcohol, cellulosic, glycolic, etc.
- Inmafeed® K1010 Al2O3 has a similar infrared spectrum to polyether type polyethylene or polypropylene glycol.

The binder infrared spectra highlight the complexity of polymeric mixing. Hence it is difficult to analyse the whole composition of each binder, especially the presence of additives such as surfactants, wetting agents or dispersants [12].



(a) (b) (c)

Fig. 2 SEM images of alumina x 8000 (a), 316L polyMIM x 1000 (b), 316L aquaMIM x 1000 (c)

	TGA-TDA: weight ratio powder-binder %	Particle average size (µm)	Average oversize factor	Density (g/cm³)	Tensile strength Rm for 316L /	Yield strength Rp_{02} (MPa)	Hardness
PolyMIM® 316L D 120 E	7.68	18	1.1669	≥ 7.90	≥ 450	≥ 140	≥ 150
AquaMIM® 316L 9023AA	7.77	23	1.1669	~7.87	≥ 430	≥ 138	≥ 140
Inmafeed® Al_2O_3 K1010	15.05	0.3-3	1.1933	≥ 3.99	≥ 3500	≥ 140	≥ 2000

Table 1 Characteristics of each feedstock

Investigations into water debinding for PIM

Indeed, there are many different polymers used to prepare feedstocks for the PIM industry. For water solvent debinding, polyethylene glycol (PEG), polyvinylpyrrolidone (PVP) isobutylene and maleic anhydride (ISOBAM), polyvinyl alcohol (PVA), polyacrylic acid (PAA) and poly(2-ethyl-2-oxazoline) PEtOx are hydrosoluble polymer components of feedstock binders [1-10].

The ceramic powder is finer than 316L powders, and the weight ratio of K1010 from Inmatec is twice that of the metallic feedstocks. The average oversize factor for the ceramic is consequently higher than for the two 316L feedstocks. Finally, the mechanical properties of the 316L polyMIM® is better than the 316L aquaMIM® (see Table 1).

The influence of temperature on water solvent debinding kinetics was studied. Figs. 3-5 present the variation of weight loss owing to temperature, and the corresponding SEM images.

Binder removal mechanism

During water binder removal the water dipole molecule dissolves the water soluble binder fraction. Starting from the moulding surface, the water penetrates gradually into the moulded sample. As the water diffuses into the powder assembly, it dissolves and extracts the polymer molecules. The dissolved polymer is transported from the inter-particle spaces into the water bath. This mechanism is driven by capillary forces and concentration gradients of dissolved binder in the moulded sample and the water bath. Binder removal takes place until an equilibrium of binder concentration is reached. This mechanism is illustrated in Fig. 6.

The objective of water debinding is to create an open porosity throughout the moulded sample. A secondary water insoluble binder fraction remains in the sample. The main function of this so-called backbone binder fraction is to strengthen the sample during and after water binder removal. The secondary binder consists of a blend of waxes and polyolefins. The backbone binder fraction prevents the powder assembly from swelling and cracking during water debinding. The SEM pictures (Figs. 3-5) illustrate this phenomenon, and Table 2 shows the results of water

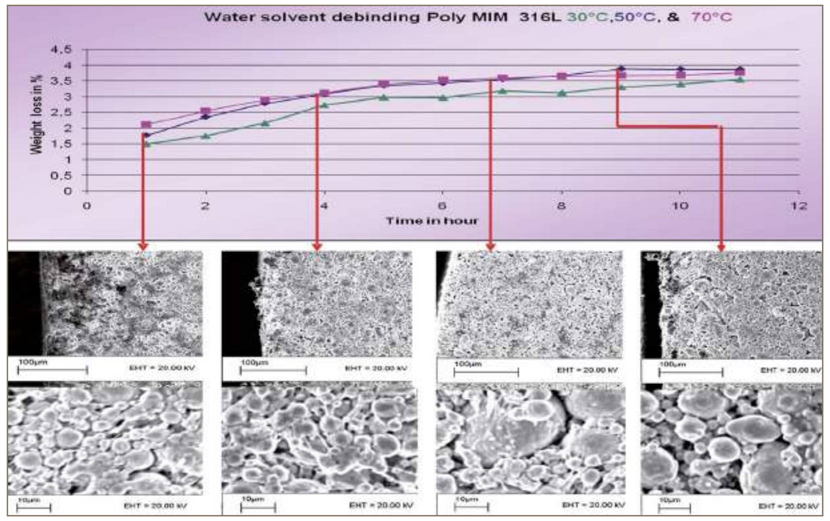


Fig. 3 PolyMIM® 316L SEM binder weight loss according to debinding water curves and corresponding SEM images at 50°C

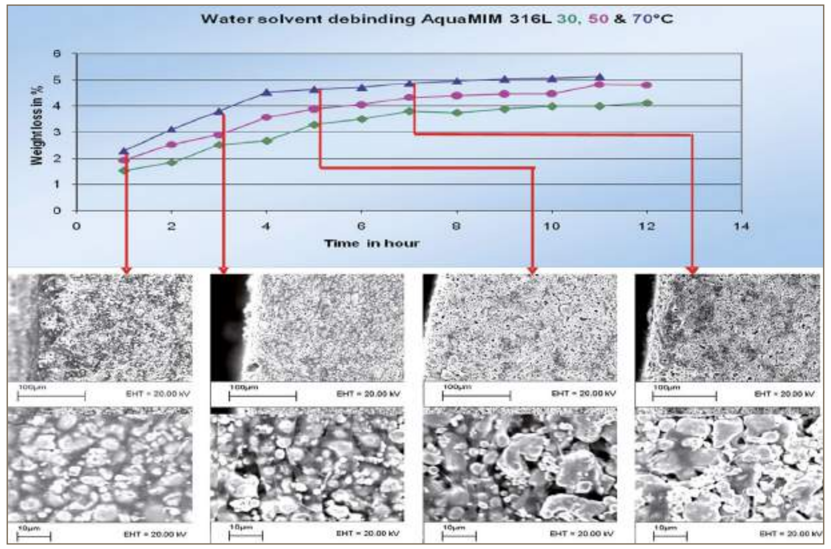


Fig. 4 AquaMIM® 316L SEM binder weight loss according to debinding water curves and corresponding SEM images at 70°C

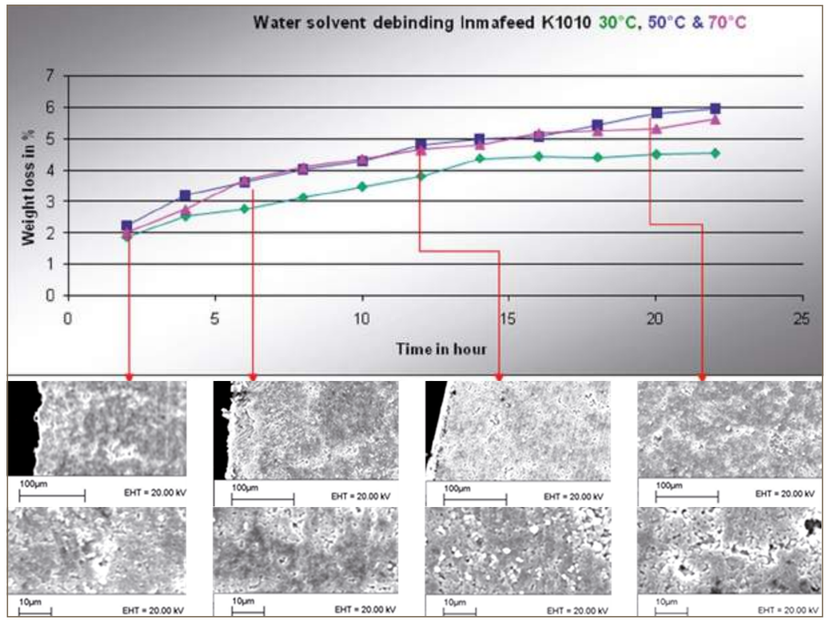


Fig. 5 Inmafeed® Al_2O_3 SEM binder weight loss according to debinding water curves and corresponding SEM images at 50°C

	Span for a 3 mm thickness	Temp.	Weight loss (%) measured	Weight loss (%) of the data sheet
PolyMIM® 316L	9 h	50°C	3.8	3.8
AquaMIM® 316L	8 h	70°C	5	5.5
Inmafeed® Al_2O_3	8 h	50°C	4	6.3
	22 h	70°C	6	

Table 2 Comparative results for water debinding

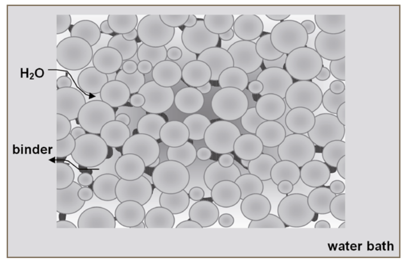


Fig. 6 Schematic illustration of water binder removal (scheme from POLYMIM)

Fig. 7 MIM Parts made of polyMIM Cu999 developed in CRITT-MDTS

solvent debinding.

The binder system which reaches the weight loss given in the data sheet is PolyMIM®, at a lower temperature than the two others. The AquaMIM® and Inmafeed® systems are less competitive because of the higher temperatures (70°C) required to achieve maximum percentage weight loss. If the customer would like to reach the weight loss specified in the data sheet, especially for Inmafeed®, the length of time for water debinding is 22 hours. The water debinding curves show that after 8 hours in water, alumina samples have lost 4% of their weight. This result is comparable to the two other products, however, the weight loss is more significant for the AquaMIM® system (5%) when debinding occurs at an elevated temperature (70°C) after 8 hours in water.

Conclusion

There are a lot of patents and producers of feedstock with water debinding solutions available on the market. Besides the technical and economical aspects, the ecological impact of industrial production is of increasing importance. Dealing with large quantities of solvents or concentrated acids presents an additional source of danger. The technical equipment is costly and requires additional safety measures. Transport, storage and handling of hazardous substances is also becoming an issue.

There is no doubt that using water as a solvent makes life much easier. There is only the question of how to dispose of the binder contaminated water. The binder used is, however, biologically degradable and non-toxic to microorganisms. In fact, this type of polymer is also used in many pharmaceutical and cosmetic products, for example PP and PEG.

The aim of this work is to help the decision making process of companies who would like to invest in PIM technology with a water solvent debinding solution. This study has compared three different water solvent debinding systems with different suppliers, binder systems and load (ceramic or metallic).

The water debinding process is easier, and the equipment is cheaper, than other technologies used for powder injection moulding. Demineralised water is primarily used and a corrosion inhibitor can be incorporated to avoid corrosion if necessary. Nevertheless the product of the chemical reaction between water and binder leads to an increasing chemical oxygen demand of the waste water [11]. Also, using corrosion inhibitors to prevent corrosion of the metal powder surface during debinding leads to an elevated pH-value [11]. Therefore a direct draining into rivers or lakes is not recommended prior to treatment of waste water in accordance with regulations.

CRITT-MDTS has been studying many more materials that can be used with water solvent debinding systems, for example titanium alloys, superalloys, copper alloys, etc. (Fig.7).

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A new approach to monitoring process temperatures during sintering

A new system developed by the Orton Ceramic Foundation, based in Westerville, Ohio, USA, offers PIM parts producers the chance to routinely record the performance of their sintering furnaces, including peak temperature and time at temperature. Such a system has been designed to supplement the use of existing thermocouples and routine temperature surveys, but with the added benefit of providing batch-to-batch data records that can be logged for improved process monitoring.

Sintering is a critical step in the production process of powder injection moulded components. Sintering furnaces benefit from Temperature Uniformity Survey's (TUS's) to meet the requirements of quality control/assurance procedures, however there is the "dark side of the moon" period between surveys in which there is the possibility of unwanted surprises from the sintering process.

If you passed your last survey, but you fail your next survey, how do you know when something changed in your sintering process? Was it two days before your bad survey or was it two days after your good survey?

Based on proven materials technology the Orton Ceramic Foundation, based in Westerville, Ohio, USA, has developed an easy to use tablet shaped product, called TempTAB, that allows users to monitor and document their thermal processes as frequently as they believe necessary.

The product can be used in both batch and continuous furnaces and is designed to work in most atmospheres including air, inert or reducing atmospheres. TempTAB's have been processed to remove any organic binders used in the forming process so there is no out gassing of any organic material and they are able to survive rapid heating and cooling cycles.

The tablets are made from blends of inorganic materials that are selected based on their predictable shrinkage when exposed to elevated temperatures. The small disc shaped tablets (28 mm x 7 mm) have a flat index surface and a hole in the centre (Fig. 1).

The tablets have been designed to be sensitive to both maximum temperature and time at temperature, the combined effect of time and temperature is often referred to as "heat work". The real proof of TempTAB sensitivity is the correlation between the readings provided by the TempTAB and the physical properties of the product being sintered.

Case study

Sintered samples were made from FC-0208-50 iron base mix and moulded at 25 bars to a green density of 6.8 g/cm³. The bars were sintered in a muffle furnace with an atmosphere of

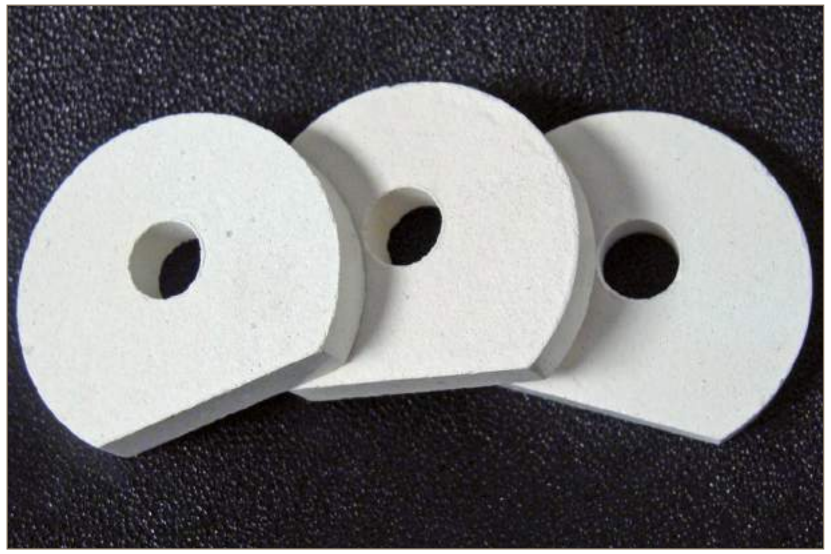

Fig. 1 TempTABs before entering the furnace

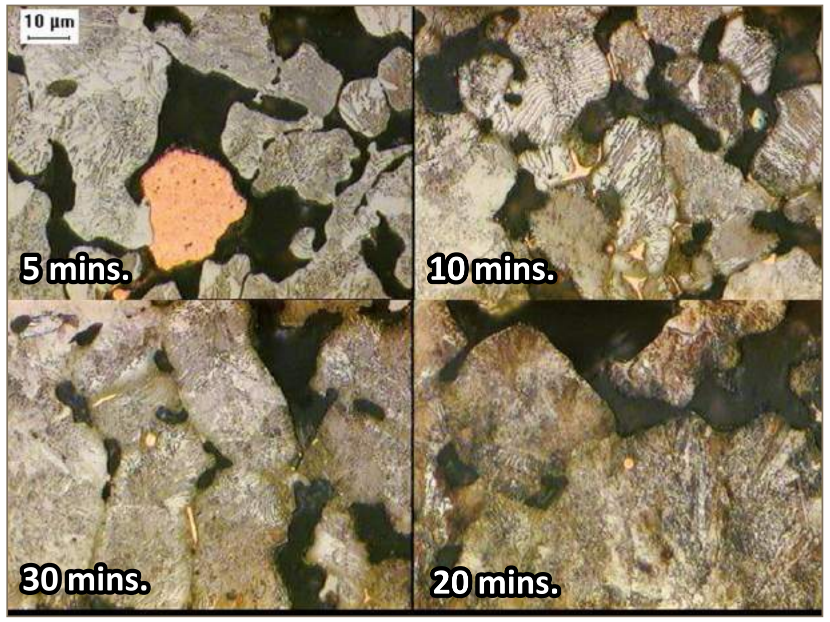

Fig. 2 The etched, 800x images reveal the degree of sintering at 2,050°F for times of 5, 10, 20 & 30 minutes (Courtesy of Powder-Tech, Inc.)

dissociated ammonia, 75% hydrogen and 25% nitrogen. The test bars were sintered at 1,121°C (2,050° F) for hold times of 5, 10, 20, 30, and 40 minutes. As can be seen in Table 1 the physical properties of this typical powder metal mix can be seen changing with increased time at temperature, and the TempTAB dimensions correlate very consistently with the varying time at temperature. The standard 2% yield strength for this mix is a minimum of 345 MPa (50,000 psi.).

The etched, 800x images shown in Fig. 2 reveal the degree of sintering at 1,120°C (2,050° F) for times of 5, 10, 20 and 30 minutes. In the 5 minute image large particles of undissolved copper can be seen, indicating the sintering process has advanced very little. The 20 and 30 minute images show the formation of pearlite indicating a normal sintering for this type mix.

Temperature variation within the furnace may be more common during sintering than variation in hold times. The impact of temperature variation within the furnace was the motivation for implementation of industry standards for TUS's. Furnace temperature variation can be caused by deteriorating thermocouples, controller calibration drift, heating element failure and heat containment insulation damage. The information in Table 2 demonstrates the sensitivity of the product properties and the correlation of the TempTAB dimensions to varying temperatures at a constant hold time.

TempTABs can report variation in temperature and/or hold time. When run within the sintering process on a routine basis TempTABs are capable of alerting the operator to any changes in the process that are likely to have a deleterious effect on the PM product.

Using TempTAB

During a normal sintering process, TempTABs can be placed alongside the product being sintered (Figs. 3-4). In a continuous process the tablets can be placed directly onto the belt or setter plates. In a batch furnace, they can be wired to the baskets or placed directly in the load.

Once the tablets have recorded the thermal process, they are gathered and labeled so that their location is easily traced. They are then measured using a digital indictor. The dimension from the digital indicator is then used to locate, in the tables provided, the process temperatures the TempTABs were exposed to. Each batch of TempTABs is provided with a calibration chart with standard heating ramp rates and multiple hold times. Orton states that as each process is different, TempTABs can if necessary be calibrated to fit individual process needs.

TempTABs were designed to help users "know" what is going on inside their furnace between surveys. TempTABs were never intended to replace thermocouples or to be used to calibrate thermocouples. Thermocouples read temperature at a specific location in real time without regard to any prior thermal history. Real time, instantaneous temperature readings from thermocouples provide vital information to digital temperature controllers and can provide the thermal profile (heating/cooling) of a periodic furnace, or if the thermocouple is allowed to travel with the parts, in a continuous furnace.

AMS 2750D and CQI-9 have specific intervals, three or six months, when temperature uniformity surveys are required. To meet the current requirements for AMS 2750D and/or CQI-9 it is necessary to place thermocouples throughout the load and record the temperatures. In a box furnace this can be accomplished by placing a self-contained data logger directly in the furnace and attaching several thermocouples that are strategically placed among the parts being treated.

If head space allows, the same type of data logger can be placed into a continuous furnace. The self-contained data logger and its heat protection package are expensive and require space within the furnace, resulting in interruption of production schedules. Of course one can accomplish the temperature uniformity survey using thermocouples and an external data logger, but this usually entails lots of wires, the possibility of broken connections and is usually labor intensive. To meet current AMS 2750D or CQI-9 requirements, for example, it is necessary to perform some variant of the above.

Time at 1,121°C / 2,050° F	TempTAB dimension	Test Bar dimension	2% Yield Strength MPa / psi	Fracture Kg / Lbs.
5 minutes	27.82 mm	89.97 mm (3.542 in.)	N/A	381 kg / 840 lb.
10 minutes	27.69 mm	89.97 mm (3.542 in.)	316 MPa /45,967 psi	1,480 kg / 3,263 lb.
20 minutes	27.38 mm	89.97 mm (3.542 in.)	388 MPa / 56,333 psi	1,778 kg / 3,920 lb.
30 minutes	27.19 mm	89.81 mm (3.536 in.)	435 MPa / 63,100 psi	1,989 kg / 4,387 lb.
40 minutes	27.02 mm	87.79 mm (3.535 in.)	439 MPa / 63,767 psi	2,116 kg / 4,677 lb.

Table 1 Constant temperature with varying hold times

Temperature 30 min. hold	TempTAB dimension	Test Bar dimension	2% Yield Strength MPa / psi	Fracture Kg / Lbs.
1,107°C / 2,025°F	27.43 mm	89.89 mm (3.539 in.)	415 MPa / 60,267 psi	1,772 kg / 3,907 lb.
1,120°C / 2,050° F	27.19 mm	89.81 mm (3.536 in.)	435 MPa / 63,100 psi	1,989 kg / 4,387 lb.
1,135°C / 2,075° F	27.00 mm	89.79 mm (3.535 in.)	455 MPa / 66,100 psi	2,035 kg / 4,487 lb.

Table 2 Constant hold time with varying temperatures

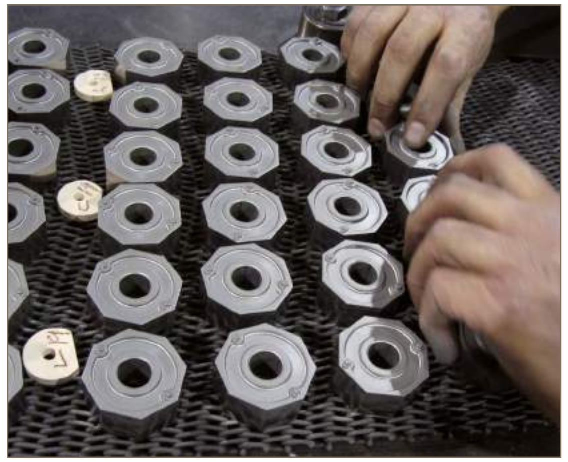

Fig. 3 TempTABs entering sintering furnace with minimal disruption to production

Fig. 4 TempTABs exiting a sintering furnace

Bringing value to the sintering process

Due to the expense, difficulty and interruption caused by a formal temperature uniformity survey, most facilities chose to only do the surveys when required to meet industry or internal standards. TempTABs can provide a simple, cost effective way to monitor the sintering process routinely without the difficulty of running a formal survey.

Benchmarking the furnace with TempTABs during a successful, formal survey establishes a baseline to compare future TempTAB readings to. Incorporating TempTABs into a quality assurance program, by establishing a routine schedule for placing TempTABs in the furnace and plotting the results in a SPC chart, will provide confidence in the consistency of the sintering process in a simple and cost effective manner. This information is not only valuable to the operator, but to the customer as well, as it provides a detailed record of the sintering process that stresses the operator's commitment to consistent, reliable quality.

Such a system has proven to be a simple, cost effective means of monitoring powder metal sintering. Data from TempTABs can be collected using special "Trakker" software that converts the dimension measured from a gauge to a process temperature and loads it into a table and graph on a connected computer (Figs. 5-6).

Between those burdensome formal surveys TempTABs provide assurance that the sintering process is in control, providing confidence in the consistency and uniformity of the sintering process.

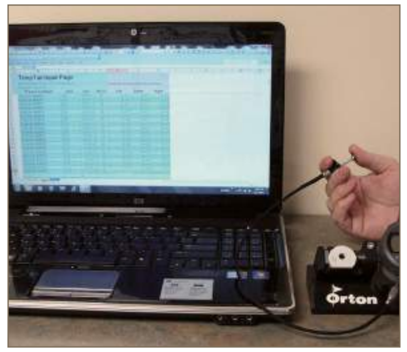

Fig. 5 TempTAB is measured after exiting the furnace and the dimension is entered into the software.

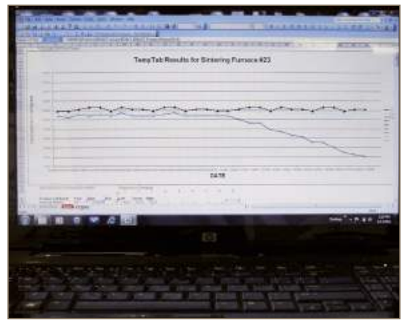

Fig. 6 TempTAB Trakker Software automatically transforms the dimensions into process temperatures and provides the data in both table and graph form for monitoring

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Global PIM Patents

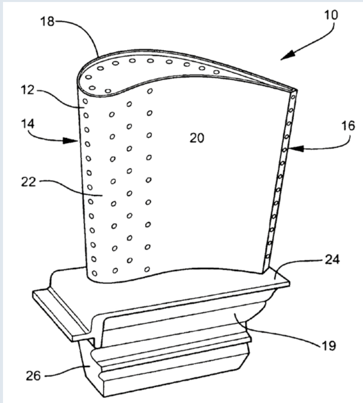
The following abstracts of PIM-related patents have been derived from the European Patent Organisation databases of patents from throughout the world.

GB2448031 (A)
METAL INJECTION MOULDING PROCESS FOR BIMETALLLIC APPLICATIONS AND AIRFOILS
Publication date: 2008-10-01
Inventor: S. J. Ferrigno, General Electric Company, USA
This invention relates to high-temperature components for gas turbine engines and more particularly to components having a composition of more than one alloy.

Current techniques for producing bimetallic components entail the use of joining processes such as tungsten inert gas welding, electron beam welding, inertia welding, brazing and similar processes. These methods are expensive, can leave weakened heat affected zones and are often difficult to inspect.

Here, the first preform is fabricated by providing a first mixture of a metallic powder of a first alloy and a binder, melting the binder and extruding the first mixture in a mould to form a first preform, and leaching the first preform to remove excess binder. The second preform is fabricated by providing a second mixture of a metallic powder of a second alloy and a binder, melting the binder and extruding the second mixture in a mould to form a second preform, and leaching the first preform to remove excess binder.

The first and second preforms are constructed through a metal injection moulding (MIM) process.



WO2008134198 (A2)
METAL INJECTION MOULDED TITANIUM ALLOY HOUSING FOR IMPLANTABLE MEDICAL DEVICES
Publication date: 2008-11-06
Inventor(s): B. Li et al, Medtronic Inc, USA
The housing of an implantable medical device is made of a titanium alloy that provides improved electrical performance, mechanical strength, and reduced MRI heating. The titanium alloy housing includes portions formed by metal injection moulding and welded together. Wall thickness of at least a portion of one major face of the housing is reduced by chemical etching a metal injected moulded housing portion.

CA2684988 (A1)
METAL INJECTION MOULDING SYSTEM AND PROCESS FOR MAKING FOAMED ALLOY
Publication date: 2008-12-24
Inventor: F. Czerwinski, Husky Injection Molding Systems, Ltd, Canada
Disclosed is: (i) a metal injection-moulding system, (ii) a metal injection-moulding system including a combining chamber, (iii) a metal injection-moulding system including a first injection mechanism and a second injection mechanism, (iv) a metal injection-moulding system including a first injection mechanism being co-operable with a second injection mechanism, (v) a mould of a metal injection moulding system, and (vi) a method of a metal injection moulding system.

CA2660484 (A1)
METAL INJECTION MOULDING METHOD
Publication date: 2008-02-14
Inventor(s): B. G. Schaffer et al, University of Queensland, Australia
This patent describes a method for forming an article by metal injection moulding of aluminium or an aluminium alloy.

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 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.

JP2008280217 (A)
ALUMINIUM NITRIDE POWDER FOR INJECTION MOULDING, ALUMINIUM NITRIDE COMPOSITION FOR INJECTION MOULDING, ALUMINIUM NITRIDE SINTERED MATERIAL, AND PRODUCTION METHOD OF ALUMINIUM NITRIDE SINTERED MATERIAL
Publication date: 2008-11-20
Inventor(s): Goto Kunihiro et al, Tokuyama Corp; Sun Arrow Kasei Co Ltd; Tomitec Corp , Japan
This patent is for an aluminium nitride powder for injection moulding, suitable for producing an aluminium nitride composition with high flowability, a green folded body with excellent shape retention and debinding properties, and an aluminium nitride sintered material with high dimensional accuracy.

Effect of a slight addition of Zr on the sintering behaviour of water-atomised 316L stainless steel powder

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The main objective of the present work is to evaluate the effects of Zr addition on various properties of SUS316L stainless steel powders produced by water-atomisation of liquid metals. It can be thought that an addition of Zr leads to an improvement in the sintering characteristics and mechanical properties by contributing to the refinement of the powder structure and Si oxide in addition to the inhibition of Si oxide formation on the powder surface.

KEYWORDS

Metal injection moulding, water atomised powder, Zr, Si, sintered density

1 Introduction

In general, powder can be sintered by either solid-phase sintering or liquid-phase sintering. In solid-phase sintering, surface diffusion acts as the driving force in the initial sintering stages; however, because of the low energy of surface diffusion, it is volume diffusion that promotes densification. The speed of volume diffusion varies depending on crystal structure, inhibiting densification in some types of steel and resulting in a deterioration of mechanical properties. This problem can be prevented by allowing for a second phase of high diffusion velocity [1-2], or generating a liquid phase by the addition of B or the like [3-4] to achieve densification.

The metal injection moulding (MIM) method is often applied to austenitic stainless steels. Some types of austenitic stainless steels are designed to form a specific alloy composition so that the δ ferrite phase, which has a higher diffusion velocity, precipitates out in order to promote sintering. However, even if the structure is fully austenitic around the sintering temperature, if the rate of cooling is slow, the δ ferrite phase will be precipitated, which may result in a deterioration in corrosion resistance.

Furthermore, for water-atomised powders, oxide film forms on the powder surface, delaying surface diffusion which in turn hampers densification.

Sawai *et al.* [5] report that when Zr has been added after the deoxidation of molten, low-sulfur steel with Mn-Si, the Mn-Si oxide and Zr-O_2 coexist with each other in cases of small amounts of Zr addition. Also, Fujikawa *et al.* [6] report that when a stabilising element such as Zr has been added to ferritic stainless steel, oxidation resistance at high temperatures is improved. In addition, it has been reported that adding Zr to stainless steel results in a decrease in grain size and an improvement in mechanical properties [7].

In this study, to examine the effect of Zr addition on the austenitic stainless steel SUS316L, which is often used for MIM method part manufacturing, we fabricated powder using the water-atomisation method. We investigated the characteristics and sintering behaviour of the powders and the corrosion resistance of the obtained sintered compacts.

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2 Experiment

2.1 Powder fabrication

We fabricated the following three kinds of powders:
1) 316L
2) 316L-Zr
3) 316L+Zr

To fabricate powder 316L, we first measured pure iron, electrolytic nickel, ferrochromium, ferromolybdenum, ferrosilicon, and electrolytic manganese to achieve the desired composition, and then melted the mixture at 1923K in a high-frequency melting furnace with a capacity of 50 kg.

To fabricate powder 316L-Zr, to the molten metal obtained from 316L we added electrolytic zirconium until the Zr concentration was 0.05 mass%.

The melts of the obtained 316L and 316L-Zr were analysed with a spark discharge optical emission spectrometer (SPECTROLAB, Japan Machinery Company) to ensure that each melt had the desired composition. Then, the molten metal was formed into powder by the water-atomisation method using a water pressure of 100 MPa. After atomisation, the powder was sieved and classified so as to obtain a mean grain diameter of 10 µm.

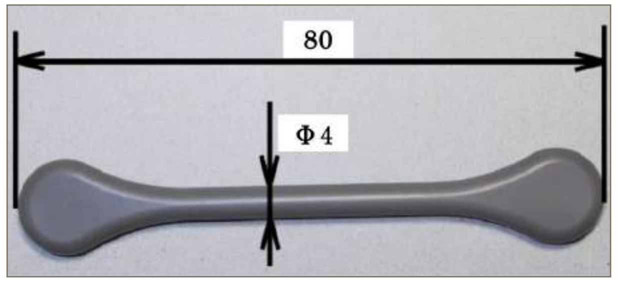


Fig. 1 Specimen shape

We prepared premixed powder 316L+Zr in order to compare it with powders 316L and 316L-Zr, which are prealloyed powders. A pure Zr powder, which was fabricated by the gas atomisation method, was sieved to 45 µm and then mixed with the previously fabricated Powder 316L until the Zr concentration was 0.05 mass%. Table 1 shows the characteristics of the powders.

2.2 Preparation of sintered compact

We mixed the three types of powders fabricated in 2.1 with binder at a volume ratio of 62.5:37.5, kneading the powders in a pressuring kneader for 3.6 ks to fabricate a feed stock. The feedstock was cooled, pulverised with a grinder, and then formed into a tensile test specimen as shown in Fig. 1 by using a moulding machine with a clamping pressure of 40 t. The test specimens were subjected to heating degreasing under conditions of 743K×3.6 ks in a nitrogen atmosphere (dew point: 193 K; oxygen concentration: 1^{-5} mass%), followed by a sintering process under conditions of 1623K×10.8 ks in an argon atmosphere (dew point: 203K; oxygen concentration: 5^{-5} mass%).

Further, to analyse the sintering mechanisms, the specimens were sintered in a vacuum for 3.6 ks at five temperatures: 973K, 1073K, 1173K, 1273K, and1373K.

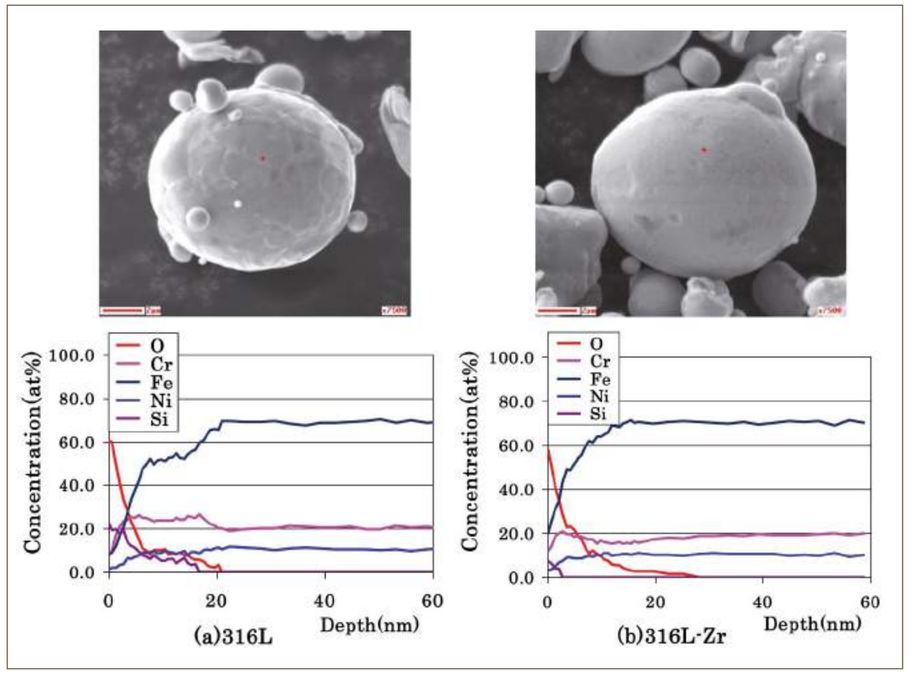


Fig. 2 Auger analysis of 316L and 316L-Zr powders

Chemical Composition (mass%)	Cr	Mo	Ni	Fe	Si	Mn	Zr	P	Hf	S	C	O	N
316L	16.51	2.13	12.45	Bal.	0.83	0.81	0	0.015	-	0.009	0.025	0.32	0.052
316L-Zr	16.48	2.15	12.51	Bal	0.81	0.77	0.052	0.012	-	0.003	0.02	0.29	0.051
Zr	0.02	-	-	0.084	-	-	Bal.	-	0.59	0.001	0.001	0.17	0.002

Physical properties	Particle size distribution (µm)			Tap density (Mg/m³)	Specific surface area (m²/Kg)
	D10	D50	D90		
316L	3.59	10.25	25.30	4.54	235
316L-Zr	3.98	9.98	24.67	4.38	240
Zr	15.54	32.07	49.50	-	45

Table 1 Chemical compositions and physical properties of powders

2.3 Evaluation

We subjected the powders fabricated in 2.1 to surface analysis by auger electron spectroscopy (JAMP-9500F; JEOL Ltd.), X-ray diffraction (XRD) analysis (RINT2500V; Rigaku Corporation), and microstructural analysis of the cross sectional surfaces and mapping analysis (JXA-8500F; JEOL Ltd.). Further, we performed state analysis on the Zr with the powders. For the total Zr, we dissolved the powder in aqua regia, and subjected the insoluble residual component to alkali fusion, mixing both. For oxide Zr, we dissolved the powder with 10 mass% Br, filtering the result, and then dissolved the residue in the mixture of sulfuric acid, phosphoric acid, and water. Each solution was analysed by ICP. X-ray photoelectron spectroscopy (XPS; Quantera SXM; ULVAC-PHI, Inc.) was also used to examine the chemical state of the Zr in 316L-Zr.

We subjected the sintered compacts obtained in 2.2 to quantitative analysis of C and O contents, density measurement (Archimedian method), measurement of mechanical properties and hardness, microstructural observation, and salt spray tests (test solution: neutral, 5 mass% salt water; test temperature: constant at 308K).

	Total Zr (mass%)	Zr as oxide (mass%)
316L-Zr	0.052	0.052

Table 2 State analysis result of Zr in powder

In addition, to investigate the sintering mechanisms, we observed inclusions in the sintered compact by the Selective Potentiostatic Etching by Electrolytic Dissolution (SPEED) method using non-aqueous solvent potentiostatic electrolysis (10 mass% acetylacetone, 1 mass% tetramethyl ammonium-methanol).

3 Experimental Results and Discussion

3.1 Powders

Fig. 2 shows the results of auger electron analysis for the surfaces of powders 316L and 316L-Zr. The thickness of the Si oxide layer is 17 nm in 316L, while it is 3 nm in 316L-Zr, suggesting that the formation of Si oxide was inhibited. As the content was below the detection limit, the Zr was not detected by the auger electron

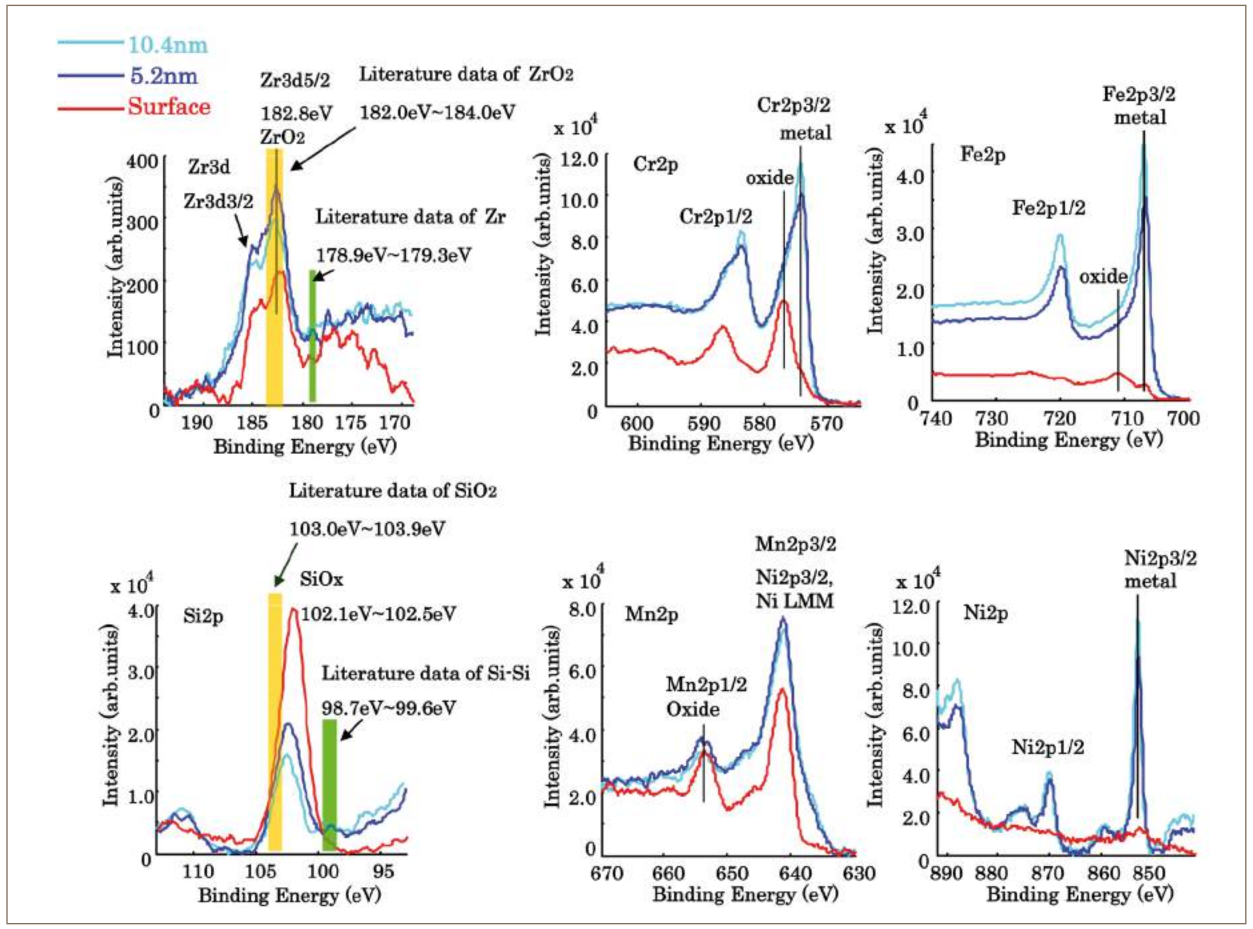


Fig. 3 XPS analysis of 316L-Zr powder

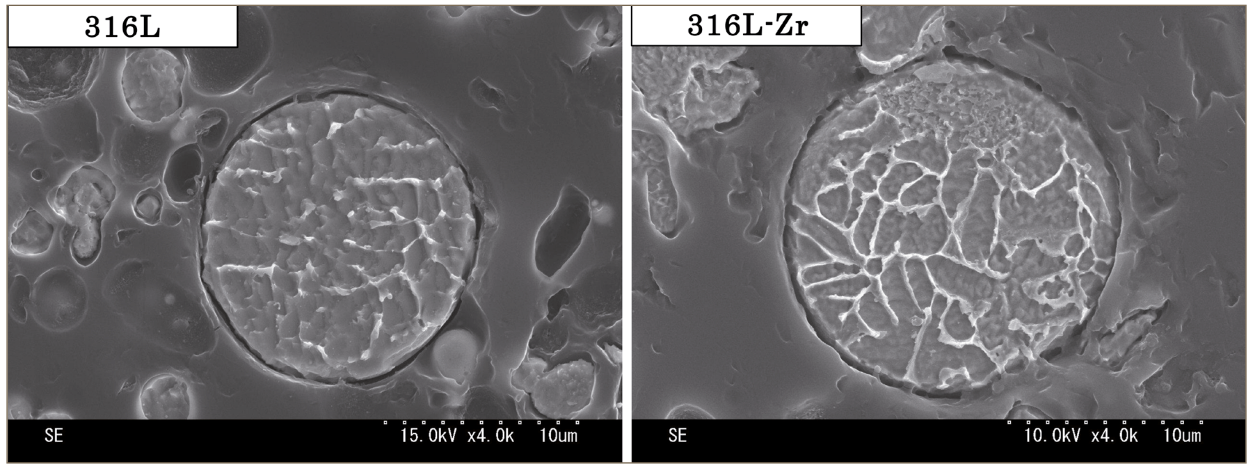


Fig. 4 Optical microstructures of 316L and 316L-Zr powders

analysis; therefore, we performed a state analysis for Zr. As shown in Table 2, Zr was detected only in oxide form. The results of XPS (Fig. 3) show that Zr is present in the form of ZrO_2 at all three depths: i.e., on the powder surface and at depths of 5.2 nm and 10.4 nm. Further, the pattern indicates a feature that appears to be a small peak in the same location as that given as the peak location for metal zirconium by the literature. Si is also present in oxide form on the surface and deep subsurface of the powder, with peak locations slightly lower than those given by the literature, suggesting that the material is in a lower valence number oxide form compared to SiO_2. We observed that the major components of SUS316L, such as Fe and Cr, are present in oxide form on the powder surface, and in metallic form in the deep subsurface of the powder.

Fig. 4 shows the microstructure of the powder. We observe clear crystal structure in 316L-Zr. Fig. 5 shows the results of mapping. A comparison of observations of the two powders suggests that while there is very little difference with regard to O, Cr, and Mo, there are differences in concentration in Si, Fe, and Ni. Specifically, Ni and Si have high concentrations in grain boundaries, while Fe has high concentrations in matrix areas. The crystal grains have been refined in 316L-Zr, which we suspect was caused by the crystal grain refinement effect of Zr.

On the other hand, Zr is known to be a ferrite-forming element, and in austenitic stainless steels such as 316L, the presence of δ ferrite phase (bcc lattice) is known to increase the sintering rate; hence, we performed XRD analyses for 316L and 316L-Zr. The results are shown in Fig. 6. Only the peaks for the γ austenite phase (fcc lattice) were detected for both powders.

In the salt water spray test carried out for the compacts of 316L and 316L-Zr, we observed no difference in corrosion resistance at any of the following exposure times: 86.4 ks, 172.8 ks, and 259.2 ks, (Fig. 8).

3.2 Sintered compacts

Table 3 shows the sintering density, content amount of C and O, mechanical properties, and hardness of the sintered compacts. The relative density values for 316L, 316L-Zr, and 316L+Zr were 95.7%, 97.7% and 95.3%, respectively. In mechanical properties, the tensile strength, 0.2% yield strength, and elongation for 316L were 487 MPa, 154 MPa, and 64%, respectively; those for 316L-Zr were 522 MPa, 169 MPa, and 64%; and those for 316L+Zr were 415 MPa, 136 MPa, and 26%. Summarily, in 316L-Zr, sintering characteristics and mechanical properties increase, while in 316L+Zr, such characteristics decrease.

Fig. 7 shows the microstructure of the compacts. The images indicate that 316L-Zr has a finer crystal size compared to 316L and 316L+Zr, with smaller pores and inclusions.

Pickering [8] suggests that there is a relationship between the mechanical properties of austenitic stainless steels and the chemical composition, crystal grain diameter, etc., that can be expressed by the following equation:

$$0.2\%\ \text{yield strength} = 15.4[4.4 + 23(C) + 1.3(Si) + 0.24(Cr) + 0.94(Mo) + 1.2(V) + 0.29(W) + 2.6(Nb) + 1.7(Ti) + 0.82(Al) + 32(N) + 0.16(\delta\ \text{ferrite}) + 0.46d^{-1/2}] \quad (1)$$

$$\text{Tensile strength} = 15.4[29 + 35(C) + 55(N) + 2.4(Si) + 0.11(Ni) + 1.2(Mo) + 5.0(Nb) + 3.0(Ti) + 1.2(Al) + 0.14(\delta\ \text{ferrite}) + 0.82t^{-1/2}] \quad (2)$$

d= mean crystal grain diameter;
t= twin spacing

According to the above equation, mechanical properties are enhanced as crystal grain diameter or twin spacing decrease. Therefore, it can be thought that in 316L-Zr, the reduction in crystal grain diameter improved the mechanical properties, and in 316L+Zr, the fact that Zr powder remained within the grain boundaries resulted in the deterioration of mechanical properties.

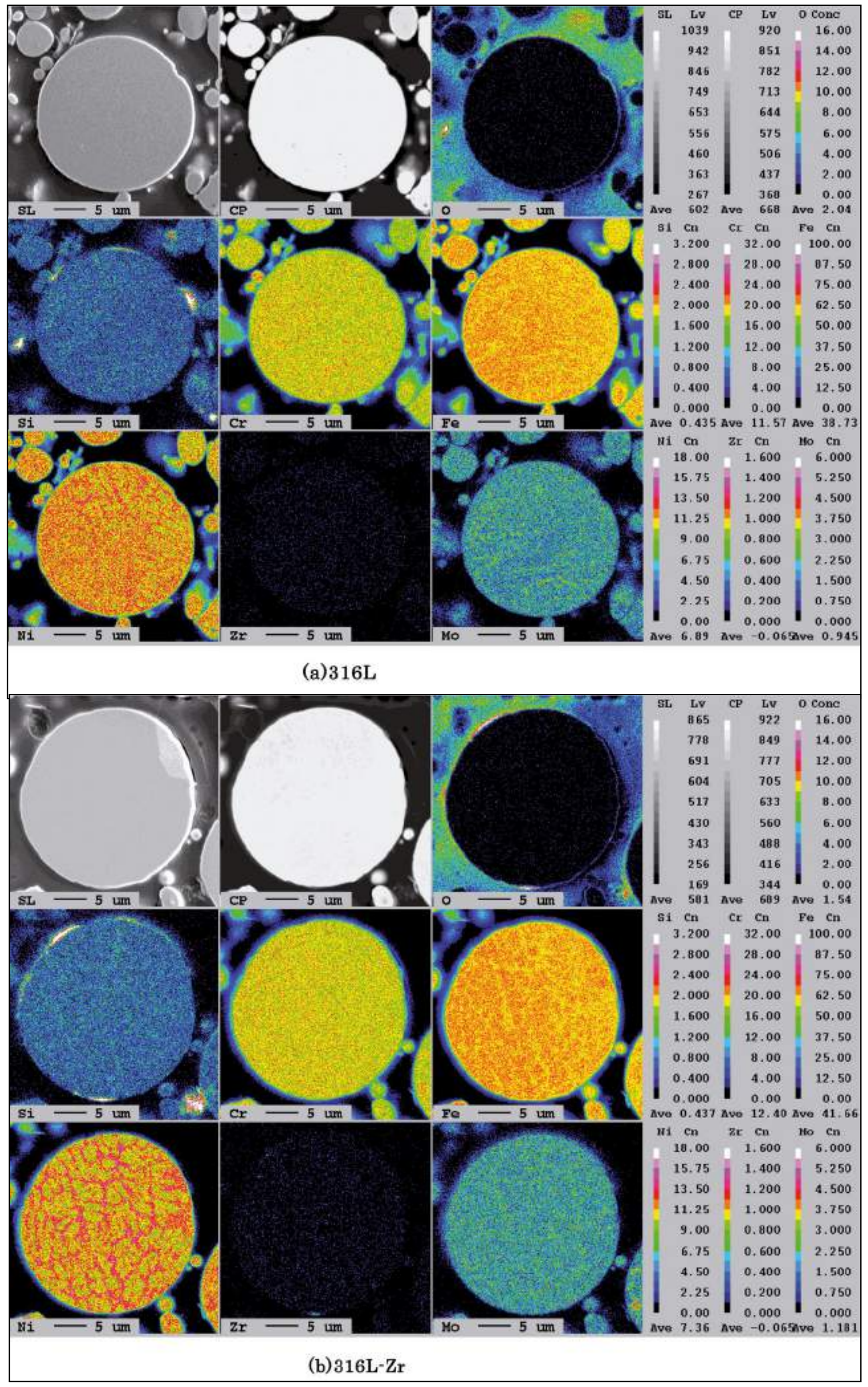


Fig. 5 Mapping analysis of 316L and 316L-Zr powders

	Relative density (%)	C content (mass%)	[O] content (mass%)	Tensile strength (MPa)	0.2% yield strength (MPa)	Elongation (%)	Hardness (HRB)
316L	95.7	0.011	0.33	487	154	64	57
316L-Zr	97.7	0.010	0.21	522	169	64	66
Zr	95.3	0.008	0.24	415	136	26	59

Table 3 Mechanical properties of sintered compacts

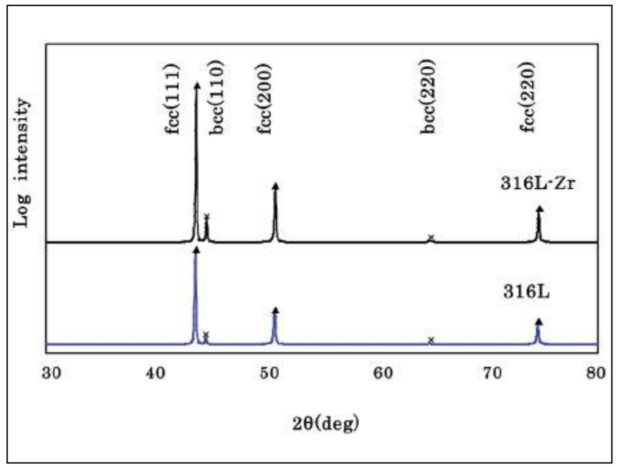


Fig. 6 XRD patterns of 316L and 316L-Zr powders

3.3 Analysis of the sintering process

Since results indicating improvements in the sintering characteristics and mechanical properties had been obtained for 316L-Zr, we investigated the changes in density during the sintering process. The results (Fig. 9) show that while in 316L and 316L+Zr almost no shrinkage occurred until 1173K, in 316L-Zr shrinkage occurred at 1073K, about 100K lower than that of 316L and 316L+Zr. In particular, in the temperature range from 1273K to 1373K, advancement of densification in 316L-Zr is faster than that of 316L and 316L+Zr by about 7-8%. Fig. 10 shows the sectional-view SEM images of the compacts at each sintering temperature. In 316L-Zr, a greater degree of progress of surface diffusion and volume diffusion is observed at 1073K, and it is clear that the diffusion initiation temperature is about 100K faster than that of the others. This indicates that surface diffusion without shrinkage begins at the initial sintering stages, which is followed by the initiation of shrinkage-accompanied volume diffusion.

3.4 Analyses of powders and sintered compacts

Fig. 11 shows the images of the compacts' (1623K) fractured surfaces after the tensile test. Examination of the fractured surface reveals that the Si oxide for 316L-Zr has a spherical shape with a diameter of 2-5 μm, while those for 316L and 316L+Zr have irregular shapes with dimensions ranging from 3-10 μm. The slightly smaller size of the Si oxide in 316L-Zr is probably attributable to the fact that, as described above, Mn-Si oxide partially melted in the molten steel, causing the oxide to establish equilibrium with the dissolved oxygen in the molten steel. In 316L, we think that the over growth of the Si oxide generated in the grain boundary of the sintered compact inhibited densification, leading to the degradation of mechanical properties and hardness. Regarding this phenomenon, Takeda *et al.* [9] suggest that Si has high levels of oxide-forming free energy, which allows the Si present inside the powder to move toward the powder surface, thereby lowering the free energy.

Fig. 12 shows the results of surveying the inclusions, which we generated by allowing the 316L-Zr compact (1623K) to be corroded by the SPEED method. Si oxide was also observed in the compacts, which confirms that Si oxide is combined with Zr oxide. In other words, it can be thought that the combined state of Si oxide and Zr oxide at the powder stage is maintained at the sintering stage. Fig. 13 shows the appearance of the sintered powder in a vacuum. For 316L, we observed that Si oxide present on the surface starts to aggregate and grow larger as the sintering temperature increases. Fig. 14 shows the state of oxides near the grain boundary triple point for 316L-Zr, which was sintered at 1173K.

We performed mapping analysis regarding Si, Zr, and O for the powder and sintered compact of 316L-Zr at 973K (Fig. 15). The results indicate that in powder form, Si is present on the surface and inside the powder, while in the compact form, Si migrates to the surface of the powder.

Fig. 16 shows the sintering mechanisms for 316L and 316-Zr, based on the above examination.

In 316L, the powder surface is covered with thick Si oxide as

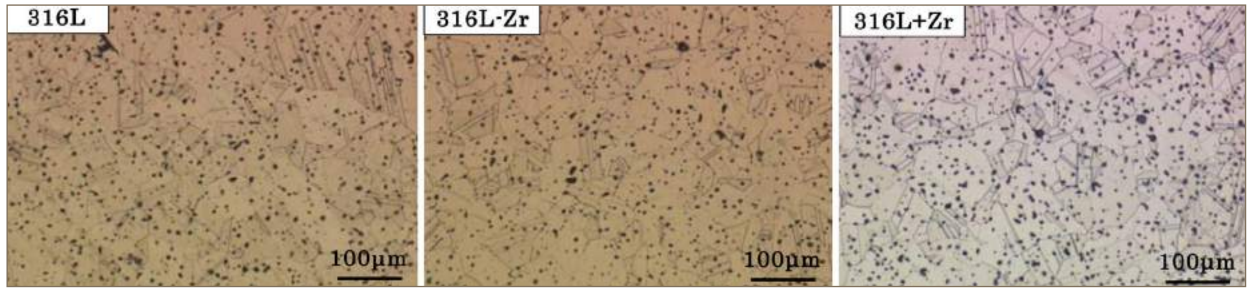


Fig. 7 Optical microstructures of sintered compacts

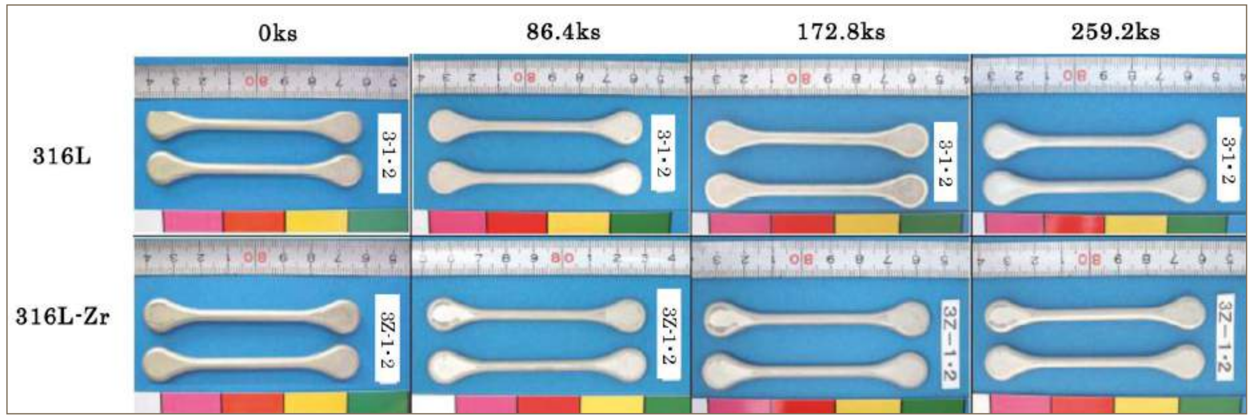


Fig. 8 Specimens after salt sprays

a result of the water-atomisation method. In contrast, in 316L-Zr, which is prealloyed with Zr, the combined oxide generated by the deoxidation reaction between Si oxide and Zr oxide at the molten steel stage is maintained through the powder stage. When Zr oxide is formed by Zr addition, the oxygen concentration in the area around the molten steel decreases, and the Si oxide partially melts into the molten steel in order to maintain equilibrium with the oxygen in the molten steel, which reduces the size. When such molten steel is atomised, Zr oxide film forms on the outermost layer of the powder, thus creating a powder in which Si oxide formation is inhibited.

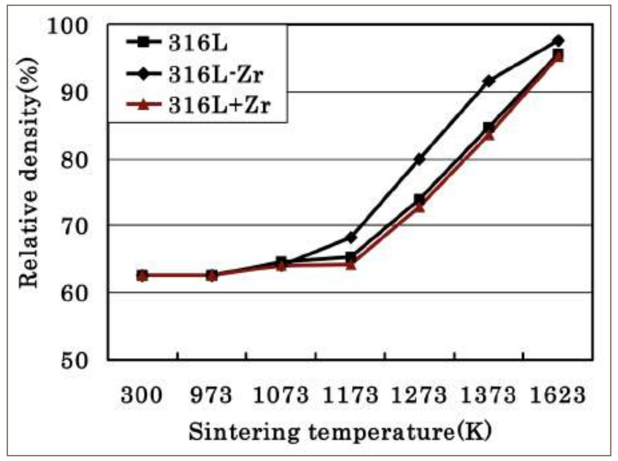


Fig. 9 Relation between sintering temperature and relative density

When such a powder is sintered by heating, as Yamada *et al.*[10] identified, activity increases in relation to the concentration. Also, the Si contained in the powder has high-oxide-forming free energy, allowing the Si to diffuse to the surface of the powder to form oxide. In this case, O is supplied by the oxides present on the powder surface, such as Fe and Cr; therefore, reduction reactions as shown in the following equations occur:

$$2FeO+Si \rightarrow 2Fe+ SiO_2 \quad (3)$$

$$2Cr_2O_3+3Si \rightarrow 4Cr+ 3SiO_2 \quad (4)$$

As a result, the surface energy of the reduced powder surface increases, and sintering proceeds by forming necks in order to lower the increased surface energy. In 316L, in which Zr was not added, the Si oxide formed in the triple point grew in size and inhibited densification; while in 316L-Zr, in which Zr was added, the finely dispersed Si oxide acted as the pinning agent, contributing to the refinement of the structure by preventing the overgrowth of crystal grains, which resulted in an improvement in the densification and mechanical properties.

As demonstrated above, it can be thought that an addition of Zr leads to an improvement in the sintering characteristics and mechanical properties by contributing to the refinement of the powder structure and Si oxide in addition to the inhibition of Si oxide formation on the powder surface.

4 Conclusions

Powder analysis and the MIM method sintering tests were performed by fabricating powders using the water-atomisation method: (1) powder 316L; (2) powder 316L-Zr in which 0.05

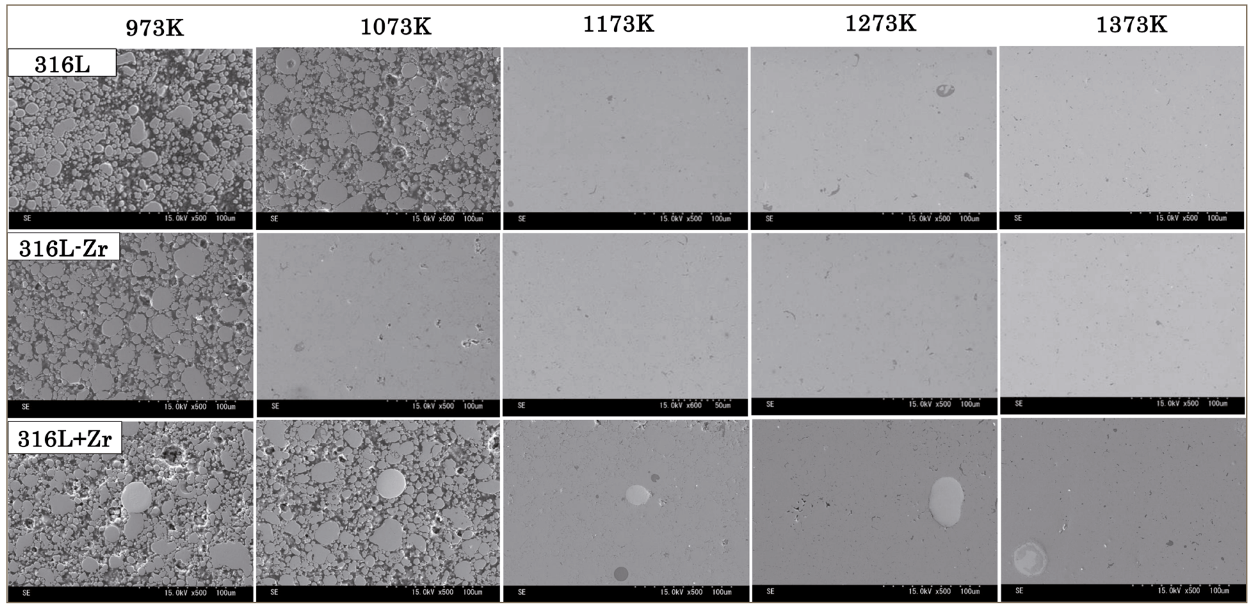


Fig. 10 Micrographs of sintered compacts by SEM

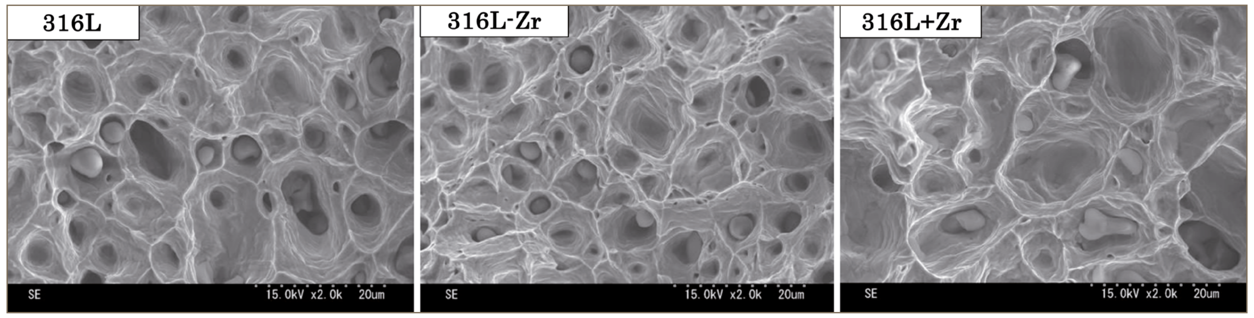


Fig. 11 Fracture surface of sintered compacts

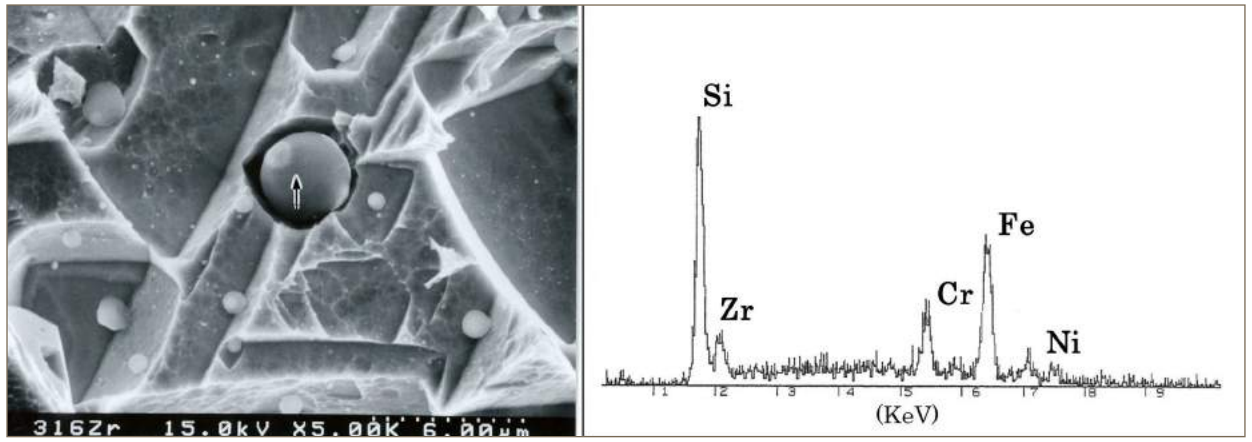


Fig. 12 Oxide discovered in SPEED-etched 316L-Zr sintered compact

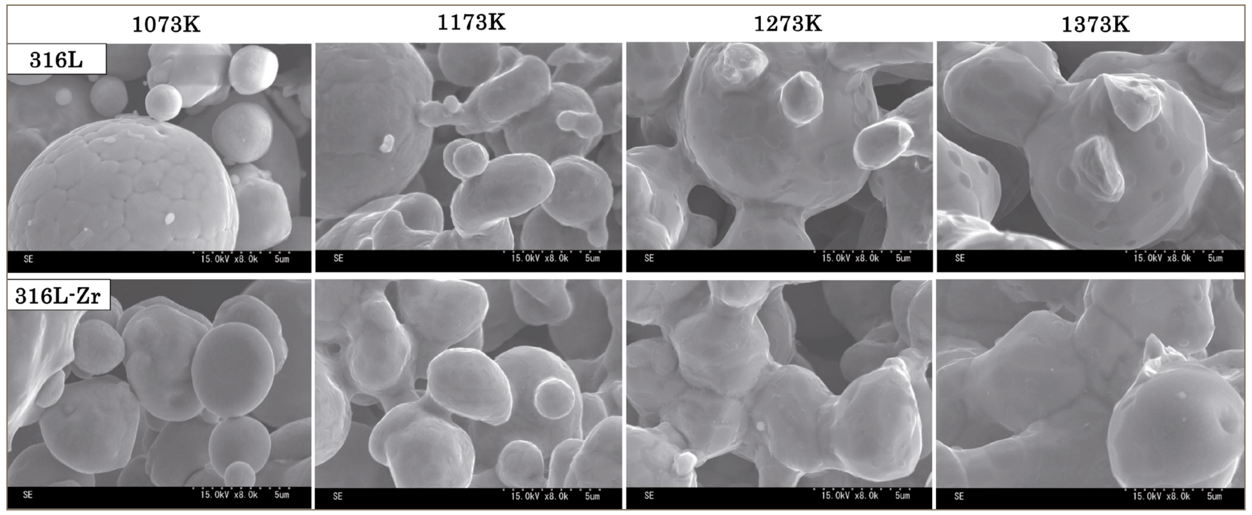
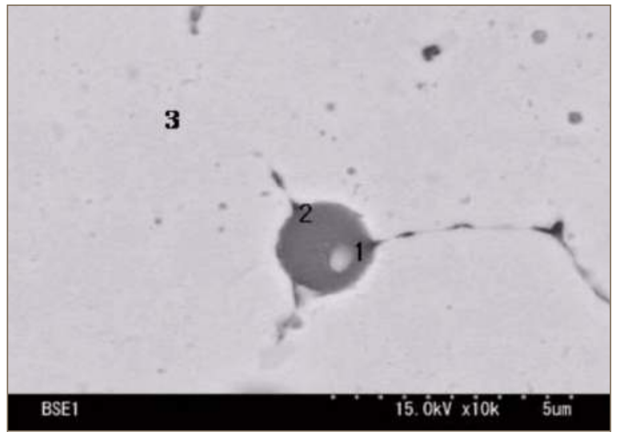


Fig. 13 Surface observation of sintered powders by SEM



	O	Si	Cr	Mn	Fe	Ni	Zr	Mo
1	31.29	9.84	13.95	6.33	12.03	1.67	24.89	
2	26.99	13.27	18.93	9.86	15.71	2.78	12.45	
3		0.67	16.19		67.67	13.31		2.17

Fig. 14 Oxide discovered on the boundaries of a 316L-Zr sintered compact

mass% of Zr was added; and (3) powder 316+Zr, in which 0.05 mass% of Zr was added to Powder 316L from (1). We established the following.

(1) While the oxide layer formed on the surface of powder 316L had a thickness of 17 nm, that of powder 316L-Zr had a thickness of 3 nm, suggesting that the Zr addition inhibited the formation of Si oxide.

(2) In powder 316L-Zr, we observed refinement in the microstructure, which we think contributes to the densification caused by volume diffusion.

(3) In powder 316L-Zr, the sintering density of the compacts increased about 2% more than powder 316L. However, in powder 316L+Zr, we observed no effects from the Zr.

(4) In the sintered compacts of powder 316L-Zr, we observed improvements in mechanical properties, while in powder 316L+Zr, the mechanical properties deteriorated.

(5) In the sintering process, we observed an increase in sintering density for powder 316L-Zr from 1073K; we observed advancement of the neck formation by structural observation from 1073K as well, indicating that the initiation temperature is lower than that of 316L by 100K.

(6) In 316L-Zr, Zr added to the molten steel lowers the oxygen concentration in the area near the molten steel by becoming Zr oxide. In powder obtained by the atomisation of the molten steel, the Si had high levels of oxide-forming free energy and high activity. Thus, Si diffuses to the powder surface at the time of sintering, becoming oxides by removing O atoms from the oxides present on the surface of the powder, such as Fe and Cr oxides. Consequently, the surface energy of the powder increases, promoting the neck formation, which we consider the cause of the improvements in sintering characteristics and mechanical properties.

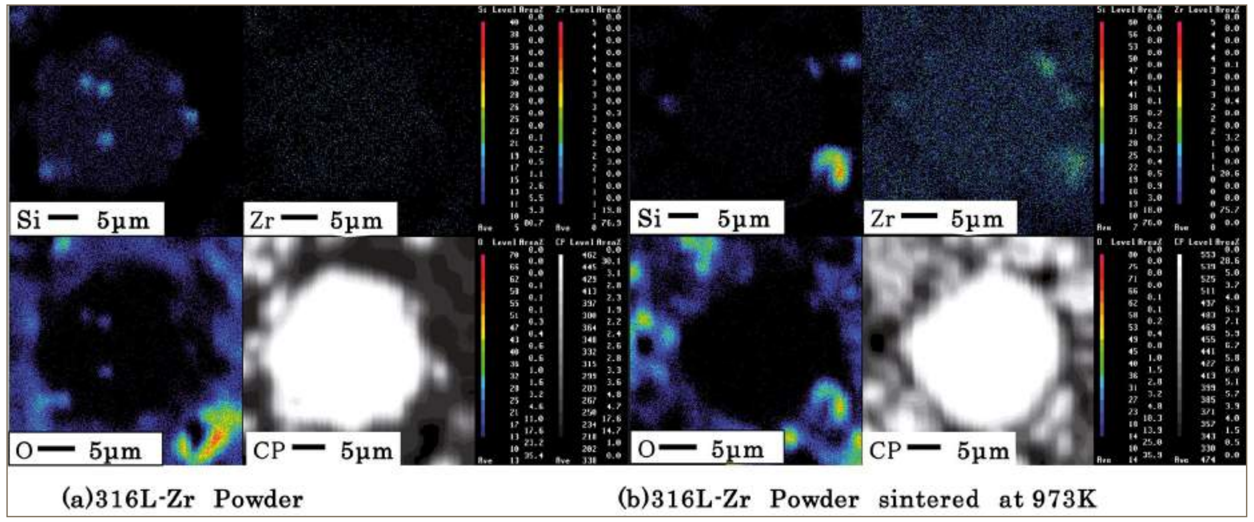


Fig. 15 Mapping analysis of Si, Zr and O in 316L-Zr powder

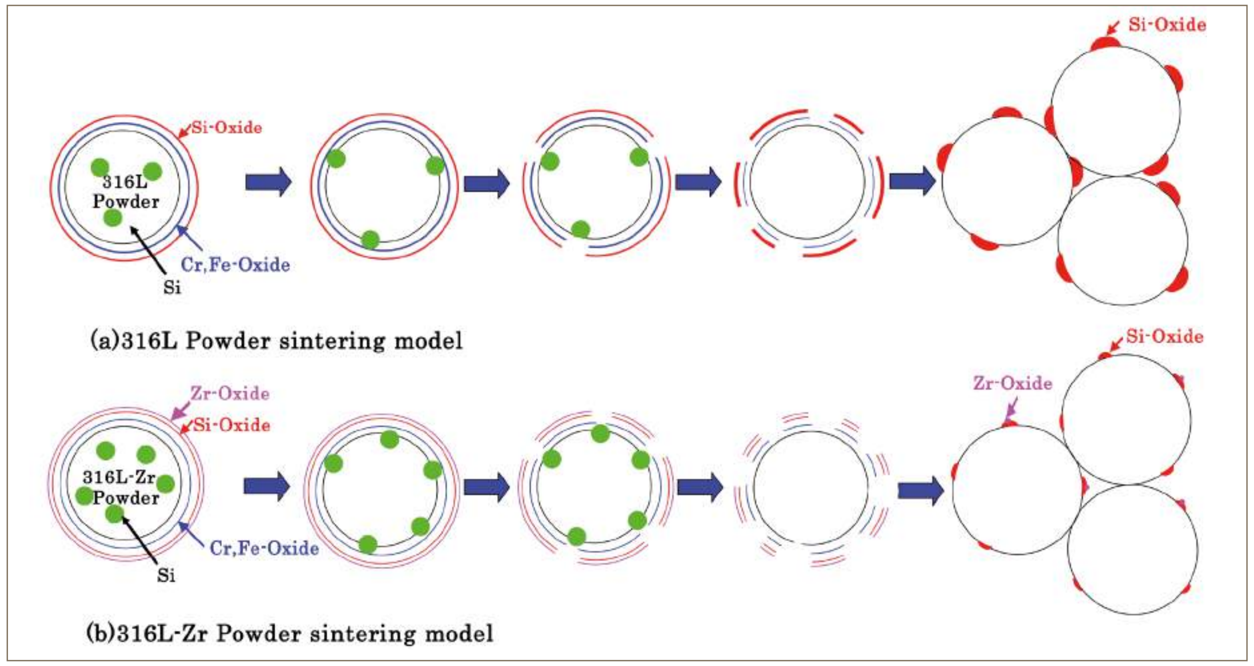


Fig. 16 Proposed sintering mechanism for 316L and 316L-Zr powders

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Technical Paper

Progress of Two-Component Micro Powder Injection Moulding (2C-MicroPIM)

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Two-component micro powder injection moulding (2C-MicroPIM) offers some very interesting opportunities to microsystem technology. It allows the combination of different features like hard - soft, magnetic - non-magnetic, electrically conductive - insulating as well as hard - tough by integrating different materials within one micro device. However, it is accompanied by complex challenges. In the presented study the availability of 2C-MicroPIM for the production of fixed, as well as, movable shaft-to-collar connections will be discussed. In detail, the influence of the materials like ceramic powders and binder components, the design of the injection moulding tool and the adjustment of the process parameters are important steps, which have to be understood and optimised. With respect to this task significant progress compared to previous trials has been achieved. The influences of these factors are presented as well as essential methods for improving 2C-MicroPIM for the production of movable and fixed connections.

Introduction

Due to the growth of microsystem technologies, high demands are made on the production of complex micro devices. Micro injection moulding represents an important facility to produce such micro devices by mass production. For plastic applications this technology is already established, while its applications for micro parts which are composed of robust materials like metals or ceramics still need further development. Micro parts composed of ceramics or metals can be produced by micro powder injection moulding (MicroPIM).

The basic requirement for MicroPIM from the materials side of view is a combination of a ceramic or metallic powder and diverse binder components. Generally the binder consists of a polymer, a diluent and one or more additives.

A further benefit can be obtained when two materials can be combined in a micro device and therefore also different functions. This can be enabled by the so-called two-component micro powder injection moulding (2C-MicroPIM), for which two injection units are used. Also for this type of injection moulding, an elementary development is necessary. Research activities on this technology began during the last few years. These activities include studies based on metals [1-4] as well as ceramics [5-7]. The potential of two-component micro powder injection moulding is being studied with the help of a shaft-to-collar-connection as demonstrator. The axle of this demonstrator is composed of alumina, while the gear wheel is made up of yttria-doped zirconia. The aim of the investigations is to realise a fixed connection as well as a movable connection, which means that the gear wheel can rotate around the alumina axle. Former works concentrated on tool design and construction [8], materials selection [9] and process development [10]. It was demonstrated that materials are suitable for movable as well as for fixed connections and feasible material combinations were found. For the process development and the materials selection some basic conditions have to be taken into consideration. For example, for a movable connection the axle has to shrink earlier and with a higher degree than the gear wheel. On the other hand, for fixed connections the shrinkage behaviour of both components should be almost identical. With the help of the demonstrator it was shown that fixed connections can be repeatedly produced and that movable connections can in principle be generated. Recent work focused on an improvement of the rotateability of the gear wheel of the shaft-to-collar connection. This includes investigations of the process as well as modifications of the tool design.

Results

Injection moulding of shaft-to-collar connections using the first tool design, which had two gatings with the shape of the arcs (C-arcs; Fig. 1) resulted in difficulties with respect to the realisation of movable connections. The reason therefore was a material movement towards this gating, when the alumina feedstock was injected in order to form the axle. The distance between the depressed gatings and the axle was too small to bear up against the injection pressure when forming the axle as a second component. As a consequence, the sintering process for these samples had to be stopped at a maximum temperature of 1300°C and rotation movements of the samples followed by a stepwise continuation of the sintering process and further rotation movements had to be conducted. A new design has been developed which now contains two point-shaped gatings instead of the C-shaped arcs (Fig. 1). The distance between axle and the gatings of the gear wheel has been increased and the depression of the gatings was reduced. As a result the wall between gatings and the axle is thicker now and therefore more stable against the pressure that is induced by the injection of the alumina feedstock. Fig. 2 shows a comparison of the former and the actual gating concept.

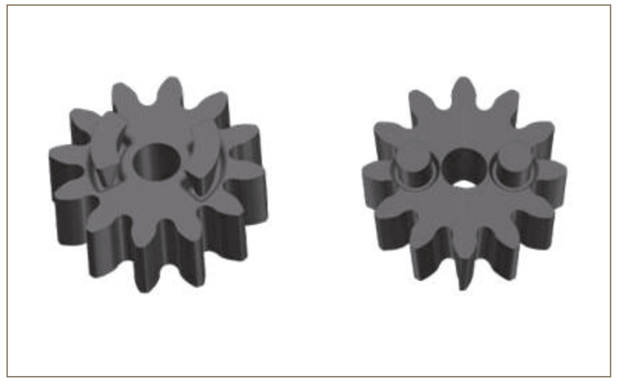

Fig. 1 Scheme of the gear wheel for the shaft-to-collar connections. Left: Former design with C-arc shaped gatings. Right: Actual design with point-shaped gatings

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After debinding and sintering (parameters are listed in Table 1), the samples, whose materials and process parameters were adapted to movable connections, show an improved rotateability. All studied samples, which have been sintered at a maximum temperature of 1450°C, were movable. Observations with scanning electron microscopy show a clearly visible gap between the alumina axle and the zirconia gear wheel (Fig. 3). However the movability can not be maintained when sintering at a maximum temperature of 1500°C. Probably diffusion processes and an increased solubility of Al_2O_3 in ZrO_2 [11] might be a reason for joining of the two components when sintering at a higher temperature. Nonetheless an interruption of the sintering process at T_{max}=1300°C, followed by rotation movements of the presintered samples and a stepwise continuation of the sintering process is no longer necessary.

Progress was also made with respect to the production of fixed connections. A material combination of the alumina powder CT3000SG (d_{50} = 0.7 µm; specific surface area = 7.3 m^2/g) from Almatis GmbH (Ludwigshafen, Germany) and the zirconia powder TZ-3YS-E (d_{50} = 0.6 µm; specific surface area = 6.6 m^2/g) from Tosoh Corp. (Tokyo, Japan) for the production of shaft-to-collar connections emerged to yield cracks in some of the examined samples. It is assumed that the different degrees and kinetics of shrinkage are responsible for these cracks since these factors cause stress in the components.

Step	Temperature	Heating rate	Duration	Remarks
Heating up	RT → 180°C	0.5K/min		
Dwell	180 °C		1 h	
Heating up	180 °C → 250°C	0.5K/min		
Dwell	250 °C		1 h	
Heating up	250 °C → 500°C	2K/min		
Dwell	500 °C		2 h	
Cooling down	500 °C → RT	-5K/min		(1)
Heating up	RT → T_{max}	5K/min		(1)
Dwell	T_{max}		1 h	
Cooling down	T_{max} → RT	-5K/min		

RT: Room temperature; T_{max}: maximum temperature (1450°C/1500°C); (1): Beside cooling down to RT after debinding a transition to the sintering process can be conducted as well after dwelling at the last step of the debinding procedure.

Table 1 Debinding and sintering parameters for movable shaft-to-collar connections

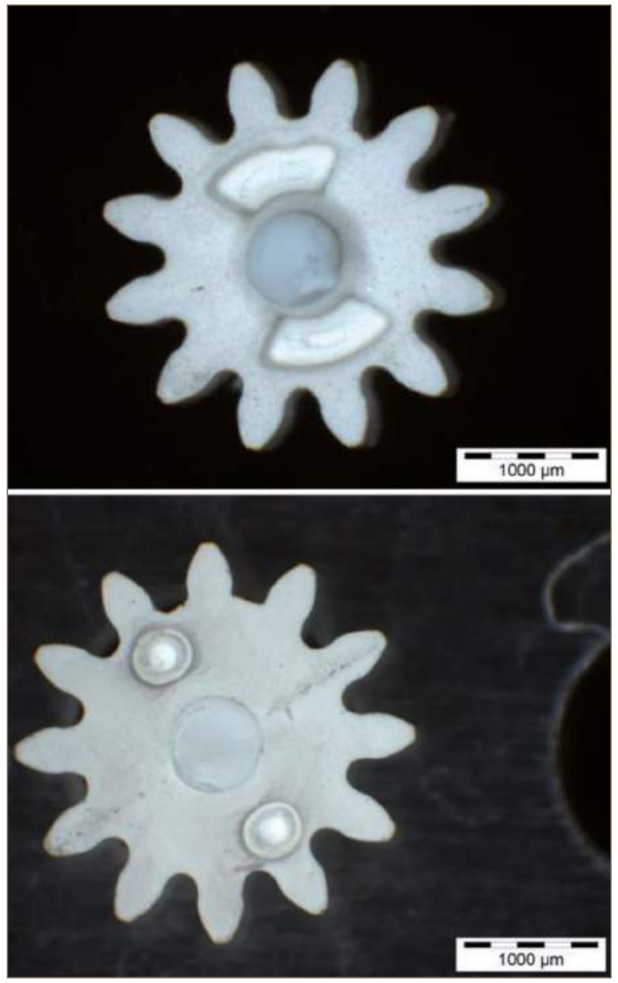

Fig. 2 Green bodies of shaft-to-collar connections. Top: Former design with C-arc shaped gatings. Bottom: Actual design with point-shaped gatings. The upper part of the axle was broken away in both cases to enable a view at the gatings of the gear wheel

Step	Temperature	Heating rate	Duration	Remarks
Heating up	RT → 180°C	0.5K/min		
Dwell	180 °C		1 h	
Heating up	180°C →250°C	0.5K/min		
Dwell	250°C		1 h	
Heating up	250°C → 500°C	2K/min		
Dwell	500°C		2 h	
Cooling down	500°C → RT	-5K/min		(2)
Heating up	RT → 1300°C	5K/min		(2)
Heating up	1300°C → 1550°C	2.5K/min		
Dwell	1550°C		2 h	
Cooling down	1550°C → RT	-5K/min		

RT: Room temperature; (2): Beside cooling down to RT after debinding a transition to the sintering process can be conducted as well after dwelling at the last step of the debinding procedure.

Table 2 Debinding and sintering parameters for fixed shaft-to-collar connections

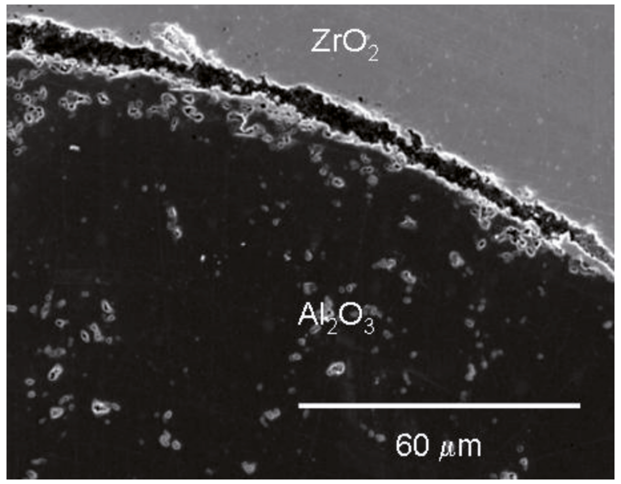

Fig. 3 Scanning electron microscopy image of the interface of a sintered shaft-to-collar connection (movable connection)

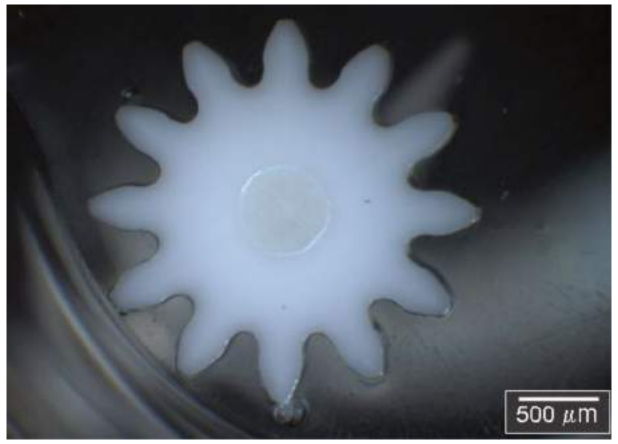

Fig. 4 Stereo microscope image of a cross section of a sintered shaft-to-collar connection (fixed connection)

A better adaptation of the two components was therefore required. Instead of the two mentioned ceramic powders the combination of the alumina powder CT3000SG from Almatis GmbH with zirconia powder PYT05.0-005H (d_{50} = 1.1 µm; specific surface area = 4.9 m^2/g) from Unitec Ceramics Ltd (at that time located in Stafford, England) was used for a new series of shaft-to-collar connections. The samples were debinded and sintered as listed in Table 2. Cross sections of the samples were prepared after sintering and studied using light and scanning electron microscopy. The investigated samples did not show any signs of cracks (Fig. 4). The new combination is therefore more suitable for the realisation of fixed shaft-to-collar connections.

Conclusions and outlook

The described investigations showed that with the help of a modified injection moulding tool with an improved gating system the producibility of movable shaft-to-collar connections can be improved significantly. However there are still processes, which affect the rotateability of a gear wheel in contact with an alumina axle negatively at higher temperatures ($T \geq 1500°C$). These processes might be diffusion processes and/or the formation of solid solutions in the system Al_2O_3-ZrO_2.

Additionally improvements could be obtained for the production of fixed connections. With the help of adaptation of the ceramic powders, i.e. the selection of other powders, the quality of the micro part was improved in a way that cracks did not occur.

However, the 2C-MicroPIM process as a production method for shaft-to-collar connections has not reached its optimum so far and further improvements are possible, but also essential if it should be suitable for industrial production. The quality of the axle after sintering is not satisfying so far, due to high porosity and the occurrence of cavities. This topic is in particular 2C-MicroPIM specific, as both components have to be adapted to each other and therefore the degrees of freedom, e.g. powder loadings or sintering temperature, are limited. Future works will focus on feedstock improvements in order to reduce or avoid these problems. Other fields of interest that should be attended are size accuracy and the behaviour of the components at higher temperatures in the system Al_2O_3-ZrO_2(Y_2O_3). It has to be proved if diffusion processes and/or phase reactions occur at the interface and if these processes are relevant for 2C-MicroPIM and especially for the relatively short period of sintering.

Acknowledgements

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