

# POWDER METALLURGY REVIEW

VOL. 3 NO. 2  
SUMMER 2014



**METAL ADDITIVE MANUFACTURING**  
**PM TITANIUM CONFERENCE REPORT**  
**NON-FERROUS POWDER PRODUCTION**



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## Submitting news and articles

We welcome contributions from both industry and academia and are always interested to hear about company news, innovative applications for PM, technology developments, research and more.

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# POWDER METALLURGY REVIEW

## Opportunities in the Additive Manufacturing revolution

It is impossible to escape the intense interest in Additive Manufacturing (AM), whether from the mainstream media or key end-user industries such as the aerospace and medical sectors. What is certain, however, is that whether you see Additive Manufacturing as a threat to conventional metal powder technologies or as an opportunity, Additive Manufacturing is here to stay. Our review of metal Additive Manufacturing processes and applications (page 41) offers an insight into the development of the technology to-date. Case studies illustrate just how revolutionary the freedom to "design for function" rather than "design for manufacture" will be on component development in the future.

From an end-user perspective, Additive Manufacturing is particularly attractive for the processing of advanced materials such as titanium, where conventional processes can be prohibitively expensive. This is of course music to the ears of titanium powder suppliers who finally are presented with a dynamic market for their products, a contrast to the relatively slow growth in conventional titanium PM and MIM. Our review of the second International Titanium Powder Processing, Consolidation and Metallurgy Conference, which took place in New Zealand, December 2-4 2013, includes the developments in both titanium PM and AM processing (page 53).

In this issue we also publish an extensive review of non-ferrous powder production technologies, including copper, aluminium, titanium and nickel powders (page 65).

With the PM2014 World Congress, Orlando, May 18-22, almost upon us, we warmly welcome our readers to visit us on booth 222. We look forward to seeing you in Florida!

Paul Whittaker  
Editor, *Powder Metallurgy Review*

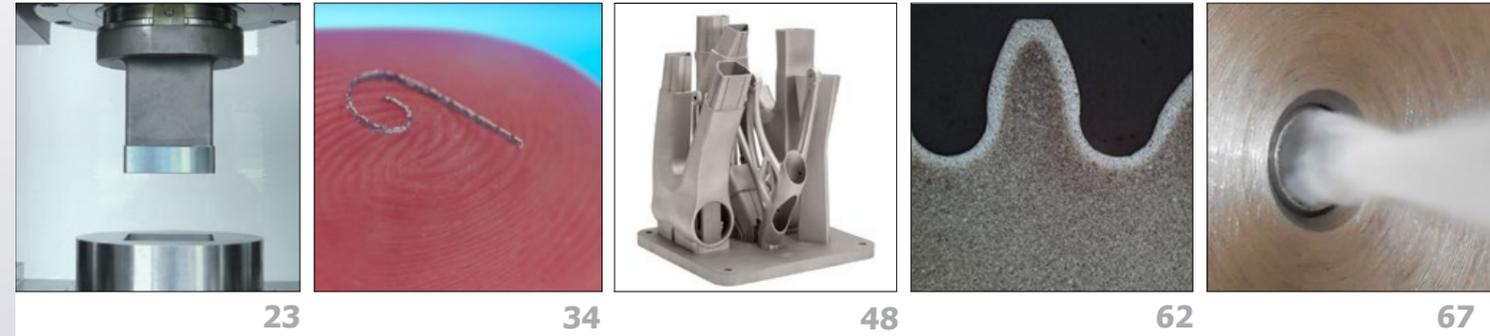


## Cover image

*Additively manufactured bracket produced by Airbus for the A350 XWB aircraft (Image ©Airbus S.A.S 2014)*



Choose the best in sintering



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## Critical Process Parameters in MIM Sintering Furnaces

The accuracy of dimensional tolerances of sintered MIM parts is limited, especially in the case of larger geometries. One reason is green density fluctuations introduced during high-pressure injection molding leads to inhomogeneous shrinkage behavior at elevated sintering temperatures. Another reason is large temperature gradients in the furnace hot zone cause geometrical distortions, even in part areas with rather homogenous density distributions.

By examining different factors for determining unwanted temperature gradients in MIM vacuum furnaces, we are able to evaluate the process parameters and present possible improvements by utilizing tight control and accurate design of heating elements and hot zones, as well as advanced process gas management systems, for commercial use in large series production environments.

Find out about critical process parameters for MIM sintering heat treatment furnaces, testing results and more by visiting the link below.



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*\*Ipsen also offers debinding and sintering furnaces in a variety of larger sizes.*

Visit [IpsenUSA.com/Critical-Process-Parameters](http://IpsenUSA.com/Critical-Process-Parameters) to view the full technical presentation.



## in this issue

### 41 Additive Manufacturing with metal powders: Design for Manufacture evolves into Design for Function

Additive Manufacturing (AM) offers the possibility to produce complex parts without the design constraints of traditional manufacturing routes. No longer solely a prototyping technology, AM is now being used for the production of series components for the most demanding applications. Juan F Isaza P and Claus Aumund-Kopp review a technology that is set to revolutionise the future use of metal powders.

### 53 Titanium PM 2013 Conference: The impact of impurities, advances in porous titanium and titanium Additive Manufacturing

The second International Titanium Powder Processing, Consolidation and Metallurgy Conference took place in Hamilton, New Zealand, from December 2-4 2013. Prof Ma Qian reports on selected highlights from the conference

### 61 Vacuum sintering and Low Pressure Carburising of PM components

France's ECM Technologies reports on an innovative new combined batch vacuum sintering and Low Pressure Carburising furnace that enables the complete thermal treatment of PM components in one step.

### 65 Non-ferrous powder production: Manufacturing methods and properties of copper, aluminium, titanium and nickel powders

This extensive review of key non-ferrous powders used in the Powder Metallurgy industry focuses on the production and properties of copper, aluminium, titanium and nickel powder. Professor Oleg Neikov identifies the methods available for manufacturing these powders including detailed descriptions of the atomisation process, mechanical grinding, and electrochemical methods. Key powder properties are also listed.

## regular features

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# industry news

To submit news for inclusion in *Powder Metallurgy Review* contact Paul Whittaker [paul@inovar-communications.com](mailto:paul@inovar-communications.com)

## GRIPM acquires majority stake in Makin Metal Powders

GRIPM Advanced Materials Co., Ltd, a producer of electrolytic copper powder based in Beijing, China, has acquired a majority stake in Makin Metal Powders (UK) Ltd, Rochdale, UK. The combined group will have production capacity of over 21,000 tonnes per annum providing a wide range of copper and other non-ferrous powders.

GRIPM is the largest supplier of electrolytic powders in China and plans to further develop its range of high quality dendritic, atomised and speciality products. Makin will continue to operate independently from its Rochdale facility whilst also working with GRIPM to expand its range of products and technical support offered.

"GRIPM is an ideal partner for Makin as they offer complementary products which will allow the combined

companies to build a truly global supplier to the Powder Metal industry," stated Dr John Boden, Managing Director of Makin Metal Powders. Boden will continue to lead the Board of Directors, supported by the existing team of senior managers and workforce.

GRIPM's parent company GRINM (General Research Institute for Nonferrous Metals) is the largest research institute in the non-ferrous metals industry in China, with a turnover of over \$1 billion.

"GRIPM have long admired Makin's expertise, products and professionalism and the two companies will work closely together to meet the growing market demands for copper and copper alloy powders. It is our intention for GRIPM and Makin to grow to become one of the best and strongest suppliers to the Metal Powder industry by utilising the extensive R&D and technical capabilities of our parent company GRINM," stated Dr Limin Wang.

[www.makin-metals.com](http://www.makin-metals.com)

## MG miniGears sold to German investment fund Finatem

An agreement for the sale of the assets of MG Mini Gears S.p.A of Padua, Italy and Suzhou, China, has been signed between parent company Carraro Group and the German investment fund Finatem. The total value of the transaction is reported as approximately €28 million and is scheduled for completion by May 31, 2014.

As a result of this transaction the two production companies, specialising in the production of PM gears, will cooperate jointly with Herzog GmbH, a company focused on components and gears based in southern Germany and already controlled by Finatem.

"This is an important step for the Group towards a further focus on our core business, which in these years registered very positive results both in terms of volumes and margins and that shows attractive growth perspectives already in the medium term," stated Enrico Carraro, Chairman of miniGears' parent company Carraro Group.

With its head office in Padua, and an operational plant in Suzhou, miniGears is specialised in the production of small Powder Metallurgy gears for a range of different sectors from automotive to professional gardening and power tools. The two plants employ a total of 526 people (280 in Padua) and reported a total turnover of €69.8 million in 2013.

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



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## GKN reports increased profits

GKN plc has reported a 20% increase in the group's operating profit to £661 million in 2013, up from £553 million in 2012. Pretax profit was stated to be £578 million, up 17%, with sales across the group up 10% on the previous year at £7,594 million.

"We have again made good progress in-line with our strategy to grow a market-leading global engineering business. Although some of our end markets were challenging, we continued to show growth and are reporting good underlying financial results, helped by our 2012 acquisition of GKN Aerospace Engine Systems, which performed strongly. We expect the Group's progress to continue in 2014," stated Nigel Stein, Chief Executive, GKN plc.

The GKN Powder Metallurgy division, comprising GKN Sinter Metals and Hoeganaes, reported that sales increased 7% to £932 million, up from

£874 million in 2012. Strong growth was reported in North America, Europe and China but with a more modest improvement in India, where vehicle markets remained volatile. Sales in Brazil fell due to weaker industrial markets. Trading profit for GKN Powder Metallurgy increased £7 million to £94 million in 2013.

GKN stated that during the year the Powder Metallurgy division continued its strong product development, particularly with advanced products and powders, and was awarded £144 million of annualised sales in new business. Notable milestones reported by GKN included the opening of a new sintering plant in Yizheng, China. Also reported were new products such as helical pulley gears for electric power steering system for ZF Group; pump components for a global double clutch transmission (DCT) programme for Getrag, high strength gear ring components for Start Stop systems for Valeo; and components for a number of variable valve timing (VVT) systems.

[www.gkn.com](http://www.gkn.com) ●●●

## PM production in China up in 2013

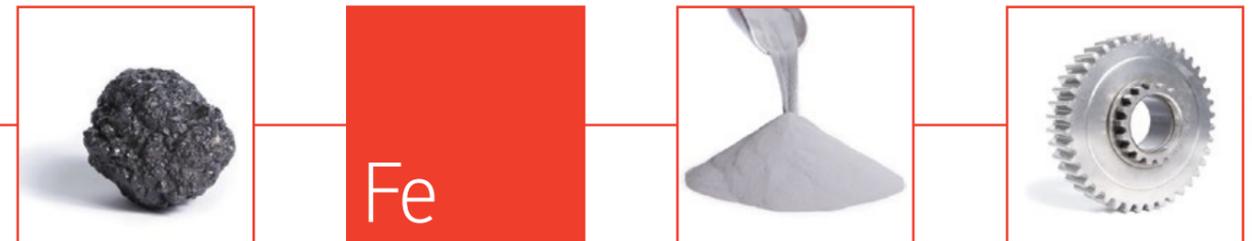
According to figures issued by the China Machine PM Association (CMPMA), production of structural PM parts in 2013 reached 180,242 metric tonnes, an increase of 11.9% over 2012. The CMPMA report states that these figures are based on 51 responding PM structural part producers.

In terms of sales value the CMPMA reported an increase of 8.9% for 2013 to Yuan 590,448 million (\$951 million) of which Yuan 79,641 million (\$128 million) represented new PM products, an increase of 9.8%.

Exports of PM structural parts decreased in 2013 by 2.9% to Yuan 60,617 million (\$97.6 million). Total profits after taxes soared by 40.4% to Yuan 37,684 million (\$60.7 million). The 51 reporting PM companies employed an average of 15,804 people over the year, representing a slight decrease (0.9%) over the previous year. ●●●

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 Email: [minyu@TDMfginc.com](mailto:minyu@TDMfginc.com)





## Cremer acquires majority share of SOF Equipment GmbH

Cremer Thermoprozessanlagen GmbH, based in Düren, Germany, has announced the acquisition of the majority of shares in SOF Equipment GmbH. Based in Eschweiler, Germany, SOF manufactures a range of furnaces for various industries including Powder Metallurgy (PM) applications.

Cremer has been a manufacturer of industrial furnaces with a focus on the Powder Metallurgy (PM) and Metal Injection Moulding (MIM) technology sectors for almost 50 years. The company produces a range of furnaces including walking beam, belt, pusher, multi-tube, drum type, batch type and rotary-hearth systems.

"The acquisition of SOF by Cremer strengthens the market position and will provide an extended product range, more powerful technology and innovation support, plus an optimum service from a single source," stated Dipl.-Ing. Ingo Cremer.

Following the transaction, SOF Equipment GmbH has been renamed as Cremer-SOF Engineering GmbH and Ingo Cremer has been appointed General Manager (CEO). [www.cremer-ofenbau.de](http://www.cremer-ofenbau.de) ●●●



**Metal Powders**  
[www.qmp-powders.com](http://www.qmp-powders.com)



## Miba triples the size of its site in China with €35 million expansion

Miba has recently celebrated the official opening of the second phase of development at its site in Suzhou Industrial Park, 90 km west of Shanghai, China. The expansion sees a tripling of the plant's production area and represents a total investment of around €35 million.

The company sees potential for major growth opportunities in China, primarily in the passenger vehicle, truck and ship markets. The site expansion will allow local production of engine bearings, sintered Powder Metallurgy components and innovative coatings. The Friction division will also begin production of friction materials at the Suzhou site in the near future.

Miba Precision Components (China) Co. Ltd. (MPCC) was founded in October 2005, with completion of the first phase of development in 2007 providing an 8,000 m<sup>2</sup> facility. The second phase expansion project began in 2012 and has now added a further 16,000 m<sup>2</sup>, bringing the site's total area to 24,000 m<sup>2</sup>.

Around 400 employees already work at the Suzhou site. The newly constructed facility will employ a total of 1000 when at capacity. In the last fiscal year, 6% of Miba's consolidated revenue was generated by the MPCC site in Suzhou.

[www.miba.com](http://www.miba.com) ●●●

## Expansion at Hart Materials

Hart Materials Limited, a UK based supplier of metallic and composite small particulate materials, has announced it is relocating to larger premises.

"Basically we have outgrown our existing office space and needed our own integral warehouse due to the expansion of our activities," stated Dr Tony Hart, Chairman of Hart Materials. "Consequently we decided to purchase premises that became available about a mile from our existing offices in Wombourne, South Staffordshire. Our intention is to broaden the activities of the company in specific areas."

For many years Hart Materials has specialised in nickel-based materials including nickel powders and composite particles such as nickel-coated graphite. This focus has, however, led the company into application areas with demands for totally different particulate materials.

The expansion will also allow Hart Materials to modify the properties of a number of existing commercially available particulate materials in-house, stated the company.

[www.hartmaterials.com](http://www.hartmaterials.com) ●●●

## Timcal announces change of name to Imerys Graphite & Carbon

Timcal Graphite & Carbon, headquartered in Bodio, Switzerland, has announced the company is changing its name to Imerys Graphite & Carbon, reflecting the group's parent company branding.

Timcal produces and markets a large variety of synthetic and natural graphite powders, conductive carbon blacks and water-based dispersions. The group lists seven manufacturing plants across the globe and employs some 450 people.

"This step to a common brand will increase the visibility of Imerys and will also improve the co-operation and communication with our customers and suppliers," stated the company.

In 1994 Timcal, at the time called Lonza G+T, was taken over by the French group Imetal. In 1999 Imetal changed its name to Imerys and the parent group now brands itself a world leader in mineral-based speciality solutions for industry.

[www.imerys-graphite-and-carbon.com](http://www.imerys-graphite-and-carbon.com) ●●●

## Asbury Graphite on course for European expansion

Asbury Carbons, a US based supplier of graphite and carbon related products, is expanding operations into Europe with the construction of a new production facility located in Maastricht, Netherlands. The new site is scheduled to begin full production in August this year.

The new facility will utilise the same base materials as the company's North American site in order to produce PM industry standard grades of graphite such as 1651, 3203, PM5, PM9, HPPM5 and HPPM9. The facility aims to obtain ISO9001 registration.

Nicholas T Mares, VP of Marketing at Asbury Carbons told Powder Metallurgy Review, "the new facility at our site in Maastricht will offer the Powder Metallurgy industry in Europe greater choice and quicker lead times for all our standard PM grade graphite."

The location of the new facility in Maastricht was carefully selected from over 30 possible sites. "Maastricht provides an ideal location for us, offering a good supply and distribution network. We are closer to our customers in Europe and can offer potential customers access to our comprehensive range of products," added Mares.

[www.asbury.com](http://www.asbury.com) ●●●



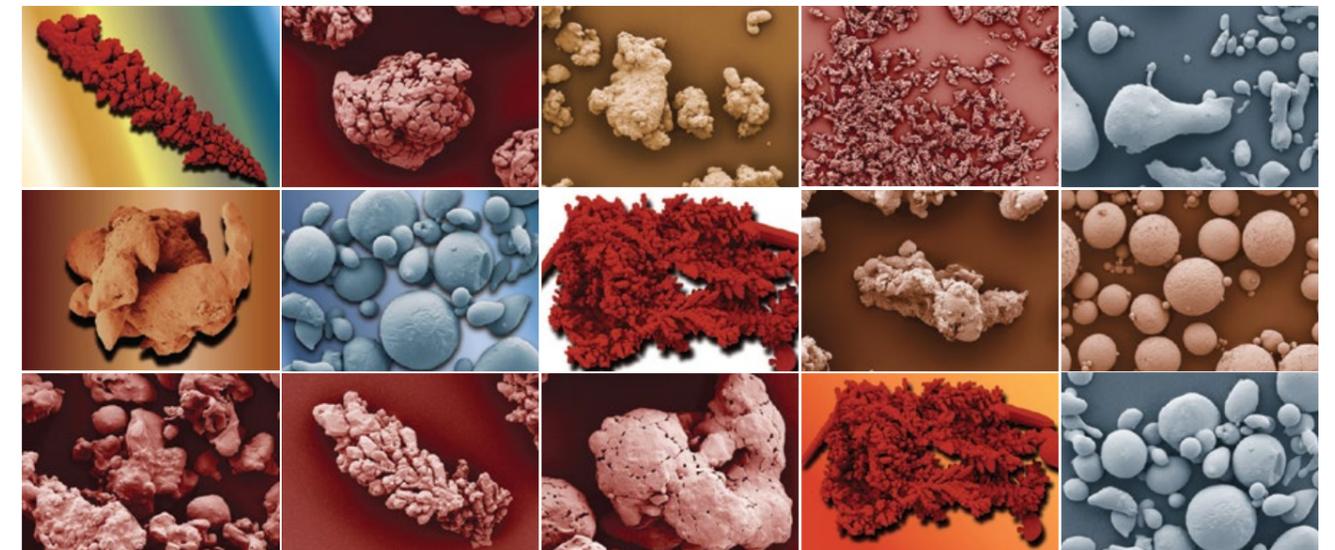
The new Asbury facility in Maastricht will begin production in August 2014



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Makin Metal Powders (UK) Ltd has achieved its current position as one of the leading Copper and Copper Alloy powder producers in Europe by supplying the powders that match customer technical specifications in the most cost effective manner on a consistent basis, batch after batch.

Makin Metal Powders (UK) Ltd

## Nickel price soars amid export ban and sanctions fear

The price of nickel, a key alloying element in low alloy steels, stainless, high alloy steels and non-ferrous alloys, broke the US\$17,000/t barrier in early April, a rise of 16% since the start of 2014. The strong performance of the nickel price is forecast to continue as a result of reduced supply of nickel ore following an Indonesian ban on exports. Until this ban, Indonesia was the world's top high-grade nickel ore supplier.

According to a report from Standard Bank quoted in the *Financial Times* on April 10th, if the export ban remains in place beyond July's presidential election in Indonesia, the global nickel market will see significant deficits of 134,000 tonnes in 2015, 106,000 tonnes in 2016 and 77,000 tonnes in 2017. "In the absence of a change in Indonesian policy, we think that by 2016, the market will get tighter than in 2006-7, when prices

traded in the \$30,000-\$50,000 a tonne range," the bank stated.

Potential sanctions by the EU and the US against Russia could impact on the supplies of nickel from Russia's Norilsk Nickel, a producer that accounts for 17% of the global nickel production. According to CEO of Norilsk Nickel, Vladimir Potanin, the company is considering measures to shield against possible sanctions from the US and EU by looking to Asia. "We have large volume of operations in the Chinese market, but the main payment currencies are dollar and euro. In principle, nobody hinders settlements in such currencies as the yuan for deliveries to China. We decided to explore this issue, to look how it'll function," Potanin told *Russia Today*.

Potanin stated, however, that he does not believe there will be tough sanctions as "they are unnecessary, uninteresting and harmful to both parties. But in a case of specific emotional actions of regulators or of certain countries - just in case - it is necessary to study what we will do in this situation."

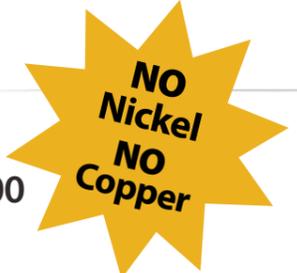
Following previous nickel supply crises and price hikes, materials suppliers to the PM industry developed a number of low nickel containing steel powder materials that used alternative alloying elements such as manganese. Traditional PM steel compositions can utilise relatively high levels of molybdenum, nickel and copper to achieve medium to high mechanical properties in the as-sintered condition, however these alloys are sensitive to price instability.

Whilst low nickel containing powders and also copper-containing materials cannot be used as a direct substitute for alloys with higher nickel contents that are currently being used because of dimensional change and mechanical property requirements, their availability offers the PM industry a strategic opportunity to, for selected PM steel materials, move away from a dependency on nickel. The latest nickel supply and price issues could, therefore, again accelerate the trend towards leaner, more cost effective alloys, some being completely free of nickel. ●●●

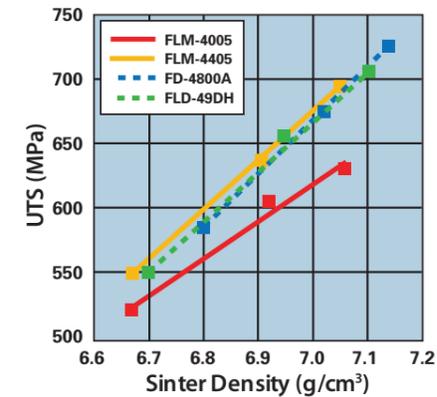


# AncorTECH™ PM Manganese Steels

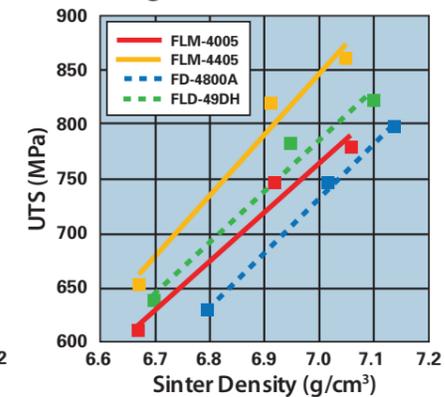
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## GKN Sinter Metals receives automotive supplier awards

### Schaeffler and Continental 'Premium Supplier' award

GKN Sinter Metals has been named as a Premium Supplier by Schaeffler and Continental, who have been cooperating in their purchasing since the beginning of 2009.

The businesses, which supply automotive manufacturers and the industrial sector, recently held their second joint Premium Supplier Day. Only 31 out of more than 1,200 strategic suppliers met the criteria for the highest recognition. These companies have qualified as Premium Suppliers due to their outstanding capabilities to offer innovations, achieve outstanding Quality and Delivery performance and to ensure excellent competitiveness on a global basis.

The recognition was handed out by Schaeffler to representatives of the GKN Sinter Metals management team in Bruneck, Italy, in March.

Peter Mölgg, President European & Asia Pacific Operations, who received the nomination, stated, "We are honoured to be part of this prestigious group of Premium Suppliers and recognise above all, the confidence of our valued customers Schaeffler and Continental in the high quality of our services and prod-



Representatives of the GKN Sinter Metals management team in Bruneck, Italy receive the award

ucts. We would like to thank every single GKN Sinter Metals employee involved for their commitment and passion."

GKN Sinter Metals supplies numerous Schaeffler and Continental locations worldwide, including Brazil, China, Germany, Italy, India and the US, with a wide variety of automotive parts made from conventional PM and MIM processes.

### Nexteer 'Perfect Quality' award

Nexteer, one of the world's top five global suppliers of steering systems, has jointly awarded GKN Sinter Metals plants in Salem, USA, and Bonn, Germany with the company's 'Perfect Quality 2013' award. The award identifies companies setting the standard of exceptional quality performance among Nexteer's supplier community.

"We are delighted to receive this award from our valued customer Nexteer, and by the outstanding performance and passion for quality that our plants are providing to our



James McConnel, Advanced Supplier Quality Engineer, Nexteer, hands over the award to the Salem plant team

customers," stated Stuart Greidanus, GKN's Senior Vice President Sales & Marketing Sinter Metals.

"This award recognises what can be achieved through collaboration with our customers, a focus on quality and global operational excellence. These attributes are fundamental to our success," added Greidanus.

Nexteer Automotive has global headquarters in Saginaw, Michigan, USA, and operates twenty manufacturing plants around the world.

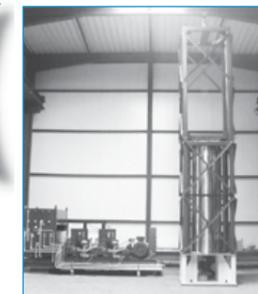
GKN's Salem plant has been supplying power steering rotors to Nexteer's USA facility in Saginaw Michigan for more than a decade. In addition, three more GKN Sinter Metals plants in the USA deliver pump rings, EPS pulleys and steering column components to Nexteer in Mexico, India and China.

GKN in Bonn supplies the Nexteer facility in Tychy, Poland, one of its long-term customers, with steering adjustment components.

www.gknsintermetals.com ●●●



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## Metaldyne recognised by GM as a 2013 Supplier of the Year winner

Metaldyne, LLC headquartered in Plymouth, Michigan, USA, has reported that it was presented with a GM Supplier of the Year award at the auto maker's recent awards ceremony held on March 12 in Detroit.

GM's Supplier of the Year awards were presented to 68 suppliers worldwide, less than 1% of GM's supply base, who have consistently exceeded GM's expectations and created outstanding value. This is the first time Metaldyne has received this award from GM.

"Our suppliers play a huge role in helping GM deliver compelling vehicles to our customers," stated Grace Lieblein, GM Vice President, Global Purchasing and Supply Chain. "Supplier of the Year winners have outstanding track records for consistently meeting our business needs, while also supporting our cultural priorities."

The award was accepted on behalf of Metaldyne and its employees by

Thomas Amato, President and CEO, Metaldyne LLC, and Stephanie Jett, Metaldyne's Vice President of Sales – Vibration Control Systems.

"Being recognised a General Motors' Supplier of the Year is an exciting acknowledgement for all Metaldyne employees," stated Amato. "All of us at Metaldyne strive to provide General Motors and all our customers unrivalled support and leading technology, and we will continue delivering the best possible products and services to our customers well into the future."

Metaldyne is a global manufacturer of highly engineered metal-based components for engine, transmission, and driveline applications in the automotive and light truck markets. Products include Powder Metallurgy engine connecting rods and engine bearing caps amongst others. Metaldyne has over \$1 billion in annual revenue and lists 26 locations in 13 countries.

www.metaldyne.com ●●●

## Miba plans further US site expansion

It has been reported that Miba plans to expand its site in McConnelsville, Morgan County, Ohio, USA. According to the *Marietta Times*, the company is in the process of expanding its Miba Sinter facility, creating 70 new jobs by 2015. Miba will also consolidate its North American back office support staff to a new service centre to be built in McConnelsville.

The expansion of the Miba Sinter facility will add around 3700 m<sup>2</sup> (40,000 square feet) of manufacturing space.

The site in McConnelsville includes Miba Sinter and Miba Bearing divisions. "In 2013, the McConnelsville facility generated \$27 million in sales. The building expansion is expected to bring an additional \$50 million in sales over the next two to three years," stated Shannon Wells, director of the Community and Business Development Office of Morgan County.

www.miba.com ●●●



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## HC Starck looks to become world's largest independent tungsten provider

In a recent statement from HC Starck the company has announced that one of its aims for 2014 will be to noticeably expand its market position in the tungsten segment, becoming the world's largest independent tungsten provider. HC Starck stated its plans to commission two major tungsten joint ventures, one in China and one in Vietnam, were under way.

"Thanks to our joint ventures, we are in an outstanding position to leverage Asia's market potential and use the demand in the tungsten segment for our growth," stated Andreas Meier, CEO of H.C. Starck. "Together with our existing tungsten powder production activities in Germany and Canada, this will make us the world's largest independent tungsten producer."

HC Starck stated that the products from the two joint ventures will be used as high-performance materials in many growth industries,

particularly tool construction, medical technology, the automotive and aviation industry, as well as in the energy sector; in wear parts for the mining, tunnel and road construction sectors; as metal for alloys, and in catalyst production for the chemicals industry.

In financial results published by HC Starck it reported that after two years of strong growth, the company posted group sales of €703.9 million in 2013, below the previous year's figure of €862.9 million. The report stated that the tungsten metal powder business was affected by a sharp decline in the European market and decreases in raw material prices, especially in the first half of the 2013. Toward the end of the year, however, this segment experienced a noticeable recovery that brought a positive start to fiscal 2014.

[www.hcstarck.com](http://www.hcstarck.com) ●●●

## Sinterite renews its ISO accreditation

Sinterite, a Gasbarre Furnace Group Company based in Pennsylvania, USA, has announced the renewal of its ISO/IEC 17025:2005 accreditation. The accreditation allows the furnace maker to perform certified calibrations for furnace users, a process required by many of the Group's customers.

The ISO 17025 accreditation is similar to the 9001 Standard but is more specific in requirements for competence. This is the primary ISO Standard used by testing and calibration laboratories. Laboratories use this standard to implement a quality system aimed at improving their ability to consistently produce valid results.

With this accreditation, Sinterite joins Gasbarre Press Division, Major Gauge & Tool and McKee Carbide Tool as Gasbarre companies with ISO Accreditation.

[www.sinterite.com](http://www.sinterite.com) ●●●

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## Global tungsten prices show signs of stabilising

Prices for tungsten, the key material used for producing cemented carbide tools, heavy metals, and other powder metallurgy products have stabilised in recent months.

According to a report in the *Nikkei Asian Review* the price of ammonium paratungstate (APT), the intermediate material of tungsten, is currently priced at around \$372-\$382 per 10 kg.

Prices declined by about 10% from peaks at the end of August last year largely because of producers rushing to unload inventory before year-end. The fall, however, has been slowing down since the end of last year as can be seen in the Fig. 1.

The price of tungsten carbide powder (3-4 micron) was also reported stable at \$46/kg (*Metal Pages*, March 2014) compared with



Fig. 1 Tungsten prices shown in dollars per 10 kg (Courtesy *Nikkei Asian Review*)

\$53/kg in August 2013.

However, as Fig. 1 shows the lowest price for tungsten in 2013 remains almost double those seen five years ago. Nevertheless, unlike some other critical and rare metals, the price of APT is relatively stable. The price of neodymium, a rare earth metal used for making high-performance magnets, has for example collapsed recently after increasing by a factor of ten over the past five years. ●●●

## GTP tungsten confirmed conflict free

Following an audit conducted in December 2013, the audit committee of the Electronic Industry Citizenship Coalition (EICC) and the Global e-Sustainability Initiative (GeSI) has confirmed that Global Tungsten & Powders (GTP), a division of the Plansee Group, does not process any tungsten sourced from conflict regions. GTP is the world's first tungsten producer to receive this certificate.

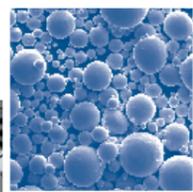
Tungsten Ore Sustainability is a fixed purchasing policy commitment throughout the Plansee Group. Global Tungsten & Powders ensures that it does not use any raw materials from socially, ethically or ecologically questionable sources.

GTP is the Plansee Group's main tungsten supplier.

[www.plansee.com](http://www.plansee.com) ●●●

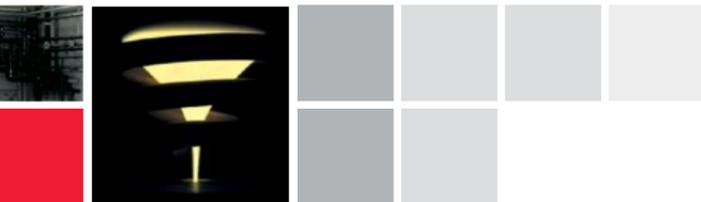
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[www.powdermetalpresses.com](http://www.powdermetalpresses.com)



## Plansee opens new office in Singapore

Materials specialist Plansee has announced the opening of a new office in Singapore to serve the Southeast Asia region.

The office will be headed by Isao Nishida who has worked for the Plansee Group for some 13 years. Nishida is an experienced specialist for molybdenum, tungsten, tantalum and niobium and all their applications.

The high-temperature strength, corrosion resistance, high density and low coefficient of thermal expansion mean that these metals have become an indispensable part of many industries, stated Plansee.

The Plansee Group is headquartered in Austria and has production plants and sales offices in more than 20 different countries.

www.plansee.com ●●●

## PyroGenesis to supply \$15 million metal powder production system to Additive Manufacturer

PyroGenesis, a developer and manufacturer of plasma waste-to-energy systems and plasma torch systems based in Montreal, Canada, has announced that it has signed a letter of intent (LOI) to supply a major international company with several plasma-based metal powder production systems for use in Additive Manufacturing. At this stage the customer's name is being withheld.

"One of the limiting factors in the full commercialisation of 3D printing for metal products is the availability of high-quality, high-purity metal powder," stated P Peter Pascali, President and CEO of PyroGenesis. "Our platform is a proven product with completed commercial sales having already taken place in North America and Europe. A successful conclusion of this LOI will result in the rapid deployment of our powder

production platform for 3D printing, thus accelerating the adoption of Additive Manufacturing world-wide."

"Our customer is looking to PyroGenesis and its plasma-based technology to ensure a strategic and continuous supply of metal powder feedstock for their own internal 3D printing production use," added Gillian Holcroft, Executive Vice President, Strategic Alliances.

"This is only at the LOI stage," cautions Pascali, "and one should consider this news in that light. However, we have made sufficient progress to be highly confident that a contract will be concluded in short order; which once again underscores the success PyroGenesis is having in deploying its core plasma technology to niche high-margin applications and in multiple units."

www.PyroGenesis.com ●●●

## Erowa workholding system offers quick changeover of PM tooling

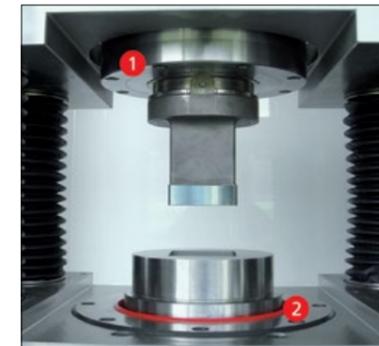


Fig. 1 Erowa PM Tooling: Press adapter with PM Tooling punch chuck (1) and die-plate chuck (2)

Powder Metallurgy (PM) parts are becoming increasingly sophisticated and the variety of product shapes is on the rise, states Swiss tooling system manufacturer Erowa AG. The frequent changeover of tooling used in powder presses

can greatly reduce valuable production time.

A workholding system is the interface between the punch, die and press adapter. With the use of the Erowa PM Tooling rapid-change workholding system the changeover times for pressing tools are kept to a minimum, claims the company.

In Erowa's system a single or multi-level powder press is equipped with the appropriate PM Tooling chucks. There are die chucks and punch chucks (Fig. 1). The chucks are integrated directly into the powder press or press adapter.

The chucks are precisely aligned to each other with the help of a specially designed alignment set with no need for further adjustments. Punches and dies can be quickly exchanged and clamped with high precision. A 0.002 mm positioning accuracy guarantees an easy start after changeover, states Erowa.

New punches and dies to be manufactured are mounted on Erowa PM pallets and produced on the PM Tooling system. The PM Tooling system forms the basis on all machines and ensures consistency throughout the production process. From milling and grinding to EDM sinking and EDM wire-cutting to pressing, the 0.002 mm positioning accuracy is reliably observed. The precisely manufactured punches and dies are then clamped in the press adapter without further alignment.

Compatibility with other Erowa workholding systems allows for the complete automation of production machines. The reference positions are given by the precisely aligned PM Tooling chucks.

The benefits claimed by Erowa include a reduction in downtime, a significant increase in the precision of the whole production process and increased reliability. PM Tooling chucks are completely sealed against dirt and guarantee highly precise clamping of punches and dies.

www.erowa.com ●●●

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## New GE plant to assemble world's first passenger jet engine with additive manufactured fuel nozzles

GE Aviation has announced it will open a new \$100 million assembly plant in Indiana, USA, to build the world's first passenger jet engine with fuel nozzles produced by Additive Manufacturing (3D Printing) technology. The company stated that additively manufactured fuel nozzles will be used in its new LEAP engine, a joint venture between GE and France's Snecma, which will enter service in 2016 and already has 6000 confirmed orders valued at \$78 billion.

The new plant will be based in Lafayette, Indiana, and will employ around 200 people by 2020. GE will operate an advanced assembly line equipped with automated vision inspection systems, radio frequency parts management and other new technologies designed to improve production.

Each LEAP engine has 19 additively manufactured fuel nozzles which are five times more durable than the previous model. Additive Manufacturing allowed engineers to use a simpler design that reduced the number of brazes and welds from 25 to just five.

GE also reported that the engine incorporates a number of next-generation materials including heat-resistant ceramic matrix composites (CMCs) and break-through carbon fibre fan blades woven in all three dimensions at once. The CMC parts help with weight and heat management. They are two-thirds lighter than the metal equivalent and can operate at temperatures 20% higher than their metallic counterparts, at levels when most alloys grow soft.



Each LEAP engine will use 19 of these additively manufactured fuel nozzles

"When you start thinking about design, the weight savings multiplier effect is much more than three to one," stated Michael Kauffman, GE Aviation Manufacturing Executive. "Your nickel alloy turbine disc does not have to be so beefy to carry all those light blades, and you can slim down the bearings and other parts too because of a smaller centrifugal force. It's just basic physics."

The new technologies allowed the design team to cut the engine's weight by hundreds of pounds compared to the same size engine built using metal parts, increase the internal temperature and make it more efficient. "We are pushing ahead in materials technology, which gives us the ability to make jet engines lighter, run them hotter, and cool them less," Kauffman added. "As result, we can make the engines, and the planes they'll power, more efficient and cheaper to operate."

The LEAP engine has benefited from GE's \$1 billion annual investment in jet propulsion R&D. Scientists at GE Global Research have spent the last two decades developing some of the most advanced parts of the new engine, including CMCs, Additive Manufacturing methods and controls systems.

[www.ge.com](http://www.ge.com) ●●●



The LEAP engine mounted on a rig and undergoing testing

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## MPIF's Vanguard Award to be presented to Dowding

Robert J Dowding, Research Manager for Materials and Manufacturing Science, U.S. Army Research Laboratory (ARL), has been selected to receive the first ever Vanguard Award from the Metal Powder Industries Federation (MPIF).

The new award from the MPIF recognises Powder Metallurgy (PM) industry champions from the end-user community whose long-time promotion of the technology has contributed to the expansion of PM applications.

During Dowding's career at ARL much of his PM based R&D work has been related to material and process development for tungsten and tungsten alloys. He has personally performed research examining strain aging in tungsten heavy alloys (WHA), the processing of novel tungsten-based compositions, and the re-spheroidisation of tungsten grains

in heavily cold-worked WHAs.

Dowding has been a strong leader in, and supporter of, the US Army's Small Business Innovative Research (SBIR) program, his involvement dating back to the program's very first days in the mid-1980s. As the contracting officer's technical representative for more than 35 Phase I and Phase II contracts, his efforts have been leveraged to support multiple mission programs in the materials science and manufacturing of protection- and lethality-related applications. He has also advanced critical mission programs in PM, tungsten research, nanomaterials, armour ceramics, and Powder Injection Moulding (PIM). [www.mpif.org](http://www.mpif.org)



Robert J Dowding will receive the Vanguard Award

## 3D Systems launches seminars to promote AM technology

3D Systems, headquartered in Rock Hill, South Carolina, USA, has launched a global series of seminars to promote the latest 3D design-to-print technologies and solutions. The seminar series consists of over 300 events at the company's reseller locations throughout the world, from North America and Europe to the Middle East and Asia.

The seminars are designed to inform, educate, train and inspire local businesses, entrepreneurs, designers and other professionals on the 3D printing powered design-to-manufacturing technology available. The seminars include demonstrations from the company's 2014 product line spanning more than 20 new printers.

[www.3DSystems.com](http://www.3DSystems.com)

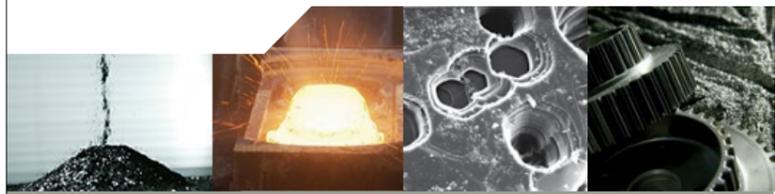
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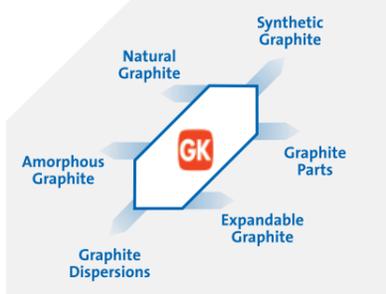
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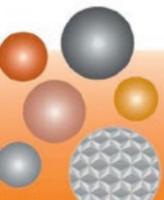


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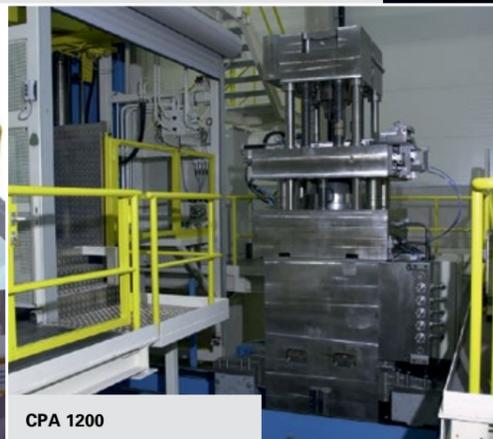
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## Abtex introduces Tri-Ten systems for deburring Powder Metallurgy parts

Abtex Corporation, based in Dresden, New York, USA, has designed its Tri-Ten deburring system for use in Powder Metallurgy applications. The system is a wet process U-Series system that deburrs both sides of PM parts in a single pass.

The compact Tri-Ten machine from Abtex is classed as a return to operator (RTO) system. Parts are loaded at one end and a magnetic conveyor transports them through an offset planetary head where three abrasive filament brushes deburr the upward facing surface.

The conveyor then carries the parts to a mechanism that flips them on to a parallel magnetized conveyor for transport back through the planetary head where the second side is deburred.

Parts are drenched with a special coolant as they are deburred. Parts are rinsed, demagnetized, and any



The Tri-Ten machine from Abtex

coolant residue is blown off before they exit the machine through an opening next to the entrance.

Abtex engineers have designed wet and dry Tri-Ten deburring systems in RTO and flow-through configuration to deburr green and sintered powder metal parts. Each system is customised for a specific application.

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

## New insert grade material from Sandvik Coromant

Sandvik Coromant has announced that it will be introducing new insert grades for steel turning and cast iron milling incorporating its Inveio branded technology. Following the introduction of insert grade GC4325 last year, the company will now offer grades GC4315 and GC4330.

Sandvik Coromant states that the performance of these grades is made possible by Inveio's uni-directional crystal orientation. Normally, the crystal orientation of the CVD alumina coating is random, however the company claims to have found a way to control the growth of the crystals, lining them up in the same direction to create a stronger, uniform structure of the coating.

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

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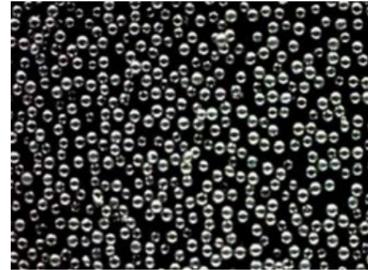
## NanoSteel introduces micro peening alloy with high hardness and durability for peening to precise specifications

The NanoSteel Company, a US based leader in nano-structured steel materials design, has announced the introduction of its proprietary EverShot™ ferrous micro peening alloy. The new material combines high hardness with a low breakage rate which, the company states, allows the shot peening of parts to precision specifications.

"Compared to both ceramic and ferrous microshot media, EverShot™ enables the shot peening of parts to tighter dimensional requirements at significantly improved uptimes," stated Harald Lemke, NanoSteel's Vice President and General Manager of Powder Metallurgy. "The media is ideal for the shot peening of small parts and parts with small radii or complex geometries such as gears, springs and threads."

In a customer test conducted by NanoSteel's development partner Superior Shot Peening in Houston, Texas, USA, the EverShot™ media with an average shot particle size of 83 microns (0.0033 inches) generated intensities equal to CW14 steel cut wire shot while providing a more uniform level of compression.

"NanoSteel's micro shot is extremely durable and generates the most uniform level of sub-surface compressive stresses that I've seen from any media other than ultra-sonic shot peening," stated Dan Spinner, Superior Shot Peening's Director of Technology. "The high hardness of the NanoSteel ferrous micro shot results in a deeper impact than existing ferrous micro shots without additional work hardening."



*The EverShot™ media is ideal for the shot peening of hard and small parts, and parts with small radii or complex geometries such as gears, springs and threads*

Competitive benchmarking shows that an EverShot™ cut lasted up to seventy times longer than ceramic and nine times longer than other high hardness ferrous micro shot. This substantially higher durability provides more consistent surface quality and improved uptime from less frequent material replacement while lowering process waste, claims the company.

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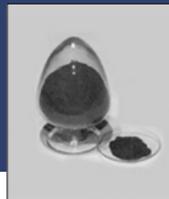


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### Airbus and Chinese University look at further applications for titanium Additive Manufacturing in commercial aviation

Airbus and China's Northwestern Polytechnical University (NPU) have signed a cooperation agreement on exploring ways to further apply Additive Manufacturing technology in the commercial aviation sector. Under the agreement NPU, located in the Chinese city of Xi'an, Shaanxi Province, will manufacture test specimens of titanium alloy parts for Airbus using its Laser Solid Forming technology.

"We are pleased to have been selected by Airbus, the world's leading aircraft manufacturer, as a partner to carry out the pilot project to explore ways of applying 3D printing technology in commercial aviation," stated NPU President Weng Zhiqian. "This project is a test for our 3D research capability and we are confident we

will deliver satisfactory results on quality and on time that will establish a solid foundation for further cooperation in this field."

Airbus stated that it is exploring the use of Additive Manufacturing (AM) to produce individual parts or even larger airframe structures for the company's line of aircraft. The test specimens produced at NPU will be manufactured according to Airbus specifications and will be measured and assessed by Airbus.

In a separate project Airbus has already produced a variety of plastic and metal brackets for use on the A350 XWB aircraft. The material and structural properties of these parts have been tested and are now incorporated on the company's fleet of developmental aircraft.

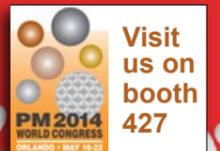


*Additively manufactured developmental bracket produced by Airbus for the A350 XWB aircraft*

Airbus is also working toward spare part solutions with this technology, which it claims is ideal for producing out-of-production aircraft spare parts on demand. This month, the first AM component, a small plastic crew seat panel, flew on an Airbus A310 customer jetliner operated by Canada's Air Transat.

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## Additive Manufacturing helps with ear implants and facial reconstruction

Germany's Laser Zentrum Hannover e.V. (LZH), an independent non-profit research institute based in Hannover, has announced it has been developing Additive Manufacturing technology to help with a number of medical procedures.

One of the projects reported is the use of Additive Manufacturing (AM) to simulate the procedure for surgeons working on hearing impaired patients. Inserting an implant into the tiny cochlea requires utmost care and during the operation the surgeon runs the risk of destroying intact sensory cells, which could result in further deterioration of the patient's hearing.

The micrometer-small cochlea replicas that the surgeons use to practice the procedure are manufactured by the LZH's Photonic System Technology Group using AM technology. The Surface Technology Group at LZH, in cooperation with the Hannover Medical School (MHH), is also developing implants that change their shape due to temperature changes during the surgery, thus making the insertion much easier.

In another project, AM has been used for producing temporary magnesium scaffolds. These magnesium scaffolds, once implanted, are slowly and gradually decomposed by the human body. The scaffolds are particularly suited for reconstructing defects of the facial skull because their shape can be matched to the face of the patient. Directly after the surgery the bioresorbable implants stabilise the tissue above. Over time they make room for new bone cell growth.

[www.lzh.de](http://www.lzh.de)



*This laser-additive manufactured micro-actor for cochlea implants can change its shape due to temperature changes*

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## Conference on deformation and fracture in PM materials to take place in Slovakia

The International Conference on Deformation and Fracture in PM Materials (DF PM 2014) will be held in Academia Congress Centre, Stará Lesná, High Tatras, Slovakia, October 26-29, 2014. The conference represents a continuation of the conferences on PM organised in the former Czechoslovakia at regular intervals since 1962.

The established orientation of the DFPM international conferences is on fundamentals of material properties. The aim of the conference is to promote information exchange between scientists, researchers and industrial engineers with the aim of improving the properties, lifetime and reliability of PM materials. Furthermore, a closer international cooperation in the field of deformation and fracture behaviour of these materials will be promoted.



The Academia Congress Centre is the venue for DF PM 2014

The main topics covered at the conference include:

- Microstructure, physical properties, failure, fracture micromechanism
- Application of PM materials under complex stress and exploitation conditions
- Modelling
- Advanced PM technologies and materials

www.imr.saske.sk ●●●

## Programme published for HIP'14 Conference

The Technical Programme for the 11th International Conference Hot Isostatic Pressing, HIP '14, Stockholm, Sweden June 9-13, can now be viewed on the conference website. Organised by the International HIP Committee (IHC) and Jernkontoret, the Swedish Steel Producers' Association, the event will also include an exhibition and offers a number of optional site visits.

The conference will focus on developments in HIP technology. Aspects related to PM processing, diffusion bonding and part densification will also be included.

The Technical Programme includes sessions focussing on oil and gas, power generation, aerospace, materials, modelling and HIP processes.

www.hip14.se ●●●

## Update to PM property database

The online Global Powder Metallurgy Property Database (GPMD) was launched in 2004 as a joint project between the European Powder Metallurgy Association (EPMA), Metal Powder Industries Federation (MPIF) and Japan Powder Metallurgy Association (JPMA).

Since its launch, the database has been refined and extended to include the non-ferrous Powder Metallurgy (PM) and Metal Injection Moulding (MIM) sectors with coverage of nearly 4,000 lines of high quality data.

Thanks to continuous growth the site now has over 10,000 registered users from countries all over the world and from a wide range of industrial sectors. In the latest development launched last week additional information has been added covering strain controlled fatigue data for:

- The pre-alloyed steel grade FL-05M1/FL-4405(0.85% Mo, 0.20% Mn, balance Fe) with a 0.5% ele-

mental carbon addition, in both the as-sintered and quench and tempered conditions.

- The "hybrid" material FLN2-4405, based on the same pre-alloyed steel grade, but with elemental additions of both 0.5% carbon and 2% nickel, in the as-sintered condition.
- The pre-alloyed steel grade FL-5305 (0.50% Mo, 0.20% Mn, 3.0% Cr, balance Fe) with a 0.5% elemental carbon addition, in the sinter-hardened and tempered condition

The data is displayed in a user-friendly format viewable in both tabular and graphic formats and it is hoped it will be of particular interest to design engineers in a range of industries.

www.pmdatabase.com ●●●

## EPMA to celebrate its 25th anniversary

This year marks 25 years since the formation of the European Powder Metallurgy Association (EPMA) in Brussels, Belgium. The EPMA will celebrate reaching this milestone with a number of special events beginning with a VIP Dinner and a 25th Anniversary Seminar to be held following the association's Annual General Assembly in London, UK, June 5 - 6, 2014.

The members only VIP Dinner will be followed by a one day seminar with presentations from international PM experts focussing on the theme of developments in the European PM industry.

Celebrations will continue later in the year at the Euro PM2014 Congress to be held September 21 - 24 in Salzburg, Austria.

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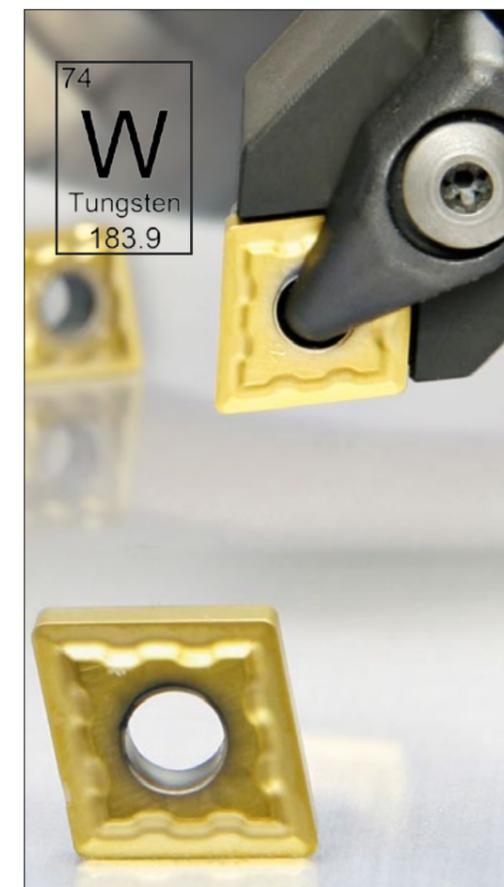
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## Dates announced for China's 7th "PM Technology and Business Forum"

China's "PM Technology and Business Forum", organised annually since 2008, is one of the largest PM industry events in China. The 7th event in the series takes place from June 12-14, 2014 in the HaiZhou Hotel, Haining, Zhejiang Province, China, and is sponsored by the China PM Business Website, [www.pmbiz.com.cn](http://www.pmbiz.com.cn)

The event organisers told Powder Metallurgy Review, "In 2013 the economic and political situation was

complex in China as well as overseas, which impacted on the PM industry. Generally, however, the PM industry in China sustained strong growth over this period. Leading businesses enjoyed the advantages of scale and more advanced technology and are playing an increasingly important role in the international supply chain."

"Whilst some small and medium scale enterprises experienced difficulties, PM enterprises in China closely follow developing trends and

are looking to integrate technology advantages, improve quality systems and seek business opportunities. The PM Technology and Business Forum provides a good platform for this."

The forum's Technical Program includes:

- Automobile keynote reports: Trends in PM parts adoption by automobile OEMs
- Materials selection, design and processing of PM moulds
- Current situation and developing trends of PM additives
- Iron powder consumption in China and its position in the world
- Cutting-edge technology of PM
- PM2014 Congress overview
- Invited keynote: Current situation of MIM industry in Europe, combined with situation in China

Discussion forums will enable the direct communications between experts and participants to solve production problems. There will also be an exhibition featuring the latest PM equipment, technology and products. A visit to a PM related enterprise is also planned. The delegate fee is RMB 1200 per person.

[www.fmyj.org](http://www.fmyj.org) ●●●

## Powder Technology course at Lund University, Sweden

A new course on Powder Technology in Pharma, Food, Chemistry and Metallurgy is scheduled to take place at Sweden's Lund University, September 10-12, 2014. The three day course is aimed at industry professionals and Ph.D students and will include presentations from lecturers at Lund University and external speakers.

The aim of the course is to provide participants with a better understanding of powder products and processes and to supply tools to stimulate new ideas for development and improvement of powder products and processes.

[www.lu.se](http://www.lu.se) ●●●

## EOS adds new titanium and stainless steel grades for Additive Manufacturing

German Additive Manufacturing systems provider EOS has expanded its range of materials for metal AM applications to include Titanium Ti64ELI and Stainless Steel 316L. The two new grades of powder will complement the company's existing range of materials suitable for the production of metal components using the DLMS process.

Parts built in EOS Titanium Ti64 have a chemical composition and mechanical properties corresponding to ASTM F136. Providing a high detail resolution it can be processed on an EOSINT M 280 (400 Watt) metal laser-sintering system. This light metal alloy shows an excellent corrosion resistance. Due to its biocompatibility and high grade of purity it is particularly suited for the additive manufacturing of medical implants,

the company stated.

The EOS Stainless Steel 316L alloy has been optimised specifically for processing on the EOSINT M 280 metal laser-sintering system. It shows a good corrosion resistance and a high ductility. Parts built from EOS Stainless Steel 316L have a chemical composition corresponding to ASTM F138 (Standard Specification for Wrought 18Cr-14Ni-2.5Mo Stainless Steel Bar and Wire for Surgical Implants UNS S31673).

In the medical industry, this alloy is particularly suited for surgical instruments, for endoscopic surgery, orthopaedics and implants. The material is also a good choice for use in the watch and jewellery industry, where the designer benefits from extensive freedom of design. Parts such as watch cases thanks to defined



Watch case manufactured using EOS StainlessSteel 316L (Courtesy EOS/CPM)

hollow spaces can be manufactured more cost-efficiently and easily, at the same time saving resources.

The material is also well suited for additive manufacturing applications such as spectacle frames or functional elements in yachts. In the aerospace industry EOS Stainless Steel is a good choice for the manufacture of clamping elements or heat exchangers. Parts manufactured from this material can be mechanically post-processed or polished.

[www.eos.info](http://www.eos.info) ●●●

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## Additive Manufacturing with metal powders: Design for Manufacture evolves into Design for Function

Additive Manufacturing offers the possibility to produce complex parts without the design constraints of traditional manufacturing routes. Components that would not have even been possible just a few years ago can now be made to high standards using a wide range of metal powders. No longer solely a prototyping technology, Additive Manufacturing is now being used for the production of series components for the most demanding applications. Juan F Isaza P and Claus Aumund-Kopp review the various processes in use as well as key application areas and present an outlook for a technology that is set to revolutionise the future use of metal powders.

Additive Manufacturing (AM) is a technology that produces three-dimensional parts layer by layer from a material, be it polymer or metal based. The method relies on a digital data file being transmitted to a machine that then builds the component. Additive Manufacturing offers many advantages in the production of parts, presenting unrivalled design freedom with the ability to manufacture single or multiple components from a wide range of materials (Fig. 1).

As the machine essentially builds the component one layer at a time the method can be described as an additive process, rather than a subtractive process that removes layers of material, such as milling. Other terms often used to describe the process include 3D Printing, Additive Fabrication, Freeform Fabrication, Fabling and Additive Layer Manufacture. Of these the most popular terminology used by the general press and a number of

designers is 3D Printing. However, it can be argued that this phrase should really only be used to describe one method of production, namely the deposition of a material using a print head or nozzle.

In this article the term Additive Manufacturing will therefore be used when describing the technology. The specific area of focus will be on the production of metal parts via the various AM routes.



Fig. 1 Metal Additive Manufacturing offers unrivalled design freedom with the ability to manufacture parts from a wide range of materials. This is a prototype of an optimised bracket for an Airbus A380 made from stainless steel powder, with conventional bracket behind (Courtesy EADS)



Fig. 2 The latest metal AM machine from EOS was introduced in 2013. The EOSINT M400 uses a 1 kW laser (Courtesy EOS)



Fig. 3 The Arcam Q20 uses an electron beam to melt the metal powder (Courtesy Arcam AB)

### A brief history of Additive Manufacturing

The early AM processes were established in the mid 1980s as a solution for faster product development. At this time the practices were called Rapid Prototyping, because the idea

(LOM) from Helisys. Selective Laser Sintering (SLS) from DTM was also introduced at this time, a process that fuses powder materials using a laser.

Metal based AM processes were developed in the 1990s and introduced to the market soon after. At this time several companies launched

its Laser-Engineered Net Shaping (LENS) metal powder system based on technology developed at Sandia National Labs, USA. In 1999 Rödgers, a German company, began marketing its Controlled Metal Buildup (CMB) machine based on technology developed at the Fraunhofer Institute for Production Technology in Germany. Also in 1999, ExtrudeHone introduced its ProMetal Rapid Tooling System RTS-300, the commercial realisation of MIT's process for manufacturing metal parts and tooling. Similar to the use of polymers and waxes in the preparation of feedstock for the Metal Injection Moulding (MIM) process, this system was able to print a binder on a powder bed, binding the metal particles and producing "green parts" which subsequently have to be debound, sintered and infiltrated to get completely dense material.

In 2002 Precision Optical Manufacturing began sales of its Direct Metal Deposition (DMD) laser-cladding systems, a process that produces and repairs parts using metal powder.

The continuing development of AM systems has enabled the fabrication of usable parts made in the desired material in a single-step process. It is now possible to manufacture almost 100% dense functional designs. Over time these systems have become more reliable and more efficient, with the range of suitable materials growing significantly.

***"Metal based AM processes were developed in the 1990s. At this time several companies launched systems for laser sintering approaches which were able to produce metal parts directly"***

was really to produce three dimensional models or mock-ups in order to check form, fit and function.

In 1987 3D Systems began the commercialisation of the plastic processing technique known as Stereolithography (SL), offering completely new possibilities to designers and engineers and supporting the fast growing market of "short life" products. The process essentially solidifies thin layers of UV light sensitive liquid polymer using a laser and was the first commercially available AM system in the world.

In the early 1990s other polymer based AM technologies began commercialisation, including fused deposition (FDM) from Stratasys, Solid Ground Curing (SGC) from Cubital and Laminated Object Manufacturing

systems for laser sintering approaches which were able to produce metal parts directly, providing an alternative to direct multi stage processes.

In 1994 EOS demonstrated their prototype EOSINT M160 machine based on direct metal laser sintering technology. In 1995 the company's EOSINT M250 was launched, enabling the rapid production of metal tools. These systems were able to manufacture metal parts by sintering the powder, but in many cases the mechanical characteristics of the materials were more comparable to composites than to metal alloys, due to the combination of a low melting material (e.g. a bronze-based matrix) with a high resistant material (e.g. stainless or tool steel).

In 1998 Optomec commercialised

### Today's Additive Manufacturing systems

There are various AM systems available and these can be classified in many different ways. It is possible to divide the processes by the feedstock state, the most common ones being solid (powder, wire or sheet) or liquid. The way the raw material is being applied is another way of defining the different approaches, this can be done with a recoater or inside a vat as well as drop wise etc. It is also possible to define groups of processes by the energy source or the way the material is being joined, for example using a binder, a laser, a heated nozzle etc. Classification is also possible by the group of materials being processed, such as plastics, metals or ceramics.

#### Powder-bed systems

Almost every powder-bed based AM system uses a powder deposition method consisting of a coating mechanism to spread a powder layer onto a substrate plate and a powder reservoir. Usually the layers have a thickness of 20 to 100  $\mu\text{m}$ . Once the powder layer is distributed, a 2D slice is either bound together, known as 3D-Printing, or melted using an energy beam applied to the powder bed (Fig. 4). In the second case the energy source is normally one high-power laser, but state-of-the-art systems use two or more lasers with different power under inert gas atmosphere.

Direct process powder-bed systems are known as laser melting processes and are commercially available under different trade names such as Selective Laser Melting (SLM), Laser Cusing and Direct Metal Laser Sintering (DMLS) (Fig. 5). The only exception to this process principle is the Electron Beam melting (EBM) process, which uses an electron beam under full vacuum. The melting process is repeated slice by slice, layer by layer, until the last layer is melted and the parts are complete. Then it is removed from the powder bed and post processed according to requirements.

Metal powder bed fusion



Fig. 4 Turbine manufacture with Inconel 718 for micro turbine applications

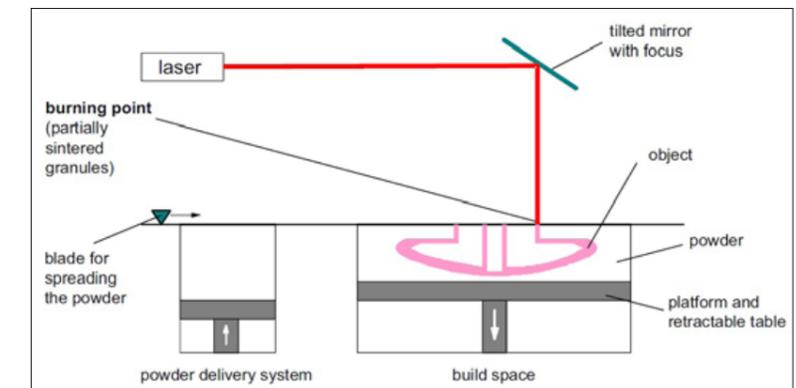


Fig. 5 Schematic diagram of the Selective Laser Melting (SLM) powder-bed process (Source VDI 3404)

machines are available today from Concept Laser GmbH, EOS GmbH, ReaLizer GmbH, Renishaw and SLM Solutions GmbH in Europe. These companies offer a variety of systems based on the similar selective laser melting principle, but giving their own processes different names. 3D-Systems, based in USA, also offers systems based on selective laser melting. The choice of the right machine is dependent on the requirements of the end user, with the type of laser unit, powder handling and build chamber being some of the main characteristics of the system to consider.

Arcam AB, based in Sweden, manufactures powder bed fusion systems that use an electron beam as the energy source for the melting process (Fig. 3). A hybrid system

that combines powder bed fusion with CNC milling is offered by the Japanese company Matsuura.

Another system using a powder bed is the Höganäs Digital Metal process. Developed by fcubic, this system uses a precision inkjet to deposit ink on a 45 micron layer of metal powder. Another 45 micron layer of powder is applied and the printing step is repeated until the component is complete. The part is then sintered to reach final size and strength. One of the benefits of this system is that the build is undertaken at room temperature without the partial melting which occurs with laser or electron beam technology. In principle there is also no need for support structures during the build as it is supported by the powder bed.



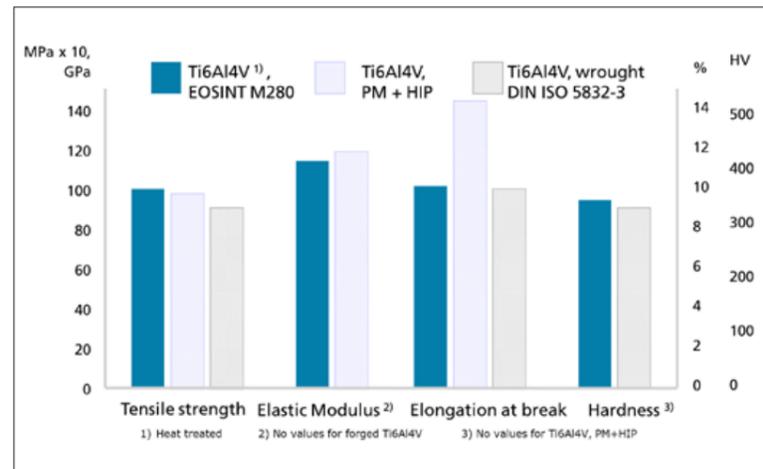


Fig. 9 Mechanical properties of Ti6Al4V



Fig. 10 Aerospace applications offer huge potential for AM. There are 19 of these AM fuel nozzles in each of GE's new LEAP passenger jet engines (Courtesy GE Aviation)

Heat treatment is often included in the process chain as well as shot peening which is used to improve the mechanical and tactile properties of the surface of AM parts.

Another post process of interest is electro polishing, as this electrochemical treatment significantly improves the surface finish of AM parts. Its first objective is to minimise micro roughness, thus reducing the risk of dirt or product residues adhering and improving the cleanability of surfaces. However, electro polishing can be also used for

deburring, brightening and passivating, particularly for surfaces exposed to abrasive media. Since electro polishing involves no mechanical, thermal or chemical impact, small and mechanically fragile parts can also be treated.

### Properties and standards

Comparing the properties of some materials resulting from different processes, it is possible to find some similarities, but is well known that depending on the processes specific properties can be improved. Not all materials behave in the same way, but in general it is possible to establish that mechanical properties resulting from AM processes are comparable to the ones resulting from casting. Fig. 9 shows some typical mechanical properties for the titanium alloy Ti6Al4V used for medical implants, illustrating the good properties of SLM parts. Parts resulting from AM processes need to be heat-treated in order to get optimal mechanical properties.

Activities supporting the development of standards for AM are being undertaken around the world. EU projects like SASAM (Support Action for Standardisation in Additive Manufacturing) are promoting the growth of AM to become efficient and sustainable industrial manufacturing processes by integrating and coordinating standardisation activities within the European community. Initiatives like STAIR AM (Standardisa-

tion, innovation and research) are also facilitating the dialogue and communication between the research and innovation communities, CEN and CENELEC members, and standardisation activities. Parallel to these activities, groups from ISO/TC261 are working in cooperation with the ASTM F42 developing standards in the field of AM concerning their processes terms and definitions, process chains, test procedures, quality parameters, supply agreements and all kinds of fundamentals. Relevant European stakeholders have been involved covering applications in the aerospace, automotive and medical industries but also for private printing purposes.

### Applications for AM technology

Initially seen as a process for concept modelling and rapid prototyping, AM has expanded over the last five years or so to include applications in many areas of our lives. From prototyping and tooling to direct part manufacturing in industrial sectors such as architectural, medical, dental, aerospace, automotive, furniture and jewellery, new and innovative applications are constantly being developed.

It can be said that AM belongs to the class of disruptive technologies, revolutionising the way we design and manufacture almost everything. From collectables and consumer goods produced in small batches to large scale manufacture in many branches, the applications of AM are vast. The number of users of these technologies has been growing constantly, from artists, designers and individuals to small companies and enterprises using AM to manufacture final products.

Typical applications for metal AM processes are summarised in Table 2. Currently metal AM is not a process for the basic mass production of millions of identical parts. However some applications, for example dental restorations, really tap the full potential of AM. In this highly individualised production process it is economically viable to use AM technologies, speeding up the

production time without inflating the costs per part. AM advantages derive from its extremely high flexibility due to the product being produced directly from a CAD model without the need for tooling. This also allows the AM process to produce almost any geometry that can be designed.

It is difficult to generally ascertain when AM production becomes a viable option for an enterprise, but for small series production (depending on the part, some thousands parts per year), functional prototypes and individual parts, AM could be a good option. AM techniques complement the vast group of production processes allowing designers and engineers the improvement of existing process chains or opening new opportunities for production. This of course motivates universities and companies to instruct their students and staff about the possibilities and capabilities of AM.

Applications in aerospace, for example the fuel nozzles for the GE LEAP engine (Fig. 10), are showing the possibilities of AM and opening the doors for new ideas and development projects. Changing the paradigm of "design for manufacturing" to "design for function" will change the way we experience improvements of future components and will improve the performance of the system.

### Design advantages

Until recently the outer geometry of a part and its function and/or strength were of main interest for the user, but AM allows the integration of additional functions and new fields of application of technical parts. One example of the ability of AM technology to integrate functionality directly into parts is in the production of moulds or tools with conformal temperature control or vacuum channels running directly below the surface of the die. An example of an extrusion tool design with this feature is described in Case Study 1 on the following page. The cooling channels in this tool cannot be produced with any other technique.

Another good example of using AM to integrate functionality can be seen in parts with repeating

Typical applications for metal Additive Manufacturing
Production of models and prototypes during a products development phase
Parts for pilot series in medical, automotive and aerospace industry
Very short series products where tooling costs for casting or injection moulding would be too high
Parts of highest geometrical complexity which could not be realised by means of conventional manufacturing (grinding, milling or even casting).

Table 2 Typical applications for metal AM processes

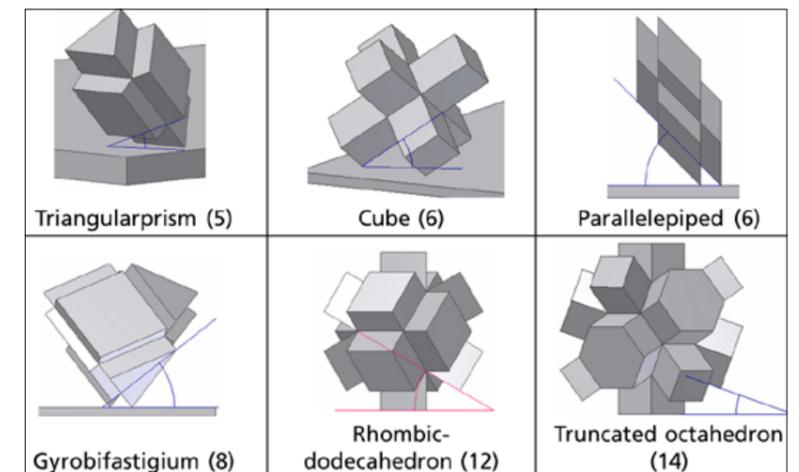


Fig. 11 basic cell geometries for structure design

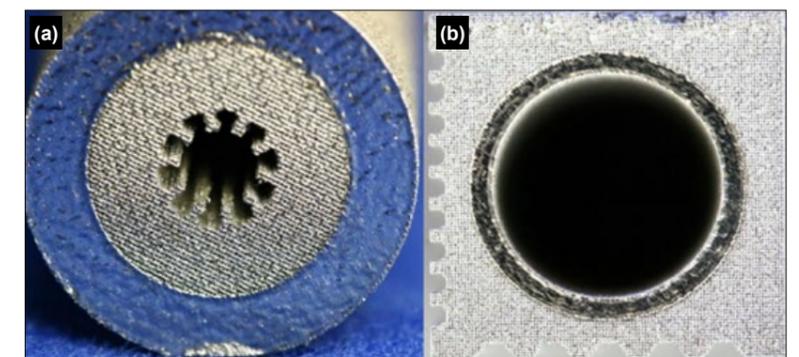


Fig. 12 Additivity manufactured study for material combinations of porous and dense areas. Shown in (a) is a dense shell with a porous core and in (b) is a porous shell with a dense core

internal patterns which enlarge inner surfaces for a better energy or mass exchange in devices for heat recovery or filtration. Some examples of unit cells for 3D tessellation are showed in Fig. 11. These structures make possible the complete filling of the space, if needed, allowing the free design of part density.

To utilise the full potential of metal AM techniques, complex internal structures can be combined with other geometric features as

outer shells. Separating parts into shell and core volumes enables new solutions not only for lightweight parts which need a dense shell and a porous core (Fig. 12 a-b) but also for parts with internal functionality. Both parts were produced in one manufacturing step, resulting in considerable savings in part weight as well as energy consumption during processing.

The design process can also incorporate the use of topological

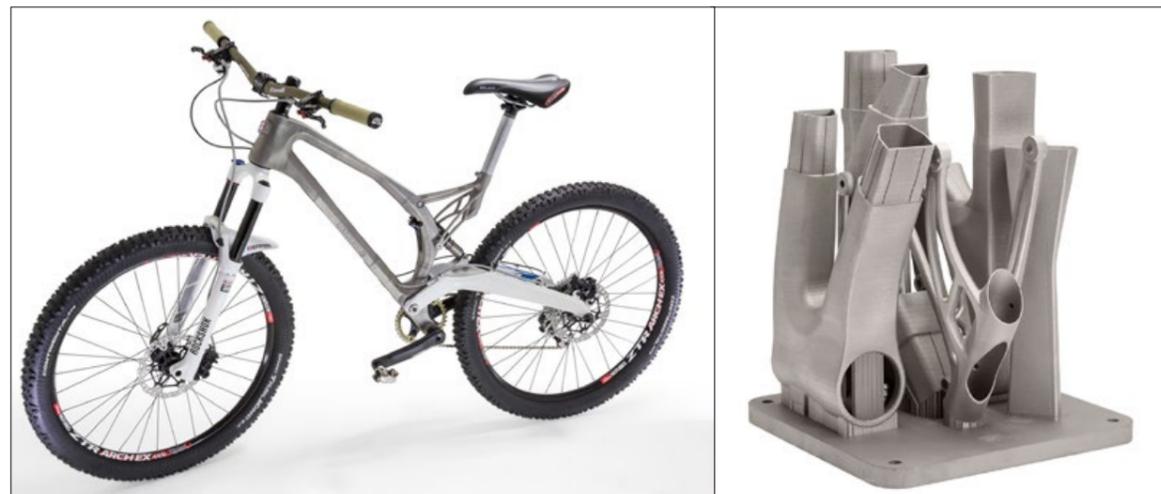


Fig. 13 The frame of this bike has been made using Additive Manufacturing. Built from Ti alloy the frame is 33% lighter than the original. The build plate (left) includes all parts for the frame (Courtesy Renishaw & Empire Cycles)

optimisation software to determine the logical place for material. Material is removed from areas of low stress until a design optimised for load bearing is finalised. The resulting component is both light and strong. This process was applied to parts of the bike frame shown in Fig. 13. The optimisation of the design and the use of titanium alloy resulted in a weight saving of 33% over the original.

Due to limited building envelopes of AM systems, the production of large structural elements by AM is restricted. The size of the current maximum build space in industrially available AM equipment is approximately 630 x 400 x 500 mm, but AM technologies are being continually improved and build space in all systems will certainly continue to grow.

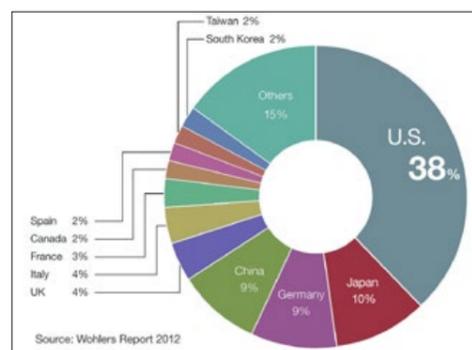


Fig. 14 Global share in terms of the number of metal powder additive manufacturing machines introduced (Source Wohlers Report 2012)

## Growth areas and market potential

Many industrial applications for AM have been developed over the last five years. It is also well known that industries such as aerospace, automotive and medical are embracing the advantages of AM and implementing the technology successfully. Table 3 shows that the value of the AM industry (all materials) is expected to grow significantly over the coming years. A regional breakdown of the introduction of metal AM machines (Fig. 14) shows that the industry is truly global.

Potential application areas in industry could be defined as ones for transportation purposes, in which lightweight engineering plays an important role as an enabler

AM industry (products and services) worldwide projected value	
2015	\$4 billion
2017	\$6 billion
2021	\$10.8 billion

Table 3 The global value of AM products and services (all materials) is expected to grow considerably (Source Wohlers Report 2013)

for better mass distribution. As mentioned before, areas offering customised products or seeing mass customisation as a new market could utilise the advantages of AM. Production lines implementing manufacturing on demand could also use the huge potential of layer based technologies. It is of course difficult to predict exactly which areas will adopt AM for the future, as in 1947 at Bell Labs it was also hard to imagine the multiple uses of the transistor.

For a better acceptance of AM some barriers need to be overcome, namely application knowledge, standards and norms for material quality. It is reasonable to think that current technologies in industry are not all going to be replaced by AM and that the vision of one machine producing any kind of shape with unlimited material variations is unlikely. This should not be the target for the technology. Growth will be seen in opening new horizons and enabling the manufacturing of components that were not possible before.

The importance of the know-how for an easier introduction to existing process lines can be compared with the introduction of laser cutting of metal sheets. It was not really the fact that laser beams can be used to cut metal sheets which pushed the idea to become a manufacturing technique. It was more the study of the process, the results and the

## CASE STUDY 1: Vacuum calibration sleeve

In the extrusion of plastics, the most commonly used types of calibration are vacuum calibration and compressed air calibration. During the vacuum calibration process the extrudate is fed from the extrusion die to the calibrating device. The calibration bore has a smaller diameter than the nozzle pushing the extrudate close to the bore. Due to the normal pressure in the pipe section and the vacuum in the calibration sleeve a negative pressure is generated. As a result, the tube pushes outward against the bores wall. Thus, the extrudate does not stick in the calibration sleeve, the surface is rapidly cooled with cooling water. Thereby the surface is hard enough so that the tubular profile can be slid through the calibration tool.

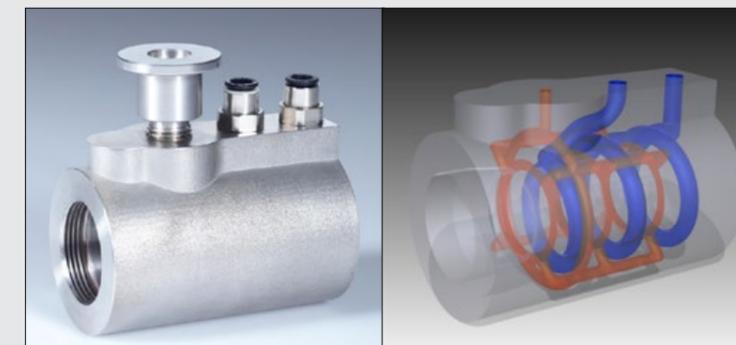


Fig. 15 Additivity manufactured calibration tool showing the internal vacuum (red) and cooling channels (blue). This part was built on an EOS M270 system at Fraunhofer IFAM

Based on this principle it was possible to manufacture a calibration tool for the extrusion of copper feedstock. As can be seen in Fig. 15 (left), the vacuum calibration can be held by an internal thread at the nozzle of the extruder. This enables a good connection, so that the extrudate can easily slide into the tool. Furthermore, a flank for the cooling water connections and the vacuum port is visible at the upper side of the device.

For a better understanding of the calibration tool, the cooling and vacuum channels are shown in Fig. 15 (right). The blue channel represents the cooling water channel (with the supply and discharge) and the red channel illustrates the vacuum channel. The diameter of the cooling water channel is 5 mm in order to ensure a good water flow (empirical value). The vacuum channel has a diameter of 3 mm.

capabilities, including resource-saving, that convinced the companies to introduce it. Without the resulting forces required to shear the material it was possible to position the shapes closely, decrease production time and maximise the output by placing different geometries together.

Metal AM offers new possibilities in the use of new materials. This is the case for a number of alloys which can only be manufactured under high cooling rates for example.

## Ongoing research activities

One notable area of research currently underway is in radio frequency identification (RFID) applications as used in logistics, especially for the purpose of tracking and managing items. Of current focus is the real-time tracking of medical instruments during a surgical operation. The metallic environment in the operating room represents

a big challenge, hampering the readability of the RFID tag. Currently transponders are joined to metal instruments by bonding or clamping inside a polymer shell, containing as little metal as possible.

Assembling the tags is a time-consuming process, which changes the usual shape of the instrument. It also represents a point of failure, because of the possibility of losing the tag during the instrument's life cycle. AM can be used to integrate the entire RFID tag in a metallic instrument while keeping the original shape. Research shows that it is possible to read RFID tags through a closed SLM metallic casing.

Successful research has been done with tags in the low frequency (LF) range. Instrument manufacturing by AM enables small wall thicknesses and a reduced temperature impact on the RFID tag during the building process. Tag integration in complex parts and readability through metal open a new dimension in the traceability and manufacturing of medical devices.

Similar research activities for the integration of electronics in polymers have been done at Fraunhofer IZM. Electronic components have been embedded in a substrate, which is manufactured using direct digital manufacturing processes. In the initial step, an assembly is manufactured, in which selected planar components are embedded, including SMD resistors, capacitors, LEDs and a microcontroller.

In the areas of material development, biomimetic, cellular materials (lattice structures) and of course process development there are groups around the world working on new ideas. For laser-based processes there are approaches to avoid residual stresses during processing by working at higher temperatures or to get better surfaces by utilisation of pulsed lasers.

Hybrid and multi-material processes offer new possibilities for industry and medical applications. On the software side some efforts are allowing faster data preparation and the use of more complex geom-

## CASE STUDY 2: Wing profile

Different departments within Fraunhofer IFAM often cooperate on research and development projects. One example of this teamwork is the manufacture of a tailored wing profile used in the development of anti-ice coatings.

In this case study a small-scale model of an aircraft wing profile used in a flexible setup for anti-icing tests also demonstrates the geometrical capabilities of AM. The part shown in Fig. 16 measures approximately 190 x 200 x 30 mm and has two separated chambers for temperature control. It was designed for the simulation of ice accretion on the leading edge and the subsequent melting of ice, including runback ice formation. An accurate aerofoil shape, the tempering channel in the leading edge (approximately 5 % of



Fig. 16 Additively manufactured wing profile polished after build-up (left) with the structure of the internal channel geometry shown (right). Designed and built on an EOS M270 system at Fraunhofer IFAM

the chord length) and thin walls for better energy exchange were some of the requirements for the functional model. The low number of produced parts and their complexity make the use of AM almost impossible to avoid. Other manufacturing techniques such as casting or machining would be too laborious and expensive for the requirements of a simulation model.

For a better distribution of the tempering fluid the rear chamber was equipped with internal channels

spreading from the inlet. The part is hollow and has a wall thickness of 3 mm for a better thermal conduction and faster response to the temperature changes. Additive Manufacturing enables the integration of additional functionality by producing parts with complex internal features and allows the manufacture of different geometries in one batch to be used for simulation purposes.

etries with higher data volumes. Simulation software could reduce the time needed for qualifying new materials and also predict some distortions during process, being an area of major interest for research activities.

An investigation at EADS Innovation Works in cooperation with the Hamburg University of Technology shows the tremendous benefits of scandium regarding the combination of high-strength properties with a reduction of density. The improvements of properties that can be achieved by adding this element to an Al alloy are impressive. The rapid solidification offered by the SLM process allows for cooling rates that are sufficient to keep all alloyed scandium in a hypereutectic Al-scandium composition. This produces high strength properties, around 500 MPa, with nearly isotropic results. Furthermore, the ductility of the additively manufactured material, with an elongation of 14% and reduction of area 20%, is remarkable.

## Summary and outlook

The outstanding feature of all AM techniques is their capability to produce parts of high geometrical complexity which can not be manufactured by any other production technique. This works because of the tool-free layer-by-layer approach of all AM processes. Parts are produced based on 3D-CAD-model-data without any tooling needed.

The number of available materials is still limited compared to other processes such as milling or injection moulding, but the number of materials qualified for polymer and metal based processes is continuing to increase.

Many AM techniques offer part qualities which are comparable to those resulting from conventional manufacturing methods. The AM produced parts can be used and post processed (milled, drilled, coated) like any other standard industrial part. Especially in metal, AM produced parts often exceed some of the mechanical property values of those machined from standard bulk material.

Another benefit is the outstanding material efficiency of most AM processes. Scrap rates for AM parts are usually below 5 %, compared to scrap rates of more than 90 % with many complex milled parts. With a decline in available raw material and rising costs this material efficiency will remain a major advantage in the long term.

Looking to the future, it can be confidently predicted that AM is set to achieve an increasing market share of production processes, helped with the introduction of faster systems with more powerful lasers and larger building chambers. A significant number of materials will be qualified for AM and over time multi-material-systems for many of the processes will become available.

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## CASE STUDY 3: Hydraulic crossing

This case study demonstrates the increased performance possible using AM techniques. Here Selective Laser Melting (SLM) allowed designers to realise significant increases in performance in hydraulics by exploiting the possibilities of this Additive Manufacturing process.

Inside the hydraulic crossing two fluid streams cross within a limited space without mixing. The conventionally produced part consists of a massive metal block where drilled and locked blind holes meet in two levels. The main bore splits into two smaller bores to keep the height and the possible mass flow constant. The weight of the conventionally produced pipe crossing is 20 kg and measures 230 x 230 x 50 mm. Without being hindered by the

limits of conventional production processes, the designer was free to optimise the functionality and in the AM produced hydraulic crossing the fluid flow was improved by adapting the internal channel geometry according to flow simulation results. Geometrical changes of cross section profiles can be manufactured without great effort. Concerning thermodynamics, fins inside the channels are feasible to improve thermal exchange processes and to increase the parts stiffness at the same time. The newly designed hydraulic crossing (Fig. 17) was produced from a stainless steel material, with internal fins supporting the part during processing. The new components dimensions are 80 x 80 x 50 mm and the total weight is only 0.7 kg, resulting in a mass reduction of around 96%.

In this study the optimisation of the

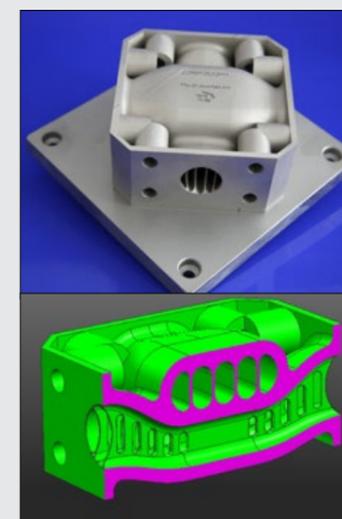


Fig. 17 An additively manufactured hydraulic crossing (left) with the internal channel geometry shown (right). Designed during EU-funded project CompoLight; built on an EOS M270 system at Fraunhofer IFAM

channel geometry has an enormous impact on the crossing's performance and thus on the entire hydraulic system. At a mass flow of 100 l/min, the pressure loss of the new design is reduced to only 20% of the pressure loss of the conventionally designed and manufactured part (Fig. 18). Additionally, without any post-processing the surface quality of the AM part was sufficient to be flanged to the connecting piping. Even at a test pressure of 1400 bar, the part showed neither plastic deformation nor leakage.

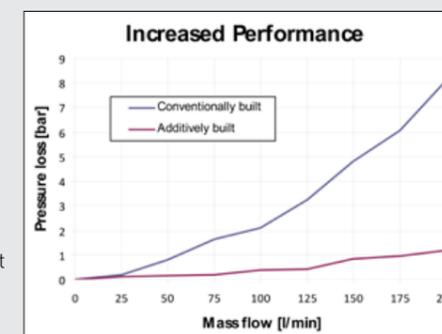


Fig. 18 Comparison of pressure loss of conventionally and additively built hydraulic crossing

## CASE STUDY 4: Turbine rotor

Nickel-based alloys, or superalloys, maintain their mechanical properties at very high temperatures. Other characteristic properties are high creep resistance and high corrosion resistance. As such they are often used for turbines or heat exchangers.

The use of nickel-based alloys with Selective Laser Melting (SLM) creates certain challenges. The process can result in thermally induced residual stresses due to the locally concentrated high energy input used to melt the powder followed by high cooling rates.

Additive production of geometrically complex and thin walled parts made of nickel-based material similar to Inconel 718 by SLM is therefore an interesting topic for AM. In this case study, micro turbines used for model aeroplanes and other purposes are the main applications. The laser-based additive production of the turbine rotor shown in Fig. 19 is an example of these parts. Particular requirements concern geometrical accuracy and high requirements concerning the material properties, working in high temperature environments (around 800°C gas temperature) and a high rotation speed (125,000 rpm).

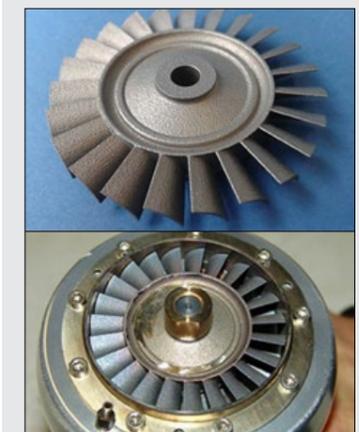


Fig. 19 The turbine rotor has an outer diameter of 68 mm (top). Lower image shows the mounted turbine rotor

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### Titanium PM 2013 Conference: The impact of impurities, advances in porous titanium and titanium Additive Manufacturing

The second International Titanium Powder Processing, Consolidation and Metallurgy Conference, PM Titanium 2013, took place in Hamilton, New Zealand, from December 2-4 2013. The event attracted over 80 participants from around the world to discuss the latest advances in the field of titanium Powder Metallurgy. Prof Ma Qian of RMIT University (Royal Melbourne Institute of Technology) reports on selected highlights from the conference on behalf of *Powder Metallurgy Review*.

The 2013 International Titanium Powder Processing, Consolidation and Metallurgy Conference (PM Titanium 2013), organised by TiDA (Titanium Industry Development Association, New Zealand) in partnership with the University of Waikato, CSIRO and GNS Science, follows on from the success of the inaugural Titanium Powder Processing, Consolidation and Metallurgy Conference, hosted by The University of Queensland, Australia, in December 2011. The conference took place in Hamilton, New Zealand from December 2-4 2013 and attracted over 80 participants. A total of 42 selected papers were presented at the conference in two parallel sessions. The two-day conference was followed by an impressive visit to TiDA and its industrial partners on December 5.

This report reviews five presentations discussed at the conference on impurities in PM titanium and titanium alloys, porous titanium and applications, and the Additive Manufacturing of titanium in New Zealand.

#### Effect of impurities on microstructural development and mechanical properties

Impurity elements need to be strictly controlled in PM Ti products. ASTM Standard Specification 988-13 for Powder Metallurgy (PM) titanium and titanium alloy structural components

specifies the impurity requirements as shown in Table 1.

The effect of oxygen on the tensile properties of PM Ti-6Al-4V (wt.%) was discussed in detail at the inaugural Titanium Powder Processing, Consolidation and Metallurgy Conference in Australia in December 2011, by Prof Hideshi Miura of Kyushu University, Japan, (Fig. 1) and Dr

Composition, Weight %	N, max	C, max	H, max	Fe	O, max	Residual max ea.
Grade 1 PM	0.03	0.08	0.015	0.20 max	0.18	0.1
Grade 2 PM	0.03	0.08	0.015	0.30 max	0.25	0.1
Grade 3 PM	0.05	0.08	0.015	0.30 max	0.35	0.1
Grade 4 PM	0.05	0.08	0.015	0.50 max	0.40	0.1
Grade 5 PM (Ti-6Al-4V)	0.05	0.08	0.015	0.40 max	0.30	0.1
Grade 9 PM (Ti-3Al-2.5V)	0.03	0.08	0.015	0.25 max	0.30	0.1
Ti-6Al-4V, L1 <sup>a</sup>	0.03	0.08	0.0125	0.25 max	0.20	0.1
Ti-6Al-6V-2Sn	0.04	0.1	0.015	0.35-1.0	0.30	0.1

Table 1 Impurity requirements for PM titanium and titanium alloys by ASTM988-13. L1: Low Interstitial

Thomas Ebel of Helmholtz-Zentrum Geesthacht, Germany (Fig. 2). Their experimental findings demonstrated that there exists a critical oxygen level for as-sintered Ti-6Al-4V, which

level, on the tensile ductility of PM Ti-6Al-4V [3]. The presentation began with a concise review of the influence of oxygen on the tensile ductility of a wide variety of titanium materials in-

dropped from 15% to 3% with increasing oxygen content from 0.25% to 0.49% (Fig. 3) while the sintered density was essentially the same (increased from 97.1% [0.25% O] to 97.6% [0.49% O] theoretical density).

Three fine oxygen-induced microstructural changes were identified in the as-sintered Ti-6Al-4V-0.49O using Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and three-dimensional atom probe tomography (3D-APT). These include [3, 4]

- The formation of fine acicular  $\alpha$  precipitates in the  $\beta$  phase
- the formation of  $\alpha_2$ -type (Ti<sub>3</sub>Al) nanometric clusters in the  $\alpha$  matrix, and
- the formation of grain boundary  $\alpha$ - $\beta$ - $\alpha$ -layered structures between the  $\alpha$  grains.

None of these microstructural features was observed in as-sintered Ti-6Al-4V-0.25O that contained oxygen below the critical level [0.33%]. A small increase in oxygen content beyond the critical level

cluding unalloyed Ti, Ti-8Al, Ti-6Al-2V, Ti-2Al-16V, Ti-1Al-8V-5Fe, Ti-10V-2Fe-3Al, high Nb or Mo containing beta Ti alloys and Ti-6Al-4V. The influence of oxygen was shown to be alloy dependent. In general, near beta and stable beta Ti alloys are much less sensitive to the detrimental influence of oxygen on ductility than other types of Ti alloys ( $\alpha$ , near- $\alpha$  and  $\alpha$ - $\beta$ ).

The study then focused on detailed microstructural characterisation of two as-sintered Ti-6Al-4V alloys which contained 0.25% O and 0.49% O, respectively. The tensile elongation

**“A small increase in oxygen content beyond the critical level can thus fundamentally change the typical microstructure of as-sintered Ti-6Al-4V”**

is around 0.33%, beyond which the tensile elongation drops dramatically to a level no longer suited to structural applications. However, the mechanism behind this critical oxygen level remained unknown.

**Understanding the effect of impurities on the microstructural development of powder metallurgy titanium and titanium alloys**

Ma Qian and Ming Yan of RMIT University, Australia, provided detailed microstructural information about the influence of oxygen, beyond the critical

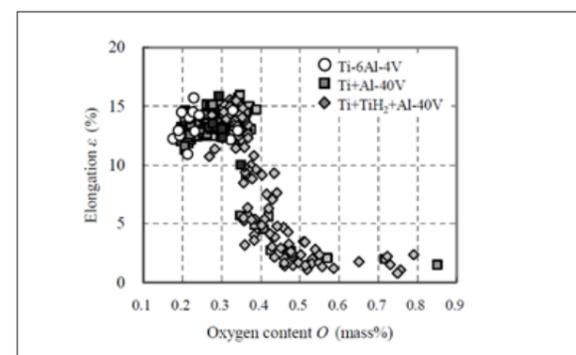


Fig. 1 Effect of oxygen on tensile elongation of as-sintered Ti-6Al-4V [1]

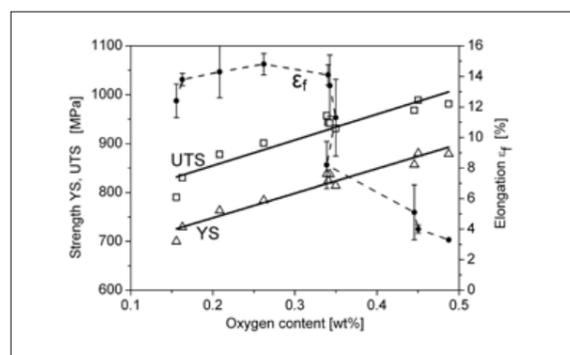


Fig. 2 Effect of oxygen on tensile properties of as-sintered Ti-6Al-4V [2]

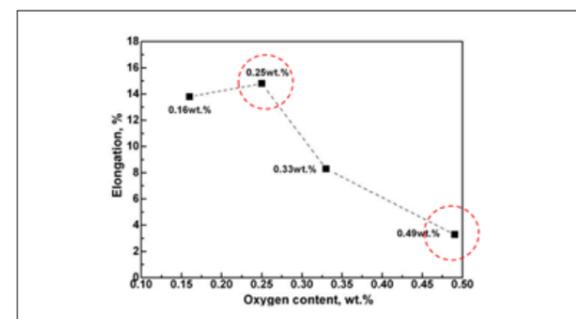


Fig. 3 Effect of oxygen on tensile elongation of as-sintered Ti-6Al-4V [3, 4]

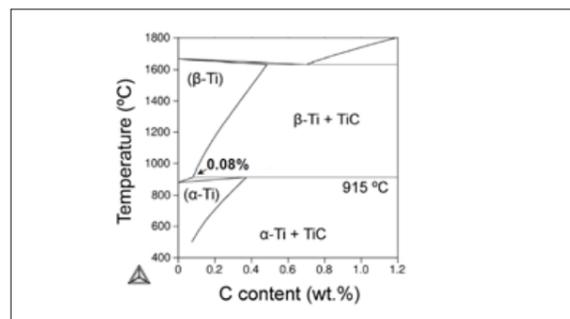


Fig. 4 Ti-C phase diagram up to 1.2 wt.% C, predicted using Thermo-Calc Software 2008 and Ti-alloys database V3 (TTT3) [3, 5]

can thus fundamentally change the typical microstructure of as-sintered Ti-6Al-4V. These oxygen-induced microstructural changes led to a sharp decrease in the tensile elongation of as-sintered Ti-6Al-4V. Details of the experimental study and discussion can be found in a recent publication [4].

Carbon [C] as an impurity in PM Ti has received less attention than oxygen. According to ASTM Standard Specification 988-13 (see Table 1), PM titanium and titanium alloys are generally allowed to contain up to 0.08 wt.% C. The binary Ti-C phase diagram predicted by Thermo-Calc, shown in Fig. 4, explains where this maximum carbon limit comes from. The solubility limit of C in  $\beta$ -Ti at 915°C is exactly 0.08 wt.% beyond which carbon will precipitate out as titanium carbides in  $\beta$ -Ti during subsequent cooling from the isothermal sintering temperature. Avoiding the formation of brittle titanium carbides is thus an important issue for control of impurity carbon in PM titanium and titanium alloys.

It seems clear that the maximum carbon content specified by ASTM Standard Specification 988-13 for PM titanium and titanium alloys did not take into account the influence of alloying elements on the solubility limit of C. On the other hand, for titanium parts that are made by Metal Injection Moulding (MIM), their carbon content will pick up after thermal debinding and isothermal sintering due to the large volume of the binder used in the process. Hence, there is a need to look at the effect of trace carbon on the microstructural development of as-sintered PM Ti and Ti alloys.

**MIM processing of titanium beta alloys**

Another focus of the above presentation was the unexpected formation of grain boundary carbides in as-sintered Ti-15Mo alloy. Coincidentally, the subsequent presentation on the MIM processing of titanium beta alloys by Thomas Ebel, Dapeng Zhao, and Firat Kafkas [6] of Helmholtz-Zentrum Geesthacht Centre for Materials and

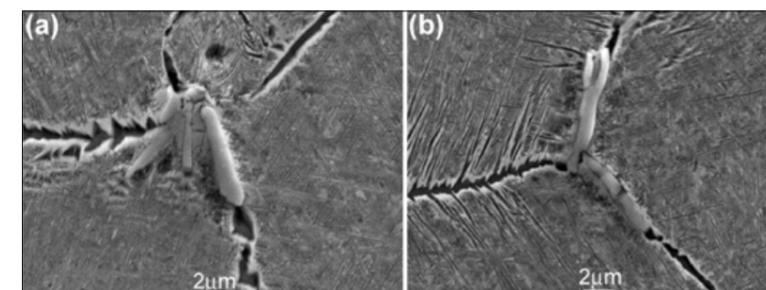


Fig. 5 SEM-SE images of grain boundary carbides observed in as-sintered Ti-15Mo containing 0.032 wt.% carbon, which is well below the specified limit of 0.10 wt. % C [3, 5]

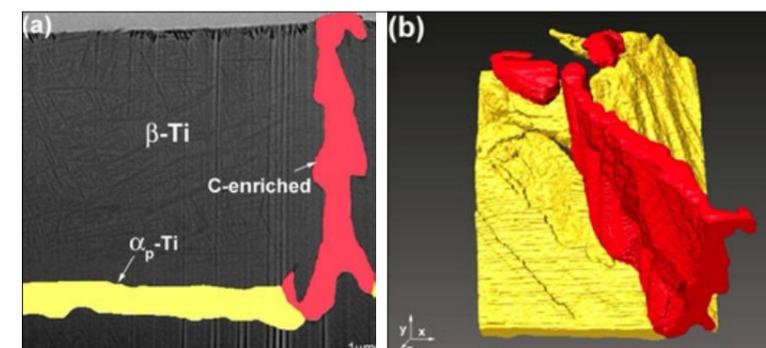


Fig. 6 (a) SEM-SE image showing GB carbide phase (red colour), primary  $\alpha$ -Ti (yellow colour) and the  $\beta$ -Ti matrix observed in as-sintered Ti-15Mo. In total 184 of such images/slices were obtained. (b) A snapshot of the 3D tomography movie to show the morphology of the carbon-enriched phase (red colour) and primary  $\alpha$ -Ti (yellow colour) [3, 5]

	O	C	N
Ti powder	0.0744	0.00469	0.0375
Nb powder	0.221	0.0152	0.0890
CP-Ti	0.175	0.0503	0.0628
Ti-10Nb	0.203	0.0562	0.0678
Ti-16Nb	0.255	0.0600	0.0525
Ti-22Nb	0.225	0.0589	0.0547

Table 2 Impurity levels of powders used for fabrication and as-sintered Ti-(16-22)Nb samples made by MIM. Compositions are given in wt.% [6, 7]

Coastal Research, Germany also focused on grain boundary carbide formation in as-sintered Ti alloys.

Fig. 5 shows the grain boundary carbides observed in as-sintered Ti-15Mo containing 0.032 wt.% C. Wrought Ti-15Mo for surgical implant applications is allowed to contain a maximum of 0.10 wt.% C (ASTM Standard Specification F 2066-08), similar to the carbon limit specified by ASTM 988-13 (see Table 1).

However, the as-sintered Ti-15Mo containing 0.032 wt.% C, which is well below the specified limit of 0.10 wt.% C, shows a noticeable presence of grain boundary carbides, with a complex morphology (Fig. 6). Thermo-Calc predictions revealed that an addition of 15 wt.% Mo reduces the solubility of C in Ti from 0.08% (Fig. 4) to 0.006% (Fig. 7). This explains the noticeable formation of grain boundary titanium carbides

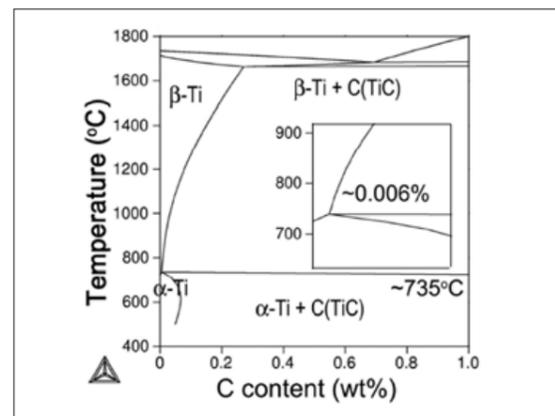


Fig. 7 (Ti-15Mo) - C phase diagram. An addition of 15 wt.% Mo decreases the solubility of C in Ti from 800 ppm to 60 ppm [3, 5]

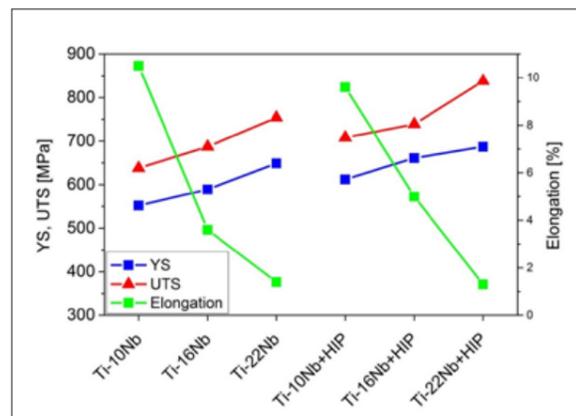


Fig. 8 Mechanical properties of as-sintered Ti-(10-22)Nb alloys with and without subsequent hot isostatic pressing (HIP) [6]

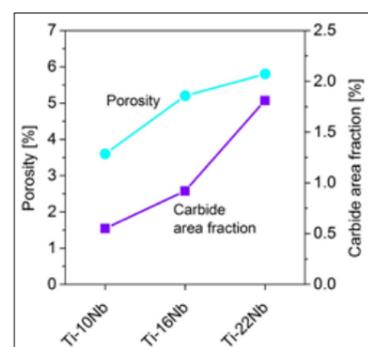


Fig. 9 Porosity and carbide area fraction in as-sintered Ti-(10-22)Nb alloys [6]

in as-sintered Ti-15Mo alloy even though the alloy contained only 0.032% C.

The paper by Thomas Ebel *et al.* [6, 7] focused on carbide formation in as-sintered Ti-(10-22)Nb (in wt.%). Direct evidence was presented of the influence of titanium carbides on the mechanical properties of as-sintered Ti-Nb alloys. Table 2 lists the impurity levels of the powders used for fabrication and the as-sintered Ti-(16-22)Nb samples made by MIM. The resulting carbon content is about 0.06% in each alloy, compared to a much lower carbon content of the powders. Fig. 8 shows the mechanical properties of the as-sintered Ti-10Nb, Ti-16Nb and Ti-22Nb alloys with and without hot isostatic pressing (HIP). The porosity level in each as-sintered alloy is plotted in Fig. 9. The results obtained after HIP suggest that the significant

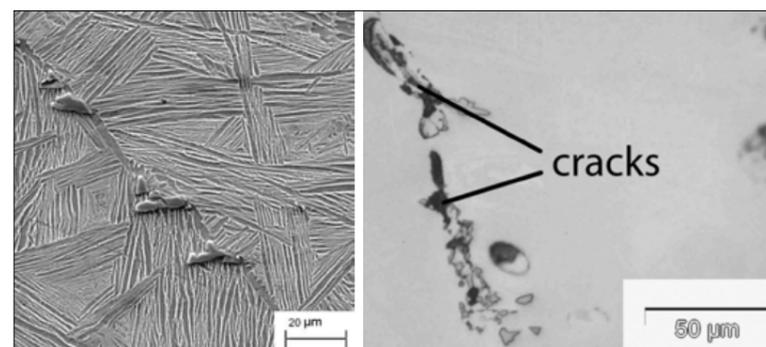


Fig. 10 [a] Carbides in as-sintered Ti-10Nb alloys and [b] cracks observed in fractured tensile samples of as-sintered Ti-10Nb alloy parallel to the tensile direction [6, 7]

drop in tensile ductility is not due to porosity. Detailed microstructural examination revealed the presence of titanium carbides in each as-sintered alloy (Fig. 10a). In addition, the area fraction of the carbides increased with increasing Nb content (Fig. 9). Cracks were found to initiate from and propagate along grain boundary carbides (Fig. 10b). Thermo-Calc predictions revealed that increasing Nb content from 10% to 22% can substantially reduce the solubility limit of carbon in  $\beta$ -Ti (Fig. 11). It was concluded that carbide formation is a critical issue for PM Ti-(10-22)Nb alloys in terms of embrittlement.

In summary, control of carbon appears to be more important than control of oxygen for PM Ti alloys that have a much reduced solubility limit of carbon due to alloying with beta stabilisers such as Mo and Nb.

#### Scavenging of oxygen and chlorine by rare earth boride and silicide during sintering of titanium and its alloys

This presentation by Y F Yang, S D Luo, G B Schaffer and M Qian [8] of the University of Queensland, Australia, showed that  $\text{CeSi}_2$  can be an effective scavenger of oxygen and chlorine for PM titanium and titanium alloys during sintering. Fig. 12 shows the formation of cerium oxides and cerium oxychlorides in as-sintered Ti-0.5CeSi<sub>2</sub> (wt.%).

The silicon introduced through the addition of 0.3-0.5%  $\text{CeSi}_2$  is limited to  $\leq 0.15\%$  (less than solubility limit of Si in  $\alpha$ -Ti, 0.45%) and its addition improves sintered density. As a result of the scavenging of oxygen together with chlorine, the ductility of the as-sintered titanium and titanium alloys was increased by about 60%. Cerium silicide shows

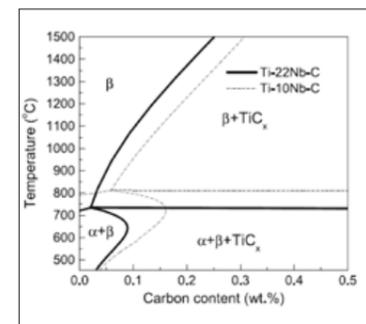


Fig. 11 Pseudo-binary phase diagrams of Ti-10Nb-C and Ti-22Nb-C alloys predicted using Thermo-Calc software [6]

the potential as an inexpensive solution to the scavenging of oxygen and chlorine for PM titanium and titanium alloys.

#### Preparation and application of porous titanium

Prof Huiping Tang, Director of the State Key Laboratory of Porous Metal Materials, Northwest Institute for Non-ferrous Metal Research (NIN), Xi'an, China, reviewed the preparation and applications of porous titanium in her keynote address "Preparation and Applications of Porous Titanium: An Overview." The last decade has clearly seen a significant increase in both the number of publications (Fig. 13a) and the number of patents (Fig. 13b) on porous titanium [10, 11].

Porous titanium can be produced via a variety of methods including the use of traditional PM processes, such as loose powder sintering, die compaction, Cold Isostatic Pressing, powder rolling and powder extrusion, and Additive Manufacturing or 3D printing. Table 3 lists the characteristics of the various methods discussed by Prof Tang. Either irregular or spherical titanium powders can be used, and a uniform pore structure is achievable in most cases. Fig. 14 shows typical microstructures of porous titanium fabricated using traditional PM processes with irregular titanium powder and spherical titanium powder. The major application fields

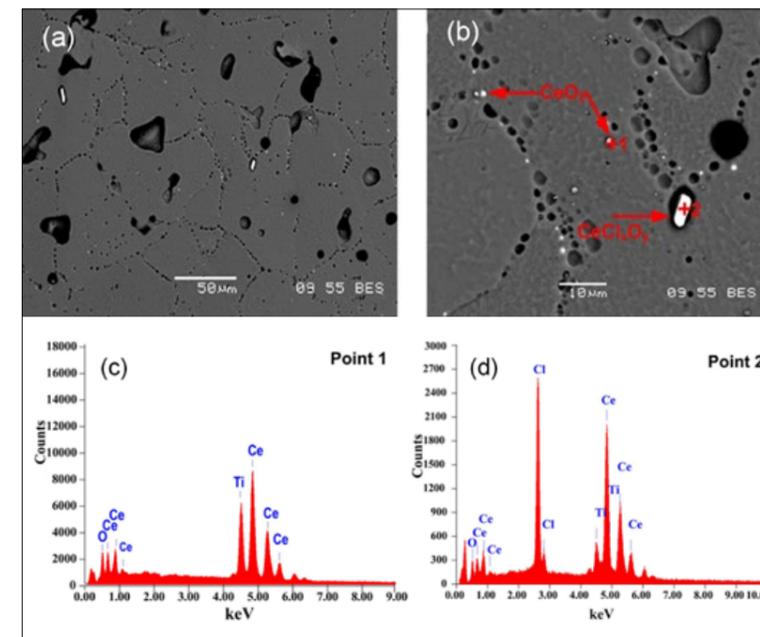


Fig. 12 As-sintered microstructure of [a] CP-Ti-0.5CeSi<sub>2</sub> (sintered at 1623 K for 120 min); [b] is a magnified view of [a]; [c] is spot EDS of a CeO<sub>2</sub> particle (point 1) in [b]; and [d] is spot EDS of a CeCl<sub>x</sub>O<sub>y</sub> particle (point 2) in [b] [8, 9]

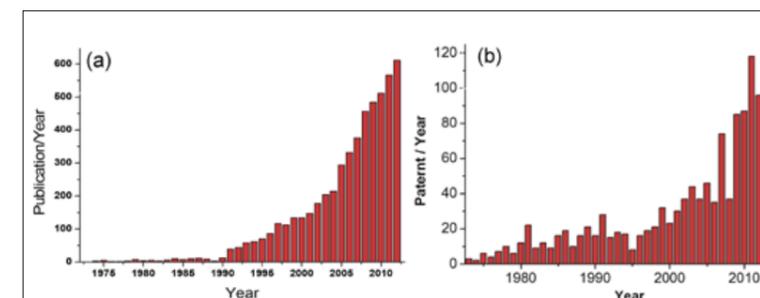


Fig. 13 [a] Annual publications on porous titanium since 1970 (Web of Science data, searched using key words of "porous titanium" or "titanium foams") and [b] annual patents on porous titanium (searched from databases of <http://patft.uspto.gov>, <http://worldwide.espacenet.com> and <http://www.pss-system.gov.cn> using key words of "porous titanium" or "titanium foams") [10, 11]

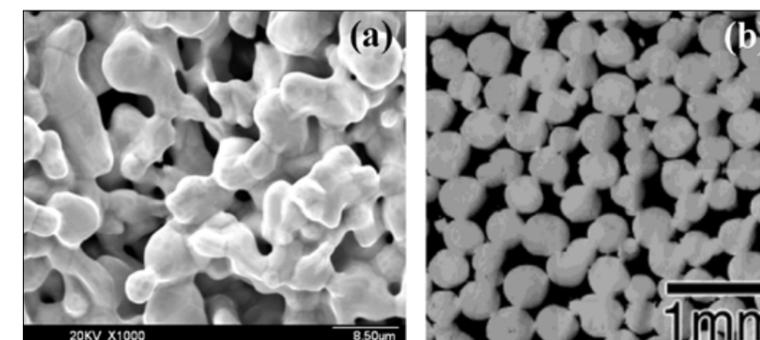


Fig. 14 Typical microstructures of porous titanium by traditional powder metallurgy processes using [a] irregular titanium powder and [b] spherical titanium powder [10, 11]

Methods	Pore structure	Porosity	Pore size	Commercial applications	
Traditional PM techniques	Uniform	≤60%	Micro pores	Yes	
Removal techniques	Space holder	Bimodal	40%-80%	Macro pores 200-500 μm Micro pores: <50 μm	Yes
	Replication		≤90%	Macro pores 0.5-1 mm Micro pores: <50 μm	Yes
Additive Manufacturing	Programmable	≤95%	≥0.5 mm	Yes	
Gas Entrapment	Open & closed	≤45%	Micro pores	No	
Metal Injection Moulding	Open & closed	≤45%	Micro pores	No	
Sintering of hollow Ti spheres	Open & closed	≤90%	Macro pores: 200-500 μm	No	

Table 3 Methods for preparing porous titanium by the pore structure created [10, 11]

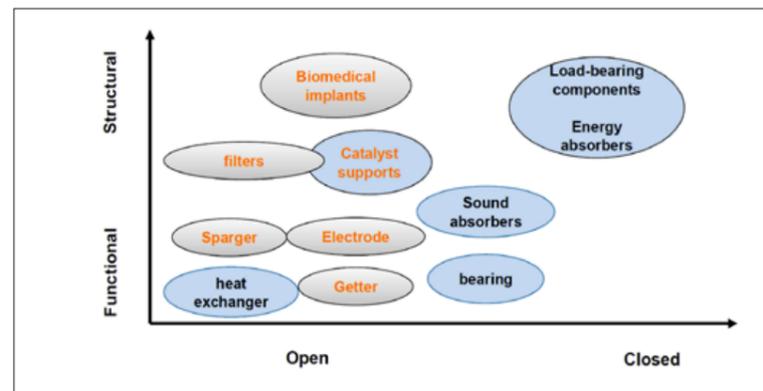


Fig. 15 Applications of porous titanium [10]

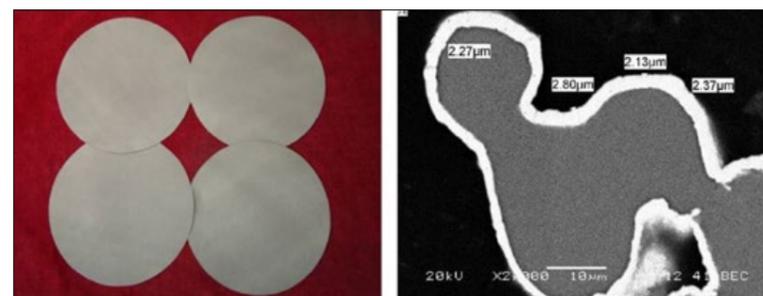


Fig. 16 Commercial porous titanium plates electroplated with platinum produced by NIN [10, 11]

of porous titanium are summarised in Fig. 15

Work on porous titanium at NIN started in 1965 and the persistent effort eventually led to the establishment of the State Key Laboratory of Porous Metals at NIN in 2007, the only one of its kind in China. To date, the State Key Laboratory of Porous Metals and its spin-off companies can produce a wide variety of porous titanium products with different degrees

of porosity, shapes and dimensions using conventional PM processes, including seamless porous titanium tubes up to 1500 mm long 200 mm in diameter and porous titanium plates/discs up to 400 mm in width/diameter with thickness being varied in the range of 0.3 mm to 4.0 mm.

Other commercial porous titanium products developed by NIN include porous titanium plates electroplated with platinum, used as current

Thickness of current collectors	0.8-2 mm
Porosity	20-50%
Pore size	5-30 μm
Particle size	25-250 μm
Gas permeability	10 <sup>-13</sup> -10 <sup>-11</sup> m <sup>2</sup>
Specific electric resistance	5-10mΩ·cm

Table 4 Typical parameters of porous titanium used for PEM water electrolyzer cell [10]

collectors in polymer electrolyte membrane (PEM) water electrolyzer cells (Fig.16 and Table 4), porous titanium used as spargers in oxygenators and water purification systems and those used as getters in many advanced electronic devices.

Since 2007 NIN has also developed a number of porous titanium parts for dental and orthopaedic applications using both conventional PM processes and selective electron beam melting (SEBM). In particular, porous titanium parts with a bimodal pore distribution manufactured using SEBM show some unique attributes. Along with the technological advances in additive manufacturing, porous titanium products are expected to find more and more industrial applications in the future according to the speaker.

### Titanium Additive Manufacturing in New Zealand

Ian Brown of Advanced Materials Group, Callaghan Innovation, Lower Hutt, New Zealand, and Warwick Downing of Titanium Industry Development Association Inc (TiDA) introduced the New Zealand Titanium Technologies Platform and the Additive Manufacturing (AM) business of titanium in New Zealand [12].

Funded by the Ministry of Business, Innovation and Employment (MBIE), the goal of the Titanium Technologies Platform is to create a multi-company, multi-sector manufacturing base for high value titanium-based export products. TiDA and subsidiary company Rapid Advanced Manufacturing Ltd (RAM), now have two Selective Laser Melting machines running from their facility in Tauranga, New Zealand. These machines are two of the few machines commercially available to offer 3D manufacturing in Ti powder and Stainless Steel, with more machines and alloys to follow.

Several carefully designed titanium products have been additively manufactured and marketed. The titanium knives made by TiDA and Victory Knives were one such example. TiDA was first contacted by Victory Knives who needed to urgently design and manufacture light-weight, yet super sharp safety knives for the sailors on board Americas Cup challenger, Emirates Team New Zealand. The entire project was an urgent six-week collaboration between TiDA, Page Macrae Coatings and Victory knives to produce a unique knife design that was ready for racing.

John Bamford, of Victory Knives, set about designing a state of the art small lightweight knife that was strong enough to cut through marine ropes. Bamford and the team at TiDA designed a knife and sheath, 3D printed in Ti-6Al-4V alloy powder, that was then super coated, to produce a knife that can cut super strong marine ropes in one blade stroke compared to the existing knife which took ten strokes (Fig. 17).



Fig. 17 Selective laser melting (SLM) at work: additively manufactured titanium knives used by sailors on board Americas Cup challenger, Emirates Team New Zealand [12]



Fig. 18 Selective laser melting (SLM) at work: a dog jaw bone replacement manufactured by TiDA. The customised jaw bone was printed from a CT scan and implanted, with a very happy pup back eating within 12 hours [12]

"The Americas Cup Safety Team were ecstatic with the fast turnaround and were amazed at the strength of the new product," stated Bamford. Such was the success this knife will be available to consumers in the near future.

TiDA's pivotal success has been in turning design projects and prototypes into manufactured parts, far quicker than ever possible with traditional manufacturing options. This fast turnaround saved the life of a dog needing an urgent jaw bone replacement. Axia Design, a New Zealand based design company, contacted TiDA with a CT scan that needed to be urgently printed in Ti-6Al-4V (Fig. 18).

The customised jaw bone was printed from a CT scan and implanted, with a very happy pup back eating within 12 hours. Surgeons commented that the implant was as close to the natural shape as ever seen, which enabled a quick recovery. This success has led to a second dog receiving a lifesaving

implant, with a view in the near future to manufacture human implants. "Humans are the next logical step for mass customisation," stated Warwick Downing, TiDA CEO. "In theory, within a few days, we could have a hip joint that is customised for the patient with a designed porous structure allowing bone growth, starting to eliminate some of the problems that traditional joints may have."

The titanium weapons suppressors designed and manufactured by TiDA and its partner Oceania Defence are another success of the additive manufacturing story of titanium in New Zealand. These uniquely designed and additively manufactured titanium weapons suppressors offer excellent performances while being 50% lighter than conventional steel weapons suppressors (Fig. 19). Design played a key role in the success. Several such products have gone into batch productions.

Client success has driven industry firsts with the technology used to solve engineering design issues.



Fig. 19 Selective Laser Melting (SLM) at work – titanium weapons suppressors designed and additively manufactured by TiDA and its partner Oceania Defence [12]

With products moving from prototyping into production, new titanium products are now being additively manufactured and are in the marketplace already. To promote the growth in research in this arena, TiDA has recently announced a Research and Technology Partnership with Callaghan Innovation, a New Zealand based Government Research Organisation. "Following the investment Callaghan has recently provided in Selective 3D machinery, New Zealand is leading the way in research that supports the growing manufacturing industry in titanium alloy metals," stated Downing. The resulting outcomes will be driven into the commercial arena, with both TiDA and Callaghan having established close links within this growing Industry. Currently, designers, engineers and researchers are working closely with clients on several titanium projects in New Zealand. Led by TiDA, the Additive Manufacturing business in New Zealand has clearly shown a good start.

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## Vacuum sintering and Low Pressure Carburising of Powder Metallurgy components

In order to improve the efficiency of the sintering and heat treatment of PM alloys for high load bearing applications such as PM transmission gears, France's ECM Technologies has developed an innovative new combined batch vacuum sintering and Low Pressure Carburising furnace. The system enables the complete thermal treatment of PM components in one step, as well as optimising the quenching and case hardening processes. ECM's Hubert Mulin, Yves Giraud and Jean-Jacques Since and Höganäs AB's Mats Larsson report for *Powder Metallurgy Review*

The sintering and heat treatment process for Powder Metallurgy (PM) materials such as Höganäs AB's Astaloy™ range requires four steps; de-waxing, high temperature sintering, carburising and surface hardening. These steps are usually achieved in dedicated atmospheric furnaces for sintering and heat treatment respectively, leading to intermediate handling operations and repeated heating and cooling cycles. Chromium alloys in particular are difficult to heat treat because chromium oxides form easily, which has a negative impact on mechanical properties.

This article presents the concept of the multi-purpose batch vacuum furnace, able to achieve all these steps in one unique cycle. The multiple benefits brought by this technology are presented and relate to considerations such as part quality, flexibility, cost savings and environmental benefits. The primary objective is to use this technology to manufacture high load transmission gears using PM materials.

#### Conventional processing steps

Today, the usual way to manufacture PM parts such as gears is divided into several steps. When the gear is shaped by die compaction, four heat treatment stages must be carried out in order to achieve all the required properties. These four stages are de-waxing, sintering, carburising and quenching. De-waxing and sintering

are typically performed in continuous belt or walking beam furnaces. The first operation, de-waxing, is intended to remove the lubricants. This is a critical step because if removal of the lubricant is incomplete, defects such as contamination or blistering may arise. Belt or walking beam furnaces are able to sinter directly after de-waxing in a single run which presents an advantage compared to the use of two dedicated furnaces. After



Fig. 1 Multi-purpose vacuum furnace installed at the Höganäs AB pilot plant

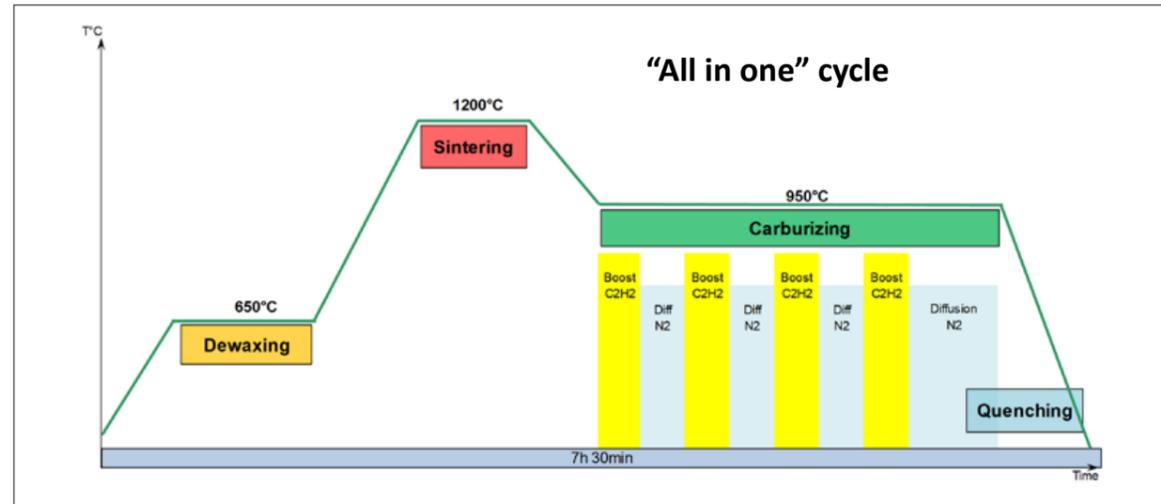


Fig. 2 Complete Treatment cycle for PM parts

sintering, the carburising treatment for a tailored case hardened profile and is generally followed by hardening, using oil quench or High Pressure Gas Quenching (HPGQ).

Typically the conventional carburising treatment is carried out in batch type furnaces. This requires intermediate handling between sintering and carburising, as well as washing and drying operations after oil quenching.

This article reviews the concept of a multi-purpose furnace which can perform the four successive steps (de-waxing, sintering, low pressure carburising and quenching) in one continuous cycle. The article will compare the traditional method of sintering and carburising to this concept.

### Furnace concept

Trials have been carried out with an industrial furnace (Fig. 1) owned by Höganäs AB, Sweden, and designed and manufactured by ECM Technologies, France. The furnace comprises two chambers, one heating cell and one gas quenching cell, separated by an intermediate leak tight and insulated door.

The front chamber is used as an airlock to load and unload the charge and also as high pressure gas quenching unit for hardening the parts. The second chamber, called a "heating cell", is the furnace itself where parts are heated and carburised. It is always maintained under low pressure (1 to 20 mbar) with back fill of a protective atmosphere.

Each chamber is equipped with independent vacuum circuits and can be operated independently. The vacuum circuits are designed to maintain the correct partial pressure inside the heating chamber and are equipped with a wax trap to collect the lubricant during the de-waxing cycle.

One internal device transfers the load back and forth between the two chambers. A service door facilitates the access to the heating chamber for periodic temperature mapping or maintenance. Fig. 2 shows the complete treatment cycle in the multi-purpose furnace.

The de-waxing step is performed at around 650°C under low pressure. At this temperature, the lubricant evaporates and is pumped out by



Fig. 3 Part etched with Nital 2%

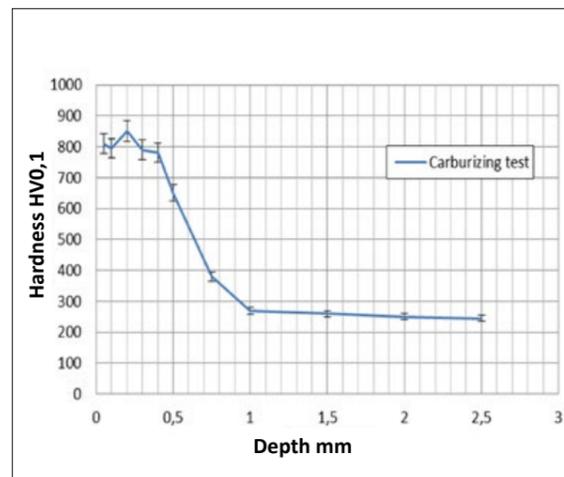


Fig. 4 The hardness profile of the part shown in Fig. 3

the vacuum circuit. It is entirely removed from the parts and collected in the wax trap. Then, the temperature is increased to reach the desired sintering temperature (up to 1250°C). At this stage, metallic bonds between particles are formed. After sintering is completed, temperature is decreased to reach the desired carburising temperature (900 to 1000°C). Then follows the patented Infracarb process [1], where the Low Pressure Carburising cycle, with alternating injections of acetylene and nitrogen, is carried out. The number of injections and cycle time is adjusted depending on the desired case depth. After final diffusion, the load is transferred back to the front chamber and is quenched with nitrogen gas at up to 20 bars. Metallurgical transformation occurs during the rapid cooling and these enhance the mechanical properties of the parts.

As an example, a 300 kg load containing small spur gears was carburised at 965°C for 74 min and the effective case depth at 550HV obtained was 0.6mm (Figs. 3, 4).

### Comparisons with conventional processing

#### De-waxing

When the parts reach a temperature above 400°C, lubricants used during die compaction evaporate. Typical lubricants, such as Amide wax, are totally decomposed between 400 and 500°C.

In the pre-heating zone of belt type furnaces, lubricant vapours are mixed with the protective atmosphere and burnt as exhaust. With conventional belt furnaces, the dewaxing time and the sintering time are linked and defined by the belt's length and the belt's speed.

In the multi-purpose furnace, partial pressure of nitrogen (1 to 20 mbar) preserves the parts from oxidation. The vaporised lubricant is condensed and collected in a trap in the vacuum circuit. The dewaxing time can be increased or decreased easily according to the load's weight.

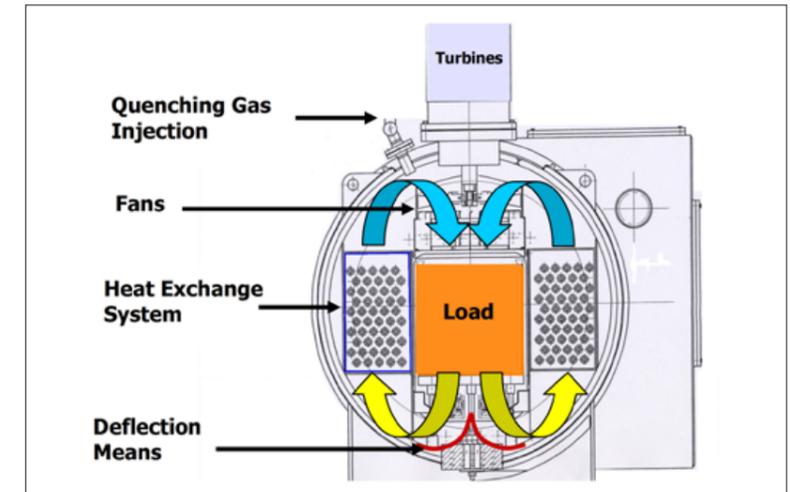


Fig. 5 Principle of gas quench chamber

The trap reduces the contaminants in the atmosphere and keeps the vacuum circuit clean. Vacuum processing is well known to be an efficient way to dewax parts and delubrication under vacuum is thus beneficial to the dewaxing rate.

chromium alloys, are prone to oxidation and precise control of atmosphere quality is required. Due to their open pore system, PM compacts are more prone to oxidation than wrought steel and residual oxidation can introduce defects

**“The absence of handling between operations in the multi-purpose furnace guarantees that the parts will not be affected by contamination or damaged between sintering and heat treatment cycles”**

#### Sintering

Many parameters are crucial during sintering, especially the time and temperature of sintering, the heating rate, the design of the fixtures and the arrangement of the parts on the trays, to name just a few.

A sintering temperature of around 1200°C is typically the maximum operating limit for traditional PM furnaces and reaching this temperature impacts on the lifetime of the heating elements. In vacuum furnaces, the lack of oxygen permits the use of graphite rods as heating elements. Graphite rods are very stable mechanically as they do not bend with temperature and their lifetime is not influenced by the working temperature.

PM compacts, especially

during sintering. A continuous furnace uses a reducing gas such as hydrogen to protect the parts, which is not necessary under vacuum. All the other parameters such as heating rate and sintering time can be easily adjusted to obtain the best sintering process.

#### Carburising

The absence of handling between operations in the multi-purpose furnace guarantees that the parts will not be affected by contamination or damaged between sintering and heat treatment cycles.

Frequently, batch furnaces are used for carburising PM parts. Carbon enrichment is controlled by O<sub>2</sub> sensors or CO/CO<sub>2</sub> ratio. In the new furnace, the carburising phase is

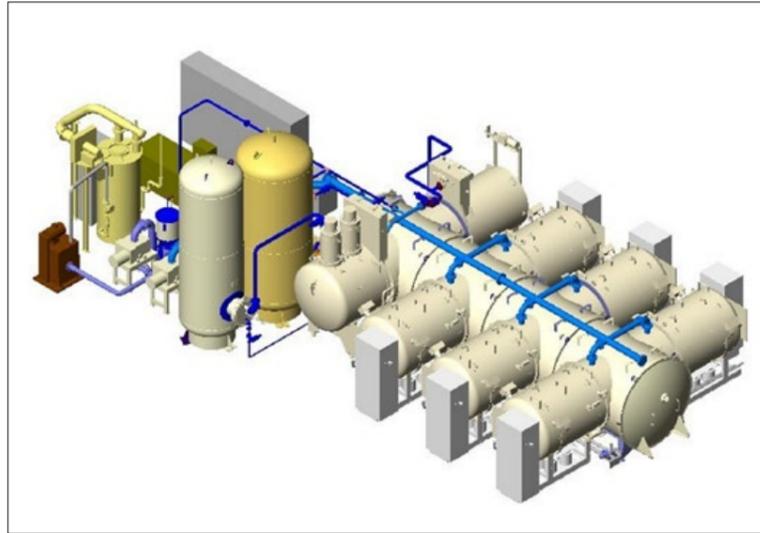


Fig. 6 Modular sintering multi-cell line type ICBP

completely controlled by "Infracarb", the patented LPC process with acetylene gas. The case depth and the carbon profile are simulated and adapted for porous materials. Low Pressure Carburising processes can be achieved at any temperature up to 1050°C. The carbon enrichment and the diffusion time can be controlled separately to achieve the required microstructure.

The cycle is shortened and the diffusion is faster than in an atmospheric carburising furnace. Moreover, there is no internal oxidation of the parts. The amount of carburising gas injected in the chamber is optimised to ensure that every part is correctly carburised. Injection is done by short boosts in order to minimise the formation of gas constituent, which leads to volatile organic components, and atmospheric rejects are reduced.

#### Quenching

The high pressure gas quench allows a range of appropriate cooling speeds (from 1 to 10°C/sec) to be reached. Fig. 5 shows the principle of the gas quench chamber.

Cold nitrogen gas is pushed down through the load, cooling it, and transferring the heat to the water when passing the heat exchanger. Gas quenching permits high flexibility and more repeatable results than oil

quenching because there is no boiling or vapour formation around the parts. With a gas quench, the pressure and also the turbine's speed can be programmed for each cycle in order to adjust the cooling rate for every type of load, thus minimising part distortion. The parts exit the furnace clean and a washing operation is not necessary.

#### Process cost estimation

For typical production of 300 kg net/hour, a modular vacuum installation with multi heating cells is required (Fig. 6). Based on an engineering cost estimate, the cost of sintering and heat treatment of parts is about 0.5€/kg. The precise cost depends on the geometry and hardenability of different kind of parts.

The main saving factor comes from the fact that carburising and gas quench steps are carried out in situ in the de-waxing /sintering equipment. The maintenance cost per year for an ICBP type furnace is around 4% or lower of the investment cost. The high modularity and restricted footprint of the furnace is also an advantage.

The global energy balance cost is positive against the conventional furnace because there is no multiple cool down and reheating of the parts for each step of the process. The

modularity of the heat treatment installation allows for further production extension by the simple addition of heating cells on the main frame without investment on a second line.

#### Conclusions

Studies have been carried out in partnership with Höganäs AB on different alloys including chromium alloys. Positive results have been achieved on:

- Control of case profiles
- Control of core hardness with base carbon content and cooling speed variation.

Low Pressure Carburising has been proven very suitable for control of process parameters without oxidation. The multi-purpose furnace potentially offers an improvement at every stage of the PM production process: It will be the tool for further optimisation to improve mechanical properties like fatigue strength; distortion reduction, and validation of the whole process for the production of high performance PM gears.

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- [1] U. S. Patent No. 6,065,964, 23 May 2000

## Non-ferrous powder production: Manufacturing methods and properties of copper, aluminium, titanium and nickel powders

This extensive review of key non-ferrous powders used in the Powder Metallurgy industry focuses on the production and properties of copper, aluminium, titanium and nickel powder. Professor Oleg Neikov, from the Institute for Problems of Materials Science in Kiev, Ukraine, identifies the methods available for manufacturing these powders including detailed descriptions of the atomisation process, mechanical grinding, and electrochemical methods. A number of key powder properties are also listed.

Non-ferrous powders are produced by mechanical, chemical, and electrochemical methods. The comminution of solid metals and alloys is done by milling in tumbling, vibration and planetary mills, attritors and jet mills. The dispersion of melts includes granulation and atomisation. General chemical methods comprise the precipitation from a gaseous phase, carbonyl and reduction methods. The electrochemical methods include electrolysis.

In this review we focus on production and properties of copper and copper alloy powders, aluminium, nickel, and titanium powders produced by various widely used and also new advanced methods. Atomisation is the most common method that allows the production of powders over a wide range of compositions and in a wide variety of powder particle sizes from a few microns to a few mm. Chemical and electrolytic methods are widely used for producing high-purity and fine

powders. One of the basic methods for the production of nanopowders is extraction from the gaseous phase. The disintegration (milling) of solid non-ferrous metals has significant limitation due to the ductility of most of them. Nevertheless, milling in high-energy apparatus such as attritors finds many applications for the mechanical alloying of powders.

#### Atomisation

Atomisation has become the prevailing production method for many non-ferrous metals and their alloys, due to the simplicity of the breakup of a liquid stream into fine droplets. Atomisation also allows the manufacture of new types of rapidly solidified powders, attaining properties not achievable by traditional cast technology [1]. The general types of atomisation processes include:

- Jet atomisation where a liquid metal is dispersed into droplets by impingement of high-pressure jets of gas, water or oil

- Centrifugal atomisation, where a liquid stream is dispersed into droplets, flakes, or ribbons by a rotating spinning disk, spinning cup, or consumable electrode
- Ultrasonic atomisation, where a liquid metal film is subjected to ultrasonic vibration

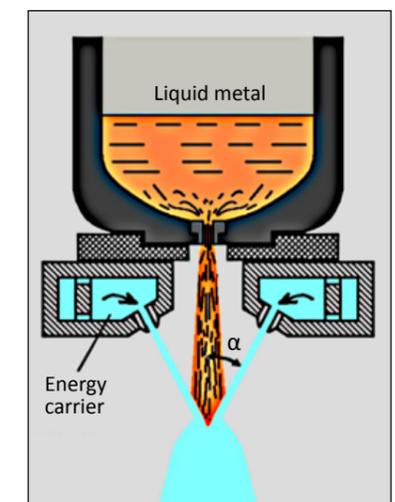


Fig. 1 Free-fall atomiser with water cone configuration

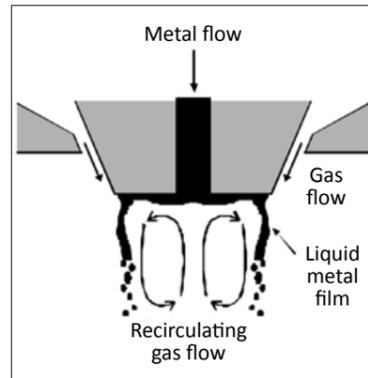


Fig. 2 Close-coupled atomiser

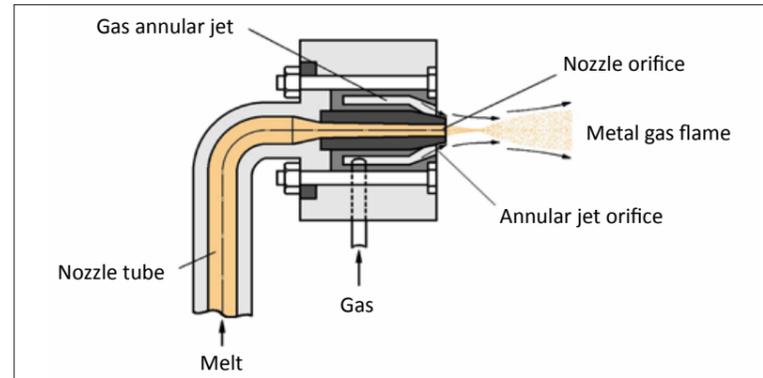


Fig. 3 Aspiration pressure atomiser type

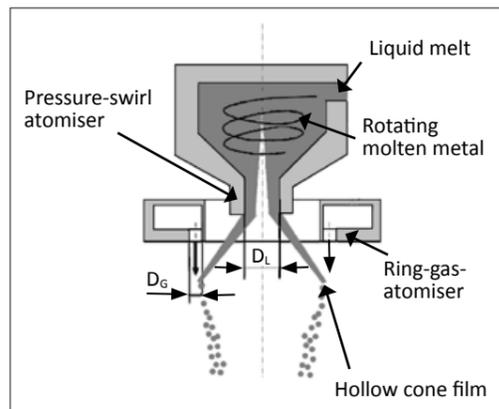


Fig. 4 Pressure-swirl hybrid prefiling atomiser

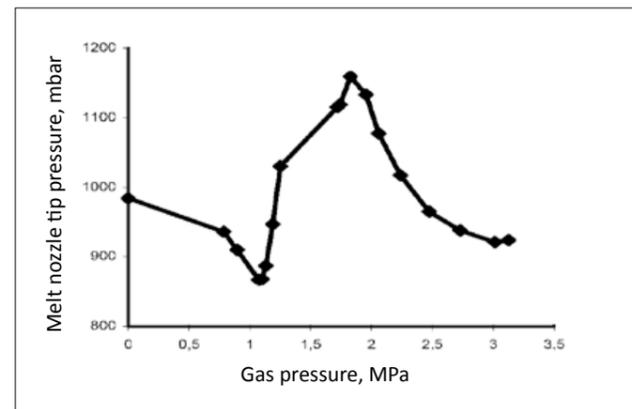


Fig. 5 The pressure formation on the nozzle tip is dependent on gas pressure in close coupled nozzle design (by the constant protrusion length 10 mm)

- Vacuum atomisation, where molten metal heavily saturated with a gas is atomised in vacuum
- Impulse atomisation, where impulses are mechanically applied to the melt

Also of interest is the CANOPUS technique, a rapid solidification method based on small-scale vapour explosion [2].

**Gas atomisation**

Gas atomisation (GA) is a process, in which the liquid metal is dispersed by a high-velocity jet of air, nitrogen, argon or helium. GA is used for commercial production of copper and its alloys, aluminium and its alloys, magnesium, zinc, titanium and its alloys, nickel-base alloys, cobalt-base alloys, lead, tin, solders, precious metals, refractory metals, and beryllium powders.

Many atomiser designs have been developed and can be classified

as 'free fall' as outlined in Fig. 1, 'confined' (or close-coupled atomiser (CCA)) represented in Fig. 2, and 'internal mixing', where the gas and the melt are mixed together before entry into the atomising chamber [3]. Fig. 3 shows an aspiration pressure nozzle.

In confined designs the circulation is created by the gas flowing coaxially to the tundish nozzle and the coherent melt stream causes, due to aspiration force action, the molten metal to spread onto the face of the nozzle edge with film formation, where it is sheared by the flowing gas. Film formation is an important condition for effective atomisation of the melt. Fine particles are formed as a result of the primary breakup of the melt film upon its interaction with supersonic gas flow at the nozzle edge. Therefore similar devices are also classified as prefiling atomisers.

A pressure-swirl hybrid prefiling

atomiser combines pressure-swirl atomisation and gas annulus atomisation. In the first stage a film is generated followed by gas jet atomisation in the second step [4]. The pressure-swirl-metal chamber, atomisation chamber, and gas-recirculation system are the main components of the powder atomisation unit (Fig. 4). Due to overpressure, the molten metal flows tangentially into the swirl chamber, leaves it through a small cylindrical hole ( $D_L$ ), and forms a swivelling hollow cone film of molten metal. The film is subsequently atomised by the gas jets through the orifices ( $D_G$ ).

Close-coupled atomisation nozzles have been the subject of numerous studies. The increased understanding of the atomisation process offers new ways to approach the problem of particle size control and atomisation efficiency [1].

Recently, high speed imaging techniques for studying gas atomisa-

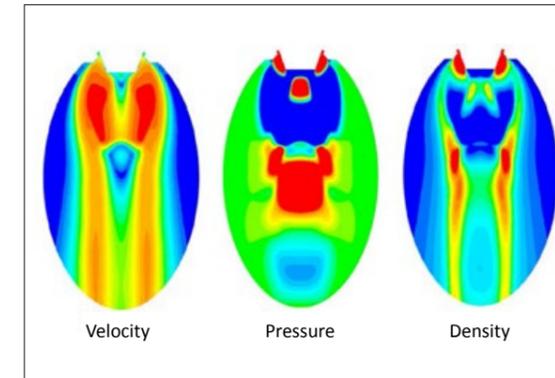


Fig. 6 Computational fluid dynamics model of the aspiration pressure nozzle type in the high pressure condition

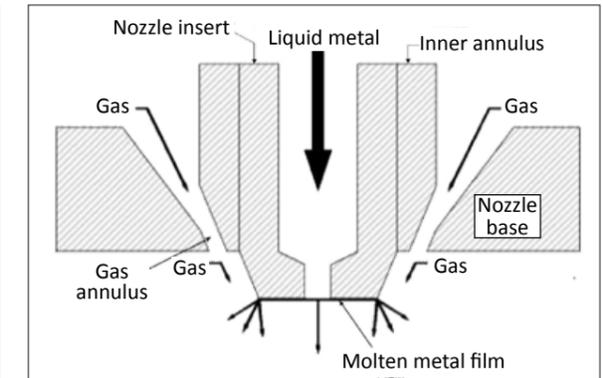


Fig. 7 Schematic of the cross section of a close-coupled atomiser annulus type nozzle

tion have been used. Techniques include both a high frame rate (18,000 fps) video camera to film a continuous sequence of images and a pulsed laser to obtain still images with an effective 6 ns exposure time [5]. The pressure value on the liquid metal nozzle tip is an important parameter in close-coupled gas atomisation. The pressure formation on the melt nozzle tip is dependent on gas pressure, as shown in Fig. 5 [1].

Computational fluid dynamics (CFD) and volume of fluid (VDF) techniques have been used to study the close-coupled atomisation process. Three different types of nozzle setup were investigated by the instrumentality of the above methods - aspiration pressure nozzle and back pressure style nozzles at low and high pressure conditions [6]. Modelling of the aspiration nozzle type shows a typical stagnation area several nozzle diameters away from the liquid metal orifice. It also shows a so-called pressure punch on the central axis close to the liquid metal orifice (Fig. 6). High pressure in the gas punch squeezes the melt to the side and forms the melt film and hollow centre in a close-coupled process.

One of the incidences that can occur during the process is the wetting of the atomisation nozzle by the liquid metal, a so called 'lick back'. Fig. 7 is a schematic of the cross section of an annulus type

nozzle showing the normal path for the melt and also the melt paths taken that define partial and full lick back situations [7]. However, lick back can also appear as a result of atomiser design features. Other factors may also lead to lick back, such as gas pressure regime, surface tension of melt and melt reaction with the insert's surface. If the melt makes contact with the inner annulus the nozzle will be destroyed.

The effect of different atomiser designs has been the subject of many studies, for example [7-10]. Studies, based on water spray modelling [7], showed that a combination of gas pressure, nozzle insert angles and extension can lead to lick back. This has been tested by examining the atomisation plume at three nozzle tip angles (Fig. 8) and at two atomisation pressure levels (arbitrarily low and high). Additional tests were performed with the insert recessed and

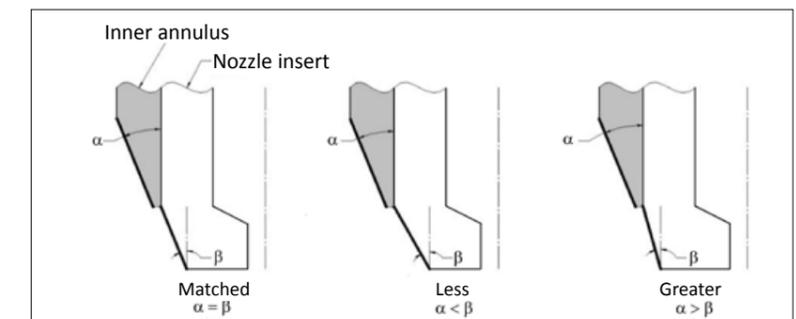


Fig. 8 Schematic of the angle matching between the atomisation nozzle and nozzle insert.

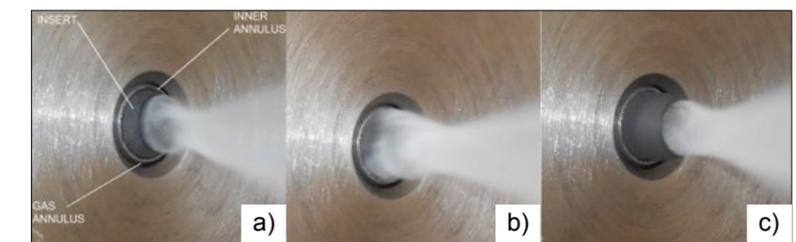


Fig. 9 Configuration of atomisation plume: a - the angles of the atomisation nozzle and insert are matched, at high gas pressure; b - the angle of the atomisation nozzle is less than the insert angle, at high gas pressure; c - the angle of the atomisation nozzle is greater than the insert angle, the nozzle insert is in a protruding placement, at low gas pressure

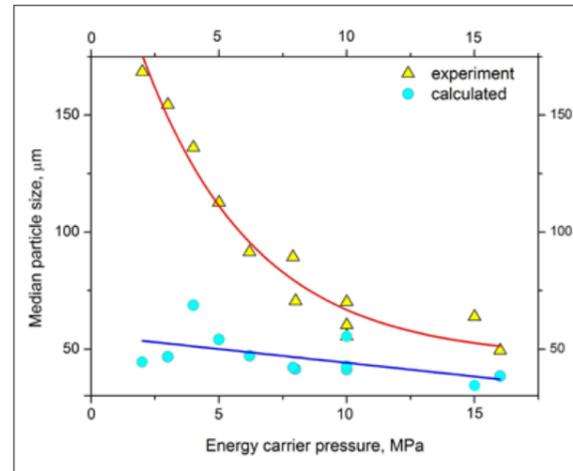


Fig. 10 The dependence of powder median diameter on energy carrier pressure at 1273 K (1000°C) temperature

protruding from its normal placement in the atomisation nozzle.

Fig. 9 shows three typical atomisation conditions: partial (a), total (b) and without lick back (c). In the case of the matched angle configuration (Fig. 8) the low pressure level shows total lick back. The high pressure level shows partial lick back. In this case increasing the pressure decreases the extent of lick back. When the nozzle jet angle is less than the nozzle insert angle total lick back is at both pressure levels. In the case where the angle of the atomisation nozzle is greater than that of the insert lick back does not occur at the low pressure level and only partial lick back occurs at the high pressure level. Among the compared nozzle insert placements the atomisation nozzle with greater angle in a protruding placement appeared more preferable.

Advanced understanding of the CCA technique allows the production of metal powders in a wide range of average particle sizes, including the production of coarse powders with a narrow standard deviation [9, 11, 12].

In conventional inert gas or air atomisation, typical metal flow rates through single orifice nozzles range from about 1 to 90 kg/min. The capability of plants varies from very small laboratory units to immense plants such as ANVAL Atomiser 1 in Sweden, designed for the production of large tonnages of superalloy and other alloy powders. Melting takes place in two 5.5 ton induction furnaces and a plasma heated tundish is used. Due to the height of the tower, powders with a diameter of up to 1mm can be produced. Very fine powder can also be produced for applications such as MIM. In conventional atomisers, typical gas flow rate ranges from 1 to 50 m<sup>3</sup>/min at pressures ranging from 350 kPa to 4 MPa. The superheating of molten metal (the temperature differential between the melting point and the temperature at which the molten metal is atomised) is generally about 75 to 150°C. In gas atomisation with inert gas, the cost of gas consumption is significant, and a means of circulation to promote gas reuse is desirable, especially in large-scale facilities.

### Water atomisation

Water atomisation (WA) is used for the commercial production of iron, copper and its alloys, nickel-base alloys, zinc powder and precious metals. WA is less expensive than other methods of atomisation and allows powders to be produced at a rate of 1 to 500 kg/min using a single nozzle. The main limitation of WA is powder purity, especially for metals and alloys inclined to oxidation. However, because of the higher cooling rate of the melt by WA, the average thickness of particle surface oxides is similar to that in gas atomised powders. This can be seen in particular in water atomised aluminium alloys [13] discussed later in this review.

The major components of a conventional water atomisation plant are the melting furnace, a tundish, an atomising chamber, a water pumping/recycling system, and the dewatering and drying units.

Conventional installations usually operate with water pressure in the range of 5 to 20 MPa, producing powders with median particles sizes of 30 to 150 µm. Significantly higher water pressures of 50 to 150 MPa are used to manufacture finer powders with median particle sizes of 1–20 µm. These finer powders are used for MIM, metallic bonds in diamond tools, PM bearings, paints, coatings and pastes.

The atomisation of melt by means of the steam explosion (CANOPUS) method allows, according to the data [2], an achievable cooling rate of the melt of 10<sup>8</sup> K/s. This was achieved on a laboratory installation. Molten metal held in a crucible flows out through a small orifice in the base, drop by drop, at a set time interval. The drop of melt entering into the stream of cooled water (or water salt solutions), causes an explosive formation of steam and results in melt atomisation. For the production of Al<sub>89</sub>Fe<sub>11</sub> alloy, the cooling liquid used was a 25% water solution of calcium chloride. Powder particle sizes obtained were in the range of 30–400 µm. However, this method has not yet been tested beyond the confines of the laboratory.

Through the existing theoretically substantiated understanding of the nature of the water atomisation process [14–18], the creation of the calculation methods for determination of the process parameters for the powder production, with defined particle shape and granulometric composition, encounters large difficulties. This is due to complications of analytical descriptions of the hydrodynamic and heat exchanging processes by the contact of high pressure water jet with liquid metal. The research studies of water atomisation are not so numerous as in the area of gas atomisation.

An advanced empirical formula that takes into account basic factors of the atomisation process, is proposed by authors [16] and is written as:

$$d = 4.97 \frac{G_{Me}^{1.24} v_{Me}^{0.35}}{G_w^{0.3} \gamma_{Me}^{0.15} D_c^{1.03} \rho_{Me}^{0.56} \rho_w^{0.25} v_w^{0.70} v_w^{0.96} (\sin \alpha)^{0.96}}$$

where  $G_w$  is the melt mass flow (kg/s),  $v_w$  is the water jet velocity (m/s),  $G_{Me}$  is the water mass flow (kg/s),  $v_{Me}$  is

the kinematic viscosity of the liquid metal (m<sup>2</sup>/s),  $\rho_{Me}$  is the density of the liquid metal (kg/m<sup>3</sup>),  $\gamma_{Me}$  is the surface tension of the liquid metal (N/m),  $D_c$  is the liquid metal squirt diameter (m),  $\rho_w$  is the water density (kg/m<sup>3</sup>),  $v_w$  is the kinematic viscosity of the water (m<sup>2</sup>/s),  $\alpha$  is the jets apex angle.

The experimental and calculated dependence curves of the powder mass median diameter values on powder pressure in the 2.0–16.0 range at 1000°C are shown in Fig. 10. The calculated median diameter values are similar to the actual size at high pressure volumes. However, the difference increases when decreasing the water pressure. At 2.0 MPa the calculated particle sizes are around three times smaller than actual values.

### Mechanical grinding

Comminution is the oldest mechanical operation for size reduction of solid materials and an important step in many processes where raw materials are converted into intermediate or final products. It is the most widely used method of powder production for hard metals and oxide powders. Secondary grinding of spongy cakes of reduced oxide, electrolytic or atomised powders, is the most common milling process; hammer crushers and rod mills are used for this purpose.

Recently this technique has become ever more important in connection with the development of new advanced alloys by means of mechanical alloying. Thereby the purpose of grinding includes particle size reduction, particle size growth, particle shape change (to flake shape), agglomeration, blending of two or more materials or mixed phases, modifying or changing of properties of a material (density, flowability, or work hardening), non-equilibrium processing of metastable phases such as amorphous alloys, extended solid solutions, and nanocrystalline structures. The drawback of mechanical grinding

is a low output-input ratio. The energy efficiency of comminution is very low and the energy required for comminution increases with a decrease in produced particle size.

Vibratory, attrition, planetary and large diameter tumbling mills may be classified as high-energy mills and, as such, may be used effectively in solid state or mechanical alloying processes. Fluid energy mills are also related to high energy mills.

Fig. 11 shows the possibilities of typical size reduction equipment for grinding a material of a given feed size to a desired product size, when the purpose is comminution of a hard and/or brittle metal or ceramic material.

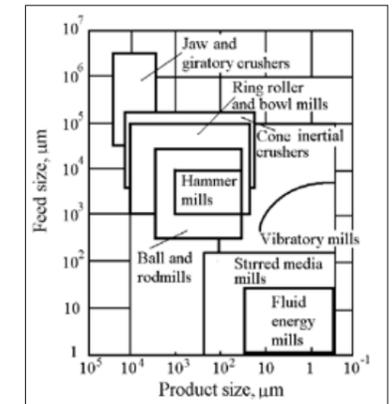


Fig. 11 Possibilities (with certain approaching) of typical size reduction equipment for grinding a material of a given feed size to a desired product size



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## Electrochemical methods

Electrochemical methods can be used to produce a variety of metal powders. They enable the production of powders with particle sizes ranging from a few nanometers (by the method of zone electrochemistry) to several millimetres (electrolysis of melts). The main properties of such powders are purity, dendritic shape of particles (Fig. 12) and good compressibility.

Electrochemical methods make it possible to control crystallisation (definite shape and size of powder particles) by selecting the process parameters: concentration of the metal and hydrogen ion exponent pH of the electrolyte, cathode potential, current density, temperature, and rate of circulation of the electrolyte, type and size of anode and cathode and their distance from each other, type and quantity of addition agents, and conditions of removing deposits at the electrodes.

Powders of the majority of metals such as copper, nickel, cobalt, cadmium, zinc and silver may be manufactured by electrodeposition of metals from aqueous solutions. This method is differentiated by the properties of the electro depositions: 'direct deposition' produces a friable or spongy deposits that can easily be disintegrated mechanically into fine particles, and 'brittle process' produces a dense, smooth, brittle layer that can be ground into powder. Both deposition types are achieved by controlling the suitable composition and operating conditions. The condition of electrodeposition impacts substantially on the dendritic deposition structure.

The mode of electrolysis at a continuous current load (galvanostatic) is realised in practice easily, but has failings. During electrolysis development of dendritic deposition occurs unevenly: speed of dendrite lengthening is diminished with time,

and superfine particles at the beginning of the process are transformed into rounded large spherulites. Such change of deposition structure is related to the decline of diffusive impediments because of development of growth of sediment surface.

Electrodeposition in controlled potentiostatic conditions allows the maintenance of the permanent level of diffusive limitations, speed of growth of sediment does not change therefore and dendrite structure is homogeneous [19]. An advanced control method for continuous video recording of the copper metal deposition growth was used. This technique is described below, in the chapter "Copper and copper alloy powders".

## Nanopowders

Powders with particle sizes less than 100 nm are referred to as nanopowders. Three types of nanoscale materials are recognised: nanocrystalline, agglomerated nanopowder, and nanosuspension.

The gas-phase methods are the conventional processes used for producing metallic nanopowders. These processes are based on the condensation of the supersaturated metal vapour in the presence of an inert gas such as argon, helium or nitrogen and produce high-quality powders. Evaporation of the material is performed by either electron beam, magnetron sputtering, or a laser, either in a resistance or induction furnace. When the vapour collides with the cold inert gas it forms the homogeneous particles. The particulate fog condenses on cooled substrates (crystallisers) in the vacuum chamber.

The main problems in production and processing of metallic nanopowders are caused by their high reactivity and explosiveness owing to very high specific area, and the agglomeration of particles. A novel technology for manufacture of semi-products directly from metal vapour, omitting the operation of producing nanopowder, is promising (Fig. 13) [21]. The simultaneous evaporating of several materials, mixing of their

vapour flows, followed by condensation on a substrate and subsequent thermomechanical treatment, allows the construction of materials with unique properties. This technology also enables the production of multilayer condensates. Laboratory and production units, developed by the EO Paton Institute, have some independent evaporators (water cooled crucibles) and corresponding electron beam guns for evaporation of three to four materials by a preset programme [21].

Ultrafine copper powders, produced by chemical precipitation from solution, find wide application in microelectronic components, multi layer ceramic capacitors (MLCCs), and  $\mu$ -Metal Injection Moulding (MicroMIM,  $\mu$ -MIM).

Micro-MIM is an advanced method of Metal Injection Moulding, using sub- $\mu$ m sized metal powders and very fine moulds to produce extremely small three dimensional structures with very high resolution, sharp edges and outstanding aspect ratios [22]. By precise selection of process parameters, including concentrations, temperatures, negative logarithm of hydrogen-ion activity (pH) value, and time, it is possible to control and adjust properties such as particle size, apparent density, specific surface, and overall morphology to customer requirements.

The plasma atomisation technique is a development for the preparation of concentrated colloidal metal solution in hydrophilic and hydrophobic circumstances [23]. The metal content in colloidal solution is as follows: for silver 52 mg/L, copper - 56 mg/L, gold - 5.5 mg/L, palladium - 3.6 mg/L. TEM images of copper and silver particles are shown in Fig. 14. Formation of aggregates of particles can be explained by the high content of particles in solution. The median size of copper, silver and gold particles is about 30 nm, and palladium about 2 nm [24].

The plasmachemical technique enables the production of refractory metal ultrafine powders (W, Mo, Ni), compounds (TiN, AlN, Al<sub>2</sub>O<sub>3</sub>,

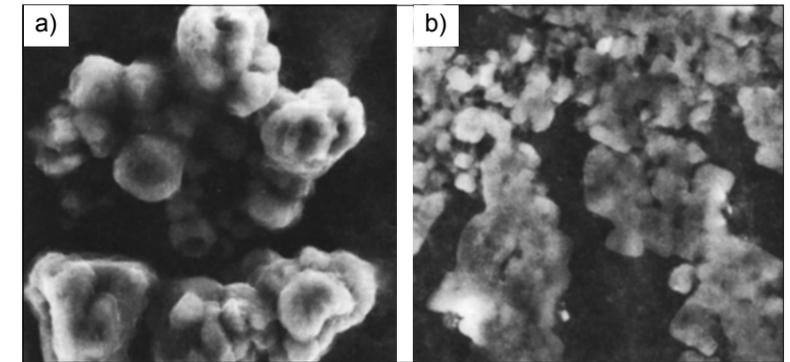


Fig. 14 TEM images of copper (a) and silver (b) nanoparticles

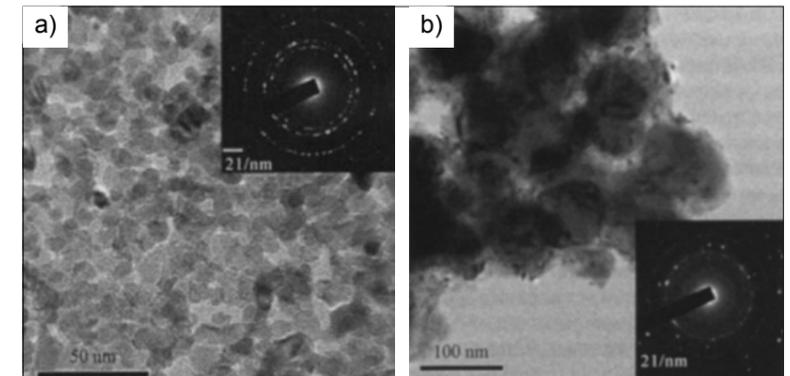


Fig. 15 TEM micrographs of NiO (a) and Ni/NiO (b) nanopowders made by thermal decomposition of nickel acetate hexamine at 673 K

SiC, Si<sub>2</sub>N<sub>4</sub>, Ti(C, N), and others), and composite material types such as Si<sub>3</sub>N<sub>4</sub> + SiC and TiB<sub>2</sub> + TiN, and others. Due to plasmachemical synthesis features (non-isothermal process, coagulation of particles, and others), the particle size distribution range is, in most cases, rather wide.

Laser pyrolysis has been developed for the production of nickel nanopowders [25, 26]. This process allows the production of nickel particles by laser-driven decomposition of a nickel precursor, such as nickel carbonyl (NiCO). Here, an infrared laser rapidly heats a dilute mixture of nickel carbonyl and a photosensitizer in a carrier gas to decompose the precursor and initiate particle nucleation. The photosensitizer is selected from the group consisting of sulphur hexafluoride, ethylene, silicon tetrafluoride and ammonia. Nickel carbonyl was generated in situ from activated nickel powder and carbon monoxide at room temperature. During the synthesis process, laser heating allows for rapid cooling

of the freshly nucleated particles by mixing with unheated gas. By varying the precursor flow rate, laser energy, and unheated gas flow rate to change the residence time, precursor concentration, and reaction temperature, the average particle size can be controlled over a range of primary diameters from 5 to 50 nm.

The thermal decomposition in air in the temperature range 573–773 K is used for synthesis of Ni/NiO nanopowders [27]. Thermal decomposition of nickel ammine complexes occurs with forming nickel hydroxide, carbonate and hydroxycarbonate ammines precursors. Composition of the precursors depends on temperature: temperature increase results in decreasing content of nickel carbonate and hydroxycarbonate ammines precursors. Formation of metal nickel phase can occur with thermal decomposition of nickel dioxide ammine only. Thermal decomposition of nickel carbonate and hydroxycarbonate ammines leads to forming NiO, Ni(OH)<sub>2</sub>, NiO

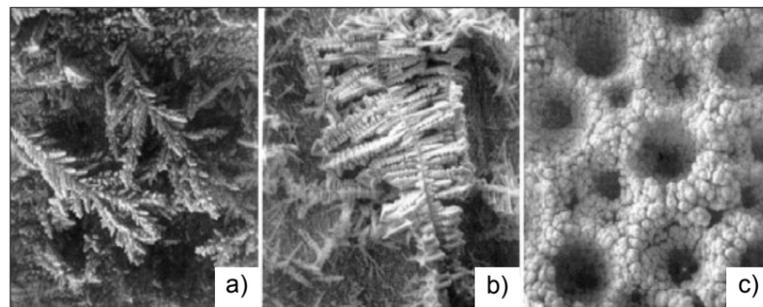


Fig. 12 Scanning electron micrographs of electrolytic dendritic sediments on the cathode in the water solution: copper (a), lead (b), nickel (c)

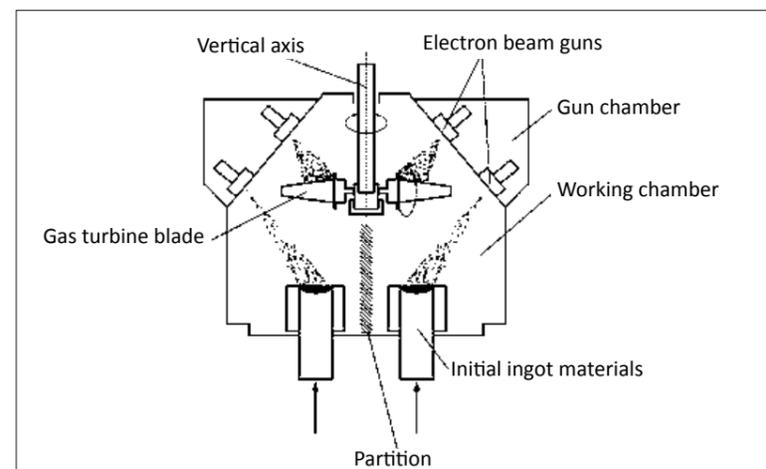


Fig. 13 Schematic of microlayered material deposition on gas turbine blades.

and Ni phases precipitation is characteristically for powders produced at 673 K temperature (Fig. 15).

Mean particle size of nickel and nickel oxide depends on temperature: in the temperature range from 623 to 773 K the particle size of nickel oxide has grown from 5 to 25 nm and nickel from 50 to 55 nm.

### Copper and copper alloy powders

Copper-based Powder Metallurgy products rank third after iron and steel and aluminium-based PM products in terms of volume. The copper-based PM products sector currently achieves annual shipments of over 60,000 mt globally. At present oxidation-reduction, atomisation, and electrolysis are used for commercial production of copper powders.

#### Oxidation-reduction process

Oxide reduction and electrolytic deposition are the primary large-scale production processes for copper powder. In this process, particulate copper oxide is transformed to copper at an elevated

temperature in reducing gases. The sintered porous cake produced is then ground to powder. Originally, the initial material used for reduction was copper mill scale or cement copper. However, as copper powder of higher purity was required, particulate copper of high purity (chopped scrap or atomised powder) was used as a starting material and oxidised to cuprous or cupric oxide or a mixture of both. Melting is usually carried out in either fuel-fired or induction furnaces. It is important to ensure a very low content of elements that form very stable oxides, such as Al and Si, which reduce the compressibility of the powder and also make it abrasive; while tin and lead additions cause difficulties in pouring the melt due to incrustation and clogging in the furnace and nozzles. In the case of applications of copper powder for electrical and friction parts, it is important to remove the impurities that have negative effects on electrical and thermal conductivity.

In commercial practice, oxidation of particulate copper is realised in air at temperatures above 923 K. Oxidation in fluidised beds or rotary kilns

accelerates the oxidation process. However, this process is more difficult to control than oxidation in a belt conveyor furnace.

Reducing atmospheres include hydrogen, dissociated ammonia, natural gas or other endothermic or exothermic gas mixtures. Hydrogen is a more effective reduction agent than carbon monoxide, especially at low temperatures. However, hydrogen is more explosive than carbon monoxide. The typical reduction temperature ranges from 698 to 723 K. At higher temperatures sintering of the product may take place. The reduced copper oxide leaves the furnace as a porous cake which is crushed to the required particle size. The resulting powders have good compressibility and green strength.

#### Electrolysis processes

In the electrolytic process copper is electrodeposited to obtain a spongy powder deposit at the cathode rather than a smooth, adherent one. Low copper ion concentration and high acid content in the electrolyte favour the formation of powder deposits. High cathode current density and the use of an electrolytically refined copper anode also facilitate the formation of powder. Along with these conditions, control of additional variables is necessary to produce powders that satisfy commercial requirements. These variable parameters include quantity and type of addition agent, temperature and circulation rate of the electrolyte, size and type of anode and cathode, electrode spacing, and brush-down interval.

One of the basic problems for industrial scale electrolysis is the ability to obtain copper powder with a homogeneous structure. The transfer of results obtained from laboratory research on the continuous electrolysis process to industrial production is difficult. The known method of full scale experiment consists of periodically removing the cathode bar from a cell section followed by the photographing of the deposits formed. However, this method is inadequate because pauses disrupt

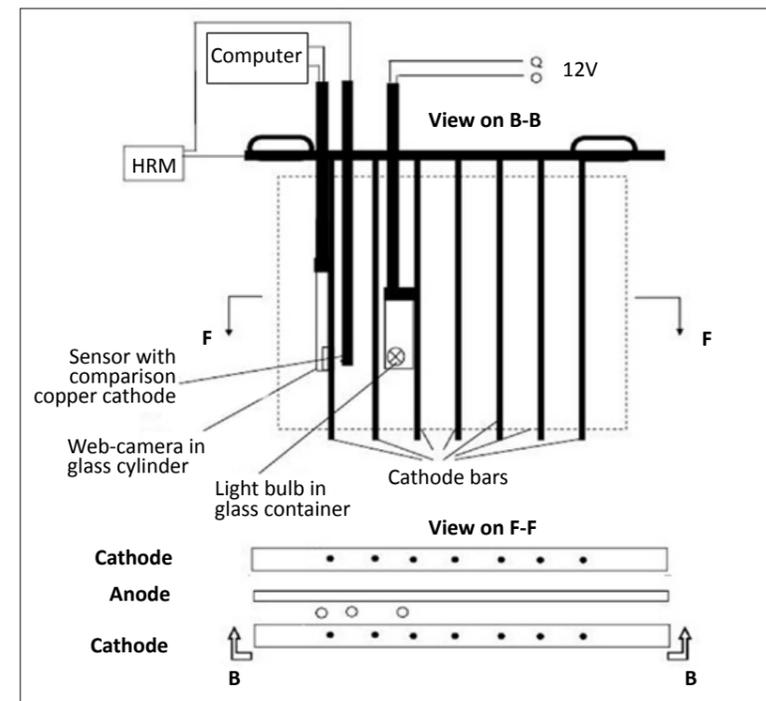


Fig. 16 Schematic of installation of the electrolysis process with continuous control on an industrial electrolyser (Courtesy Prof. Murashova)

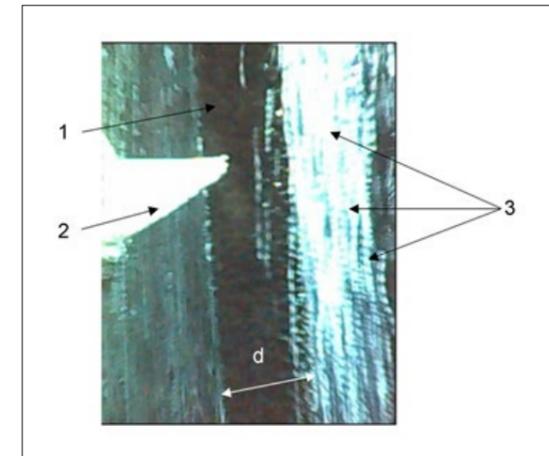


Fig. 17 Fragment of cathode bar immediately after energising: 1 – cathode, 2 – sensor tip, 3 – routes of emitted hydrogen bubbles

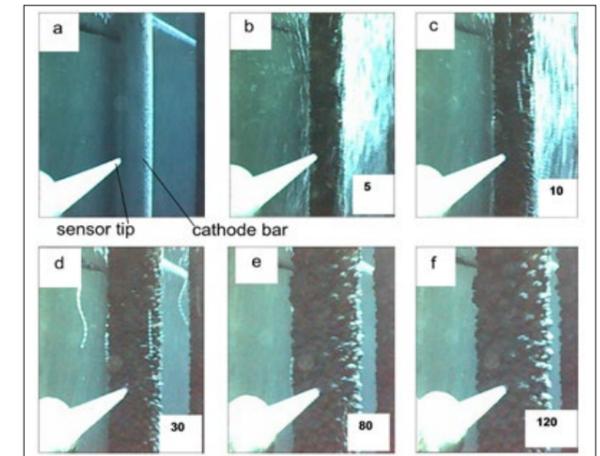


Fig. 18 Formation process of dendritic deposit layer (Courtesy Prof. Murashova)

the kinetics of the electrolysis process. An advanced control method that could be used on an industrial scale utilises a video recording of the copper deposit growth. This technique, with simultaneous recording of time history cathode overstrain, provides the possibility of improving electrolysis efficiency and controlling the conditions for optimal results [28].

A schematic illustrating the installation of a continuous electrolysis process on a commercial scale, with a submersible web camera is shown in Fig. 16. Formation of dendritic copper sediment is accompanied by the release of hydrogen. Hydrogen is especially intensively emitted after energising, as can be seen in Fig. 17. Fig. 18 also shows the routes of escaping hydrogen bubbles as they travel along the cathode bar surface. After 5 minutes the bubbles form a thick "fog" (Fig. 18b) but the release of hydrogen is appreciably decreased 30 minutes after energising.

The properties of electrolytic copper powder depend on the various parameters of the operation and, therefore, can be adjusted by modification of certain process variables. Properties of typical commercial grades of electrolytic powders are shown in Table 1, which lists various grades of electrolytic copper powders. The purity of electrolytic powder is high, with a copper content usually exceeding 99.5%. Apparent density (AD) values range from 0.65 to 4.5 g/cm<sup>3</sup>. Flow rate strongly depends on the AD. As a rule, powders with densities less than about 1.3 g/cm<sup>3</sup> do not flow and powders with densities of 1.3 to 2.3 g/cm<sup>3</sup> have poor free flow. High compressibility is characteristic for electrolytic copper powders, which have found a wide usage in many electrical and electronic applications.

#### Atomised copper powders

In this process inert gas atomisation produces spherical particles, while the shape of water atomised powders can be regulated from irregular to nearly spherical by controlling the interaction conditions

between the water jet and the metal stream, including liquid flow rate, water pressure, and atomiser design. Particle shapes of gas and water atomised copper powder are shown in Fig. 19.

To produce a powder with a particle size of less than 150 μm, water pressures of 10 to 15 MPa are required. Subsequent reduction of the water atomised powders improves their compressibility due to agglomeration

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and pore generation. The removal of oxygen requires reduction temperatures of 973 K or higher. AD of atomised Cu powders can be decreased by the addition of small amounts, up to 0.2%, of certain elements, such as magnesium and lithium, which help to lower the surface tension of the copper melt. The additions of a small amount (0.1 to 0.3%) of phosphorus when gas atomising Cu powder allows the production of spherical particles with very low oxygen content. During atomisation phosphorus is oxidised and forms a protective phosphorus pentoxide film. Large-scale atomisation is usually run as a continuous process. Molten copper may be discharged through a tube in the

side of the furnace wall and directly atomised, or through a tundish. Properties of typical commercial grades of water and gas atomised powders are shown in Table 2.

**Copper alloys**

Copper alloys, including brasses, bronzes, and nickel silvers are commercially produced mainly using atomisation methods as for copper. High-purity ingot materials are required in order to avoid the subsequent reduction of oxides. Properties of typical commercial copper alloy powders are given in Table 3. Water atomised bronzes are used for bearings manufacture. Spherical gas atomised bronzes are used to make filters. The diffusion-alloyed or

partially prealloyed bronze powder 90%Cu-10%Sn is used for microbearings, filters, friction materials, and diamond tools. Particle shapes of brass and bronze powders are shown in Fig. 20 [29].

**Aluminium and aluminium alloy powders**

Advanced processes such as rapid solidification, mechanical alloying, and spray forming create powders that, upon subsequent consolidation, provide significant improvements in room and elevated temperature strength, fracture toughness, fatigue life, and corrosion resistance. The real advantage of Powder Metallurgy processing is in the production of new alloys and composites with

Properties	Unit	Source / Standard grades													
		GGR				ESP		UEM		MMP		Pom		Pell	
		FLL2 light	FL medium	SSM heavy	GG grits	C100	C270	PML2	SA	SFG16-200	SFG22	LP0	SB		
<b>Nominal composition:</b>															
Hydrogen	wt%	-	-	-	-	0.15	0.08	0.2 max	0.2 max	0.2 max	0.2 max	0.40 max	0.15 max	0.15 max	0.20 max
Acid insoluble	wt%	<0.17 <sup>a</sup>	<0.17 <sup>a</sup>	<0.10 <sup>a</sup>	-	0.02	0.02	0.04 max	0.04 max	-	-	-	-	0.06 max	0.06 max
Copper	wt%	99.8 min	99.75 min	99.8 min	99.85 min	99.7	99.9	99.6	99.7 min	99.4 min	99.5 min	99.8 min	99.8 min	99.7 min	
<b>Sieve analysis:</b>															
>250 µm	wt%	-	-	-	-	-	-	-	nil	-	-	-	nil	-	-
>150 µm	wt%	-	-	-	-	-	-	-	3-9	-	nil	-	5.0 max	1.0 max	-
>100 µm	wt%	-	-	-	-	-	-	-	25-35	nil	20 max	nil	15-25	6.0 max	-
>75 µm	wt%	-	-	-	-	-	-	-	-	3.0 max	10-25	0.2 max	38-48	-	traces
>45 µm	wt%	-	-	-	-	-	-	-	80-90	-	15-45	12 max	-	50-60	10 max
<45 µm	wt%	-	-	-	-	-	-	90 min	10-20	75-85	30-45	-	37-43	40-50	90 min
Apparent density	g/cm <sup>3</sup>	0.8	1.3	2.4	3.0	1.0	2.7	0.90-1.10	2.25-2.55	1.50-1.70	none	0.65-0.75	2.35-2.55	2.3-2.5	0.9-1.1
Hall flow rate	s/50g	none	none	-	-	none	26	none	none	45 max	45 max	none	40 max	37	none
<b>Compacting properties at 165 MPa</b>															
Green density	g/cm <sup>3</sup>	-	-	-	-	5.9	6.1	-	-	6.4	6.4	-	-	6.4	6.0
Green strength	MPa	-	-	-	-	27.5	10.3	-	-	15 min	5 min	-	-	14	18

GGP Metalpowder AG; ECP – ElectroCopper Products Ltd; UEM – Uralelectromed; PM – Pometon; MMP – Makin Metal Powders Ltd  
<sup>a</sup> – oxygen content; Pell – Pelletiers, T.W., Berry, D.F., Production of copper alloy powders. Article in ASM Handbook, ASM International Publishers, Vol 7, 1998, pp 143-145

Table 1 Properties of typical commercial grades of copper powders produced by the electrolytic process

Properties	Unit	Source/Grades						
		GGP			Eckagranules			
		GCW-150 (w)	GCW-45 (w)	GCMM-45/106 (w)	AK<0.5 mm (g)	AK<0.16 mm (g)	AK<0.045 mm (g)	WS-U<0.1 mm
Copper	wt%	>99.6	≥99.4	>99.2	...	...	...	...
Shape		irregular	irregular	irregular	spherical	spherical	spherical	irregular
<b>Sieve analysis:</b>								
<500 µm	wt%	-	-	-	100			
<160 µm	wt%	-	-	-	-	100		
>150 µm	wt%	0	-	-	-	-		
>106 µm	wt%	<10	-	<0.5	-	-		
<100 µm	wt%	-	-	-	-	-		100
>63 µm	wt%	-	≤0.2	<15.0	-	-		
>45 µm	wt%	30-40	≤10	20-40	-	-	100	
<45 µm	wt%	-	90-100	<60-80	-	-	-	
Apparent density	g/cm <sup>3</sup>	3.1-3.5	3.0-3.8	2.0-2.4	5.2	5.0	4.4	4.2

GGP Metalpowder AG; Eckagranules Metal Powder Technologies; (w) – water atomised; (g) – gas atomised

Table 2 Properties of typical commercial grades of copper powders produced by atomisation techniques

structures and compositions that cannot be produced by ingot metallurgy. Rapid solidification extends the solubility of alloying elements, particularly transition and rare earth elements, and refines the structure of intermetallic phases responsible for improved mechanical properties.

A number of methods for obtaining rapidly crystallised aluminium alloys are known. The highest melt cooling rate (up to 10<sup>8</sup> K/s) can be achieved when there is contact of the melt with a metallic surface which discharges the heat (rapidly rotating wheels, drums, rollers etc.). However the powders produced are in the shape of scales and such powders are not suitable for the PM process because of their poor compressibility. These methods are not widely used in industry and typically remain the methods of laboratory investigations.

Centrifugal atomisation of the melt allows granules of 0.1 to 5 mm to be obtained. The drawbacks of the process are the relatively low melt cooling rate (not higher than 10<sup>3</sup> K/s) and complexity of the equipment.

When melt is atomised by compressed gas the cooling rate

reaches the value of 10<sup>5</sup> K/s. This process is currently the main industrial method for manufacturing aluminium and aluminium alloy powders. However in order to make the process explosion proof, the melt is dispersed by compressed

inert gases (nitrogen, argon or air enriched with inert gas) that considerably increase the expense of the process.

The well-known water atomisation (WA) process allows one to increase the cooling rate and is more econom-

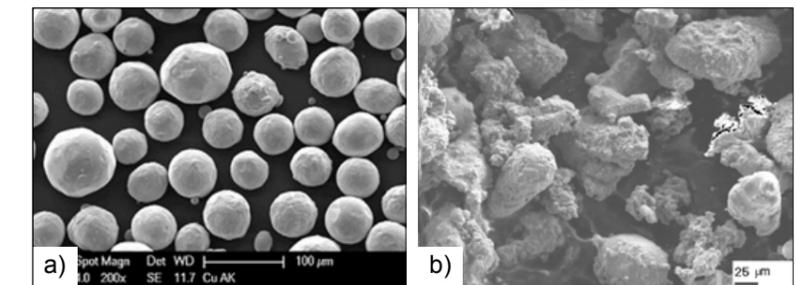


Fig. 19 Scanning electron micrographs of air atomised (a) and water atomised (b) copper powders

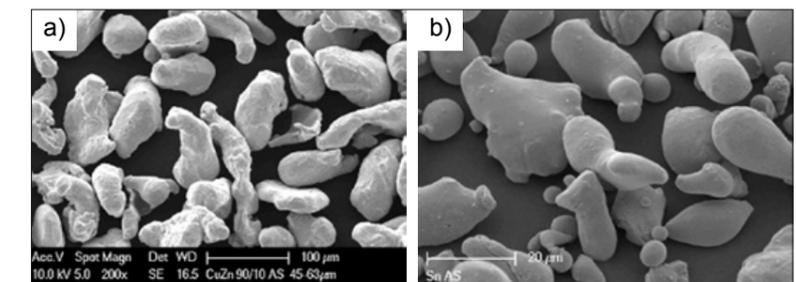


Fig. 20 Particle shape of air atomised: (a) brass powder ECKA CuZn40 and (b) bronze powder ECKA SnAS

Grade	Process	Chemical and physical properties									Source
		Composition, wt%	Shape	AD, g/cm <sup>3</sup>	Flow rate, s/50g	Sieve size, µm/wt%					
						>200	<200 >100	<100 >63	<63 >40	<40	
<b>Brass</b>											
CuZn30	WA	Cu70-Zn30	irregular	2.9-3.5	45 max	0.0	10-25	-	-	60-90	PM
CuZn40	WA	Cu60-Zn40	irregular	3.3-3.5	26 max	3-5	15-35	<100 >75 10-20 wt%	<75 >45 5-20 wt%	<40 25-40	UEM
25 GT 50/50 - 100	WA	Cu50-Zn50	irregular	2.5	-	-	-	-	-	<160	PO
60 AS < 0.1mm	AA	Cu60-Zn40-Si0.3	irregular	4.0	-	-	-	-	-	<100	ECK
V AS 0,16 - 0,5 mm	AA	Cu71-Zn28.5-Al0.5	irregular	3.3	-	-	-	-	-	160-500	ESK
V AS <0,063 mm	AA	Cu71-Zn28.5-Al0.5	irregular	3.6	-	-	-	-	-	<63	ESK
<b>Bronze</b>											
CuSn 11	GA	Cu89-Sn11	spherical	4.5-5.5	45 max	0.0	0.0	<75 >45 20 wt% max	80 min	MMP	
89/11 AK <0,2 mm	GA	Cu89-Sn10,7-P0,3	spherical	≥4.5	excellent	-	-	<200	-	ESK	
89/11 AK 0,045-0,063 mm	GA	Cu89-Sn10,7-P0,3	spherical	≥4.5	excellent	0.0	0.0	0.0	0,045-0,063 100wt%	ESK	
CuSn 20	WA	Cu80-Sn20	irregular	2.55-3.25	none	-	-	-	-	Median diameter 5.1 µm	NIPP
CuNi 20	WA	Cu80-Ni20	irregular	4.9 [a]	none	-	-	-	-	Median diameter 10 µm	NIPP

WA - water atomisation; AA - air atomisation; GA - gas atomisation; [a] tap density; PM - Pometon; UEM - Uralelectromed; PO - Poudmet; MMP - Makin Metal Powders Ltd; NIPP - Nippon Atomised Metal Powders Co.

Table 3 Properties of typical commercial grades of copper alloy powders

ically efficient. But water atomisation is not commercially applied to aluminium alloys for two reasons: [1] explosion risk during atomisation due to rapid hydrogen emission through powder-water interaction, and [2] severe oxidation of the surface of Al powders. However, recent studies have shown that WA under optimised conditions can be used for production of high-quality Al alloy powders. The problems of safe operation in Al alloy powder production and of powder quality were solved by the use of inhibitors in the water, by the control of suspension temperature and hydrogen ion exponent (pH), by the hydraulic classification of atomised products, and by the optimisation

of the dehydration procedure. The method for obtaining water atomised powders developed at the IPMS is explosion proof and provides the cooling rate of 10<sup>4</sup> K/s [30-32]. The use of rapid solidification (RS) allows one to increase the solubility of insoluble alloying elements. As shown [33-35], the use of technology based on high-pressure water atomisation allowed the production of Al alloy powders for various applications. In particular, ultrahigh-strength, high-strength weldable, and elevated temperature PM alloys were found to exceed high levels of mechanical properties as compared, for example, with ingot alloys of similar compositions. The excellent

compressibility of WA powders simplifies the procedure of powder consolidation which is based on hot extrusion of cold pressed degassed powders. Recent improvements in key steps of WA technology, namely the classification of powder by size and drying of wet powders, have been made [Fig. 21]. The technological process of powder production in this way includes the following operations. A charge, prepared with the use of ligature alloys and containing all of the alloying elements, is melted in an induction furnace. The prepared melt is poured into a tundish, from where it flows out through a calibrated

opening in the base. The gravity stream of melt in the atomisation chamber is atomised by jets of modified high-pressure water (controlling temperature, content of inhibitor and stabiliser). A suspended atomised powder from the atomisation chamber enters into a suspension reservoir, from where it is continuously pumped and fed to hydraulic classification by size in a hydrocyclone (Fig.22). The suspended solid with coarse powder fractions goes back into the suspension reservoir; powder is settled in this underbody and subjected to mechanical dehydration by filtration under a vacuum. Suspension with the selected fine fractions is settled in a thickener or particles are separated in a filter. After dehydration the moist powders are exposed to vacuum drying. The developed hydraulic classification system provides the powder separation of different fractions in the ranges of 0-40 µm, beginning with 0-10 µm.

An improved drying process of wet powders was applied [37]. Disintegration of powder particle aggregates, continuous immixture and even distribution of particles on a heat supplying surface were achieved by means of a contact-convection drying process with a high drying efficiency for drying chemically active substance and preventing powder oxidation and their thermal self-ignitions [38]. Fig. 23 shows the inside view of a contact-convection dryer.

Ultimate tensile strength of the widely known Al-Zn-Mg-Cu alloys is comparable with the strength of high-strength steels but amounts to only 1/3 of the weight, which makes them especially attractive for light weight products such as those used in aircraft construction. However, the high strength of these alloys is accompanied by increasing tendency to corrosion and rising notch sensitivity. Al-Mg system alloys show promising properties with a combination of high strength and good corrosion resistance. The latter can be considerably strengthened by additional alloying both by soluble

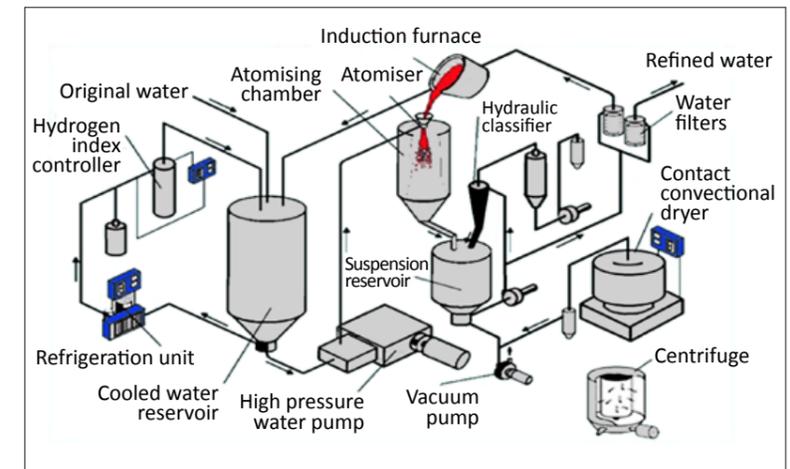


Fig. 21 Schematic diagram of the pilot plant to produce WA aluminium alloy powders

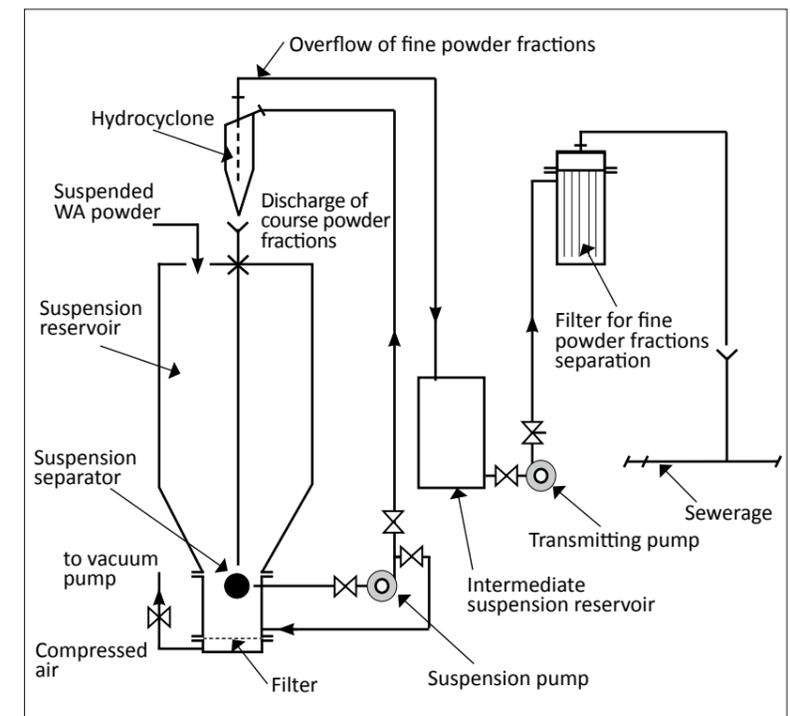


Fig. 22 Scheme of hydraulic classification system on particle size fractions



Fig. 23 Inside view of contact-convection dryer

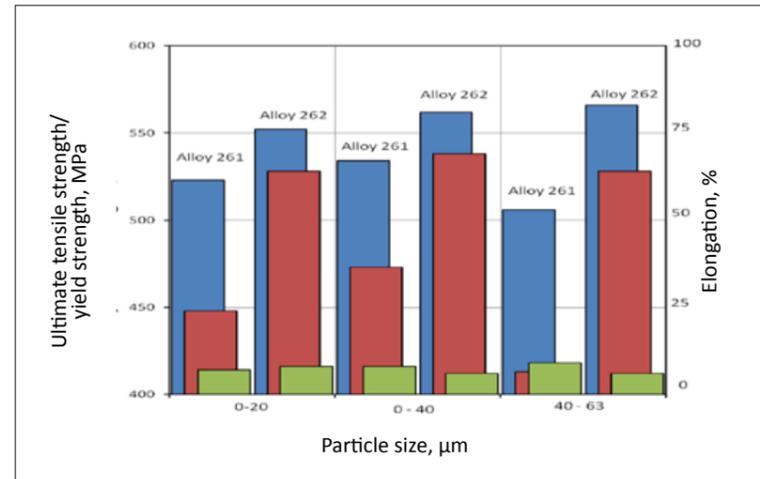


Fig. 24 Tensile strength data dependence on powder particle size fractions from which Al-4Mg-1.0Sc (#261) and Al-5Mg-0.7Sc-0.3Zr (#262) alloys are made

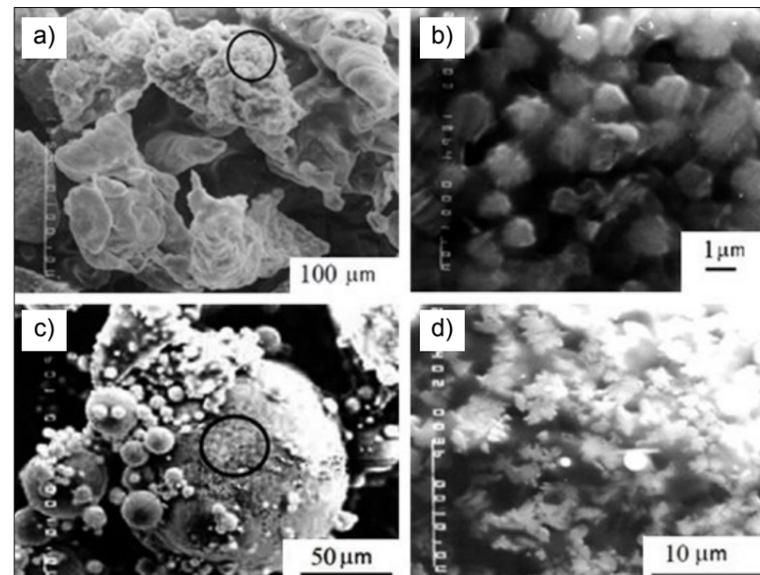


Fig. 25 SEM images of surface morphology and microstructure of powder particles of the Al-8.5Cr-1.5Fe alloy: powder particles (a) and separate powder particle (b) of WA powder; powder particles (c) and separate powder particle (d) of GA powder

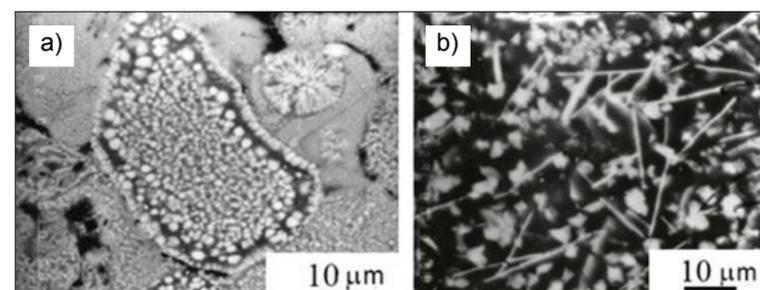


Fig. 26 SEM images of microstructure of polished section of heat pressed powder briquette: WA powder particles (a), GA powder particles (b)

(on condition of rapid solidification, i.e. formative supersaturated solid solution on the basis of aluminium) transitional metals (manganese, chrome, vanadium), and practically insoluble (iron, nickel, cobalt, rare-earth metals) elements [38, 39]. It is also possible to considerably enhance the efficiency of the alloying procedure of aluminium alloys using scandium [40-42]. The highest ultimate tensile strength of the developed alloys [39] is up to 524-562 MPa at sufficient plasticity. Tensile strength data dependence on powder particle size fractions, from which Al-4Mg-1.0Sc (#261) and Al-5Mg-0.7Sc-0.3Zr (#262) alloys are made, shown in Fig. 24.

The Al-Fe-Cr(Mn), Al-Cr-Zr(Mn), Al-Fe-V(Mo) and Al-Fe-Ce system alloys can be related to the elevated temperature alloys, where most high properties are currently reached. Among them the chromium containing alloys are more attractive. Thus Al-8Cr alloy retains the strength after 100 hours aging at 573 K [43]. Along with this the present alloy system possesses good machinability. The treatment of alloys at elevated temperatures offers the possibility, by means of additional heat treatment, to promote their mechanical properties. The Al-8.5Cr-1.5Fe alloy showed high strength. Both gas atomised and water atomised powders are used for making semi-product specimens.

The powders with typical surface morphology are shown in Fig. 25. The water atomised particles are highly irregular in shape and possess uneven surface in contrast with gas atomised powders which have spherical shape. Fig. 25b shows that the grain sizes equivalent to dendritic parameters is in the range of 0.5 to 2.0 µm, with an average of 1.0 µm, which corresponds to a melt cooling rate of about  $10^4$  K/s, calculated in accordance with [44]. The grain size of the gas atomised powder particles is relatively larger (Fig. 25d). X-ray diffraction studies showed that in both water and gas atomised powder particles of Al-8.5Cr-1.5Fe alloy the structure consisting of the aluminium

matrix and strengthened phases of mixed composition are inclusive of quasicrystals. Crystal intermetallics ( $Al_{13}Cr_3$ ,  $Al_5Cr_3$ ) are observed. SEM studies show the principal difference in microstructure between water and gas atomised particles pressed in briquettes (Fig. 26).

The water atomised powder particles structure is represented as a matrix with spherical particles of the strengthening phases (Fig. 26a). A structure consisting of the matrix with a mixture of spherical and needle-like particles of the strengthening phases is evident in the gas atomised powder particles (Fig. 26b). As SEM studies showed, the quasicrystalline particles have always spherical shape and some of them a five-point star shape. As is shown (Fig. 26), the quasicrystalline phase particles of water atomised powder are more dispersible and their content is higher as compared with gas atomised powders. An essential difference of the water and gas atomised powder alloys is evident. As is shown, the strengthening phase particles (light particles) are more dispersed in rod from WA powder as compared with rod from GA powder. Along the fibre boundaries the particles of oxides (dark particles) are shown. There are particles of ground powder particles surface oxide films as a result of plastic deformation during extrusion. The highest tensile strength is obtained in alloy made on the basis of water atomised powders. The ultimate tensile strength is on a level 491, 368 and 261 MPa at sufficient plasticity for 293, 463 and 573 K, respectively.

A strip-casting process, termed "spray rolling" (also "spray strip casting"), combines elements of spray forming and twin-roll casting [45]. Thus the powder metallurgical process of a semi-product in the form of strips is accomplished omitting the cold solid powder state and billet compacts. In this process, molten metal is atomised with a high velocity inert gas, the resultant droplets are quenched in flight, and the spray is deposited onto mill rolls (Fig. 27). In-flight convection heat

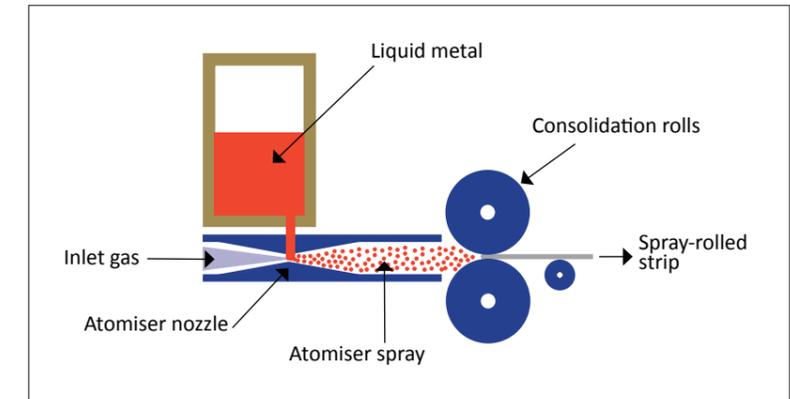


Fig. 27 Schematic of the spray rolling process principle

transfer extracts most of the metal's latent heat and conduction heat transfer at the rolls rapidly removes the remaining heat. Hot deformation of the semi-solid material by the rolls completely consolidates the rapidly solidified product. The high solidification rates in spray rolling allow a wider range of alloys to be processed, and at higher production rates than is currently possible in commercial twin-roll casting practices [46]. Direct strip-casting techniques are attractive because they improve the economics of manufacturing aluminium sheet products by eliminating the ingot casting, homogenisation, and hot-rolling unit operations in conventional ingot processing.

Commercial 2124-T851 and 7050-T7651 plates are remelted and spray rolled. The alloys were induction melted under a nitrogen atmosphere, heated about 100°C above the liquidus temperature, and pressure fed into an atomiser. Atomised droplets were directed horizontally and deposited into the roll gap of a 200 mm×300 mm 2-HI Fenn rolling mill, operating at a roll surface speed of 4.1 m/min, to produce strips measuring about 100 mm wide and 2.5 mm thick. The production rate was 4100 kg/(h·m). The tensile properties of spray rolled 2124-T851 produced under gas to metal mass flow rate at 0.30 compare favourably with those of commercial conventional material. Overall tensile properties of the spray rolled and heat treated 7050 are similar to those of the commercial product.

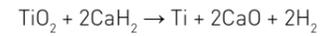
### Titanium powder

Titanium and its alloys are extensively used in aerospace, biomedical and other industrial applications due to their low density, excellent mechanical properties at room temperature as well as at elevated temperature and outstanding corrosion resistance. Ingot titanium is expensive to produce and fabricate. Another problem is connected with segregation in highly alloyed materials. PM technology can prevent the recrystallisation of such alloys due to rapid solidification of the melt. At the same time, PM allows the processing of Ti alloys to near-net shape, reducing material losses and costs respectively. A limiting factor to the widespread use of titanium alloys is their high cost compared to competing materials. Currently, chemical reduction, hydride/dehydride (HDH), gas atomisation, plasma-rotating electrode, and plasma atomisation processes are primarily used for the commercial production of Ti and Ti alloy powders. The characteristics of the titanium powders produced by various techniques are shown in Table 4.

### Chemical reduction process

The chemical reduction process comprises the chlorination of rutile,  $TiO_2$ , in the presence of carbon. The resulting titanium tetrachloride is reduced by the magnesium thermic process (Kroll process) or by the sodium thermic process (Hunter process). Sponge fines are the

primary product of titanium powder (Fig. 28). The remaining chloride is removed by vacuum distillation or by water leaching. The calcium hydride process was used in Russia, where commercially pure titanium is produced from titanium dioxide by reduction with calcium hydride [48] as follows:



The reduction is fulfilled at temperatures ranging from 1373 K to 1473 K.

**The HDH process**

The HDH process is based on the reversible interaction of titanium and hydrogen. Hydrogenated titanium in possession of angular shape (Fig. 29) is very brittle and can be simply ground to a fine powder. The minimum hydrogenation temperature for commercially pure powder is 400°C with subsequent dehydrogenation at 700–800°C. Careful removal of hydrogen is required because of the heightened

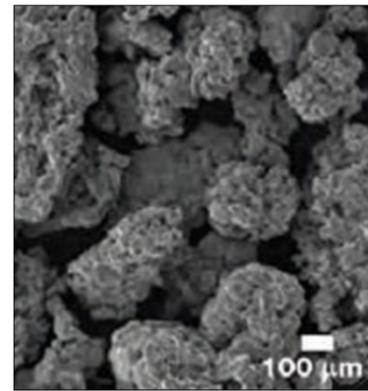


Fig. 28 SEM photomicrograph of sponge fines made by the Kroll process [47]

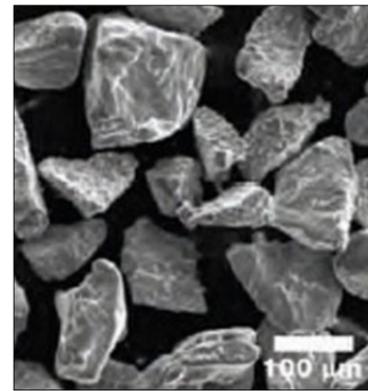


Fig. 29 SEM photomicrograph of angular HDH titanium powder [47]

sensitivity of titanium to any remaining hydrogen. Ingot, billet and scrap of various purity can be used as a starting material. The HDH process was used for reducing the titanium powder size from ingot feedstock and sodium powder. Owing to moderate HDH powder cost the expense was justified [49, 50].

**Atomisation**

The plasma atomisation process was developed to produce fine spherical titanium powder using titanium wire as the starting material [51]. In this process the wire is fed into the apex of three plasma torches, where it is melted and atomised in an argon atmosphere (Fig. 30). Droplets are

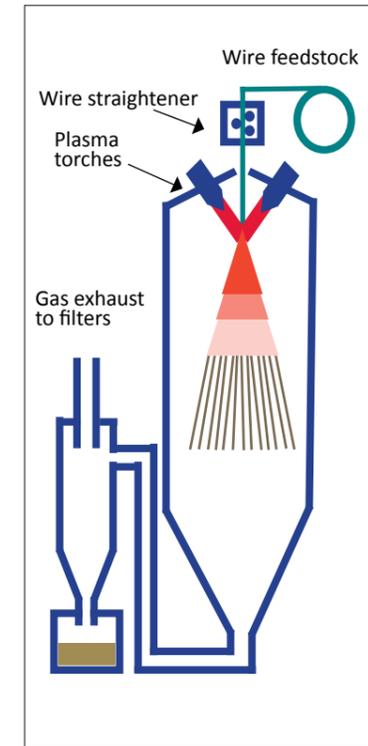


Fig. 30 Schematic of the plasma atomisation process developed to produce fine spherical titanium powder from titanium wire [51]

then cooled during their flight in argon with a cooling rate in the range of 10<sup>2</sup>–10<sup>3</sup> K/s and solidified, shaped like spherical powder particles (Fig. 31a) [52]. Spherical titanium and titanium alloy powders with particle sizes from 0–25 up to 0–180 μm have been commercialised in six size grades (Fig. 31b). Apparent density depending on size fraction is in the 2.52–2.75 g/cm<sup>3</sup> range. The oxygen content depends on particle size and is in the range 0.1–0.3 wt%. Plasma atomised titanium powders are used for the production of porous filters, MIM, thermal spray processes and biomedical applications.

The gas atomised prealloyed Ti-6Al-4V alloy has characteristic spherical shape (Fig. 32).

**Plasma-rotating electrode process (PREP)**

The plasma-rotating electrode process (PREP) is a centrifugal atomisation process where the end of a consumable metal bar is

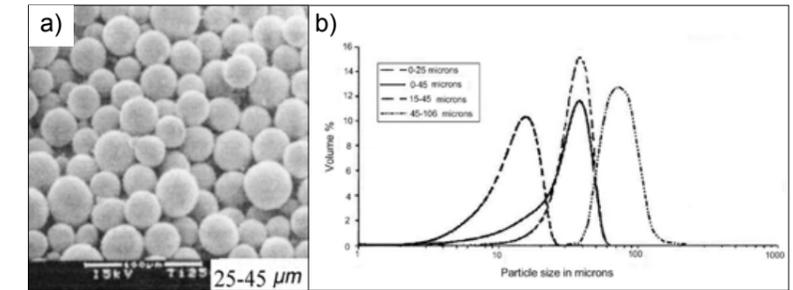


Fig. 31 Plasma atomised powders (a) SEM photomicrograph of 25–45 μm size fraction (b) Differential curves of particle size distribution of four size fraction [51]

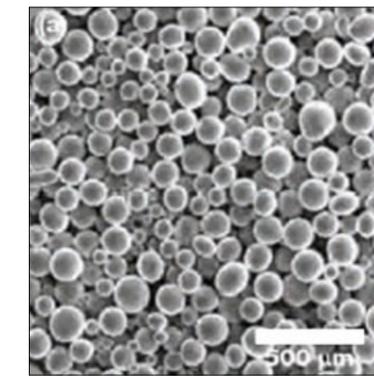


Fig. 32 SEM photomicrograph of a gas atomised prealloyed spherical Ti-6Al-4V [47]

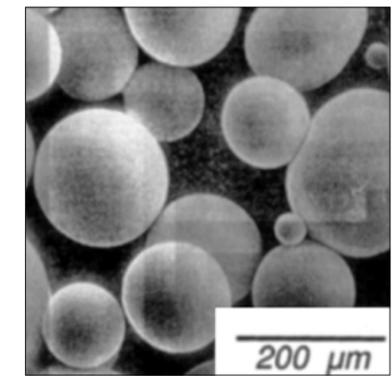


Fig. 33 Ti-6Al-4V powder produced by the plasma rotating electrode process (PREP)

melted while being rotated about its longitudinal axis. The consumable electrode can be melted by various energy sources, but specifically by helium plasma in this case[53]. When an electric arc is used the process is identified by the abbreviation REP. Molten metal is centrifugally atomised in droplet form and solidifies in an inert gas atmosphere to spherical powder particles. Typical sizes for Ti-6Al-4V powders are between 100 and 300 μm with a median particle size of about 175 μm (Fig. 33). The PREP powder has good flow characteristics, and a tap density about 65% of theoretical.

**Tekna technique**

Teknas's induction plasma spheroidisation process converts irregular shaped titanium powders to a spherical shape. Typically an irregular powder of <150 >38 μm size fraction is converted to a spherical powder at the same size

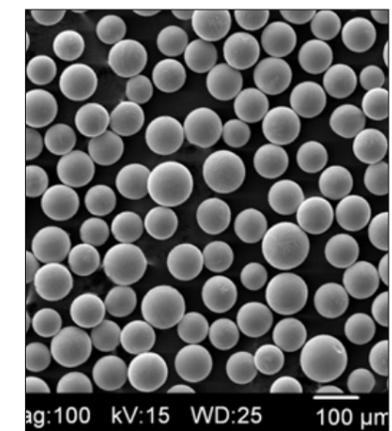


Fig. 34 SEM photomicrograph of spherical powder produced by processing angular HDH titanium to a spherical shape using the Tekna technique [47]

range. Fig. 34 shows spherical powder made by the Tekna technique processing angular HDH titanium to a spherical shape.

Process	Powder type	Advantages	Condition/disadvantages
Hunter process (pure sodium)	Elemental	Low cost; excellent for cold press and sinter	Limited availability; high chloride
Kroll process (magnesium thermic process)	Elemental	Lower cost; good compactability; readily available; low chloride	
Calcium hydride reduction process (a)	Elemental	Lower cost; good compactability; readily available; low chloride	Elevated temperature
HDH powder produced from alloys	Prealloyed	Simply available; amenable to cold compaction	High cost
Atomised	Prealloyed	High purity; available	High cost; not cold compactable
REP/PREP (b)	Prealloyed	High purity	High cost; not cold compactable
ITP/Armstrong (c)	Elemental & prealloyed	Compactable; moderate cost	Processibility/quality; production scale up
Electrolysis & electrolytic refining	Elemental & prealloyed	High purity; readily available; good compactability	High cost
MER electrolytic process (d)	Elemental & prealloyed	To be determined	Developmental
CSIRO (e)	Elemental & prealloyed	To be determined	Developmental

(a) – produced in Russia [48]; (b) – Rotating electrode process/plasma rotating electrode process; (c) – International Titanium Powder; (d) – Materials & Electrochemical Research [MER] Corp., UA; (e) – Commonwealth Scientific and Industrial Research Organisation, Australia

Table 4 The powder characteristics of the titanium powders produced by various techniques (modified Table 2 [47])

**Electrolysis of titanium compounds**

The technological flow of the electrolytic process is shown in Fig. 35 [54]. By anode dissolution of titanium metal, its waste products or alloys and cathode sedimentation of crystalline titanium, a titanium powder characterised by low content of impurities (Fe, Si, C, O, N, etc.) is produced. There are conditions where powders alloyed with aluminium, chromium, vanadium, manganese, zirconium and other metals can be produced. Raw materials for anodes include substandard titanium sponge, alloys based on titanium and conducting products of titanium raw material reduction (oxycarbides, oxycarbonytrides, etc.). The electrolyte is

molten NaCl, or NaCl + KCl, or NaCl + KCl + MgCl<sub>2</sub>. The preparation of the electrolyte consists of tetrachloride TiCl<sub>4</sub> reduction in molten NaCl and KCl using titanium scrap or sodium. Titanium ions are reduced to metal stepwise on the cathode. On the anode titanium is oxidised up to Ti<sup>2+</sup> and Ti<sup>3+</sup>. An average degree of oxidation (valency of titanium ions) in the electrolyte is 2.2–2.3.

Table 5 contains typical chemical analysis of chemical reduction process and electrolysis process titanium sponge fines and sinter compacted Ti-6Al-4V produced from these fines and Al-V alloy powder.

**MER electrolytic method**

The MER process uses a composite anode of TiO<sub>2</sub>, a reducing agent and an electrolyte mixed with fused halides (Fig. 35). The composite anode approach containing stoichiometric carbon with the reduced TiO<sub>2</sub> is scaled to a daily output of 227 kg. The oxygen level of the batch products is below 500 ppm wt [56]. Predictions are for titanium production at a lower cost than the conventional Kroll process.

**CSIRO technology**

Commercially pure titanium is produced in a continuous fluidised bed in which titanium tetrachloride is reacted with molten magnesium [47]. Production of various alloys including

Elements	Sodium thermic process (Hunter process)			Calcium thermic process			Hydride/dehydride process		Electrolytic process
	CP (a)	(b)	CP (c)	CP (a)	(d)	CP (e)	P6/4-3 (f)	CP (g)	CP (g)
Al	-	6.2	-	-	4.65	-	5.09	-	-
V	-	4.1	-	-	3.80	-	4.17	-	-
O	0.13	0.24	0.05-0.30	0.19	0.20	0.25-0.30	0.131	0.15-0.30	0.05-0.30
N	0.03	0.016	0.02-0.03	0.06	0.06	0.05-0.06	0.006	0.03-0.05	0.02-0.03
H	0.07	0.002	0.02-0.03	0.34	0.30	0.20-0.40	0.0015	0.06-0.15	0.02-0.04
C	0.02	0.02	0.01 max	0.03	-	0.04-0.05	0.02	0.015-0.020	<0.02
Fe	0.02	0.18	0.01-0.02	0.11	-	0.15-0.20	0.1	0.07-0.10	0.02-0.08
Na	0.10	0.10	-	-	-	-	-	-	-
Cl	0.13	0.12	0.10-0.13	0.004	0.003	0.003-0.005	-	0.06-0.08	0.03-0.08
Si	-	-	0.006 max	0.05	-	0.06-0.07	0.02	0.01-0.02	<0.02
Ca	-	-	-	0.04	0.06	0.08	-	-	-
Ni	-	-	-	0.07	-	-	-	-	-
Cu	-	-	-	-	-	-	<0.01	-	-
Sn	-	-	-	-	-	-	0.01	-	-
Nb	-	-	-	-	-	-	1.17	-	-
Mo	-	-	-	-	-	-	0.01	-	-
W	-	-	-	-	-	-	0.01	-	-
Cr	-	-	-	-	-	-	0.02	-	-
Ti	bal	bal	bal	bal	bal	bal	bal	bal	bal

CP – commercially pure titanium; (a) – source: Ref 53; (b) – sinter compacted Ti-6Al-4V produced from these fines and Al-V master alloy powder, [Ref 53] source; (c) <630 >180 μm powder sizes, [Ref 55]; (d) – sinter compacted Ti-6Al-4V produced from these fines, [Ref 53]; (e) <100 >40 μm [25 %], <40 μm [75 %] powder sizes, [Ref 55]; (f) – composition of alloys synthesized from blends of CP and master alloy powders; (g) <630 >180 μm powder sizes [Ref 55].

Table 5 Typical chemical analysis of chemical reduction and electrolysis processes titanium sponge fines and sinter compacted Ti-6Al-4V produced from these fines and Al-V master alloy powder

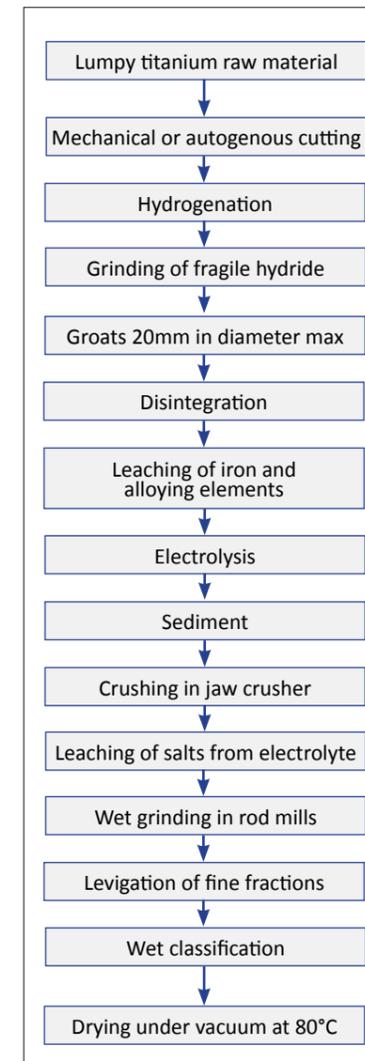


Fig. 35 The electrolytic process

aluminides and Ti-6Al-4V has been made on a large laboratory scale. The commercially pure titanium powder produced has been used to fabricate extrusions and thin sheet by continuous rolling. Commercialisation of the process is now in the planning stage with the move to the pilot plant stage.

**ITP/Armstrong process**

This process [47] is continuous and uses molten sodium to reduce titanium tetrachloride, which is injected as a vapour. The resultant powder does not need further purification and can be used in the conventional Powder Metallurgy technique. ITR currently operates an R&D facility in Lockport, Illinois, USA.

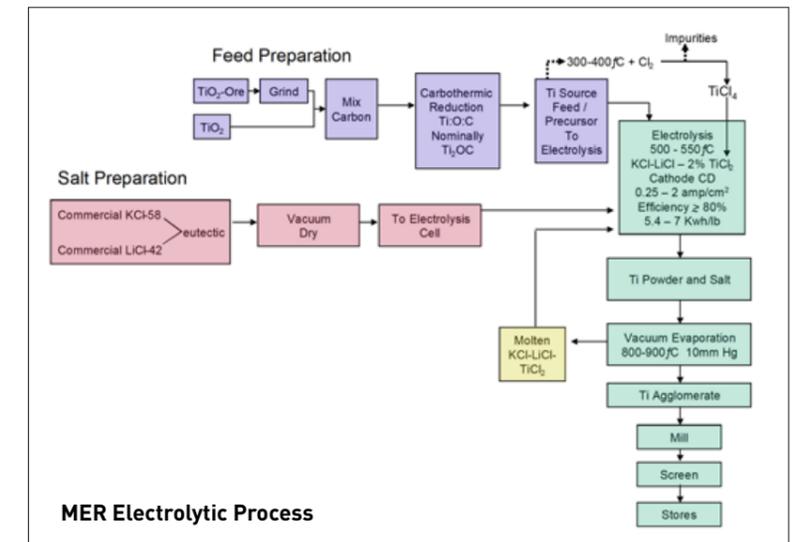


Fig. 36 MER overall process to produce titanium powders from a TiO<sub>2</sub> [47]

**Nickel and nickel alloy powders**

The two main processes for production of nickel powder are the carbonyl process, where nickel carbonyl is decomposed, and the hydrometallurgical process, where the reduction of an aqueous solution of a nickel salt with hydrogen under pressure is undertaken (the Sherritt process). Nickel and Nickel alloy powders can also be produced by inert gas or water atomisation, the electrolytic method and by mechanical alloying.

**Carbonyl processes**

Vale's Clydach Refinery in South Wales, UK, [formerly Inco Ltd] uses the Mond-Langer process [57], originally designed as a method of metal refining. Vale produces high-purity nickel powders from a unique carbonyl gas refining process. Gaseous nickel tetracarbonyl is formed by reacting carbon monoxide with nickel concentrates under controlled conditions. Subsequent thermal decomposition of the gas permits recovery of the nickel as a fine metallic powder and nickel pellets. Basic characteristics of Vale types and Norilsk Nickel types of carbonyl nickel powders are shown in Table 6.

Vale heavy powders are marked by "100" series and light powders

by "200". Acicular particles are the characteristic type of nickel powder produced by carbonyl decomposition (Inco types 120–123, and Norilsk Nickel type IL5). They are fine and regular in shape, with rough surfaces [58]. The minimum purity of the nickel powders is 99.9%. Typical microstructures of both groups (heavy and light) are shown in Fig. 37 and Fig. 38. Apparent densities range from 3.5 to 1.41 g/cm<sup>3</sup> for "heavy" and from 1.4 to 0.45 g/cm<sup>3</sup> for "light" powders. The median particle size of "heavy" is about 15 μm. Type 110 contains extra-fine, discrete spherical particles of nickel that are tightly sized in the range of 1–2 μm, the finest Ni powder commercially available for PM. Generally, the requirements of nickel containing composition powders for PM applications, consist of high purity, high rate diffusion transfer by interaction with the surrounding particles of other powders, high specific surface area, and a reasonably narrow particle size distribution. These powders are fine and regular in quasi-cubic shape, with rough surface projections. Typical of such powders is Norilsk Nickel type UT3.

The Vale powder typically contains 700 to 900 ppm (0.07 to 0.09 wt%) oxygen, 3 to 5 ppm (0.0003 to 0.0005 wt%) iron, max 1 ppm (0.0001 wt%) sulphur, and 600 to 700 ppm (0.06

to 0.07 wt%) graphitic carbon. The Fisher subsieve size is 3 to 7  $\mu\text{m}$ , apparent density is 1.8 to 2.7  $\text{g}/\text{cm}^3$ , and surface area amounts to 0.4  $\text{m}^2/\text{g}$  (BET). Chain-like nickel powders (Inco types 255, 287, etc., Norilsk Nickel type 1L7, 1L8, etc.) are characterised by their acicular, chain-like structure of fine particles, which make them fluffy in character, with low apparent density, 0.45 to 1.0  $\text{g}/\text{cm}^3$ , and large specific surface area, 0.6 to 0.7  $\text{m}^2/\text{g}$  (BET). The strongly linked chain morphology in combination with a low apparent density and the high surface area found in ultra fine carbonyl nickel powders is favourable for the production of sintered plates which have a porosity approaching 90% that is strong enough to withstand the stresses during charging and discharging of rechargeable batteries.

Nickel powders (Norilsk Nickel types S) are characterised by their unique, chain-like, highly convoluted structure, with low apparent density range from 0.52 to 0.56  $\text{g}/\text{cm}^3$  and

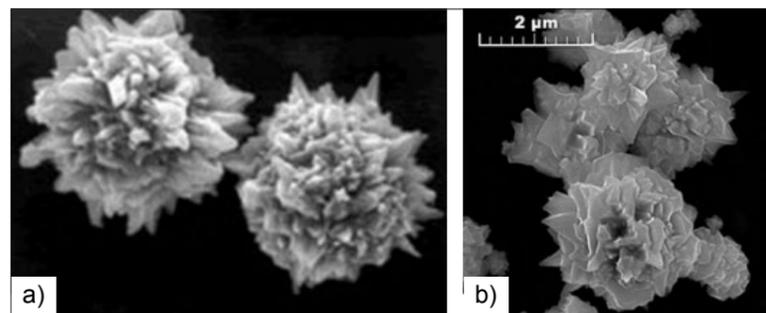


Fig. 37 SEM photomicrographs of typical acicular shape "heavy" nickel powder produced by carbonyl decomposition: (a) – Vale type 123; (b) – type IL5 Norilsk Nickel

specific surface area range from 1.5 to 2.0  $\text{m}^2/\text{g}$ , which make them suitable for the manufacturing of rechargeable batteries. Ultra fine nickel powders (e.g., Inco type 210, Norilsk Nickel type S10) also have chain-like morphology, but the particles are finer, the Fisher subsieve size is 0.5 to 1.0  $\mu\text{m}$ , and specific surface areas range from 1.5 to 6  $\text{m}^2/\text{g}$  depending on the grade.

The other manufacturing method is used by Vale's Copper Cliff Nickel Refinery for recovery of nickel from

a variety of nickel concentrates, including the removal of nickel from copper, cobalt, and precious metals. By the same basic technology this refinery generates tetracarbonyl at high pressure, due to the need to extract nickel in the presence of high copper concentrations. The refinery produces pellets and powder of 99.9% min nickel purity.

Application areas for carbonyl powders include batteries and fuel-cell electrodes, PM and MIM parts, catalysts, pigments and coatings,

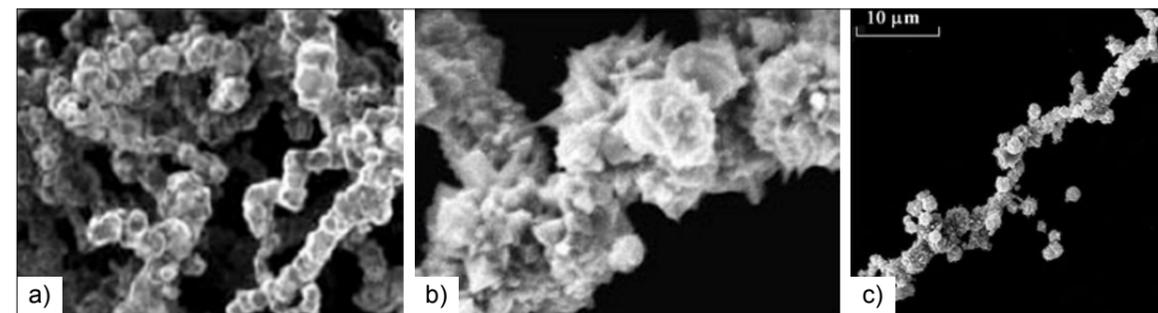


Fig. 38 SEM photomicrographs of typical filamentary shape "light" nickel powder produced by carbonyl decomposition: (a) – Vale type 210; (b) – Vale type 255; (c) – type S-30 Norilsk Nickel

chemicals, electronic alloys, and magnets.

#### Hydrometallurgical processing

This process comprises leaching, solution purification and metal recovery operations. The process can use a wide assortment of different feedstock, predominantly the sulphide concentrates. In the final metal recovery step nickel is precipitated from solution as a metal powder through a hydrogen reduction process. Currently, the main feed to the Sherritt refinery is a nickel and cobalt sulphide concentrate [59]. The sulphide ore is high in iron along with cobalt. The processing of this ore is accomplished by means of high pressure leaching with sulphuric acid. The key operation in this process is the separation of nickel and cobalt from iron, which typically constitutes 50 wt% of the ore. It is possible to achieve the selective dissolution of nickel and cobalt with less than 1 g/L iron in the leach liquor. The typical chemical composition of nickel-cobalt sulphide concentrate includes 51–56 wt% Ni, 5.0–6.0 wt% Co, 0.5–0.8 wt% Fe, 0.1–0.2 wt% Cu, 1.0–1.3 wt% Zn, 32–37 wt% S. The first stage in the refining processing is the leaching of the metal content from the sulphide feed. The leaching process is carried out at elevated temperature and pressure in continuous conditions during interaction between the fine sulphide concentrate and ammonium sulphate in the autoclaves [55].

The next step of the refining process is the cobalt separation. After the removal of cobalt, the

nickel-rich solution is subjected to a copper removal process. The solution remaining after copper elimination contains an appreciable quantity of sulfamate and other forms of unsaturated sulphur compounds. These sulphur remains are removed in the oxidation and hydrolysis stage. In the final stage of the Sherritt process nickel is precipitated from solution as a metal powder by hydrogen.

#### Atomisation

Atomised nickel-based alloy powders are used mainly for hardfacing and aerospace components. Nickel-based hardfacing powders are produced by gas and water atomisation. Most nickel-based hardfacing powders are of the Ni-Cr-B-Si type. Ni-based superalloy powders are used to produce turbine disks in engines for aircraft and power generation. These powders are made by inert gas atomisation using vacuum induction melting in order to minimise oxygen and nitrogen contents. PM superalloy powder grades have also been developed for production of gas turbine engine disks [60]. Another important powder production method is based on centrifugal atomisation. Here the melt is disintegrated by rotating under vacuum or protective atmosphere.

The MIM process is one of the near net-shape forming routes applied for fabricating superalloy Inconel 718 compacts using gas and water atomised powders. Sintering was performed at 1200–1250°C in a vacuum (0.1 Pa). The obtained relative density was in the range of 98–99%;

the tensile strength was 1000 MPa and elongation around 10%. Water atomised powder alloys were shown to be more promising due to their ductility and the highest possibility to increase alloying element content for improvement of mechanical properties [61, 62]. Other MIM processed superalloys (Inconel 713C and Udimet 720) showed high temperature properties. The proof included tensile tests at temperatures up to 900°C and evolution of oxidation resistance up to 1100°C [63].

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Type	Average particle size, $\mu\text{m}$	Particle shape	Apparent density, $\text{g}/\text{cm}^3$	Angle of repose, (degree)	Content of impurities, wt%			
					O	C	Fe	S
<b>Vale powders*</b>								
210	0.5-1.0	Chain-like	< 0.5	-	0.10	0.05-0.15	< 0.01	0.002
255	2.6-3.4	Chain-like	0.45-0.6	72	0.10	0.05-0.15	< 0.01	0.0002
287	2.9-3.6	Chain-like	0.8-1.0	66	0.10	0.05-0.15	< 0.01	0.0002
100	3-5	Acicular	1.6-2.1	65	0.10	0.05-0.20	< 0.01	0.0002
122	4-7	Acicular	1.9-2.5	60	0.10	0.05-0.10	< 0.01	0.0002
128	7-9	Acicular	2.5-3.0	48	0.10	0.05-0.10	< 0.01	0.0002
337	40-300	Semi smooth high density	3.5-4.0	-	0.25	0.08-0.10	0.01-0.05	< 0.001
<b>Norilsk Nickel powders and pellets**</b>								
S 20	1.85-2.2	Chain-like	0.51-0.64	-	-	0.09	0.0015	0.0007
S 30	2.91-3.5	Chain-like	0.6-0.8	-	-	0.28	0.002	0.001
1L5	2.1-2.4	Acicular	1.01-1.4	-	-	0.28	0.002	0.001
1L8	1.4-1.6	Chain-like	0.45-0.6	-	-	0.001	0.002	0.001
UT1	7-8.5	High density	3.0-3.5	-	0.02	0.1	0.003	0.0001
UT3	3-6	Quasi-cubic shape	1.91-2.5	-	0.02	0.072	0.003	0.0001
DNK-0	7-13 mm	Spheroidal	-	-	-	0.015	0.015	0.001

\* Content of other impurities, wt%: Zn, Zr < 0.01; Al, B, Mg < 0.005; Bi, Co, Cr, Cu, Mn, Mo, Pb, Si, Sn, Ti < 0.001

\*\* Content of other impurities < 0.006 wt%

Table 6 Basic characteristics of Inco and Norilsk Nickel carbonyl nickel powders

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www.mpif.org

#### Additive Manufacturing with Powder Metallurgy

(co-located with PM2014 World Congress)  
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#### HIP 2014, 11th International HIP Conference

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www.hip14.se

#### 7th Powder Metallurgy Conference and Exhibition, TPM7

June 24 - 28  
Ankara, Turkey  
www.turkishpm.org

#### Sintering 2014

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www.sintering2014.com

#### Euro PM2014

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