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POWDER METALLURGY REVIEW

Changing dynamics in carbonyl metal powder production

The rapid growth of manufacturing in China has resulted in an increased need for raw materials to supply many different industries. Whilst in some cases this extra demand has resulted in the need to import these materials, it has also given China the opportunity to further develop its own domestic supply chain.

One group of materials that has been impacted by this changing dynamic is carbonyl metal powders. In a little over ten years, China has become the leading producer of carbonyl iron powder as well as a major player in the carbonyl nickel powder business. The changing global landscape of carbonyl metal powder production is discussed in detail in our report on page 39.

As a result of the tremendous growth of China's Powder Metallurgy industry, the country is home to what is now the world's largest Powder Metallurgy exhibition. PM China 2017 takes place in Shanghai from April 26-28 and Inovar Communications will again have a booth in the exhibition hall. Call by booth number B002 and pick up your free copy of *PM Review* and our sister publications *PIM International* and *Metal AM*.

Paul Whittaker
Editor, *Powder Metallurgy Review*

Cover image
Graphite is added as a lubricant in brake friction materials, decreasing the friction coefficient and wear rate of products (courtesy Graphit Kropfmühl GmbH)

Spring 2017

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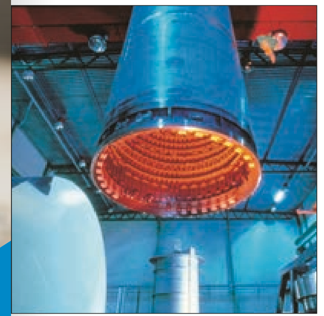
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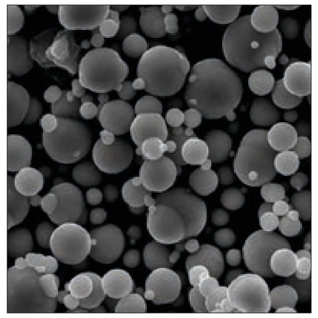
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To submit news for inclusion in *Powder Metallurgy Review* contact Paul Whittaker, paul@inovar-communications.com

Linde and Praxair agree merger to form $65 billion company

Linde AG and Praxair, Inc. have announced that the companies intend to combine in a merger of equals under a new holding company. The companies have signed a non-binding term sheet and expect to execute a definitive Business Combination Agreement as soon as practicable.

Based on 2015 reported results, the combination of Linde and Praxair would create a company with pro forma revenues of approximately $30 billion, prior to any divestitures, and a current market value in excess of $65 billion. Under the proposed terms of the transaction, current Linde and Praxair shareholders would each own approximately 50% of the combined company.

"The strategic combination between Linde and Praxair would leverage the complementary strengths of each across a larger global footprint and create a more resilient portfolio with increased exposure to long-term macro growth trends," stated Steve Angel, Praxair's Chairman and CEO. "We consider this to be a true strategic merger, as it brings together the capabilities, talented people and best-in-class processes of both companies, creating a unique and compelling opportunity for all of our stakeholders."

The combined company would adopt the globally-recognised Linde name and be listed on both the New York Stock Exchange and the Frankfurt Stock Exchange. The new company will seek inclusion in the S&P 500 and DAX indices.

"Under the Linde brand, we want to combine our companies' business and technology capabilities and form a global industrial gas leader. Beyond the strategic fit, the compelling, value-creating combination would achieve a robust balance sheet and cash flow and generate financial flexibility to invest in our future," stated Professor Dr Aldo Belloni, CEO of Linde.

The combined company would be governed by a single Board of Directors with equal representation from Linde and Praxair. Linde's Supervisory Board Chairman, Professor Dr Wolfgang Reitzle, would become Chairman of the new company's Board. Praxair's Chairman and CEO, Steve Angel, would become CEO and a member of the Board of Directors.

www.praxair.com
www.linde.com

Carpenter Technology completes acquisition of Puris LLC in $35 million deal

Carpenter Technology Corporation, headquartered in Philadelphia, Pennsylvania, USA, has completed its acquisition of the assets and business of Puris LLC, based in Bruceton Mills, West Virgina, USA, a producer of titanium powder. The reported purchase price was $35 million, with the acquisition including Puris' manufacturing assets, patents and related intellectual property.

"This acquisition will provide Carpenter with immediate entry into the rapidly expanding titanium powder market and is consistent with our strategic focus on strengthening our leadership position in important growth areas," stated Tony Thene, Carpenter's President and CEO. "Puris brings industry leading technology and processes for the production of titanium powder, Additive Manufacturing part production capabilities, a talented team, attractive intellectual property and established customer relationships. The strengths of Puris, coupled with Carpenter's reputation as an industry leading producer of premium alloys and our global commercial reach, will allow us to further deliver on the growing needs of our customers," added Thene.

Stephen Peskosky, Vice President of Corporate Development at Carpenter stated, "The addition of titanium powder to Carpenter's existing capabilities is significant due to the current and anticipated demand increases from the Additive Manufacturing industry, which produces mission critical parts supplied to Aerospace and Medical markets, as well as other markets."

Puris is a leading producer of titanium powder for AM and other applications. In addition, the flexibility of Puris' production capacity and process enables fulfilment of both high volume demands, as well as custom lots. Since its founding in 2014, Puris has successfully built leading capabilities, established advanced technology procedures and earned valuable quality approvals and accreditations. Operations will continue at Puris' existing production site in Bruceton Mills, West Virginia, USA, with the facility operating as a functional unit of Carpenter Powder Products.

www.cartech.com

GKN reports increase in 2016 sales

GKN plc has reported its results for the year ended 31 December 2016, showing sales for the whole group increased by 22%, to £9,414 million, with organic sales increasing by £209 million (up 2%). Trading profit was up 14% to £773 million. The group's Powder Metallurgy division reported growth in sales of 14% to £1,032 million, however organic sales were flat following the £11 million pass through to customers of lower steel prices and other surcharges.

"This is a good set of results with GKN continuing to make underlying progress in line with our expectations. We performed well against our key markets, overcoming some demand weakness and demonstrating once again the strength of our businesses, strong market positions and leading technology. Strategically we made good progress, including smoothly integrating Fokker and completing the disposal of Stromag – evidence of our sharper focus on capital allocation towards Aerospace and Automotive markets," stated Nigel Stein, Chief Executive of GKN.

The GKN Powder Metallurgy division comprises GKN Sinter Metals and Hoeganaes. GKN Sinter Metals is the world's leading manufacturer of precision automotive sintered components as well as components for industrial and consumer applications. Hoeganaes is one of the world's leading manufacturers of metal powder, the essential raw material for Powder Metallurgy. Underlying growth (before raw material pass through) was reported to be 1%, lower than global light vehicle production which was up 5%, due to under-representation in the strong China market. Underlying sales growth above the market was achieved in China, Europe and Brazil but sales in North America fell slightly due to weaker demand from the division's largest automotive customer. The organic reduction in trading profit was £4 million, including a £3 million restructuring charge as part of the Group-wide programme. There was a £2 million reduction from start-up losses in the new powder business acquired in China and the gain from currency translation was £15 million.

During the year, GKN Powder Metallurgy achieved a number of important milestones, which included winning around £200 million of annualised sales in new and replacement business. The division commenced production of high quality automotive grade powders in China for the Asian market and formed a new business to manufacture titanium powders in North America for Additive Manufacturing (AM) applications. "We expect 2017 to be another year of further growth, helped by the benefits of the actions taken in 2016 and GKN's constant focus on continuous improvement," added Stein.

www.gkn.com

Umicore to purchase Eurotungstene

Umicore has announced that it has signed a share purchase agreement to acquire 100% of Eurotungstene, a company within the Eramet Group specialised in developing, manufacturing and marketing metal powders used in diamond tools and hard metal applications.

It was stated that the acquisition of Eurotungstene, which will be integrated into the Cobalt & Specialty Materials business unit, brings together two businesses with complementary strengths. It will allow Umicore to broaden its product portfolio to better serve the needs of its diamond tool customers by relying on Eurotungstene's in-depth technical know-how, operational experience and diversified product portfolio. It will also allow Umicore to offer tungsten-based materials to hard metal customers.

Umicore Cobalt & Specialty Materials produces a variety of cobalt and nickel-based chemicals for a wide range of applications. One of its product groups is cobalt and pre-alloyed powders for the production of diamond tools and hard metal applications used for stone cutting and construction, metal cutting tools and wear parts. "We are very pleased with this acquisition which is an excellent strategic fit for the tool materials activity of our Cobalt & Specialty Materials business unit. This transaction further supports our ability to grow and deliver value to our customers," stated Benjamin Schmoker, Business Director of Umicore.

Eurotungstene, based in Grenoble, France, is specialised in processing metal bonding powders, tungsten powders and tungsten carbide powders for use in diamond tools and hard metal applications. In 2015, Eurotungstene generated a turnover of almost €42 million and employed 127 people.

www.eurotungstene.com
www.umicore.com

Höganäs reorganises business areas and appoints new head of global operations

Höganäs AB, Sweden, has reported that it is reorganising its business into three global business areas, namely Automotive, Industrial and Environmental. The company stated that operations will be globalised and it has also appointed Magnus Grönborg as its Chief Operations Officer. Prior to joining Höganäs, Grönborg was Group Operations Director for the product area Products & Solutions at Lindab.

"The purpose of this reorganisation is to better allocate and focus our resources on our customers' needs and to develop sustainable products and solutions for the future," stated Melker Jernberg, CEO at Höganäs."I am pleased that Magnus has accepted to join Höganäs. He brings with him an extensive experience from building successful cross-border teams that optimise operations in terms of safety, quality and cost."

Höganäs announced that it has appointed three Business Area Managers for Automotive. Dean Howard, former Senior Vice President Sales at North American Höganäs, takes responsibility for Automotive Americas. Marie Samuelsson, Vice President Commercial, will head up Automotive Europe and Fredrik Emilson, the company's Head of Asia, assumes responsibility for Automotive APAC (Asia Pacific).

Avinash Gore, currently Head of Region North America, has been appointed Business Area Manager for Environmental, with Dean Howard assuming Gore's role as Managing Director of North American Höganäs.For the Industrial business area, Höganäs has begun a recruitment process with Hans Söderhjelm, Vice President R&D, taking interim responsibility.

www.hoganas.com

Federal-Mogul reports full-year 2016 results

Federal-Mogul Holdings LLC, headquartered in Southfield, Michigan, USA, has announced financial results for the full year ended December 31, 2016. The company reported net sales were $7,434 million, a $15 million increase compared with the prior-year period. Sales from acquired businesses, as well as higher organic OE sales in both divisions, were largely offset by $107 million of negative impact from currency fluctuations.

Net income from continuing operations attributable to Federal-Mogul for the full-year 2016 was $82 million compared with a net loss from continuing operations attributable to Federal-Mogul of $117 million in 2015. Adjusted net income for full-year 2016 was $141 million. Operational EBITDA for full-year 2016 was $744 million (10.0% of net sales), an increase of $100 million (+16%) compared to 2015, including a $12 million negative impact from currency exchange rate fluctuations.

Federal-Mogul's Powertrain division reported revenue of $4,463 million, $13 million higher than in 2015. The year-over-year comparison was impacted by $57 million of negative currency exchange. In constant dollars, revenue increased by $70 million or 1.6%. Operational EBITDA for full-year 2016 was $473 million compared to $428 million in the prior year.

Federal-Mogul's Motorparts division reported revenue of $3,215 million for the full year, compared to $3,253 million in the prior-year period, including $50 million of negative impact from currency exchange rate fluctuations. EBITDA was $271 million compared to $216 million in 2015.

www.federalmogul.com

Hilti reports 2016 continued growth

The Hilti Group, headquartered in Schaan, Lichtenstein, has reported that in the 2016 financial year it achieved sales of CHF 4.6 billion, a growth of 5.7%. In what was stated as being a challenging environment, the group reported that in local currencies sales were up 5.3% and after adjusting for the divestment of US-based solar affiliate Unirac, sales in Swiss Francs increased by 7.1%.

The development in North America was highlighted as being particularly noteworthy with Hilti showing a strong performance in a consistently dynamic construction market (up 11.6% in local currencies, excluding the Unirac effect). The European region was up 6.2%, with Southern European markets displaying growing momentum.

As a result of the persistent economic crisis in Brazil, the Latin American region was slightly below the previous year's performance (down 1.2%). Continued sales increases were achieved in the regions of Eastern Europe / Middle East / Africa (up 5.6%) and Asia / Pacific (up 3.3%).

"These figures prove that our major investments made over the past few years are now materialising. Sales growth was particularly spurred by our enhanced R&D activities as well as the further expansion of our sales capacity. Thus, we are happy to look back on 2016 with satisfaction," stated CEO Christoph Loos. "We are confident we will outperform the market in 2017 once again. In order to achieve this, we will continue to invest in our products, services and software and further expand our sales team."

www.hilti.com

New President at Gasbarre

Gasbarre Products, Inc. has announced that Alex Gasbarre has been appointed COO and President, Press & Technologies, at Gasbarre Products, Inc., St. Marys, Pensyvannia, USA. The company also announced that Heath Jenkins, also of Gasbarre Products, Inc., has been promoted to VP Sales & Marketing.

Alex Gasbarre began his career with the company in 2005 as a Production Manager. He is also currently a member of the Powder Metallurgy Equipment Association Board of Directors and serves on Metal Powder Industries Federation's Industry Development Board.

Founded in 1973, Gasbarre manufactures a range of mechanical powder compaction & sizing presses and ancillary equipment.

www.gasbarre.com

Ceratizit acquires Becker Diamantwerkzeuge and takes majority share in Best Carbide Cutting Tools

Ceratizit Group has announced it has acquired Germany's Becker Diamantwerkzeuge, a producer of wear resistant tools for the automotive, mechanical engineering, medical technology and aerospace industries. Both companies agreed not to disclose financial details of the transaction.

Becker Diamantwerkzeuge employs 70 people in Puchheim near Munich, Landsberg am Lech and Idar-Oberstein. It was stated that the transaction will not affect the company's customers and dealers. Alexander Becker, the former co-owner, will lead the company as Managing Director. Becker's tools are designed for use with materials that are exotic and difficult to machine such as aluminium, carbon fibre reinforced plastics or high alloy steel. In order to minimise wear whilst machining, the cutting edges of the carbide tools are reinforced with diamonds or boron nitride.

Ceratizit also announced that its wholly-owned subsidiary Ceratizit USA, Inc. has signed an agreement to acquire a majority stake of the Californian solid carbide round cutting tool manufacturer Best Carbide Cutting Tools, LLC, a manufacturer of solid carbide round cutting tools located in California, USA. The company is housed in a 46,000 ft^2 state-of-the-art facility and currently has around 90 employees.

"Best Carbide Cutting Tools gives Ceratizit an exceptional chance to strengthen our network in the USA and build on nationwide top distributors. Best Carbide is known for the quality of its tools, with a strong focus on high-end micro tools," stated Jacques Lanners, Co-Chairman of the Ceratizit Executive Board.

"Through this partnership, Best Carbide will have access to Ceratizit technologies and expertise that will help the company to improve its manufacturing process, technical capabilities and quality of tooling – with the goal of ultimately adding greater value for our customers. For the first time in its 37-year history, Best Carbide becomes part of a global cutting tool organisation. We believe this will have substantial benefit for Best Carbide's stability, growth and advancement. The agreement is a win for both companies. Best Carbide Cutting Tools will continue to operate as it does today, but now with full support from the Ceratizit Group," Mark Nunez, President of Best Carbide added.

www.ceratizit.com

AP&C's new state-of-the-art titanium powder factory on target

Arcam AB has reported that the new titanium powder factory at AP&C, its metal powder manufacturing subsidiary in Montreal, Canada, is progressing well and is on plan.

With its new state-of-the-art plant, it was claimed that AP&C will be the first dedicated AM material manufacturer to have multiple production sites. The company added that the new production facility is a significant commitment to the business and growth strategies for the future.

"The need for high end titanium powder is driven by the fast growth and adoption of Additive Manufacturing. Arcam is determined to serve the industry through cost efficient solutions thus converting traditional manufacturing into Additive Manufacturing. A requisite is to offer highest quality powder for production at competitive cost and sufficient volumes," stated Magnus René, CEO of Arcam.


Alain Dupont, President of AP&C, at the construction site

Arcam also reported that AP&C recently received an order for 30 tons of titanium powder for Metal Injection Moulding (MIM) applications. This order, stated the company, also establishes AP&C as a major supplier to the MIM market. The order for Ti6Al4V MIM powder will be delivered during 2017.

www.arcam.com

Bodycote site earns highest aerospace accreditation

Bodycote, the world's largest heat treating services provider, has announced that its Chesterfield, UK, Hot Isostatic Pressing (HIP) facility has once again earned the highest level of Nadcap accreditation following a recent Nadcap audit. The official approval was awarded in February 2017.

As one of the original HIP facilities to achieve this standard, the company is now targeting to further extend its Merit status. This is building on a long history of supplying Hot Isostatic Pressing to the world's aerospace prime manufacturers and their first tier suppliers.


A HIP vessel being lowered into place

Moving forward, Bodycote stated that the Chesterfield site continues to play a strategic, long term role for new aerospace high technology programmes. The company added that it will continue to invest in resources and capital for development and operations to meet the demands required for this future growth.

www.bodycote.com

Seco/Vacuum Technologies LLC formed to serve North American market

Seco/Warwick has announced the creation of Seco/Vacuum Technologies LLC (SVT), a new company designed to provide standard and custom vacuum furnaces and related professional services to the North America market.

With nearly 400 vacuum furnaces installed in North America alone, Seco/Warwick is already a well-established vacuum furnace brand. Through the creation of a company dedicated to the unique requirements of its North American customers, Seco/Vacuum Technologies aims to improve delivery of the furnace technology and configure it for seamless compatibility to American standard components and controls.

"Seco/Vacuum Technologies is positioned to deliver improved products and services to established customers and new companies as well, by adapting some of Seco/Warwick's core products and capabilities for better compatibility with North American expectations," stated SVT's Managing Director, Piotr Zawistowski.

"In addition to providing the world's best technology, our key to this strategy will be to compete aggressively on both price and delivery. For example, our Value Incentive Program, or VIP for short, is a platform that offers several standard Vector® furnace models configured for fast delivery to US customers," added Zawistowski.

www.secovacusa.com

Submitting news..

To submit news to PM Review please contact Paul Whittaker: paul@inovar-communications.com

Update to Powder Forged Steel Parts standard

The Metal Powder Industries Federation (MPIF) has issued a revised and expanded version of the MPIF Standard 35-PF Powder Forged Steel Parts. The 2017 edition of the standard has been developed for the powder forging (PF) commercial parts manufacturing industry and provides the design and materials engineer with the latest engineering property data and information available.

Property data are included for four PF material systems, namely Carbon Steel, Copper Steel, Low Alloy PF-42XX and PF-46XX Steels in the normalised and quenched & tempered conditions. Chemical composition and minimum density requirements are cited.

Data tables (Inch-Pound and SI Units) include typical property values for Ultimate Strength, Yield Strength, Elongation, Reduction of Area, Rockwell Hardness, Impact Energy, Compressive Yield Strength and Mean Fatigue Limit.

This standard is said to provide the design and materials engineer with the latest engineering property data and information available in order to specify materials made by the powder forging process.

www.mpig.org

Bosch and MAHLE plan sale of joint turbocharger business

The Bosch Group and the MAHLE Group have announced the decision to seek a buyer for their joint venture, Bosch Mahle Turbo Systems (BMTS). The joint subsidiary was founded in 2008 and produces turbochargers for manufacturers of passenger cars and commercial vehicles.

With a total of around 1,400 employees, production is largely based in St. Michael, Austria, and in Shanghai, China. In Germany, BMTS also has a presence at two further locations in Stuttgart and Blaichach.

Further investment required

The international market for turbochargers is expected to continue to grow in the years ahead as the trend toward smaller engines fitted with turbochargers, especially for hybrid drives, remains strong. BMTS has already benefited from this market trend, however it was stated that the company is not yet large enough to achieve sustainable success in this market environment. "Large production quantities and the associated economies of scale represent a critical competitive advantage," stated Dr Rolf Bulander, Chairman of the Business Sector Mobility Solutions at Bosch.

"Despite bulk orders received from renowned customers, the associated high capacity utilisation at our plants and above-average growth rates, present sales volumes at BMTS are still too low in comparison with our larger competitors and must be further expanded," added Bulander.

For this reason, further investments are needed to achieve the company size required in the market. Bosch and MAHLE said they do not intend to drive forward the further expansion of BMTS themselves, because both parent companies need to focus their investments chiefly on new areas of development. "Thanks to our state-of-the-art locations and BMTS's good products, we are currently confident of finding a prospective buyer who will successfully expand the business," stated Wolf-Henning Scheider, Chairman of the MAHLE Management Board and CEO of the MAHLE Group.

Employee representatives and employees have been informed about the intention to sell the joint venture. Discussions are taking place with the relevant employee representatives to prepare the next steps in the sale process.

www.bmturbosystems.com

Tungsten parts manufacturer opens new US facility

Tungsten Heavy Powder & Parts (THPP) has begun operations at its new facility in Laramie, Wyoming, USA. As construction of the new site nears completion, it has been reported that the company has started operating its CNC machining line and plans to open its Powder Metallurgy line and general machine shop in the coming weeks.

The company, headquartered in San Diego, California, manufactures a wide range of tungsten components and is moving production to the new site in Wyoming. As well as being the largest supplier of military fragments in the world, THPP also manufactures fragment subassemblies, penetrators of all kinds, sabots, tails and various tungsten alloy rods. The company primarily serves the aerospace, aviation and military industries.

In the CNC section, penetrating tips are machined for military grade ammunition. "Tungsten is nice in this application because it's armour piercing. Our customers are the big defence contractors," General Manager Eric Riley told the Laramie Boomerang.

With the first of three process lines underway the company can now focus on getting its PM process up and running. "In a few weeks time we hope to start up the Powder Metallurgy section," stated the company's CEO and owner Joseph Sery.

"When the [PM operation] starts, it will produce 2,750 parts per minute. When manufacturing at that rate, quality is a major part. Because when you make a bent part at that rate, you've made 100 bent parts before you've sneezed," added Sery.

Tungsten parts manufactured at the company's new facility (Image: Shannon Broderick/Boomerang photographer)

As Tungsten Heavy Powder & Parts starts more applications at its Laramie branch, Sery said it will move all of its manufacturing stateside. The San Diego, California, branch will remain open as a centre for administrative and sales purposes, Riley said.

tungstenheavypowder.com

Abbott Furnace acquires intellectual property of Drever Furnace

Abbott Furnace Company, a manufacturer of continuous industrial furnaces and thermal processing equipment based in St. Marys, Pennsylvania, USA, has announced that it has acquired the intellectual property of Drever Furnace from The Rose Corporation. The Drever designs will be integrated into Abbott Furnace Company's current product offering and manufactured in St. Marys.

"We are very pleased with this acquisition and the value it brings to our company. We look forward to working with the current Drever customer base to bring them a full complement of products and the customer service for which Abbott Furnace Company is so well known," stated Ed Gaffney, CEO of Abbott Furnace Company.

Abbott Furnace Company manufacture a range of continuous belt furnaces suited to Powder Metallurgy applications. The company designs each furnace around customer's needs and product characteristics.

www.abbottfurnace.com

Metaldyne Performance Group reports 2016 year end profits

Metaldyne Performance Group (MPG) has reported net sales of $2,791 million for the year end 2016, compared to $3,047 million in 2015. Gross profits were reported at $469.2 million, a decrease of 9% from $516 million in 2015, with a net income of $96.9 million, down from $125.8 million in the previous year.

The company reported a strong cash flow that resulted in net debt reduction of approximately $60 million, achieved through dividend payments, share repurchases and the acquisition of Brilliant Iron Works for $14 million.

Among the year's financial highlights for MPG was the booking of nearly $700 million of new business awards, surpassing the 2016 target of $400 million.

Commenting on the Company's results, George Thanopoulos, Chief Executive Officer of MPG, stated, "We are very pleased with our 2016 results which reflect our ability to generate significant cash flow and strong margins. Our cash flow allowed us to reinvest in the business in addition to increasing shareholder value through debt reduction, share repurchases and increased dividends in 2016. These results also reflect our ability to drive value for our customers, who have rewarded us with significant new business awards, doubling our net new business backlog."

MPG provides highly-engineered components for use in powertrain and suspension applications for the global light, commercial and industrial vehicle markets.

www.mpgdriven.com

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Global vehicle market reaches record high in 2016

It has been reported that more vehicles were sold in 2016 than ever before, with growth coming from every world region except South America. According to WardsAuto, global vehicle sales grew by 5.4% in 2016 to an all-time high of 93.57 million.

European vehicle sales up 5.3%

WardsAuto stated that sales of vehicles in Europe ended 5.3% higher in 2016, reaching 20.29 million, of which 15.13 million were for passenger cars and light vehicles. Car production was up 5% in Europe in 2016 to 18.99 million, although Russia saw a further decline in its car production of 11% to 1.425 million. Residual pent-up demand and overall economic recovery is said to bode well for the European auto industry in 2017.

North America sees record annual sales

North America posted record annual sales for the second year in a row with 21.5 million vehicles sold, some 1.5% higher than 2015. Vehicle production in North America also saw a modest increase of 1.2% to 18.16 million with the USA showing an increase of 0.8% to 12.19 million, Canada an increase of 3.8% to 2.37 million and Mexico an increase of 0.9% to 3.59 million.

Asia/Pacific up 9.5%

The biggest annual increase came unsurprisingly from China which now has a 30% market share in vehicle sales. Sales in China were reported to have increased by an estimated 14.4% to 28.15 million with similar increase in vehicle production. Overall the Pacific-Asia region grew by 9.5% in 2016 to around 47 million giving the region a 50% share of the global automotive market.

The Japan Automobile Manufacturers Association (JAMA) reported a drop of 1.5% in total vehicle sales to 4.97 million, but a more modest decline of 0.5% year on year in passenger car production to 7.87 million.

The Korea Automobile Manufacturers Association (KAMA) reported that the country produced 7.3% fewer vehicles in 2016 reaching 4.22 million. The decline was mainly attributed to production disruption suffered by the leading automaker Hyundai Motor Co. and its smaller affiliate Kia Motors Corp. India is estimated to have produced 4.5 million vehicles in 2016 with a 7.1% increase in vehicle sales to 3.71 million.

South America down 12.5%

The South American region ended the year 12.5% down on 2015 with 3.82 million vehicles sold. Brazil saw vehicle sales drop by 20.1% to 2.05 million, whilst Argentina reported a healthy 10.5% increase to 700,000 units and Chile an increase of 7.3% to 320,000.

Good prospects for 2017 and growth forecast for electric vehicles

Speaking at the German Association of Automobile Industry (VDA) New Year reception in Berlin on January 25, VDA President Matthias Wissmann forecast continued growth for the global car industry in 2017, with the passenger car market alone set to reach sales of nearly 85 million units. He further stated that by 2020 European, and in particular German manufacturers, will more than treble their range of electric cars from the current 30 to nearly 100 models.

In 2019 electric drive will be present in practically all series, in the form of plug-in hybrids or purely battery-driven vehicles. The German automotive industry will invest over €40 billion in alternative powertrains by 2020, said Wissmann.

However, in parallel to electric mobility, development of classical powertrains will continue, he said. "Reductions in fuel consumption of 10 to 15% are possible and we are convinced that gasoline cars and diesels will still be needed. The global market for passenger cars will increase to 91 million new vehicles by 2020. This means that sales of cars with internal combustion engines will rise, even if the proportion of electric vehicles grows more strongly," added Wissmann.

Bories to succeed Buffet as Eramet CEO

Eramet has announced that Christel Bories will succeed Patrick Buffet as the company's CEO later in 2017. Bories was announced Deputy CEO in February and will be proposed as successor to Buffet as Eramet's Chairman & Chief Executive Officer at the conclusion of the company's General Meeting in May.

Departing CEO Patrick Buffet has been at the helm of Eramet for over ten years, through both a strong development phase and, more recently, a crisis in the raw materials sector. The Board of Directors' decision to replace Buffet follows signs of recovery for Eramet in its 2016 financial reports.

Bories, a graduate of HEC Paris, is expected to lead the group in a new growth cycle. Prior to joining

Eramet, she held high-level positions at multiple major corporations, including as CEO of manufacturing group Constellium in Schiphol-Rijk, Netherlands, from 2011-2013 and Deputy CEO of French pharmaceutical company Ipsen from 2013-2016. She is Independent Director of Legrand and Smurfit Kappa.

"I am delighted to join Eramet," stated Bories. "I believe this group has very solid assets to successfully develop technological and R&D know-how, recognised expertise and commitment from its teams and a stable shareholder base which is focused on the future."

www.eramet.com

Höganäs joins UN sustainability initiative

Höganäs AB has reported that it has joined the UN Global Compact, the world's largest corporate sustainability initiative. Based on a set of ten principles, participants pledge to adjust their business so that they, in a sustainable way, contribute to the development of societies and economies.

"Sustainability is a strategic priority for Höganäs and by joining the Global Compact we demonstrate our clear commitment," stated Nicklas Lång, Vice President and head of Höganäs' Sustainability department.

Participating companies operate in ways that meet fundamental responsibilities in the areas of human rights, labour, environment and anti-corruption.

www.hoganas.com

ACuPowder International, ECKA Granules and SCM Metal Products rebranded to form Kymera International

ACuPowder International, ECKA Granules and SCM Metal Products have been rebranded to form Kymera International. The three companies, owned by investment firm Platinum Equity, are leaders in aluminium and copper based powders with over one hundred years of history.

"While all three companies continue to thrive and grow, it was challenging for our customers, suppliers and even at times our employees to appreciate that we were all part of one organisation," stated Barton White, CEO.

The company stated that Kymera is an adaptation of Chimera and represents the idea of multiple forces coming together to create one strong entity. White and his team believe that this is exactly what having ACuPowder, ECKA Granules and SCM Metal Products under one common brand name will achieve and that it will no doubt benefit all of its global supply chain partners.

It was stated that the management team recognises that ACuPowder, ECKA Granules and SCM are all known and respected brands and so they will continue to use these names under the Kymera umbrella until its global supply chain partners become familiar with the new name. Kymera International will be introduced slowly to the marketplace and the company was keen to highlight that no immediate changes will occur regarding billing (e.g. invoicing and banking), contact details such as email, or packaging/labelling.

www.kymerainternational.com

Sumitomo Electric receives 2016 Top 100 Global Innovators Award

Sumitomo Electric Industries, Ltd., headquartered in Osaka, Japan, has received a 2016 Top 100 Global Innovator Award from Clarivate Analytics, formerly the Intellectual Property & Science business of Thomson Reuters. The award recognises the most innovative corporations and institutions in the world and it is the fifth time Sumitomo Electric has received the honour.

The methodology behind the award is to use objective analysis of patent volume, patent-grant success rates, global reach and invention influence to identify, without bias, the world's most innovative organisations.

The company designs and manufactures a wide range of industrial products including numerous Powder Metallurgy components and hard materials. In 2016 Sumitomo Electric also acquired US based Keystone Powdered Metal Company.

www.global-sei.com

Future manufacturing hub for advanced powder processes launched in UK

MAPP, the EPSRC Future Manufacturing Hub in Manufacture using Advanced Powder Processes, is a £20 million research hub led by the University of Sheffield, UK. The hub brings together expertise from the Universities of Sheffield, Leeds, Manchester, Oxford and Imperial College London, together with 17 industry partners and the UK's High Value Manufacturing Catapult. The aim of MAPP is to realise the potential of advanced powder processes to provide low energy, low cost and low waste high value manufacturing routes and products to secure UK manufacturing productivity and growth.

The launch event was attended by 150 delegates from across industry, universities, Catapult centres and sponsors. MAPP Director and RAEng Chair Professor Iain Todd, gave an overview of MAPP's vision and how it aims to address the challenges and opportunities surrounding advanced powder processes through an ambitious interdisciplinary research programme.

"It's an exciting time for powder based processes with new opportunities opening up rapidly in a range of key sectors including aerospace, energy, automotive and healthcare. However, there are still some fundamental scientific issues to be addressed before these technologies can be adopted more widely," stated Todd. "MAPP brings together leading UK researchers, industry and the High Value Manufacturing Catapult to achieve right first time manufacturing for advanced powder processes and develop the next generation of manufacturing technologies. The EPSRC Future Manufacturing Hubs are key element in our approach to tackling the UK's productivity gap and solving some of the longer term challenges faced by the UK's manufacturing industry."

Presentations from MAPP's academic partners highlighted how leading edge research is being applied to provide new insights on advanced powder processes, leading to improved outcomes for UK manufacturing. Professor Peter Lee, from the University of Manchester, spoke about how experiments at the UK's Diamond Light Source are providing new information on the fundamental physics and chemistry of technologies such as Additive Manufacturing. Professor Andrew Bayly, University of Leeds, discussed how physical models can give a better understanding of the dynamic behaviour of powders in processes and some of the challenges associated with powder processing.

Presentations from MAPP's industry partners outlined the opportunities for advanced powder processes, some of the challenges which need to be overcome and how the research in MAPP, together with aligned programmes funded by industry and UK Government, are overcoming the challenges and delivering benefit for UK productivity.

www.mapp.ac.uk

European distributer for stainless steel powder from China's Shijiazhuang Daye Metal Powder Co

Diamond Plastics GmbH, Nuremberg, Germany, an established producer of thermoplastic powder for the SLS market, has announced it is has expanded its range to include metal powders. The company is now representing Shijiazhuang Daye Metal Powder Co., Ltd for the supply of stainless steel powder to the European market.

Shijiazhuang Daye Metal Powder Co., Ltd was established in 1998 and specialises in the research, manufacturing and sale of metal powders. The company uses high pressure water atomisation and gas atomisation technology to produce a range of metal powders suited to numerous applications.

Stainless steel grades are available for Powder Metallurgy applications including porous and structural parts, Metal Injection Moulding (MIM), metal Additive Manufacturing and cold spray processes.

www.diamond-plastics.de
www.dayepowder.com

Metal Powder Industries Federation appoints new Executive Director

The Metal Powder Industries Federation (MPIF) and APMI International (APMI) have formally confirmed that James P Adams has succeeded C James Trombino as Executive Director/CEO, effective immediately.

Adams has worked in the Powder Metallurgy industry for more than 30 years following graduation from Hennepin Technical College in 1985. He began his career with MPIF in 2004 as Director of Technical Services, working closely with the MPIF Technical Board, where he has been responsible for Federation publications, professional development programs and conference technical programming. Under his direction, the Metal Injection Molding and Additive Manufacturing with Powder Metallurgy conferences were developed. He has also served as administrative director for APMI International and the Center for Powder Metallurgy Technology (CPMT).

Adams took on additional roles as administrative director for the Powder Metallurgy Parts Association (PMPA), Metal Powder Producers Association (MPPA), Powder Metallurgy Equipment Association (PMEA), and Isostatic Pressing Association (IPA), all affiliated associations within the MPIF umbrella. Additionally, he has also been MPIF's representative for the Lightweight Innovations for Tomorrow, a National Network for Manufacturing Innovation Institute, to aid in the promotion of lightweight technology development.

"MPIF has been a global leader and voice for the North American Powder Metallurgy industry for nearly 75 years and to be its fourth Executive Director is an honour and privilege," stated Adams. "Jim Trombino has left MPIF positioned for the future and I look forward to serving the current industry while advancing emerging technologies such as metal Additive Manufacturing."

www.mpif.org

James P Adams, above, has succeeded C James Trombino as the MPIF's Executive Director and CEO

China's J-20 stealth fighter jet engines to incorporate PM superalloy turbine disks

China's J-20 stealth fighter jet (courtesy Wikipedia)

China's J-20 stealth fighter jet is set to receive locally produced engines that incorporate Powder Metallurgy superalloy turbine disks, reports *China Daily*. The jet currently relies on Russian-produced engines, but China plans to replace these with its own designed and built engines by 2018-2019.

The use of Powder Metallurgy superalloy turbine disks will allow the J-20's engines to operate at extreme temperatures. A senior scientist for Aero Engine Corporation of China (AECC) is reported to have said that development is underway on domestically produced engines featuring single crystal superalloy turbine blades as well as PM superalloy turbine disks.

"The engine's development is proceeding well. We also have begun to design a next-generation aviation engine with a thrust-to-weight ratio that is much higher than that of current types," stated Chen Xiangbao, an official with AECC.

The J-20 stealth fighter is produced by Chengdu Aerospace Corporation and currently relies on Russian-produced engines. Russia, alongside the US, is one of the top jet engine producers globally. Chinese scientists and engineers have been reported to be working on solutions for local engine production since the 1990s, with the company's aerospace industry lagging behind global development of cutting-edge aviation engines.

Due to the secrecy surrounding China's efforts to develop modern fighter jets in line with modern technology, little more is known about the status of the J-20's development, though Tang Changhong, Chief Designer of China's Y-20 transport plane and member of the Chinese People's Political Consultative Conference National Committee, told West China City Daily that the Y-20 will be equipped with Chinese developed engines between 2018-2019. It is expected that the J-20 stealth fighter will receive its own locally produced engines during the same period.

www.chinadaily.com

Report outlines tungsten market

Tungsten is one of the key refractory metals and this high melting point material has numerous applications, including incandescent light bulb filaments, X-ray tubes, electrodes in TIG welding, superalloys and radiation shielding. Tungsten's hardness and high density give it military applications in penetrating projectiles and is the key ingredient in tungsten carbide-based tools.

Roskill Information Services Ltd has published its assessment of the tungsten market in terms of supply, pricing, demand in key application areas and future outlook. Roskill states that China has increased its share of world tungsten use from just under 30% in 1996 to nearly 60% in 2016 when world consumption of contained tungsten reached just over 100,000 tonnes. China is also still the world's main producer of primary tungsten accounting for almost 80% of output in 2016.

In terms of end uses, cemented carbides remained the largest consuming sector in 2016 at more than 50% of total tungsten demand. This sector grew by more than 4% per annum between 2008 and 2016, mainly propelled by growth in China. Recent trends in cemented carbides have included the development of nano-grained WC products showing both improved hardness and fracture toughness.

The 'Tungsten: Market Outlook to 2026', 12th Edition, is available from Roskill Information Services Ltd,

www.roskill.com

Excalibure to create PM centre of excellence in France

France's PNB, The University of Burgundy and Ecosphère Agency have announced the official creation of Excalibure, a new association of industrial and academic partners collaborating in various Powder Metallurgy projects. During a recent meeting held at the Pavillon de L'Industrie in Le Creusot, France, Jean-Claude Lenain, Chairman of PNB, was announced as the association's first President.

The reported objective of Excalibure is to create a chain of industrial partners, facilitating the sharing of research and development and the growth of the industry. Since its creation, companies Areva, Arcelor Mittal, Solcera and Lisi Aèrospace have committed to a relationship with the association and will work in partnership with the University of Burgundy, Ecosphere and the PNB on future projects.

Plans were also announced to have a centre of excellence in Powder Metallurgy established in Creusot early 2019. The centre will make it possible for the partnered companies to pool their investment costs, diminishing individual economic risk. The total planned investment for the project is around €10 million, including €7 million for equipment which will also include a new Hot Isostatic Press.

Stéphanie Corre, head of the Centre for the Research of Materials in Creusot, stated, "The sharing of the Excalibure platform allows us not to invest alone in this major project, which reduces our risk- taking and allows us to be confident in the viability of this project."

www.polenucleairebourgogne.fr
en.u-bourgogne.fr
www.ecosphere.fr

Ford details $4.5 billion investment plans in electric vehicle push

Ford has announced further details of its plan to invest $4.5 billion in electrified vehicles by 2020. The automaker has listed seven of the 13 new global electrified vehicles it plans to introduce, including hybrid versions of its F-150 pickup and Mustang in the USA, a plug-in hybrid Transit Custom van in Europe and a fully electric SUV with an expected range of at least 300 miles for customers globally.

"As more and more consumers around the world become interested in electrified vehicles, Ford is committed to being a leader in providing consumers with a broad range of electrified vehicles, services and solutions that make people's lives better," stated Mark Fields, Ford President and CEO. "Our investments and expanding line-up reflect our view that global offerings of electrified vehicles will exceed gasoline-powered vehicles within the next 15 years."

Ford also announced it plans to spend $700 million expanding its Flat Rock Assembly Plant in Michigan to create a factory that will build high-tech autonomous and electric vehicles along with the Mustang and Lincoln Continental. The investment in Flat Rock comes from $1.6 billion the company had previously planned to invest in a new plant in San Luis Potosi, Mexico, which has now been cancelled.

Ford added that, to improve company profitability and ensure the financial as well as commercial success of this vehicle, the next-generation Focus will be built at an existing plant in Hermosillo, Mexico. This will make way for two new iconic products at Michigan Assembly Plant in Wayne, Michigan, where Focus is manufactured today – safeguarding approximately 3,500 U.S. jobs.

"I am thrilled that we have been able to secure additional UAW-Ford jobs for American workers," stated Jimmy Settles, UAW vice president, National Ford Department. "The men and women of Flat Rock Assembly have shown a great commitment to manufacturing quality products and we look forward to their continued success with a new generation of high-tech vehicles."

Ford is said to be focusing its EV plan on its areas of strength by electrifying its most popular, high-volume commercial vehicles, trucks, SUVs and performance vehicles to make them even more capable, productive and fun to drive.

www.ford.com

A hybrid version of Ford's best selling F-150 will be available by 2020

Scanning lasers offer potential for surface hardening of steel components

Researchers at the UPV/EHU-University of the Basque Country, Spain, have announced that they have validated the use of scanning optics for laser hardening, which allows the process to be adapted to the shape of the part.

The UPV/EHU's High Performance Manufacturing group has conducted the study and tuning of an innovative technology to carry out this process. It involves using laser, but, unlike the traditional system, it uses scanning optics, which gives the thickness of the part to be treated great capacity for adaptation.

When hardening is carried out using a highly localised heat source, such as a laser, it enables only the surface to be hardened, leaving the core of the parts in their original state. "The parts are not so brittle and as little heat is inserted, the part does not become as distorted. In the end, what the heat does is deform the part and that means it has to be finished using other methods," stated Aitzol Lamikiz, Professor of the UPV/EHU's department of Mechanical Engineering and member of the High Performance Manufacturing group that carried out the research.

In industry the laser hardening process has been used since around 2000, however, according to Lamikiz, it has a limitation. "The laser sweeps a constant bandwidth so the hardened zone thus ends up with a constant thickness." In order to make the technology more flexible, this research group at the UPV/EHU decided to assess the viability of incorporating moving, scanning optics into this process.

The team used a galvanometric scanner which moves a very small laser at great speed, sweeping the surface line by line. That way, the hardening width can be adapted simply by changing the program parameters. Drawing an analogy between the hardening treatment and the process of painting a wall, Lamikiz explained that conventional laser hardening "would be like painting the wall with a roller, so the width that is painted corresponds to that of the roller. However, with the new technique, we substitute the roller for a marker with the finest point."

"It was possible to use this technique to carry out the hardening. Then we gradually saw how the result of the treatment changed according to the speed of the laser movement, the power used, etc. According to our tests, when the laser moves very fast, the results are similar to those of the conventional process," Lamikiz stated.

Promising results

Exploring the possibility of using this methodology further, the UPV/EHU's department of Mechanical Engineering ran a project known as Hardlas in collaboration with companies in the Basque Country and Piedmont, Italy, to see how far the process was viable. "We can say that the project was a success as we saw that it was viable and that it could be transferred to industry," said the researcher.

The laser hardened test part (Courtesy Aitzol Lamikiz)

Although they have tested the viability of the process, there are still steps to be taken to get as far as industrial production. One of the main difficulties they came up against was controlling the process. "It is very important to get the material treated to the necessary temperature so that the treatment takes place, but it must not be exceeded otherwise we would melt the material. In our process, as the laser is constantly moving, control is more complex," explained Lamikiz.

The tests were carried out at the university using lab equipment. "To use the process on an industrial scale, it would be important to try it out with more powerful lasers, different types of lasers, on other materials, etc.," added Lamikiz.

www.ehu.eus

POWDERMET2017 conference programme published and student grant support announced

The Metal Powers Industry Federation (MPIF) has published the Conference Programme and opened registration for POWDERMET2017, Las Vegas, Nevada, USA, June 13 – 16, 2017. The International Conference on Powder Metallurgy and Particulate Materials will feature presentations from over 200 worldwide PM industry experts on PM,

The conference will be held at the Bellagio Hotel in Las Vegas

particulate materials and metal Additive Manufacturing."This year's conference hosts a packed technical program with the latest innovations in PM, particulate materials and metal Additive Manufacturing," stated Dan Messina, Technical Manager, MPIF.

The conference opening general session will feature a keynote presentation from Todd Grimm, T.A. Grimm & Associates, on 'Navigating the Metal Additive Manufacturing Landscape'. The author will blend industry updates, trends and insights, helping to cut through the hype and identify when, where and why metal Additive Manufacturing makes sense.

POWDERMET2017 shares several events and an exhibit hall with the co-located AMPM2017, the fourth annual Additive Manufacturing with Powder Metallurgy conference. Over 100 exhibitors will showcase PM and metal AM processing equipment, powders and products.

It was also announced that the US National Science Foundation has approved a grant programme to support forty students from US institutions to attend the POWDERMET2017 and AMPM2017 conferences. The awards will cover the conference registration fee and three nights hotel accommodation.

"We continually see that there is a shortage of skilled workers. This is a huge opportunity for students interested in pursuing careers in engineering or materials science," stated MPIF Executive Director and CEO Jim Adams. "Students will be able to meet and learn from some of the best engineers and component designers in the industry by attending technical sessions, special interest programs, walking the exhibition hall and networking during industry luncheons and evening events."

www.mpif.org

Chemical company looks to become off-road auto manufacturer

One of the world's largest chemical groups, UK based Ineos, has announced plans to build a brand new 'uncompromising 4x4 off-roader'. Whilst the global supplier of petrochemicals, speciality chemicals and oil products, is one of the largest manufacturers in the world, it will be the first time that the company has produced a vehicle.

Following the completion of a six month feasibility study, Ineos stated that it sees a gap in the market for the off-roader following Jaguar Land Rover's decision to cease production of its Land Rover Defender. "This is a fantastically exciting project," stated Jim Ratcliffe, Ineos Chairman. "We want to build the world's purest 4x4 and are aiming it at explorers, farmers and off-road enthusiasts across the globe."

The company is expecting to spend many hundreds of millions on the project and is determined that the vehicle will not only fill a gap in the market vacated by the Defender but also provide a step change improvement in build quality and reliability. Identification of a suitable manufacturing location will be a part of the next phase of the project and the company said it will look at UK sites alongside other European options.

The project is to be run by Dirk Heilmann, formerly head of Engineering and Technology at Ineos. Heilmann, now CEO of Ineos Automotive, has already started recruiting a team of automotive experts and stated, "This is an amazing project for everyone involved. Our job is to create the world's best 4x4 and we are already moving forward with our plans."

The vehicle will not be a replica of the Defender but will reflect its philosophy. The target market is global and includes agriculture and forestry workers, explorers and adventurers as well as traditional Defender fans that simply enjoy an authentic 4x4 driving experience.

"Our job is to create the world's best 4 x 4 and we are already moving forward with our plans," stated Jim Ratcliffe, Ineos Chairman

"I am a great admirer of the old Land Rover Defender and have enormous respect for its off road capability," added Ratcliffe, "Our new 4x4 has been inspired by it. But whilst our off-roader might share its spirit, our new car will be a major improvement on previous models."

www.ineos.com

New laboratory furnaces from Linn High Therm

Germany's Linn High Therm has added to its range of laboratory furnaces. The company is offering the VMK furnace for temperatures up to 1800°C as well as the VMK-S and VMK-Vac options suited for up to 1200°C. Applications include brazing, annealing, ashing, tempering, sintering, reducing, pyrolysis etc.

Special features of the furnaces are said to include stainless steel housings for use in corrosive environments, easy operation, fast heating and fast cooling cycles. Fibre insulation is used for lab and high temperature furnaces (VMK) with a heat resistant gas-tight muffle insert (1.4841/Inconel). A water cooled door flange enables protective gas and/or vacuum operation (VMK-Vac) in case of protective gas/vacuum furnaces (VMK-S).

Linn standard laboratory furnaces are available in sizes 1–25 l and 0.6–5.0 kW.

www.linn-high-therm.de

Inert to debut new powder handling glove box

Inert, a leading manufacturer of hermetic enclosures and related systems based in Amesbury, Massachusetts, USA, has announced it will debut its new standard glove box enclosure for powder handling from its booth at this year's Rapid + TCT. Designed for the unique requirements of Additive Manufacturing materials, Inert's new powder handling system offers a safe and efficient way to work with, package and store metal powders.

The company stated that it collaborated with several key AM/3D printing companies in 2015 and 2016. "We've taken the solutions Inert engineered for our customers in Additive Manufacturing and combined them into a new, standard glove box model," stated Inert President, Daniel Clay.

"Companies working with metal powders like titanium understand that an inert environment is essential for structurally solid printed results. Our new glove box system has several additional features that make it the ideal solution to the problems these companies face with powder handling and storage," added Clay.

The new powder handling glove box is equipped with a rotating tilt table, ultrasonic vibration, argon gun for de-powdering, unidirectional flow to funnel powders to a collection well and powder storage kegs. The debut of this new system will be coupled with Inert's Argon-2, a closed loop gas management system that provides a continuous recirculation, purification and analysis of the argon environment inside the hermetically sealed glove box. The Argon-2 is one model in a suite of gas management systems offered by Inert to provide safe working conditions for oxygen and water sensitive applications.

Rapid + TCT takes place May 9-11 at The David L Lawrence Convention Centre in Pittsburgh, Pennsylvania, USA. Inert can be found on booth 1147.

www.rapid3devent.com
www.inerttechnology.com

Sinterite installs new furnace at Air Liquide Research & Technology Centre

Sinterite, a Gasbarre Furnace Group Company, has announced the installation of a continuous belt furnace at the Air Liquide's Shanghai Research & Technology Centre in China. The centre, opened in 2016, houses offices, showrooms, laboratories and a pilot demonstration department.

Sinterite's nine-zone, 305 mm wide furnace will be used in the development of Air Liquide's sintering, brazing, annealing and heat treatment applications. The electrically-heated furnace can be used with multiple configurations of atmosphere gas set-up (H_2, N_2, NH_3, O_2 and CO_2) and can be heated to 1150°C (2100°F) and rapidly cooled using the Sinterite HyperCooler.

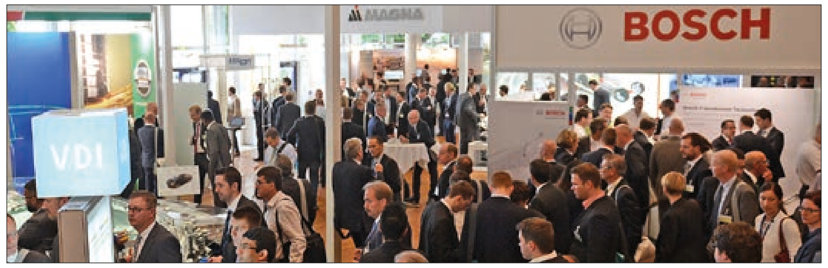
The continuous belt furnace is reported to have advanced monitoring and recording capabilities for process development and is designed primarily for Powder Metallurgy sintering processes, with dedicated delubrication, sintering and cooling sections.

Air Liquide, headquartered in Paris, France, produces gases, technologies and services for Industry with a presence in 80 countries. The company employs approximately 67,000 staff and in 2016 reported revenue of €18.1 billion.

www.sinterite.com
www.airliquide.com

Sinterite furnace designs are modular and configurable to meet specific sintering needs

Drivetrain for Vehicles congress announces move to Bonn



Following many successful events in Friedrichshafen, this year's International VDI Congress 'Drivetrain for Vehicles' moves to Bonn, Germany. Taking place at the World Conference Centre, July 5-6, 2017, the 17th event in the series will once again offer a conference and exhibition focussed on all aspects of transmission and powertrain technology.

The exhibition, which over the years has grown to become a major marketplace, will see over 100 organisations from around the world present their solutions covering all aspects of the automotive transmission and its components.

Likewise, the International Congress will be accompanied by several events. The VDI conference 'Control and Regulation of Transmissions' where the optimisation potential in transmission control (e.g. optimised driving properties, making gearshift systems more comfortable, reduced fuel consumption and lower noise level) will be addressed.

Key topics at the 'Drivetrains for Vehicles' congress include:

- Hybrid concepts and hybrid transmissions
- 48V hybridisation
- Simulation/testing technology
- Dual clutch transmission
- Automation transmission/CVT
- Control and regulation of transmissions
- Drivetrains for Commercial vehicles
- Electric drive

Running in parallel will be the VDI conference 'Drivetrains for Commercial Vehicles'. In this conference experts will share the latest innovations and industry trends, showcase current development achievements and give an overview of the present market situation.

Organised by the Association of German Engineers (VDI), the two day congress is expected to attract over 1,500 participants.

www.vdi-wissensforum.de

CleanTech laser system offers cleaning and surface preparation

Fonon Corporation, Orlando, Florida, USA, has announced the release of its CleanTech™ product line for surface preparation, paint removal and surface cleaning. Marketed under the Laser Photonics™ brand, the CleanTech laser systems offer a non-abrasive cleaning process that is said to be safer and more eco-friendly than traditional methods.

The CleanTech product line can be used for various applications across a multitude of industries including mould cleaning, weld preparation, metal parts cleaning and degreasing, amongst others. The five axis system incorporates three mechanical XYZ axes coupled with two optical XY axes. It processes a wide range of materials with special attention to highly-reflective metals.

"CleanTech laser systems remove coatings, contaminants and residues with the use of high energy laser sources and are the most cost-effective and, to a degree, revolutionary method of industrial cleaning and surface preparation," stated Dmitriy Nikitin, CEO of Fonon.

www.laserphotonics.us

Rare earths market outlook to 2026

Roskill Information Services Ltd, London, UK, recently published its market outlook for rare earth metals to 2026 with the focus on applications in the permanent magnet and catalyst market sectors. The report states that catalysts will continue to drive growth in the light rare earth elements lanthanum and cerium, whilst neodymium, praseodymium and dysprosium will see growth in permanent magnets. Supply of some rare earths is said to be far greater than that of others as a result of production methods and there is a discontinuity between supply and demand across the different elements. Despite growth in catalyst demand (and, to a lesser extent, polishing and nickel metal hydride battery), cerium and lanthanum will remain in substantial surplus to 2026. However, it was stated that demand for neodymium is beginning to outstrip supply.

Roskill states that in the short term to 2021, neodymium-iron-boron (NdFeB) permanent magnet demand is forecast to grow strongly. The traditional consumer electronics and automotive sectors currently account for the majority of NdFeB demand, but growth is also expected from the emerging green technologies such as wind turbines and new energy vehicles (NEVs). Between 2016 and 2021, global NdFeB magnet production is forecast to grow by 4–5% per annum. Global NEV production is forecast to rise to around 3.5 – 4.0 million vehicles in the same period, while global wind power installations could increase by 0.4M MW according to Roskill's report.

As a result of increasing consumption, neodymium (Nd) was expected have fallen into supply deficit in 2016, and this deficit is forecast to continue increasing to 2021. This would make continued growth of NdFeB magnets unsustainable, despite efforts by rare earth producers to increase neodymium supply. This is expected to result in price rises for Nd to a point where magnet consumers will begin to replace NdFeB magnet technologies with substitute materials. The green energy sector is the most vulnerable to NdFeB price rises because of the large size of magnets used in wind turbines. Technologies already in use in this industry include induction/synchronous generators in wind turbines as an alternative to permanent magnet motors, and induction motors in NEVs.

Roskill states that by 2021, it is expected that the high price of Nd and concerns over supply availability will make projected growth rates of NdFeB permanent magnets unsustainable, and demand for these magnets is forecast to fall rapidly from 2022, before stabilising at a much lower growth rate. Overall, NdFeB magnet growth between 2021 and 2026 is forecast to be flat, possibly falling by up to -1% per annum, but Nd prices will continue to rise. The price increase for most other rare earth metals will, however, be limited due to supply surpluses.

www.roskill.com

Seco/Warwick patents ground-breaking temperature control solution for heat treating furnaces

Seco/Warwick, Poland, has announced plans to patent a new temperature control solution for heat treating furnaces. Working to meet the stringent requirements of the aerospace industry, engineers at the company are reported to have designed a method enabling continuous control over the heating and cooling of the entire heat treatment cycle in multi-chambered furnaces.

The reported solution addresses the inclusion of thermocouples during the heat treatment cycle, which move between chambers attached to the tray. Temperature control is achieved based on a special construction that allows the operator to monitor the temperature in both chambers, providing full temperature control of processed parts in multi-chamber vacuum furnaces during the entire process.

The solution has been successfully implemented in a two-chamber vacuum furnace, Seco/Warwick's CaseMaster Evolution®, which is used for vacuum case hardening in the aerospace, automotive machinery, wind energy, transmission and commercial heat treatment industries.

"All of our customers set the bar high and expect the equipment of the highest quality that will meet the strict industry standards and their expectations in terms of technology implementation. Nevertheless, the aerospace industry sets the bar the highest. Meeting such requirements would not be possible without the regular introduction of product and technology innovations, like this one," stated Katarzyna Sawka, Global Marketing Director at Seco/Warwick. "The combination of the rich, long-term experience and expertise of Seco/Warwick with the latest technologies leads to the introduction of another ground-breaking solution that, by enabling constant temperature control, provides customers with a heat treatment process of even higher quality and efficiency."

www.secowarwick.com

The system has been successfully built into a CaseMaster Evolution vacuum furnace

The changing landscape of carbonyl iron and nickel powder production

Until recently, much of the global production of carbonyl metal powder was centred on manufacturers in Europe and North America. Although China has produced these powders since the 1960s, the last ten years or so has seen a rapid increase in the volume of both carbonyl nickel and carbonyl iron powders produced in this region. In this report, Jun Shu and Lou Koehler describe the production processes and look at the changing landscape of carbonyl metal powder production.

Carbonyl nickel refining was first commercialised in 1902 by the Mond Nickel Company Ltd, with carbonyl iron powder production being developed in 1925 by Germany's BASF (formerly I G Farben). Today, carbonyl nickel powders are widely used in Powder Metallurgy, battery and fuel cell electrodes, hardmetal binders, welding rods, high-temperature filters, conducting additives, electronic materials, anti-seize lubricants, chemicals and catalysts (Fig. 1). Typical applications of carbonyl iron powders include Metal Injection Moulding (MIM), magnetic cores, hardmetal binders, radar absorption materials, magneto-rheological fluids for shock/vibration damping, precision polishing, industrial diamond synthesis and health supplements.

Global carbonyl nickel powder production capacity has reached around 37,000 metric tons per year, with the main producers being Vale in Canada and Wales, China's Jinchuan and Norilsk in Russia. Carbonyl iron powder production capacity is reported to be around 29,000 metric tons per year, with BASF being the largest manufacturer. However, nearly half of the current carbonyl iron powder capacity is represented by recent additions from China.

Carbonyls of nickel and iron, of molecular forms Ni(CO)$_4$ and Fe(CO)$_5$, were discovered by Dr Ludwig Mond and his colleagues in 1890 and 1891, respectively [1, 2]. Subsequently, carbonyl nickel refining, or the Mond process, was first commercialised in 1902 by the Mond Nickel Company in Clydach, South Wales, to produce high-purity nickel pellets [3]. Carbonyl nickel powder

Fig. 1 Commercial applications of discrete carbonyl nickel powder

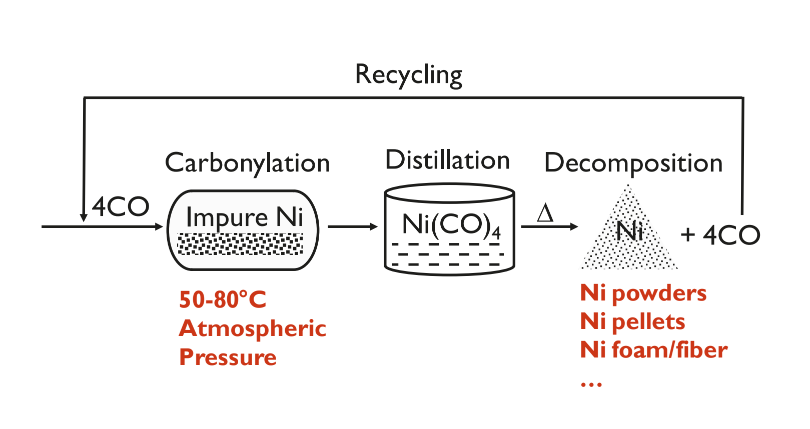


Fig. 2 Schematic diagram of carbonyl nickel extractive metallurgy

production was an invention of the German company BASF in the late 1920s with nickel matte feedstock acquired from the Mond Nickel Company [4].

The International Nickel Company (Inco) acquired the Mond Nickel plant in the 1920s and, in 1943, commercial nickel powder production began. In 1973, Inco opened a second carbonyl plant in Sudbury, Canada, the site of its major nickel mining operations [5]. These plants have been continually modernised and now operate under the ownership of the Brazilian mining giant Vale SA. This mature process is acknowledged as the best available technology for refining pure nickel. The three main reasons for this are the ability to produce a very high purity product, the low energy levels consumed in the process and the fact that, although the process uses highly toxic process intermediates, there are no polluting waste products, as virtually all the carbon monoxide gas is recycled and emissions of toxic carbonyls are controlled in the parts-per-billion concentration range.

The original carbonyl process, still used at Vale's Wales refinery, harnesses the ability of nickel in an impure form to be extracted into a nickel carbonyl gas (boiling point 43°C) at ordinary temperatures and then restored to a pure metallic state by gentle heating. Production begins with a nickel oxide feedstock. The nickel oxide is continuously fed into a reduction kiln, where it is tumbled in a stream of pure hydrogen at ~230°C to produce impure nickel in granular form. In the second stage, the volatilisation kiln, the nickel reacts with carbon monoxide at close to atmospheric pressure to form nickel carbonyl gas, or nickel tetracarbonyl $Ni(CO)_4$:

$$\mathrm{Ni} + 4\mathrm{CO} \underset{180-250℃}{\overset{50℃}{\rightleftharpoons}} Ni(CO)_4$$

$$K_{eq}\ (50℃) = 3.78\mathrm{x}10^4 \qquad (1)$$

The nickel carbonyl gas is then piped to an adjacent plant for thermal decomposition into pure nickel pellets or powders. To produce powder, the nickel carbonyl gas is injected at a metered rate into the top of the decomposer towers. The walls of the towers are heated to 300-500°C. The gas decomposes instantly to form nickel powder which settles at the bottom of the unit. The powder is collected, blended for uniformity, screened and packaged. A schematic of the carbonyl nickel extractive metallurgy is shown in Fig. 2.

When an iron component is present in the feedstock, iron carbonylation occurs to form iron carbonyl, or iron pentacarbonyl $Fe(CO)_5$:

$$\mathrm{Fe} + 5\mathrm{CO} \underset{190-300℃}{\overset{150-200℃,\,25\,MPa}{\rightleftharpoons}} Fe(CO)_5$$

$$K_{eq}\ (50℃) = 5.67\mathrm{x}10^{-2} \qquad (2)$$

At ambient pressure, as practised at Vale's Wales refinery, the iron carbonyl formation equilibrium is negligible in comparison with nickel carbonyl formation. However, this parallel reaction equilibrium increases with pressure, which occurs at the more modern facilities in Vale Canada, Norilsk and Jinchuan, thus making it necessary to separate nickel carbonyl from iron carbonyl in a high pressure carbonyl process in order to produce pure nickel products. In fact, high pressure is fundamental to carbonyl iron synthesis in order to produce carbonyl iron powders upon thermal decomposition.

With the increase of decomposition temperature, a side reaction of CO disproportionation, or the Boudouard reaction, becomes important, resulting in residual carbon deposition on nickel and/or iron particles in specific refining processes:

$$2\mathrm{CO} \underset{850℃}{\overset{450℃}{\rightleftharpoons}} C + CO_2$$

$$K_{eq}\ (250℃) = 9.53\mathrm{x}10^7 \qquad (3)$$

Although the Boudouard reaction is thermodynamically favourable at low temperature, its reaction rate is kinetically low until about 450°C in the atmospheric pressure decomposition process. Increasing pressure shifts the position of equilibrium towards the right-hand side, resulting in more residual carbon formation.

The beauty of carbonyl metal refining (for nickel or iron) is that, following the metal extraction and distillation, gaseous metal carbonyl can be thermally decomposed back to high purity metal and carbon monoxide. Under precisely controlled thermal decomposition conditions such as temperature, feed rate, carbonyl concentration, partial pressure and introduction of additives, carbonyl nickel, iron or ferronickel powders can be produced in different morphologies and particle size distributions ranging from micron size powders to centimetre size pellets.

Carbonyl nickel powder production

Worldwide carbonyl nickel powder production capacity trends since the new millennium are shown in Fig. 3. Driven by booming industrial needs, Chinese carbonyl metal refineries have added nearly 20% carbonyl nickel powder capacity in the last ten years. It should be noted that, although world capacity is in excess of 37,000 metric tons per year, demand for carbonyl nickel powders is in the 25,000 metric tons per year range.

Carbonyl refining requires strict process safety measures, throughout the entire refinery operation, due to the extremely toxic nature of nickel carbonyl. For many decades, carbonyl nickel refining was the exclusive domain of the mining companies Inco Limited in Canada and Wales (acquired by Vale in 2006) and Norilsk Nickel in Russia. In 2015, a third mining company, Jinchuan in China, also commercialised carbonyl refining of nickel pellets and powders [6]. Below is a brief description of existing carbonyl nickel refining operations, with a comparison of characteristic operating parameters listed in Table 1.

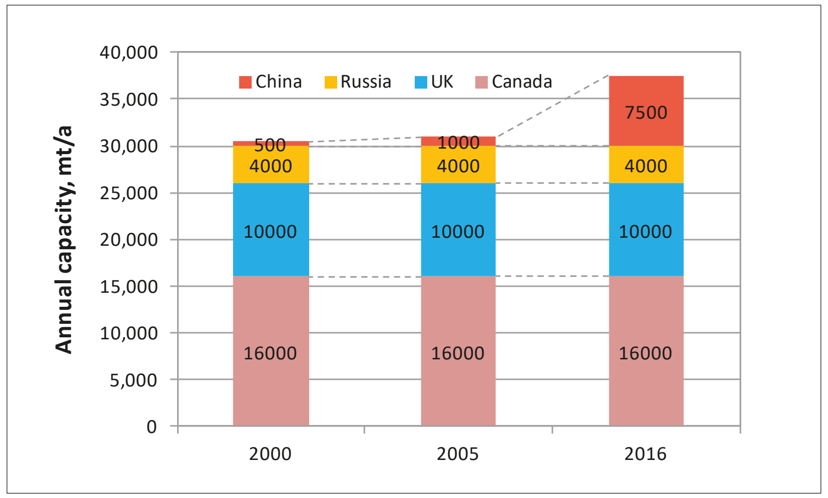


Fig. 3 Worldwide carbonyl nickel powder production capacity

United Kingdom: Vale Clydach Refinery

The first ever carbonyl refinery still uses the atmospheric pressure carbonyl refining process. Production begins with a nickel oxide feedstock received from mines in Sudbury, Canada. Powder is produced in the final stage of the process by injecting nickel carbonyl gas at a metered rate into the top of one of eight decomposer towers, each ten metres in height by two metres in diameter [7]. By adjusting feed rates and temperatures, the powder particle morphology can be adjusted.

Two separate primary shapes are produced by different decomposition conditions; a spiky discrete powder and a filamentary powder. Combined nickel pellet and powder production capacity at Clydach is estimated at 45,000 metric tons per year, of which 10,000 metric tons are produced in powder form.

Canada: Vale Sudbury Refinery

This refinery was commissioned in 1973 using an intermediate pressure carbonyl refining process to increase the nickel extraction yield. In intermediate pressure nickel carbonyl refining, nickel containing metallics are batch-charged into a rotating carbonylation reactor and allowed to react with carbon monoxide at ~7.0 MPa and 170°C [7, 8]. Nickel carbonyl and a fraction of iron carbonyl are condensed for storage, followed by distillation to separate nickel carbonyl and iron carbonyl based on their different boiling temperatures. Pure nickel carbonyl from the top of the distillation column is the feed for production of carbonyl nickel powders and pellets. Powder is produced in ten decomposer towers, each ten metres in height by two metres in diameter [7]. The mixture of nickel carbonyl and iron carbonyl from the bottom of the distillation column is the feed for production of FeNi pellets.

Refinery	Vale Clydach Nickel Refinery	Vale Sudbury Nickel Refinery	Norilsk Nickel Kola MMC	Jinchuan Carbonyl Refinery
Feed	Impure Ni oxide	Impure Ni metallics	Ni metallics, Off-spec Ni	Ni metallics, Off-spec Ni
Pressure (MPa)	Atmospheric	7.0	22.5	7.0-9.0
Temperature (°C)	50-60	170	150-250	150-220
Annual capacity (mt/a)	45,000 (10,000 powder)	60,000 (16,000 powder)	5,000 (4,000 powder)	10,000 (5,000 powder)
Ni extraction (%)	90	97-98	97-98	97-98
Commissioning year	1902	1973	Early 1960s	2015
Typical products	Ni powders, Ni pellets	Ni powders, Ni pellets FeNi pellets	Ni powders, Ni pellets	Ni powders, FeNi powders, Fe powders, Ni pellets

Table 1 Comparison of commercial carbonyl nickel refining processes

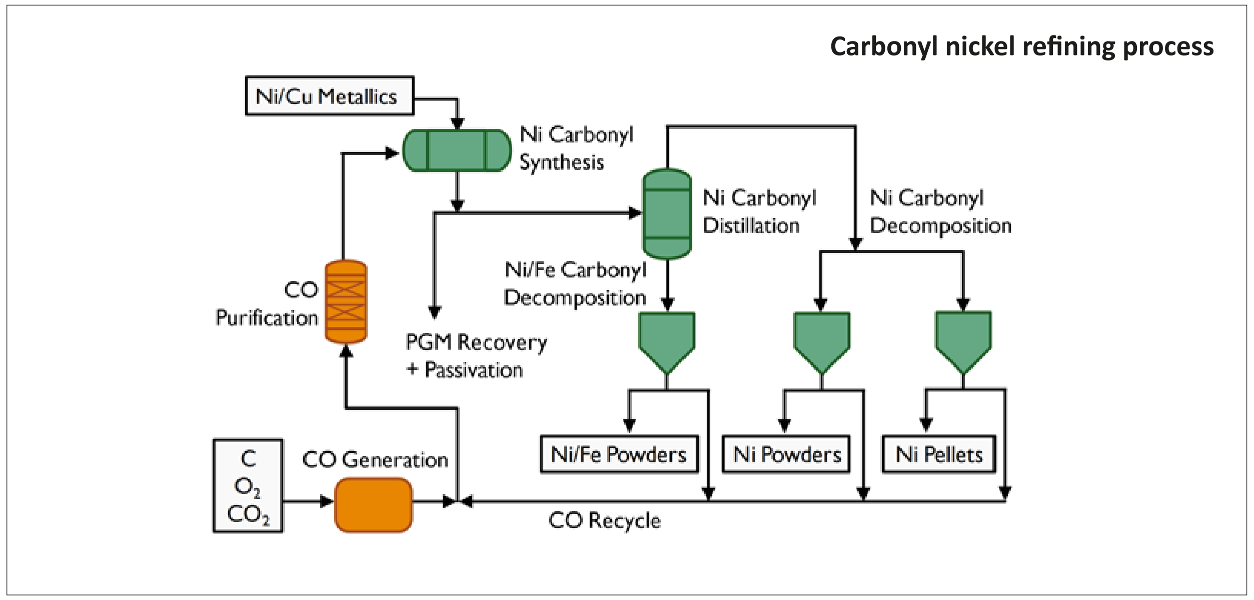


Fig. 4 Jinchuan nickel carbonyl refining process flow-sheet

It should be mentioned that, in the original design of the Vale Sudbury refinery, this stream was used to produce FeNi powder [9]. Carbon monoxide from the decomposers is compressed and cycled back to the carbonylation reactor. Estimated annual capacity in Sudbury is around 60,000 metric tons, of which 16,000 metric tons is in powder form. Powder capacity is higher here than at Clydach due to higher carbonyl gas strength (~40% versus ~14% [7]) generated by intermediate pressure refining.

Russia: Norilsk Monchegorsk Refinery

Carbonyl nickel refining in Russia can be traced back to the initial German activities at I G Farben before World War II. Two small carbonyl plants were developed by I G Farben in Germany in the late 1920s for the production of carbonyl iron and carbonyl nickel powders. As an extension of its original iron carbonylation technology, I G Farben conducted nickel carbonylation at a high pressure of ~20.0 MPa and 200°C. Carbonyl nickel powder production promoted the development of nickel-cadmium batteries suitable for tanks and military aircraft during the 1930s [4]. After the end of World War II, Russia is said to have dismantled and moved one of the I G Farben carbonyl plants, located in the Soviet Occupation Zone, to Monchegorsk on the Kola Peninsula (now Norilsk Nickel) in north-western Russia and constructed a commercial plant for production of carbonyl nickel powders in the early 1960s [10].

Norilsk Nickel practises high pressure carbonylation at around 22.5 MPa and 150-250°C. Off-spec electrolytic nickel cuts and/or granular nickel metallics are batch charged in a fixed carbonylation tower to react with a continuous carbon monoxide feed, followed by fractional distillation of nickel and iron carbonyls. Subsequent decomposition of nickel carbonyl in one-metre diameter decomposer towers yields nickel powder and nickel pellets [10]. Estimated capacity at Norilsk is around 5,000 metric tons per annum, of which 4,000 metric tons is in powder form.

China: Jiangyou Hebao Nanomaterials Co Ltd

The Chinese carbonyl nickel industry debuted in the early 1960s with the development of the nuclear industry. The former state-owned 857 Factory was built in the mountainous region of Jiangyou, Sichuan Province for the production of carbonyl nickel powders under the sponsorship of the Chinese Ministry of Nuclear Industry. This plant developed a high pressure carbonylation process at about 150-180°C and 15.0-20.0 MPa, with scrap nickel as the feedstock. Annual production capacity of carbonyl nickel powders was up to a few hundred metric tons for China's nuclear industry use [11,12]. With technological advancement in the nuclear industry and the increased availability of imported carbonyl nickel powders into China, the former state-owned 857 Factory was gradually phased out at the beginning of the new millennium. Its core carbonyl technology business was then restructured as Jiangyou Hebao Nanomaterials Co., Ltd., for the production of small volumes of carbonyl nickel powders and carbonyl iron powders for niche markets.

China: Jinchuan Carbonyl Refinery

Development of carbonyl nickel refining technology at Jinchuan Group started in the late 1990s following the Chinese economic boom. A pilot plant with a designed carbonyl nickel capacity of 500 metric tons was built in the early 2000s in order to establish the engineering basis for a commercial plant. Various technical issues were solved, ranging from carbonylation to subsequent carbonyl decomposition into high purity nickel products, as reflected by over 70 Chinese patent applications. In 2015, Jinchuan Group commissioned a new carbonyl refinery with a design capacity of 10,000 metric tons of carbonyl nickel products, of which half are in powder form. Fig. 4 shows the basic flow-sheet of Jinchuan carbonyl nickel refining.

Jinchuan's nickel resource is sulphide ores, similar to Vale and Norilsk. Their carbonyl refining unit operations are based on similar principles. Process deviations are mainly driven by availabilities of feedstocks at Jinchuan Group. In the Jinchuan carbonyl refining process, the starting raw materials include Ni/Cu metallics from smelting, nickel shot and electrolytic nickel scrap. Carbon monoxide is generated from coal burning and purification. Initially, the pilot plant adopted the high pressure carbonylation route [11]. Through extensive assessment and improvement of the technology, the operating pressure was decreased to the intermediate pressure range (refer to Table 1) and applied to the commercial refining process [12]. Due to the simultaneous carbonylation of iron impurities, distillation is necessary to separate nickel carbonyl from iron carbonyl before the final thermal decomposition into carbonyl nickel products such as nickel powders and nickel pellets. Precise control of nickel powder particle size distribution and morphology is readily achieved through sophisticated modern process control systems.

China: Jien Nickel

It should be mentioned that carbonyl nickel refining has also been practised at Jilin Jien Nickel Industry Co., Ltd., in the north eastern Chinese Province of Jilin since 2004, utilising the consulting services of the Canadian CVMR Corporation [13]. An atmospheric pressure carbonylation process was adopted in this refinery, using imported nickel oxide as feedstock. The design capacity of carbonyl nickel powders at Jien Nickel was 2,000 metric tons per year. Due to limitations in feed material supplies, Jien Nickel later tested a nickel hydroxide feed derived from a lateritic ore leaching process [14, 15]. While Jien is still optimising the atmospheric pressure carbonylation process, their production activity remains minor on the global carbonyl nickel powder supply market.

Characteristics of carbonyl nickel powder

In general, carbonyl nickel powders with an apparent density lower than 1.0 g/cm^3 are referred to as light nickel powders, often in the form of filamentary morphology (three-dimensional chain of fine particles fused together), while carbonyl nickel powders with an apparent density over 1.0 g/cm^3 are referred to as heavy nickel powders, often in the form of discrete particle shapes. Fig. 5 shows typical microscopic images of filamentary and discrete carbonyl nickel powders produced by the Jinchuan Group. Their N series carbonyl nickel grades are named in relation to their apparent density (bulk density), e.g., N24™ is for nickel powder with an average apparent density of 2.4 g/cm^3, and N06™ with an average apparent density of 0.6 g/cm^3. Jinchuan filamentary carbonyl nickel powder N06™ is equivalent to Vale Type 255™ nickel powder, and their discrete nickel powder N24™ is equivalent to Vale Type 123™ nickel powder [16] and Norilsk UT3™ nickel powders.

Table 2 gives a comparison of typical physical and chemical properties of commercially available carbonyl nickel powders [6,17,18]. For general descriptions of carbonyl nickel powder properties, refer to reference [6]. Due to their high purity and well-defined microstructures, carbonyl nickel powders have found growing industrial applications. A list of primary applications for filamentary and discrete carbonyl nickel powders is as follows:

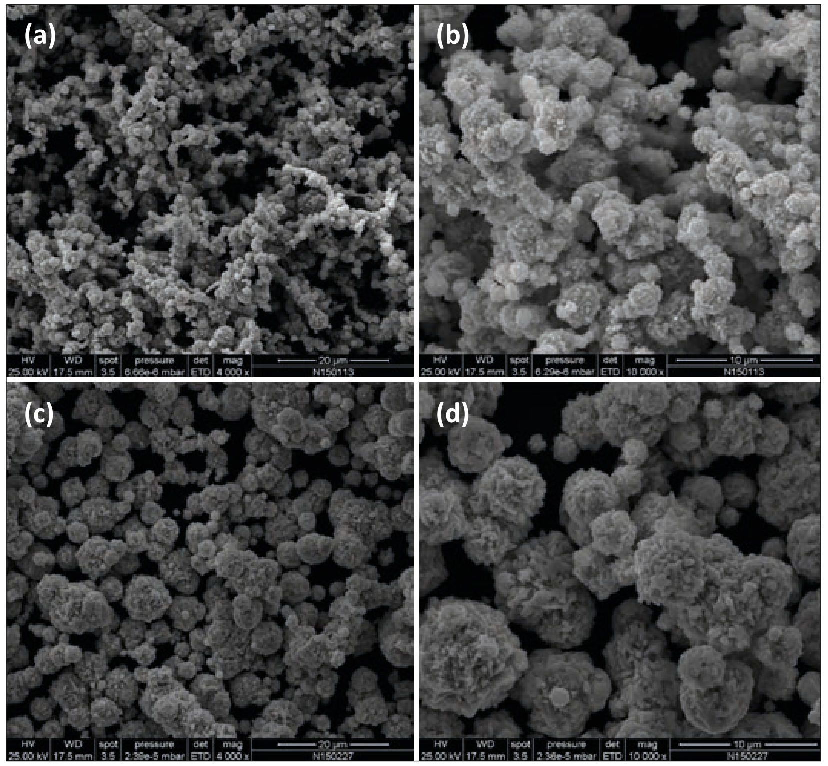


Fig. 5 Typical SEM images of carbonyl nickel powders produced by the Jinchuan Group (a) N06™ filamentary powder, 4,000x (b) N06™ filamentary powder, 10,000x (c) N24™ discrete powder, 4,000x (d) N24™ discrete powder, 10,000x

Powder type	A.D. g/cm³	FSSS* µm	Ni wt%	Fe wt%	C wt%	O wt%	S wt%
Jinchuan N06™	0.50-0.65	2.0-2.8	>99.8	<0.0015	<0.150	<0.1500	<0.0010
Vale T255™	0.50-0.58	2.2-2.6	>99.7	<0.0030	<0.2000	<0.0750	<0.0002
Jinchuan N24™	1.8-3.0	2.3-4.0	>99.8	<0.0015	<0.100	<0.1000	<0.0010
Vale T123™	1.9-2.3	3.5-4.0	>99.8	<0.0010	<0.075	<0.0800	<0.0001
Norilsk UT3™	1.9-2.5	3.0-6.0	>99.8	<0.0015	<0.090	N/A	0.0007

*FSSS – Fisher Sub-Sieve Sizer, refer to ASTM Standard B330

Table 2 Comparison of typical properties of commercial carbonyl nickel powders

Filamentary carbonyl nickel powders (light nickel powders):

- Sintered electrodes for batteries and fuel cells
- Hard metal binders
- Powder Metallurgy
- Sintered filters
- Conducting additives for electronic applications

Discrete carbonyl nickel powders (heavy nickel powders):

- Powder Metallurgy
- Hard metal binders
- Welding rods
- High purity nickel strips
- Industrial diamond synthesis
- Anti-seize lubricant
- Electronic materials
- Chemicals and catalysts

Carbonyl iron powder production

As mentioned earlier, I G Farben pioneered the production of both carbonyl nickel powders and carbonyl iron powders nearly 90 years ago [19]. The iron carbonylation (reaction 2) was not favourable at atmospheric pressure, due to a lower equilibrium reaction constant versus nickel carbonylation (reaction 1). Therefore, carbonyl iron synthesis was done under high pressure, a process that in the 1920s I G Farben had experience of through various high pressure chemical process operations. In its iron carbonyl synthesis, hydrogen reduced iron granules (or sponge iron) are used to react with carbon monoxide under a pressure of ~25.0 MPa and 150-200°C, resulting in an iron extraction of ~65% from a batch operation of about 120 hours. The pale-yellow liquid carbonyl released from the carbonylation reactor has a boiling point of 103°C and is then purified through distillation. Upon evaporation and dilution with CO, iron carbonyl is introduced to the top of a cylindrical decomposer to produce ultrafine carbonyl iron powder at a temperature ranging from 250-300°C, together with adding ammonia (NH_3) as an inhibitor to minimise the CO disproportionation rate (reaction 3). The carbonyl iron powder is removed from the decomposer bottom, with the released CO recycled back for further iron extraction.

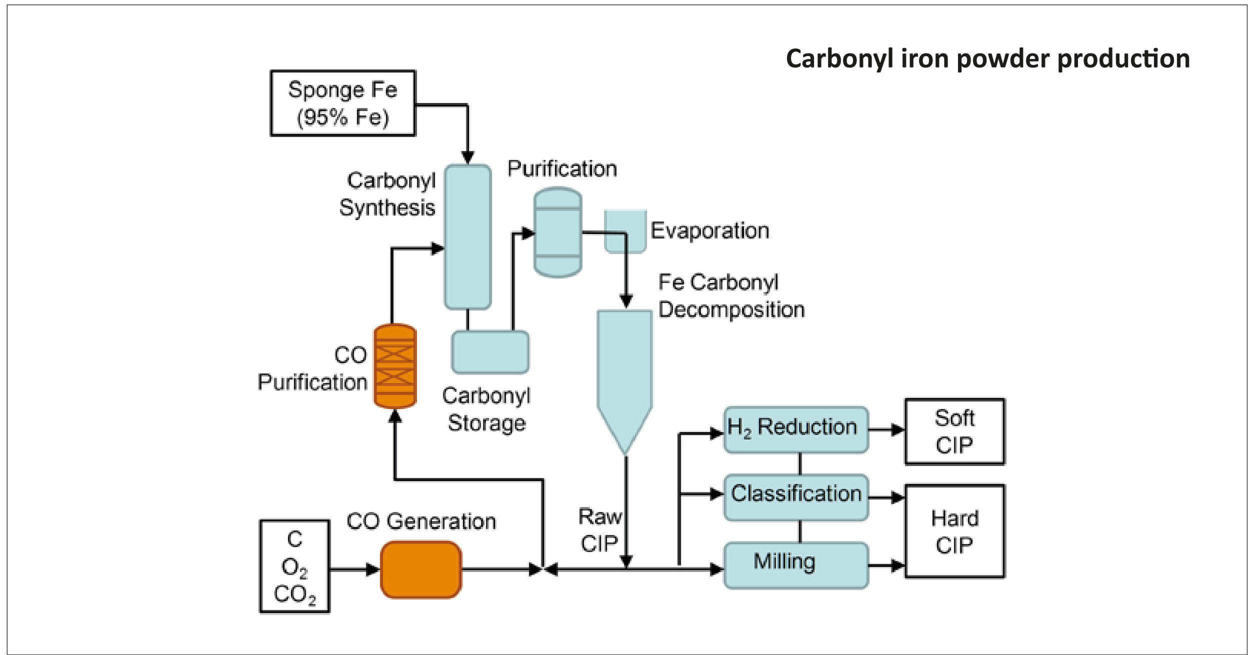


Fig. 6 A typical carbonyl iron powder production process flow-sheet

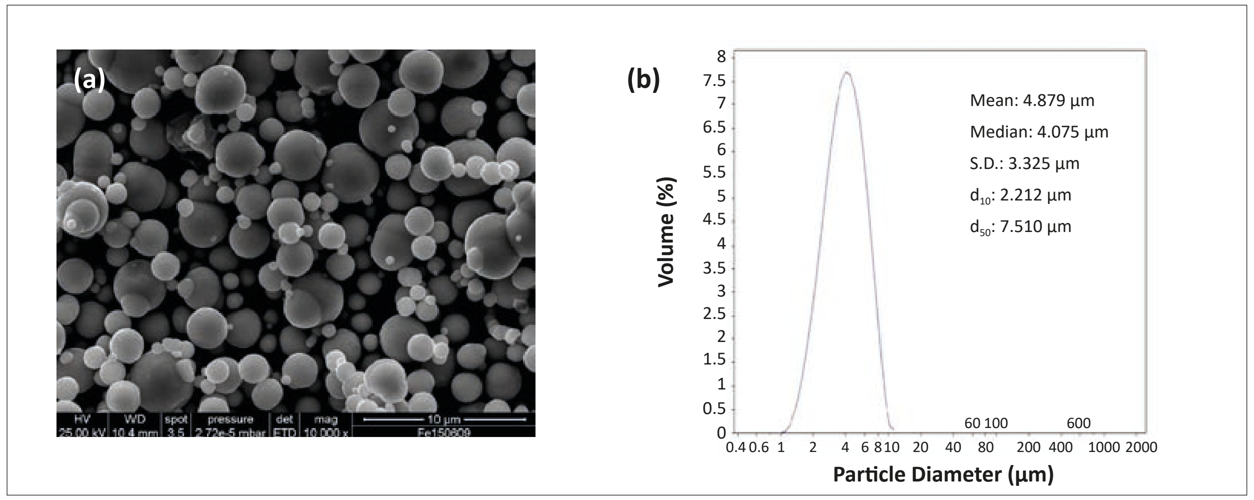


Fig. 7 Typical SEM image and particle size distribution of carbonyl iron powders from Jinchuan Group (a) Hard CIP (b) CIP particle size distribution

As-decomposed carbonyl iron powders are mostly spherical, with a particle size distribution in the range of 1-10 microns. They contain approximately 97% iron, with non-metallic elements of residual carbon (≤1.0 wt.%), nitrogen species (≤1.0 wt.%, mostly in the form of iron nitride [20] and a small amount of chemisorbed ammonia), and oxide (≤ 0.5 wt.%) from the passivation step. The residual carbon is enriched in an onion-skin layered structure due to the endothermic nature of iron carbonyl decomposition and the formation of carbon species on fresh iron particles, being mechanically hard (as in hard powder) due to tension between onion-skin layers.

Jet milling is often practised in order to produce agglomerate-free primary particles, followed by classification in a protective nitrogen atmosphere to cut carbonyl iron powders into fractions with the required particle size range as per customer needs and specific applications. To reduce residual carbon content, as-decomposed carbonyl iron powder may be subject to hydrogen reduction to yield a new carbonyl iron powder category of soft grades. This can be done in H_2 at 400-600°C for 1-4 hours to make soft grades with Fe content >99.5 wt.%, and low contents of C, N, O [21]. The new soft grade carbonyl iron powder is mechanically soft (as in soft powder) with excellent compaction properties. Fig. 6 is a schematic flow chart showing various carbonyl iron powder production steps.

Production in Europe and North America

In the last century, BASF was the dominant manufacturer of carbonyl iron powders (CIP) with an annual capacity of around 12,000 mt. Two other players were International Specialty Products (ISP), since acquired by Ashland Inc., in the USA and Sintez-CIP in Russia [22, 23], each with around 1500 mt CIP capacity per year based on their website claims. Ashland carbonyl iron powder production can be traced back to the iron carbonyl plant established by GAF Corporation in the US with patents and know-how acquired from I G Farben [24]. Despite several ownership transfers in the past, carbonyl iron powder production continues in the US [25].

Growth in Chinese production

China has had limited activities in carbonyl iron powder production since the early 1960s, with the former state-owned 857 Factory and another small chemical company (now known as Shaanxi Xinghua Group) producing carbonyl iron products for military uses. Following the restructuring of the 857 Factory in the new millennium, its knowledge accumulated from the early iron carbonyl synthesis promoted the development of several carbonyl iron powder projects in such newly formed companies as Tianyi Ultrafine Metal Powder Co., Ltd. [26] and Yuean Superfine Metal Co., Ltd. [27].

In 2001, Tianyi Ultrafine Metal Powder Co., Ltd. was founded in the Province of Jiangsu to test a pilot scale carbonyl iron powder plant. This start-up company survived the proverbial "valley of death" in its first two years and gradually ramped up the carbonyl iron powder production to a new level of around 3,000 mt per year in 2009, subject to further expansion. Iron carbonylation in the Tianyi process is done at a pressure below 20.0 MPa and at 140-160°C.

Yuean Superfine Metal Co., Ltd. in the Province of Jiangxi is a joint venture by Yuelong Superfine Metal Co., Ltd. and other investors. The holding company, Yuelong, was founded in 2003 for the new carbonyl iron powder business development. They achieved a successful iron carbonylation process under a pressure of 15.0-18.0 MPa and temperature ranging from 100-200°C in a three day batch operation [28]. In 2005, a new joint venture (Yuean) was set up to further expand carbonyl iron powder production capacity to ~2,000 mt and this now stands at 3,000 mt per year. Their carbonyl iron powder products are marketed under the trademark of Yuelong Powder [29].

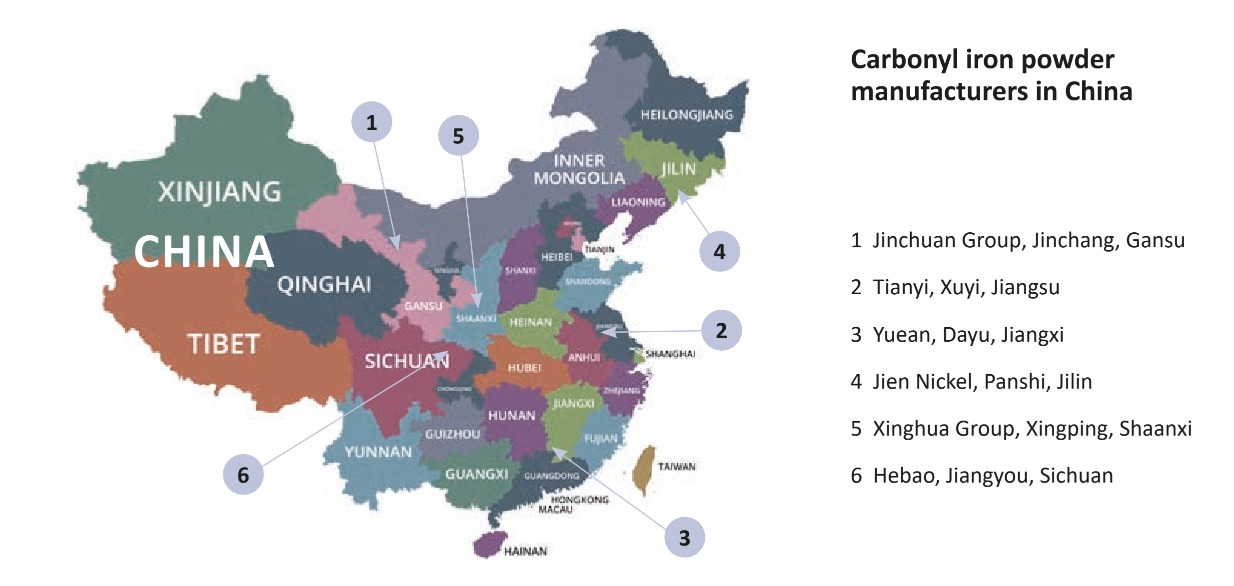


Fig. 8 Locations of Chinese carbonyl iron powder manufacturers

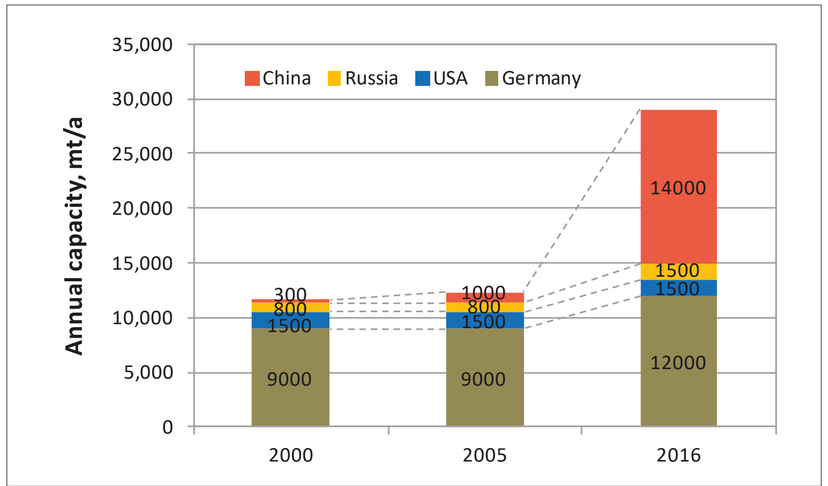


Fig. 9 Worldwide carbonyl iron powder production capacity

While developing nickel carbonyl refining technology, Jinchuan Group also developed its own carbonyl iron synthesis technology. Starting from a sponge iron feedstock, the iron carbonyl synthesis is done in a batch operation (~3 days) at a pressure between 16.0-20.0 MPa and temperature ranging from 200-250°C [30]. Jinchuan's carbonyl iron powder production capacity is about 5,000 mt per year, being the largest in China. A typical SEM image of Jinchuan carbonyl iron powder is shown in Fig. 7, with a particle size distribution curve shown on the right-hand side.

Another Chinese nickel company, Jien Nickel, also commissioned a carbonyl iron powder plant with the help of the Lanzhou Branch of The Chinese Academy of Science. Under an intermediate pressure of 3.0-8.5 MPa and a temperature in the range of 120-250°C, iron extraction reached ~75% after 60 hours batch carbonylation [31].

Small carbonyl iron powder manufacturers also include the Shaanxi Xinghua Group and the spin-off company from the former state-owned 857 Factory (several hundred metric tons each). Fig. 8 shows the geological locations of the above Chinese carbonyl iron powder manufacturers. As of today, the combined total production capacity of carbonyl iron powders from China is ca. 14,000 mt/year as per each manufacturer's claim - nearly half of worldwide total production. Real production figures of Chinese carbonyl iron powder might be lower and fluctuating depending on the market digestion of the newly added CIP capacities. A global landscape of current carbonyl iron powder production is shown in Fig. 9.

A comparison of major iron carbonyl synthesis processes in the world as of 2017 is shown in Table 3. Technologically, there are common features in the production of carbonyl iron powders and carbonyl nickel powders by different suppliers. Business extensions were often seen to produce carbonyl powders along both series of iron and nickel, as practised earlier at Inco and BASF (before 1970).

Characteristics of carbonyl iron powder

In comparison with the original BASF high pressure iron carbonyl process (over 20.0 MPa), a majority of Chinese iron carbonylation processes operate in the lower pressure range (8.0~20.0 MPa). The pressure decrease in iron carbonylation not only lowers the operation cost, but brings in higher iron extraction (75% in Chinese processes versus 65% in the BASF process). It is now understood that, during iron carbonylation at over 200°C and at

Producer, Country	BASF, Germany	Ashland, USA	Sintez, Russia	Jinchuan, China	Tianyi, China	Yuean (Yuelong), China	Jien, China
Pressure (MPa)	~25.0	13-18	20	16-20	13-20	15-18	3.0~8.5
Temperature (°C)	150-200	170-200	180-200	200-250		100-200	120-250
Annual capacity (mt/a)	12,000	1,500	1,500	5,000	3,000	3,000	2,000
Batch hrs/Fe extraction, %	120h/~65%			72h/		72h/	60h/~75%
Commissioning year	1925	1942	1953	2012	2005-2008	2005-2009	2009
Typical products	Hard/soft CIP, coated CIP	Hard/soft CIP	Hard/soft CIP	Hard/soft CIP	Hard/soft/ coated CIP other metal carbonyls	Hard/ soft CIP, liquid iron carbonyl	Hard/soft CIP

Table 3 Comparison of iron carbonyl synthesis at major carbonyl iron powder manufacturers

high pressure, CO disproportionation could be promoted on the fresh iron surface, resulting in carbon deposition on iron, which gradually decelerates the carbonylation reaction rate and inhibits further iron extraction. In comparison with nickel carbonylation, the effect of CO disproportionation under high pressure is particularly pronounced in iron carbonylation, as iron is a more active catalyst towards CO disproportionation. This can be easily understood by the fact that residual carbon content in as-decomposed carbonyl iron powders (<1.0%) is several times higher than that in as-decomposed carbonyl nickel powders (<0.2%).

Carbonyl iron powder manufacturers can produce various grades of spherical iron powders in the size range of 1-10 microns from atmospheric decomposition operations. Table 4 lists typical carbonyl iron powder products from a selection of major manufacturers. The fundamental categories are actually hard grades (as-decomposed) and soft grades (H_2 reduced). Further deviations, in such product specifications as particle size distribution and bulk density, are achievable through post processing by milling and classification to meet specific application requirements. Carbonyl iron powders have found wide applications in Metal Injection Moulding (MIM, ca. 30-35%, being the largest among others), magnetic cores for high-frequency coils, hard metal binders, radar absorption materials (RAM), magneto-rheological fluids (MRF) for shock/vibration damping, clutch and brake systems, precision polishing (for example, to produce the smooth surface finish found on the Jet Black iPhone 7), industrial diamond synthesis and food iron supplements. Excellent literature references with regard to specific applications can be found on BASF's corporate website [32].

It should be mentioned that, similar to carbonyl nickel powder products, the production of light weight filamentary iron powder and nano iron powders is also possible under special decomposition conditions. As an example, Ashland used to offer a filamentary carbonyl iron powder with characteristics of <0.2 μm diameter and average 4 μm in length.

Company	Grade	D_{50} μm	A.D. g/cm³	T.D. g/cm³	Fe_{min} %	C_{max} %	O_{max} %	N_{max} %
BASF	CIP OM	3.9-5.2			97.8	0.75-0.9	0.15-0.40	0.65-0.90
Jinchuan	F01	1-6	1.0-3.5		97.5	0.8	0.5	1.0
Tianyi	YMIM90	<5.0	<3.0	≥4.0	Bal.	0.76-0.90	0.6	0.9
Yuean	01S	3.5		4.0-4.1	97.0	0.6-0.9	0.35-0.65	
BASF	CIP CC	3.8-5.3			99.5	0.05	0.18-0.35	0.01
Jinchuan	F02	<5.5	2.5-3.2	3.5-4.5	99.0	0.1	0.3	0.1
Tianyi	RMIM20	<6.0	<3.0	≥4.0	Bal.	0.06-0.15	0.2	0.05
Yuean	HY1	5.5			99.5	0.03	0.3	

Table 4 Comparison of typical commercial carbonyl iron powders for MIM

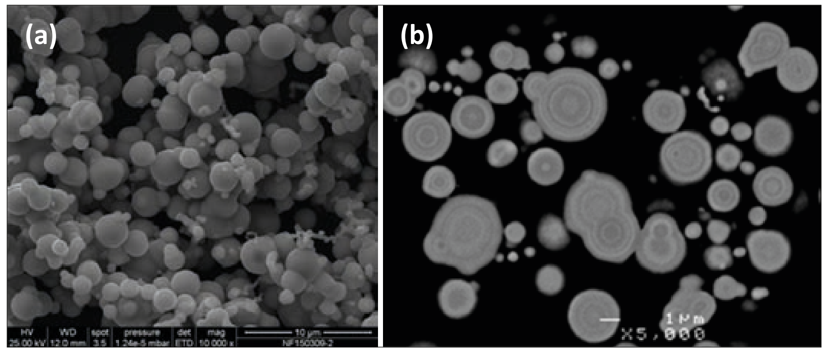


Fig. 10 Typical SEM images of (a) carbonyl ferronickel powder and (b) cross-sectional view of internal onion-skin structure

Carbonyl ferronickel powders

The Chinese Jinchuan Group produces carbonyl ferronickel powders with mixtures starting from liquid iron carbonyl and the bottom fraction of nickel carbonyl distillation columns. The mixture fraction of iron carbonyl and nickel carbonyl is easily adjusted to the required Fe/Ni ratio for production of ferronickel powders. Currently, five ferronickel powder grades are commercially available from Jinchuan Group, being NF19^{TM}, NF28^{TM}, NF37^{TM}, NF46^{TM} and NF55^{TM}, containing 10 wt.%, 20 wt.%, 30 wt.%, 40 wt.% and 50 wt.% nickel, respectively.

Typical SEM images of carbonyl ferronickel powder NF37^{TM} are shown in Fig. 10. They are, in general, micron-size spherical particles with an internal onion-skin structure (Fig. 10b). Similar to carbonyl iron powders, the formation of an onion-skin microstructure inside carbonyl ferronickel particles results from the endothermic decomposition of iron carbonyl and the CO disproportionation on the more catalytically active iron surface. The alternate layers appear to be very fine inhomogeneous mixture of elemental iron and elemental nickel, as evidenced by X-ray diffraction. Carbonyl iron is in a body centred cubic (bcc) crystalline structure, whereas carbonyl nickel is in a face centred cubic (fcc) crystalline structure. The interface between the alternate iron and nickel layers might exhibit interesting ferromagnetic behaviour, which is yet to be investigated. Applications of carbonyl ferronickel powders include Power Metallurgy, magneto-rheological fluids, radar absorbing materials, EMI/RFI shielding, industrial diamond synthesis, etc.

Conclusion

In summary, the availability and varieties of carbonyl metal powder products are expanding, especially following the recent technological progress in the Chinese carbonyl metal refining industry. With continuous carbonyl refining technology development, more carbonyl nickel and ferronickel powders will no doubt become available in the near future.

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Graphite and its applications in the Powder Metallurgy industry

With its unique structure and properties, graphite is a versatile raw material used in a wide range of industries. Graphite offers excellent electrical and thermal conductivity as well as outstanding lubrication properties and high resistance to temperature and oxidation. In the Powder Metallurgy industry, graphite is an essential additive in metal powder mixes for the production of sintered parts. In this article, Dr Robert Feher, R&D Director at Graphit Kropfmühl GmbH, Germany, reviews the use of graphite in the Powder Metallurgy process as well as its various applications.

Large quantities of carbon can be found in the Sun and in the atmospheres of many known planets. Although the Earth is less than 0.1% carbon, it forms the basis of organic chemistry and of life itself. This is because of carbon's ability to form compounds with other elements such as oxygen, hydrogen and nitrogen. Crystalline carbon is only known in four allotropic forms; graphite, diamond, pure carbon and fullerene.

Graphite is chemically inert, eco-friendly and safe. It is classified as microcrystalline and macro-crystalline natural graphite and synthetic graphite. Six hexagonally arranged carbon atoms form the basic unit of the graphite crystal. This crystal structure results in a number of special properties that make graphite a raw material with a wide variety of applications. Graphite offers excellent electrical and thermal conductivity, outstanding lubrication properties, high resistance to temperature and oxidation and the ability to form intercalation compounds.

Graphite is used in various aspects of Powder Metallurgy. It can be employed as an alloying element to increase the strength of sintered parts. It can also be used as a second phase material in metal bonded particulate composites. Composite materials are heterogeneous mixtures of two or more homogeneous phases which have been bonded together. In composites,

Fig. 1 Graphite ore is used to make graphite powder for Powder Metallurgy applications

Fig. 2 Graphite brush materials are composed of graphite bonded with resin or pitch to form a soft brush material

properties can be attained which could not be obtained if the constituents were used separately. Composites can either be classified according to morphology of reinforcements (fibre, particulate and laminate composites) or to matrix materials (metal, ceramic and polymer matrix). Another important feature of graphite is its application for lubrication and friction moderation in bearings. For each of these applications, a different tailor-made graphite is necessary to comply with the demands of the specific process.

Composites

Carbon brush materials

Particulate composites consist of nearly spherical particles embedded into a homogeneous matrix. Mostly, they are used to increase the hardness of the composite material. A typical example is electrically conducting composites, which are used for electrical contacts such as brushes on motors (Fig. 2).

Electrical brushes and brush materials are used in conjunction with slip rings, commutators or other contact surfaces to maintain an electrical connection in rotary and linear sliding contact applications. Graphite brush materials are composed of graphite bonded with resin or pitch to form a soft brush material. The ash inside natural graphite leads to an abrasive action. The fast filming of these brushes is beneficial in protecting the commutator ring during operation in contaminated atmospheres.

Electrographitic brushes are free from ash and are sintered at temperatures in excess of 2400°C. The porous material is treated with organic resins that have a lubricating effect and thus increase brush life. For low voltage and high brush current applications, the resistivity of the brush material is decisive. In this case, 50-100 µm particles of graphite (5-70%), as a soft phase, are dispersed in copper, bronze or silver.

The conductivity of the PM product is directly affected by porosity. The greater the void content, the lower the conductivity and the lower the tensile strength. Thus, a high final sintered density is desirable. Densification can be increased by additional operations such as double pressing/double sintering or forging. The dense combination of copper and graphite benefits from the low friction capability of graphite and the high conductivity of copper to ensure reliable contact.

Fig. 3 Brake friction materials

Friction materials

Another prominent example of composites is brake friction materials (Fig. 3). Brake pads have to maintain sufficiently high friction coefficient with the brake disc and must not decompose or break down at high temperatures so that the friction coefficient is compromised. The brake pad typically consists of the following materials: frictional additives, fillers, binders and reinforcing fibres [3]. Depending on the manufacturer, the composition of brake pads can vary over very wide ranges of the sub-components.

Graphite is added as a lubricant which rapidly forms a layer on the opposing counter friction material and thus decreases the friction coefficient and wear rate. It can be used in flake or powder form. Generally, graphite in flake form has improved lubrication properties while, in powder form, it can dissipate the heat generated during braking more efficiently.

The amount of graphite used is limited due to the weak bonding between graphite and phenolic resin, which is the cheapest and most common binder used in brake friction materials. This weakness affects the shear strength negatively and can be avoided by using newer types of resins, such as condensed polynuclear aromatic resins, which show a high structural affinity with graphite. Using these resins, higher shear strength can be achieved, diminishing the wear of the friction material.

Since graphite is a very good heat conductor, the overall heat dissipation of the friction material is very high and this guards against overheating and decomposition of the material. However, measures have to be taken to further conduct the heat away from the system in order to prevent overheating of the braking fluid.

Fig. 4 Graphite is added as a separate powder which is admixed with the metal powders used for PM structural parts

Self lubricating bearings

For friction moderation in bearings, other rules apply. In this case, larger graphite grain sizes of d_{50} > 20 µm are used and a high resulting pore volume is desired after the sintering process. In iron carbon graphite materials, only up to 0.5% carbon is dissolved in the iron phase, whereas 2.0–3.5% of compact graphite with high tap density is dispersed. The open porosity of the system is about 20%, resulting in a network of interconnected pores which is saturated with a suitable oil as lubricant. The excess graphite in the material has the function of improving the noise behaviour as well as providing an emergency functioning property for the bearing.

"The excess graphite in the material has the function of improving the noise behaviour as well as providing an emergency functioning property for the bearing"

Alloying elements in steel and iron powders

Metal compacts

Several different techniques are used to add the alloying elements in steel and iron powders. In premixes, the most important alloying elements are Cu, Ni and P (as Fe_3P), mixed in as separate powders. Carbon is added in the form of graphite. Diffusion alloyed systems consist of Mo, Cu and Ni particles that have been diffusion alloyed on the surface of iron particles. Finally, pre-alloyed materials consist of Mo, Ni, Mn and Cr alloyed with iron. When atomised to metal powders, the alloying elements are homogeneously distributed in the particle. For both diffusion alloyed and pre-alloyed powders,

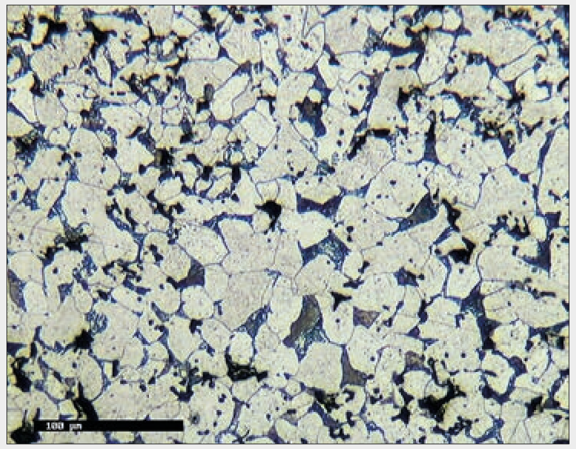

Fig. 5a Fe + 0.2% GK graphite after sintering. Density 7.0 g/cm^3, hardness (HV10) 80 and tensile strength 220 MPa [7]

Fig. 5b Fe + 0.8% GK graphite after sintering. Density 7.0 g/cm^3, hardness (HV10) 130 and tensile strength 380 MPa [7]

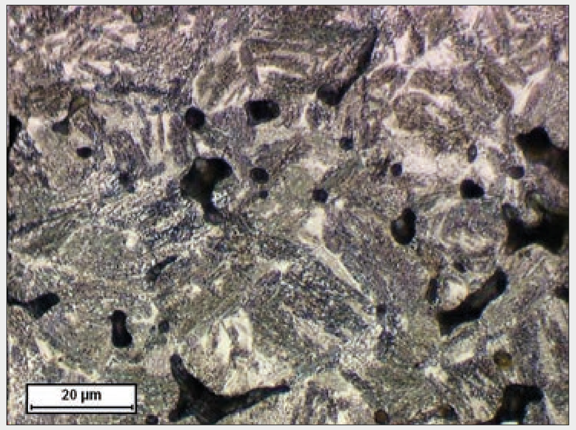

Fig. 6a Fe + 3% Cr + 0.5% Mo + 0.4% C (GK graphite) after sintering. Cooling rate 0.5°C/s, density 7.0 g/cm^3, hardness (HV10) 240 and tensile strength 800 MPa. Upper bainite and approx. 1% martensite [7]

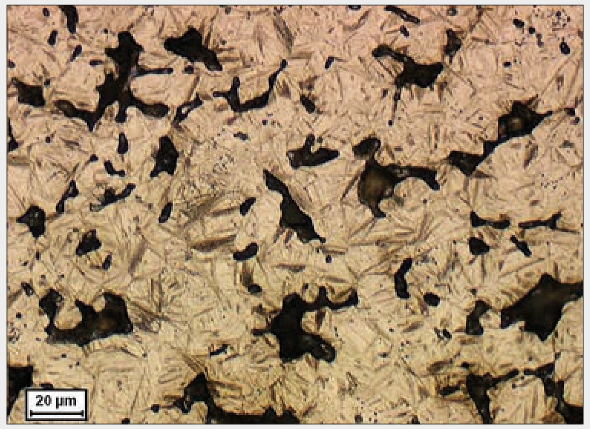

Fig. 6b Fe + 3% Cr + 0.5% Mo + 0.4% C (GK graphite) after sintering. Cooling rate 2.5°C/s, density 7.0 g/cm^3, hardness (HV10) 330 and tensile strength 1030 MPa. Martensite >95% with lower bainite as balance [7]

the graphite is added as a separate powder that is admixed with the metal powders.

For many metal compacts, such as copper-tin or iron-copper, growth or shrinkage can be modified by the addition of carbon in the form of graphite. Thus, graphite can be used to control dimensional changes in these systems [4]. For copper/bronze bearings, small to medium-sized graphite particles with a d_{50}-value of 8-30 µm are mainly used. For the production of low alloyed steels, high purity graphite is commonly used, mostly in a d_{50}-range from 5-11 µm. The amount of graphite added depends on the requested material properties.

Sintered steel

As an example, the effect of carbon on the properties of sintered steel is illustrated in Figs. 5a and 5b, showing the microstructure for two different addition levels of carbon in iron. The amount of pearlite increases with increased carbon content and, consequently, the hardness and tensile strength of the material increases.

The cooling rate after sintering will affect the distribution of the carbon in the metal matrix and generate different mechanical properties, even though the carbon content is held constant. With a cooling rate of 0.5°C/s, upper bainite is mainly obtained in an iron based alloy with 3% Cr, 0.5% Mo and 0.4% C, as illustrated in Fig. 6a. By increasing the cooling rate to 2.5°C/s, a microstructure consisting mainly of martensite is obtained (Fig. 6b).

"The main issue in PM in general is that a constant graphite quality is required to secure absolute process stability"

The effect of high amounts of carbon on the properties of sintered steel is examined in [5]. The mechanical properties of PM components are adversely affected by porosity and these properties are dependent on the size and shape of the pores in the sintered steel. Therefore, as high a density as possible is favourable. This can be achieved either by high compaction pressures or liquid phase sintering. The amount of liquid phase formation, on the other hand, can be increased by the introduction of elements such as phosphorus and boron, which lower the eutectic temperature. This leads to an increased density and hardness. As a negative effect, these elements tend to segregate to grain boundaries and therefore cause brittleness. By also adding carbon to this system, the liquid phase sintering is further facilitated.

A possible solution to the formation of iron carbides and iron phosphides at higher carbon contents is the alloying of iron with high levels of carbon and phosphorus to achieve a high volume shrinkage. With a post-sintering decarburisation, the carbon can diffuse into the liquid and be partly depleted from the material, allowing the phosphorus to dissolve in the iron matrix.

Experiments varying the graphite, phosphorus and copper content show that only higher amounts of carbon (greater than 1.1-1.3%) together with the appropriate P-level lead to higher relative densities. This is because a certain amount of graphite is dissolved in the iron powder before melting takes place. Only with an excess of graphite are grains left, which take place in the eutectic reaction and lead to a high amount of persistent liquid phase. Thus, in the system Fe-P-C-Cu, the best result with regard to density is obtained by single stage die compaction at 500-800 MPa with sintering at 1220–1250°C. The sample is then is decarburised at 1100°C with a gas mixture of 10% H_2 and 90% N_2 and, later, additional moisture content at 1200°C. The ultimate tensile strength of the solid material depends on the sintered density, the pearlite content given by the amount of carbon and the presence of brittle phase. For blends with higher carbon contents, tensile strength rises with decarburisation for iron matrices, which have brittle phases that are effectively reduced by this step.

Fig. 7 The purification of graphite

Post-sintering decarburisation also has an influence on the elongation to fracture, where most alloys having a high original carbon content lose their brittleness after decarburisation. Thus, by fine tuning the relative amounts of phosphorus and surplus carbon before sintering, in combination with post-sintering decarburisation, a high density non-brittle steel part with high tensile strength can be designed.

Special applications

Special high performance metals are needed for some electrical applications, such as switching devices for high voltages and currents. In this area, Ag-C alloys have the highest resistance against fusing, caused by the start-up peak, and the lowest contact resistance. It has been found that resistance against fusing increases with higher carbon amounts [6].

However, the tendency to form migrating electric arcs as well as brittleness deteriorated with higher carbon percentages. In practice, amounts of 3-5% are used.

Properties of graphite in Powder Metallurgy applications

There are various essential requirements for the use of graphite in the Powder Metallurgy industry. The main issue in PM in general is that a constant graphite quality is required to secure absolute process stability for the PM producer and obtain the ultimate consistency in the quality of the sintered part. This can only result from a stable raw material source and controlled refining steps such as purification (Fig. 7), micronising and good quality control.

When looking specifically at different applications within the PM industry, parameters of graphite such as particle size, purity, particle

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shape and surface show that here very specific solutions or tailor-made graphite grades are generally needed.

Summary

Generally, for metal-graphite alloys, depending on the sintering process, graphite with different grain sizes should be employed. The particle size of the graphite should have an optimal relationship to the metallic particle size. If the graphite is too fine, the carbon loss for reaction with other elements before the start of the sintering process might be too high.

For composite materials such as brushes, medium to large-sized graphite particles are used, which form separate soft phases that have a lubrication effect. In brake friction materials, the shape of the graphite particles used (for example, flake, potato or needle-like shape) determines the lubricating or heat conducting properties.

In special alloys such as high voltage switching devices, small-sized graphite is used to minimise contact resistance in the alloy. For low alloyed steels, a high purity graphite source with fast reactivity is necessary to achieve short production times and as high a density as possible, together with the high tensile strength of the finished product.

Graphite can also improve the performance of the PM process itself, as shown in the Fe-P-C-Cu system where it serves to facilitate the processing conditions leading to an optimised density.

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Innovation recognised in Japan's PM award winning parts, materials and processes

The winners of the Japan Powder Metallurgy Association's (JPMA) 2016 Powder Metallurgy Awards showcase the continuing developments being made to further expand the range of applications for Powder Metallurgy. The winners showcase innovations not only in component design, but also in new materials and advanced manufacturing processes.

Development prizes: New design

Sintered bearing with dimple on inner diameter for high efficiency motors

Motors, whether large or small, need to be improved in their running efficiency. In order to achieve this, the friction caused by sintered bearings needs to be reduced. Porite Corporation received an award for a sintered oil impregnated bearing with a dimple that reduces the friction coefficient in order to replace the ball bearing on the inner diameter.

Generally, ball bearings were selected in the early stages of development due to their low friction characteristics, especially for high-efficiency motors. On the other hand, in some cases, sintered bearings were not adopted because of the differences in friction between sintered bearings and ball bearings. This remained the case even though improvements were made in the bearing material, impregnation oil and optimisation of clearance between shaft and bearing, and so on. Porite, therefore, developed the concept of using Dimple technology on the inner diameter, which had previously been used to reduce the friction of engine cylinders and hydraulic equipment. The variation of size and depth of the dimple and the

Fig. 1 These sintered bearings from Porite Corporation reduce the friction coefficient through the incorporation of a dimple on the inner diameter (Courtesy JPMA)

Fig. 2 A multilayered sintered bearing from NTN Advanced Materials Corporation. The inner layer is composed of Fe-Ni-Mo-C alloy steel, improving wear resistance (Courtesy JPMA)

Fig. 3 A Hybrid magnetic material reactor core by NTN Advanced Materials Corporation made from amorphous magnetic material (Courtesy JPMA)

unevenness of the dimple pattern creates differences in the effectiveness of reducing friction.

In order to stabilise the dimple process, the inner diameter of the unprocessed part was optimised and the process was designed to both form dimples and finish the inner diameter at the same time. This avoided the risk of the dimpling process making the inner diameter uneven. In addition, for the improvement of the dimple process tool's life, a suitable material and a suitable process for surface finishing of the tool were chosen. Also, an automatic lubricator on the auto dimple forming machine helped to improve the tool's longevity.

As a result of these improvements, Porite has been able to form dimples on the inner diameter of sintered bearings, with diameters below 5 mm, that are used for miniature motors. The friction coefficient of the sintered bearings is then more closely matched to that of ball bearings because of the reduction of the contact area and the retention of oil in the dimple.

Development of a multilayer sintered bearing

NTN Advanced Materials Corporation received an award for a multilayer bearing to be used in joint parts of construction machinery, for example a hydraulic shovel. The operating conditions require low friction and wear resistance characteristics under harsh environments characterised by low speed, heavy load, vibration and shock load.

Oil-impregnated sintered bearings, conventionally mass produced, are made from materials that achieve sliding properties and high hardness. The production adopts "heat treatment to maintain both strength and hardness" and "finish machining to maintain dimensional precision". Therefore, cost is increased in terms of both material and production. Also, in terms of performance, there is a risk of cracking caused by a reduction in impact strength by the heat treatment. The developed multilayer structure was able to solve these problems.

The inner layer (bearing side) is composed of Fe-Ni-Mo-C alloy steel to improve wear resistance. Also, Cu is added to improve sliding properties and remove the need to machine the inner dimension, as size/shape correction can be achieved by sizing. Low melting point metal is added to Fe-Cu-C material in order to provide high strength and high toughness on the outer layer.

NTN has succeeded in developing a bearing that can reduce cost, that has excellent sliding properties and wear resistance and is strengthened against shock by moulding these different materials into a multilayer bearing using separate tools.

Fig. 4 Matrix toughened material is used in these valve seats for improved anti-adhesive wear properties. Fine Sinter Co. Ltd and Toyota Motor Corporation developed this material by distributing fine, hard particles in the matrix (Courtesy JPMA)

Development of a hybrid magnetic material reactor core for a booster application

NTN Advanced Materials Corporation received a second award for a Hybrid core made from amorphous magnetic material. There is a trend for choke coils for power supply circuits to require increased current and power beyond several kW, along with the improvement of medical equipment such as MRI scanners. Conventional magnetic materials needed a large cubic volume to meet these current requirements because they readily cause magnetic saturation. High drive frequencies in the circuits used for these applications is growing in response to the need for high-speed operation of the equipment. However, conventional materials show degradation of properties, such as inductance and iron loss, at high frequencies.

NTN has developed a Hybrid core, which adopts amorphous compression material on the inner and injection formed magnetic material, filled with amorphous material, on the outer in order to achieve both high current/frequency use and miniaturisation. An amorphous injection core alone shows too low an absolute inductance value, whilst; on the other hand, an amorphous dust core alone does not have a sufficient DC bias characteristic. The hybrid structure of both of the parts can meet the required characteristic, i.e. that magnetic saturation is not caused even in a high magnetic field.

This has the effect of reducing the inductance decreasing rate to half, compared with ferrite, in high current use (300 A) and losses are reduced to 1/10, compared with Fe-Si, at high frequencies (100 kHz). In addition to this, the apparatus's maximum current is increased to 260 A (or 2.6 times 100 A) and circuit operating frequencies are increased to 50 kHz (or 5 times 10 kHz).

Development prize: New materials

Matrix toughened valve seat material with improved anti-adhesive wear properties

Fine Sinter Co. Ltd and Toyota Motor Corporation received a prize for the development of a valve seat material with improved anti-adhesive wear properties by distributing fine hard particles in the matrix. Valve seats for automobile engines are required to have high anti-wear properties and hard particles are added and distributed in the matrix to obtain a "cobblestone effect" to achieve this requirement. Also, the combustion heat from engine operation produces oxides, whose major components are Fe, on the surface of the valve seat. These oxides improve the anti-adhesive wear with the valve and this creates a synergetic effect with the conventional hard particles. However, recent improvements in engine high-efficiency technology

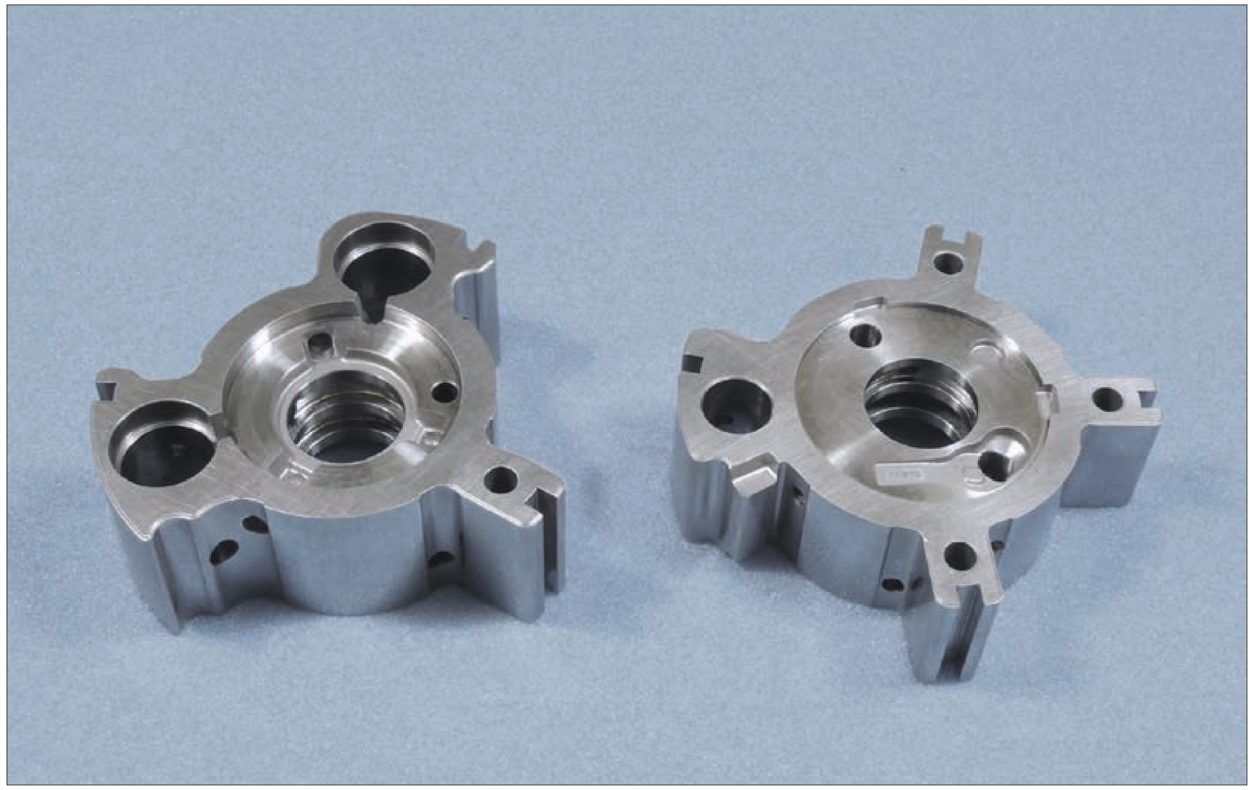

Fig. 5 Sumitomo Electric Industries Ltd's VVT parts manufacturing-line allows the simultaneous green machining of holes and grooves (Courtesy JPMA)

have created difficult conditions for Fe oxide formation. Accordingly, strengthening the valve seat against the adhesive wear by other means is required. To acquire high adhesive wear properties, the usual option is to increase hard particle content. However, this option is severely limited, as a large quantity of hard particles could cause degradations in compactability, machinability and machined surface roughness.

This development studied the method for distributing the fine hard particles in the matrix. Furthermore, hard particles have been developed with an increased austenite diffusion phase compared to conventional materials. The hard particle type developed improves adhesive properties with the matrix, solves the concerns over surface roughness after machining by detachment of hard particles and provides a 35% cost reduction compared with the conventional material.

Development prize: New materials

A VVT parts manufacturing line, which provides the simultaneous green machining of holes and grooves and the attachment of a two-dimensional barcode

Sumitomo Electric Industries Ltd. received a process development prize for a manufacturing line for Variable Valve Timing (VVT) parts, which provides the simultaneous green machining of holes and grooves and the attachment of a two-dimensional barcode to ensure high quality. Also, the line provides successful touch-less manufacturing of parts, from compacting to sizing.

VVT systems control the timing of valve opening and closing by changing the phase of the rotor with oil pressure, enhancing power output and reducing fuel consumption. VVT parts are rapidly developing, with, for example, the rotor becoming unified with oil control valve, and the demand for holes and grooves, which cannot be moulded in compaction, has increased. Therefore, the increase in machining cost, including deburring after sintering, has become a problem area.

To solve this problem, Sumitomo Electric has applied green machining, enabling rapid machining without burr formation, and has sought to decrease cost. However, previous green machining processes have had concerns regarding cracks and chips. Also, the machining was not linked with compaction and sintering and therefore created problems in terms of productivity. Therefore, the company has now developed the multi-machining technology of holes and grooves in one operation and succeeded in building a consistent higher productivity line. This line can prevent the risk of cracks and chips, due to offline handling, by in-lining the green machining equipment, linking it with the compaction process, and machining with a touch-less and stockless concept. In addition, traceability has been enhanced by attaching a two-dimensional barcode by laser beam to each product before sintering, enabling the tracing of product quality and manufacturing information, and the risk of cracks and chips in green machining has been decreased.

Fig. 6 A laser quenched multistage complex side plate, adapted to the high torque control system of a VVT rotor (Courtesy JPMA)

Effort prize

Development of a multistage complicated shape side plate, laser quenched on the nonconsecutive surface

Sumitomo Electric Industries Ltd. was awarded an effort prize for the development of a laser quenched multistage complicated shape side plate adapted to the high torque phase control system of a VVT rotor.

High torque was necessary for the controlling phase of the rotor and, in the conventional model, several springs were attached to the rotor itself to control phase. However, in the developed model, a system which controls phase from outside the unit was adopted.

Therefore, a large spring, which slides with the side plate, was attached, and the replacement of the aluminium casting with a sintered iron part was demanded. Regarding the product shape, the side plate was difficult to compact, due to the many tangs required to fix the spring to the surface and the deep oil groove on the opposite surface. In order to maximise the benefits of using PM, multistage compaction was successfully designed in at an early stage. Furthermore, the side surface of each tang demanded wear resistance, due to the sliding of the spring. Though the surface was uneven, laser quenching was applied, which achieved the required hardness without decreasing accuracy by using a simple jig and program, instead of induction hardening with a complicated coil design.

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Hot Isostatic Pressing at World PM2016: Developments in production and processing

Papers in the three technical sessions dedicated to Hot Isostatic Pressing technology at the World PM2016 Congress, held in Hamburg, Germany, 9-13 October 2016, addressed developments in production techniques and processing issues. In this article, Dr David Whittaker reviews three papers from these sessions that describe a novel capsule-free HIP method, the near net shape HIP fabrication of an impeller and the phase transformation under isostatic pressure in HIP.

WORLDPM2016 congress and exhibition

Reaching full density of 100Cr6 PM steel by capsule free HIP of high-velocity compacted material

A paper from Hans Magnusson and Karin Frisk (Swerea KIMAB AB, Sweden), Maheswaran Vattur Sundaram and Eduard Hryha (Chalmers University of Technology, Sweden), Christer Åslund (Metalvalue, Sweden), Björn-Olof Bengtsson and Magnus Ahlfors (Quintus Technologies, Sweden) and Sören Wiberg (Linde Gas, Sweden) described a novel capsule-free HIP production method.

Producing fully dense PM components through HIP normally requires a capsule or can, in order to give the product its shape. After densification, the capsule has to be removed by machining or acid pickling. The cost of capsule production and handling per component can be so high as to make the production of small components uneconomic and there is, therefore, a strong motivation to develop capsule-free HIP routes.

The production route used in the reported work was the MMS-Scanpac® process, developed by Christer Aslund of Metalvalue, and comprised:

- Agglomeration of the initially spherical gas atomised powder, to enhance achievable green strength
- Conventional die compaction
- Low temperature sintering (at 1120°C)
- Re-striking using the high velocity adiabatic compaction process
- Capsule-free HIP for three hours at 1150°C, using a pressure of 1000 bar

Fig. 1 More than 1900 participants attended the World PM2016 (©World PM2016 Andrew McLeish)

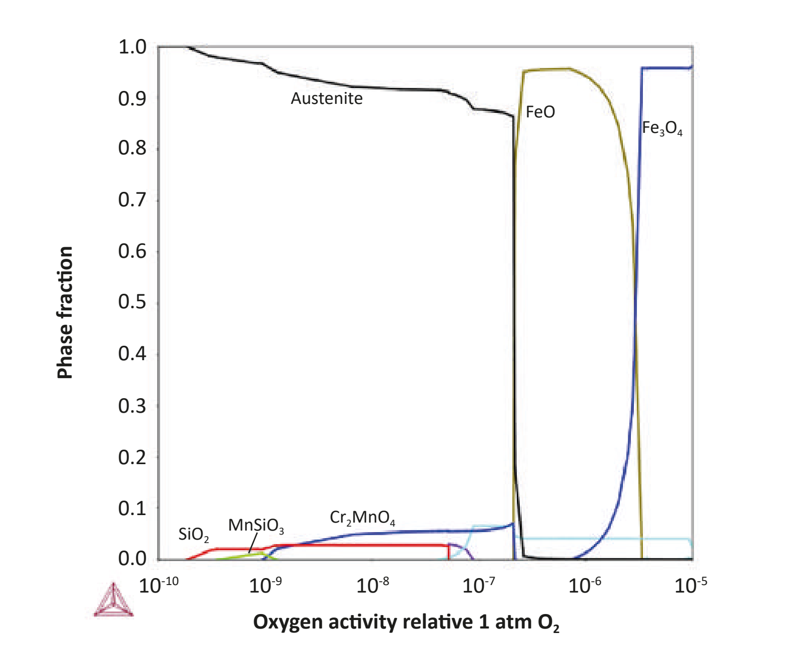
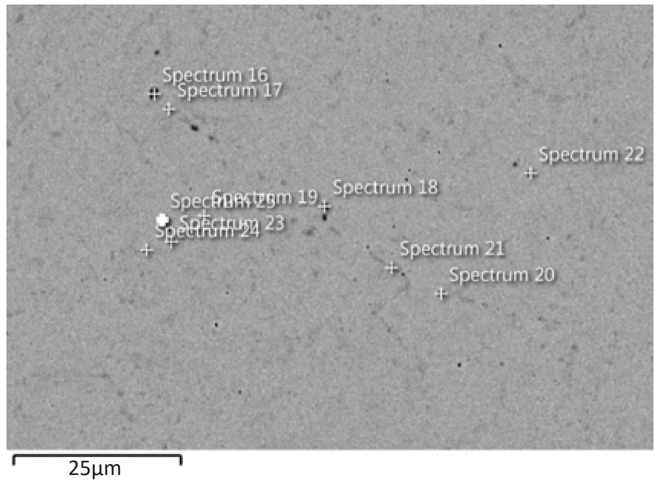


Fig. 2 Stable oxides as a function of oxygen activity at 1100°C [1]



Spectrum	O	Si	Cr	Fe
Spectrum 17	7.6	5.5	1.7	85.2
Spectrum 21	5.0	5.1	2.9	87.1
Spectrum 23	8.3	5.9	1.4	84.5
Spectrum 24	7.8	5.3	1.2	85.7

Fig. 3 Micrograph and EDS analysis of the HIPped material. Concentrations are given in weight percentages [1]

Grade	C	Si	Mn	Cr	Co	Fe
100Cr6	0.98	0.5	0.4	1.5	0.2	bal.

Table 1 Composition of the 100Cr6 steel powder [1]

This route was applied to the processing of 100Cr6 steel powder, whose composition is shown in Table 1. After re-striking, the relative density of the material was around 97% (see Table 2) i.e. well above the level at which any porosity in the structure becomes closed rather than open to the surface, thus obviating the need for encapsulation.

Table 2 shows the typical density levels achieved at various stages in the process route: after low temperature sintering, after re-striking, after HIPping and after HIPping and hardening. The HIPped relative density was in excess of 99%.

The most critical issues in the process route were defined as being the control of oxygen and carbon potentials in the sintering atmosphere, as 100Cr6 contains a number of strong oxide-forming elements (Si, Mn, Cr) and the carbon level in the final HIPped product has a strong influence on the achievable hardness and strength.

Using thermodynamic calculations, made with Thermo-Calc version 4.1, the stable oxides have been evaluated as a function of oxygen activity for 100Cr6 at 1100°C, as shown in Fig. 2. The oxygen activity is the square root of the partial pressure of oxygen in the sintering atmosphere. The oxide that is stable at the lowest activity is the most stable oxide i.e. quartz SiO_2. Other stable oxides are rhodonite $MnSiO_3$ and the spinel type of oxide Cr_2MnO_4. An oxygen activity of 10^{-9} at 1100°C corresponds to a dry hydrogen gas mixture with -55°C dew-point.

Metallographic examination of the final (as-HIPped) microstructure showed a fully dense material with few oxides. Any oxides present were typically smaller than 1 µm and EDS analysis (Fig. 3) showed a clear increase in silicon content, indicating that these oxides were probably of the quartz SiO_2 type.

Water vapour in the sintering atmosphere will accelerate decarburisation, but this can be compensated by the addition of methane. Methane is a low temperature stable gas species, which will decompose at high temperature increasing the carbon potential of the sintering gas at temperatures close to sintering. The increase in carbon potential, or carbon activity, with temperature for N_2/H_2, a gas mixture including methane, is presented in Fig. 4. The calculation was made using Thermo-Calc. The steel powder 100Cr6 contains 1% carbon. The solubility of carbon in the steel will increase with temperature and, as a consequence, its carbon activity will fall with temperature, as shown in the figure. By optimising the methane content, it is possible to have identical carbon activities in the material and the gas at sintering temperature. In a theoretical situation, without oxygen in the furnace, approximately 0.14% methane is needed for carbon control. A furnace with oxygen, or moisture, will require additional methane. This illustrates how different oxygen contents in the gas mixture can be compensated by additional methane. 0.5% O corresponds to -3°C dew-point. The minor difference at around 800°C is due to the additional carburising effect of CO, which is relatively weak compared to that of methane.

The relation between dew-point *T* in K and moisture content is given by the following equation:

$$H^{H_2O}_{gas} = 3.76 \cdot 10^6 \exp\left(-\frac{46000}{RT}\right)$$

where *R* is the gas constant. This expression has been evaluated based on thermodynamic data for water and water vapour. In the reported work, the moisture content was continuously monitored using a dew-point meter and, based on this information, methane was added. Utilising such a carbon control methodology, sintered specimens with similar carbon content to the original powder were obtained.

Process state	Measured density, g/cm³	Theoretical density, g/cm³	Fraction of theoretical density
LT-sintered	~6.3	7.84	~80%
Re-strike	7.57-7.62	7.84	96.5-97.2%
HIP (ferritic + carbides)	7.79	7.84	99.4%
HIP + hardening (martensitic)	7.67	7.71	99.5%

Table 2 Measured and theoretical densities for different process states [1]

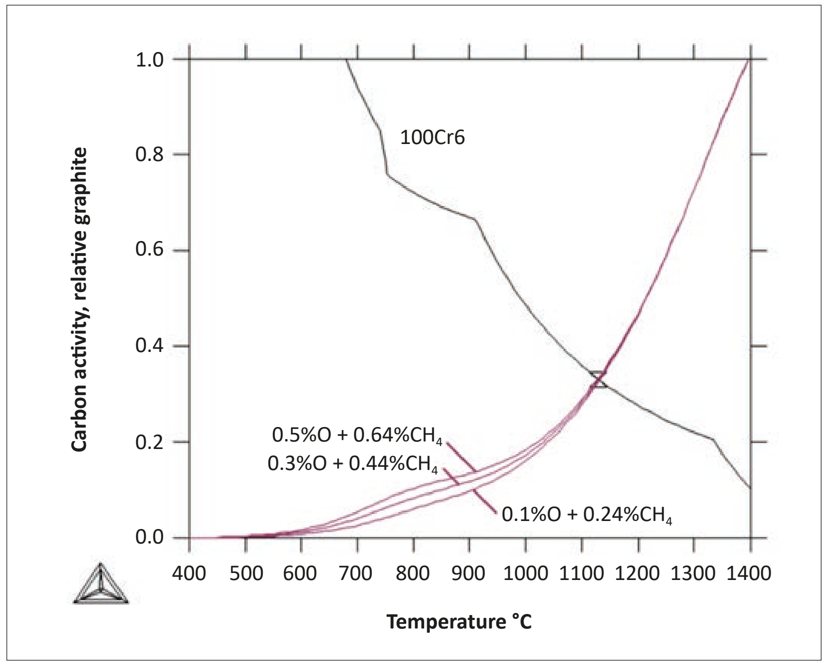


Fig. 4 Carbon activity in 100Cr6 and three different atmospheres with carbon control [1]

Near net shape fabrication of an impeller by Hot Isostatic Pressing

The development of a production route and material for the near net shape HIP (NNS-HIP) fabrication of an impeller for a nuclear power reactor coolant pump was the subject of a paper from Thomas Geneves (AREVA NP, France), Benjamin Picque (Aubert & Duval, France), Cyril Delon (COMPOSE, France), Frederic Bernard (Université de Bourgogne, France), Richard Chatain (Arts, France), Guillaume Ernoult (Ventana Group, France), Guilhem Roux (CEA, France) and Robert Ferriere (METAL-SCAN, France).

The AREVA group produces reactor coolant pumps (RCP) for nuclear power plants worldwide. Large pump components with complex shapes, such as impellers, have traditionally been cast. However, in recent years, alternative approaches consisting of machining impellers from a forged ingot have appeared in the marketplace. This solution offers an improvement in mechanical properties but has the disadvantage of a high material scrap rate, as high as 80% in the case of this type of impeller.

On the basis of this analysis of the current status quo, the reported HIPPI project was aimed at establishing the manufacturing sequence for a large-dimension impeller in a new material grade by means of Near net shape Powder Metallurgy processing. The project was also aimed at generating gains in production time and costs combined with

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Fig. 5 Fabrication of a primary pump impeller at AREVA [2]

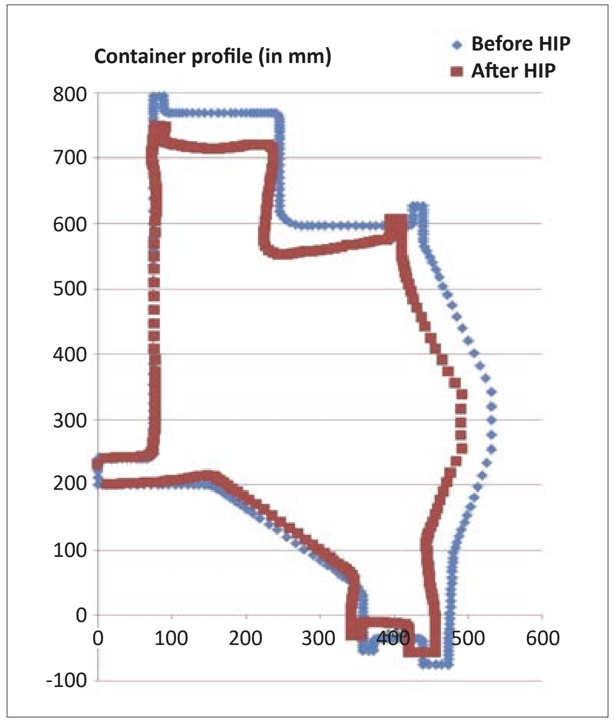

Fig. 6 2D modelling of the small dimension impeller [2]

reduced maintenance, in the context of seeking increased safety in nuclear power plants. To meet these objectives, AREVA and their partners have selected HIP as the unique mature PM process, which is able to manufacture large parts.

The presented paper reviewed the development of the required manufacturing sequence, the contribution of numerical simulation or modelling and the characterisation of a new super-austenitic stainless steel material applied to the fabrication of an RCP impeller (Fig. 5). The developed NNS-HIP manufacturing route began with the design and manufacture of the tooling (or container) by Aubert & Duval. The design procedure comprised 2D modelling to define the geometry prior to HIPping and the use of a specific internal tool and a methodology to define the 3D geometry details, such as channels, based on the results of the 2D modelling.

Following this design procedure, the initial tooling envelope was divided into different low carbon steel elements that could be machined at low cost and then readily assembled. At this stage, great attention was paid to the positioning of the weld areas of the different elements. These welds must guarantee the seal of the cavity during consolidation and maintain the equipment during the various handling operations.

After the low-carbon steel container was fully manufactured, it was filled with powder and prepared according to classical HIP capsule preparation procedures. The HIP cycle was then applied, using the parameters of 1100-1200°C and 1000-1100 bar pressure for approximately three hours. Because of the shrinkage of the system during the HIP cycle, a control step was required to relocate the impeller inside the container. This step involved an ultrasonic inspection that consisted of recording the echo from acoustic measurements, corresponding to the difference of acoustic impedance between the tool material and the impeller. The 3D software processing of these data allowed the simultaneous location of the impeller inside the container and the control of the quality of the impeller. This information was used to guide the rough machining of the container following HIPping. At the end of this stage, the remaining container material was finally dissolved by immersion in an acid bath. This approach was aimed at minimising the machining and finishing operations after HIP, in particular on the blades of the impeller, to limit the final cost of the part and reduce the fabrication time.

Since the prediction of the final shape obtained during the HIP process is a difficult issue for components with 3D shape or for non-constant radius axisymmetric parts, CEA has developed a HIP simulation procedure, involving the modelling of the powder's thermo-mechanical behaviour and experimental bench testing. Powder behaviour was considered to be elasto-viscoplastic, using the Abouaf Law. Identification of the relevant parameters in this law was carried out at different densification stages on samples obtained in interrupted HIP experiments, all tested in compression at a range of temperatures and deformation rates. The mechanical behaviour as a function of temperature of other materials (core and container) was identified with a standard method using tensile test-pieces.

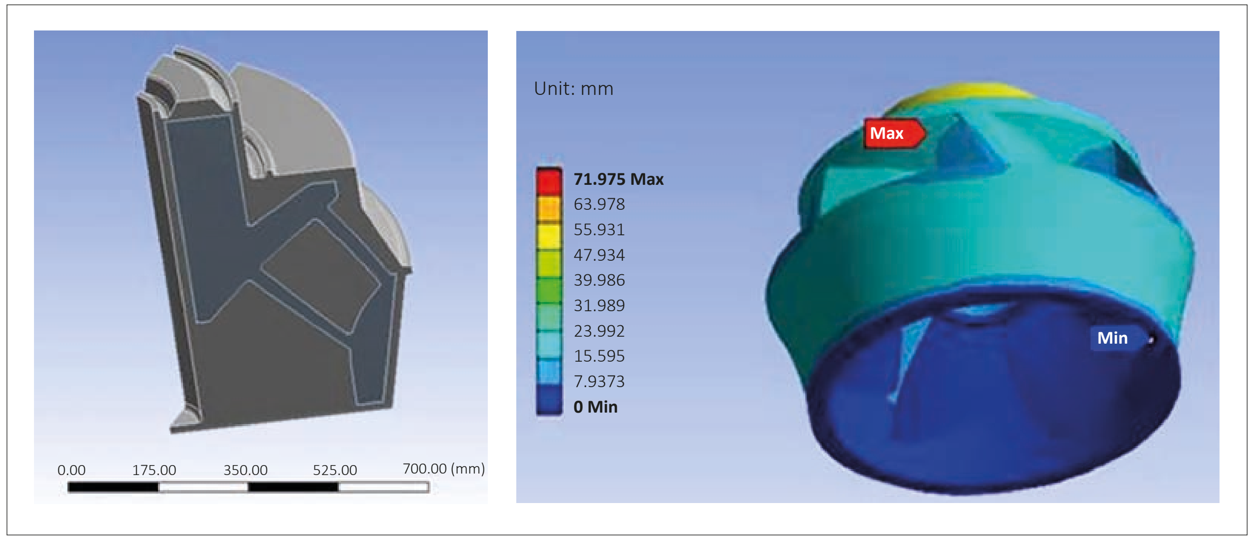

Fig. 7 3D modelling of the impeller (left: initial geometry; right: final geometry after chemical dissolution) [2]

Elements(%)	C	Si	Mn	P	S	Cr	Ni	Cu	N	Mo	W
S 31266	≤ 0.03	≤1.00	2.0 to 4.0	≤0.0350	≤0.020	23.0 to 25.0	21.0 to 24.0	1.00 to 2.50	0.35 to 0.60	5.2 to 6.2	1.50 to 2.50
Powder	0.029	0,37	3.1	0.018	0.009	23.7	21.0	1.47	0.45	5.2	1.47

Table 3 Chemical composition of the super-austenitic powder prepared by Erasteel [2]

In this project, a first axisymmetric small-dimension impeller was manufactured and compared to the simulation results. An example of the initial/final shape of the sample during the HIP process is presented in Fig. 6. A real 3D complex impeller was also simulated (Fig. 7) and compared to the initial concept of Aubert & Duval. 3D simulation of such a component required optimisation of both local material behaviour integration and global convergence scheme. The simulations were carried out in Ansys Workbench©, using routines dedicated to powder material integration and density-dependent thermal parameters. A further difficulty lay in the fact that the chemical dissolution of the container after HIP was complex to simulate. For this purpose, a "killing element" technique with an advanced relaxation scheme was used with success.

The UNS J92700 material grade, traditionally chosen for the manufacture of cast impellers, exhibits a relative sensitivity to thermal ageing and the improvement of material properties is of major interest in the lifetime extension of pressurised water reactors (PWR). AREVA therefore decided to focus on an austenitic stainless steel displaying improved mechanical characteristics as well as a good resistance to corrosion, in particular erosion corrosion and cavitation corrosion. After an extensive literature search, the super-austenitic stainless steel UNS S31266 emerged as a material of interest.

The use of the gas atomisation process for the powder production made it difficult to preserve the high nitrogen levels required by this grade. The consequence of a lack of nitrogen is the formation of sigma intermetallic phase at the grains boundaries during the cooling of the part. In order to limit the occurrence of this phenomenon, the composition of the powder was adjusted within the allowable limits of the specification, by reducing the presence of sigma phase forming elements (Cr, Mo, W, Si). These changes permitted Erasteel to achieve the composition presented in Table 3.

A study of the achievable microstructures and mechanical properties of this material in the HIPped condition was carried out on a block consolidated by HIP. The material state studied corresponded to a solution anneal at 1150°C and an air quench with a cooling rate of 20°C.min^{-1} at 800°C. In relation to the sensitivity of the grade to sigma phase precipitation, these conditions are conservative. The microstructure of the HIPped material was revealed by electrolytic etching in an aqueous solution containing 10% nitric acid and 5% acetic acid. This type of etching, in particular, reveals the intermetallic phases. An austenitic structure with twinned grains was observed. The average grain size was 60 µm and the hardness was homogeneous and around 290 $HV_{0.05}$. A moderate precipitation of intermetallic phases was also observed and subsequent EDS analyses confirmed these as being enriched in chromium.

Tensile tests were carried out at room temperature and 350°C, whereas impact strength tests were conducted only at room temperature, both in agreement with the

Sample	Temperature (°C)	0.2% Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	5d Elongation (%)
RCC-M requirements	20	≥210	≥480	≥35
	350	≥120	≥350	-
S 31266	20	499	873	57.3
	350	373	693	64.7

Sample	Temperature (°C)	KCV Impac (J/cm²)
RCC-M requirements	20	≥80
S 31266	20	162

Table 4 Tensile properties (top) and impact strength (bottom) of PM-HIPped S31266 compared to RCC-M code requirements [2]

requirements of RCC-M, the French Nuclear Industry design code for mechanical components in PWRs. The test results are presented in Table 4. PM-HIPped S31266 exhibits outstanding YS and UTS values at room temperature and at 350°C, combined with a remarkable level of elongation. These results exceed the RCC-M requirements. The tensile properties are all the more interesting, as they were combined with high levels of impact strength. Further work is required to extend the validation data to include fatigue, wear and stress corrosion tests and characterisations in the aged condition.

However, a costing analysis for the developed manufacturing route has shown that the machining costs of the container represent a significant expense in the overall production cost of the impeller. Therefore, the authors have concluded that it would be advisable to limit this method to the use of high added value materials that require the use of Powder Metallurgy processes. The overall conclusions drawn from this project were that the technical feasibility of manufacturing a large and complex component with an NNS-HIP approach has been demonstrated and that the super-austenitic S31266 grade has been revealed as a promising material for PM and HIP applications.

Phase transformation under isostatic pressure in Hot Isostatic Pressing

Finally, one of the seven designated keynote papers in the congress, presented by Alexander Angré, Oskar Karlsson and Erik Claesson (Swerea KIMAB, Sweden), turned attention to the influence of HIP process parameters and, specifically, described a study of the influence of the isostatic pressure in HIP on the phase transformations induced in a combined HIP/heat treatment cycle.

New HIP cooling systems enable very fast cooling rates under isostatic pressure. This not only enables shorter HIP cycles, but also allows complete heat treatment cycles to be performed in one HIP cycle. The new URQ HIP cooling systems give the opportunity to investigate the impact of pressures up to 2000 bar on phase transformation time dependency.

The principle of the forced uniform rapid quenching (URQ) cooling is to utilise the cooler gas outside the furnace. During cooling, the hot gas inside the furnace is moved to the outside of the furnace, where mixing of hot and cold gas occurs at the same time as the colder mixed gas is pushed into the furnace chamber. This moving of the gas is performed by a fan or a compressor. The mixed gas on the outside of the furnace is cooled even further by a heat exchanger, situated outside the furnace but inside the pressure vessel, before the gas is introduced to the furnace chamber again. The mixed gas outside the furnace is also cooled down by the pressure vessel walls. This gas flow loop is what enables the very fast cooling of a URQ HIP unit and continues throughout the cooling sequence, until the temperature in the furnace is sufficiently low.

The fact that the cooling gas in the HIP is initially at the same temperature as the HIPped workpiece results in low temperature differences between the gas and the workpiece and this in turn results in low thermal gradients and therefore significantly lower thermal stresses throughout the cooling cycle.

A number of previously reported studies have shown that extremely high pressures during cooling (i.e. 20-42 kbar) push the phase transformation from austenite to pearlite and bainite towards longer times, hence increasing hardenability. The objective of the reported study was to investigate whether a more typical hot isostatic pressing (HIP) pressure of 1700 bar would be sufficient to create the same effect in shifting the austenite to pearlite phase transformation to longer times and, if so, by what order of magnitude.

Two different materials were selected for the study:

Firstly, 4340, also designated as 34CrNiMo6 (in EN specifications) and SS 2541, is a steel designed for quenching and tempering. The chemical composition of the 4340 material used in the study is given in Table 5.

The second selected material was AstaloyTM Mo, a pre-alloyed Fe-Mo material for metal powder component production produced by Höganäs AB. The chemical composition of the Astaloy Mo material used in the study is given in Table 6. This material was water atomised, uniaxially pressed and sintered into samples with the density 7.47 g/cm³ and dimensions of Ø = 25 mm and h = 25 mm at Höganäs AB, Höganäs, Sweden.

Available TTT diagrams suggested that the pearlite nose temperatures for these two materials are quite similar, indicating that simultaneous HIPping of the two materials might be feasible. TTT diagrams for 4340 were found in the literature and, additionally, a diagram was calculated using JMatPro. As can be seen from Figs. 8 and 9, these TTT diagrams varied quite significantly from one another and this had to be taken into account when deciding on isothermal hold time intervals.

For Astaloy Mo, TTT diagrams for different carbon contents were calculated using JMatPro. Increased carbon content shifts the curves towards lower temperatures and longer times. A carbon content of 0.6% was chosen, mainly due its offering sufficient time to pearlite start and because, at this carbon content, there was no evidence of the occurrence of any ferrite transformation prior to the pearlite transformation (Fig. 10). Calculations using different grain size values in JMatPro showed a significant grain size effect on hardenability, with finer grain size pushing the curves towards shorter times. The actual measured grain sizes of the samples were therefore used as input data.

The isothermal transformation trials were planned so that a suitable isothermal temperature was selected by consulting the available TTT diagrams for the two materials. Subsequently, suitable hold times for each material were selected. The HIP cycle was designed so that the materials were initially subjected to an austenitisation treatment at 850°C for 15 minutes, followed by fast cooling down to the selected isothermal temperature, 650°C, where the material was held for the chosen hold time, followed by rapid quenching to room temperature. All HIP cycles were performed at low and high pressure separately. All HIP trials were performed with solid cylindrical specimens of the materials with a size of 25 mm x 25 mm. All HIP cycles were performed in the QIH9 URQ HIP at Quintus Technologies AB, Västerås, Sweden, permitting cooling

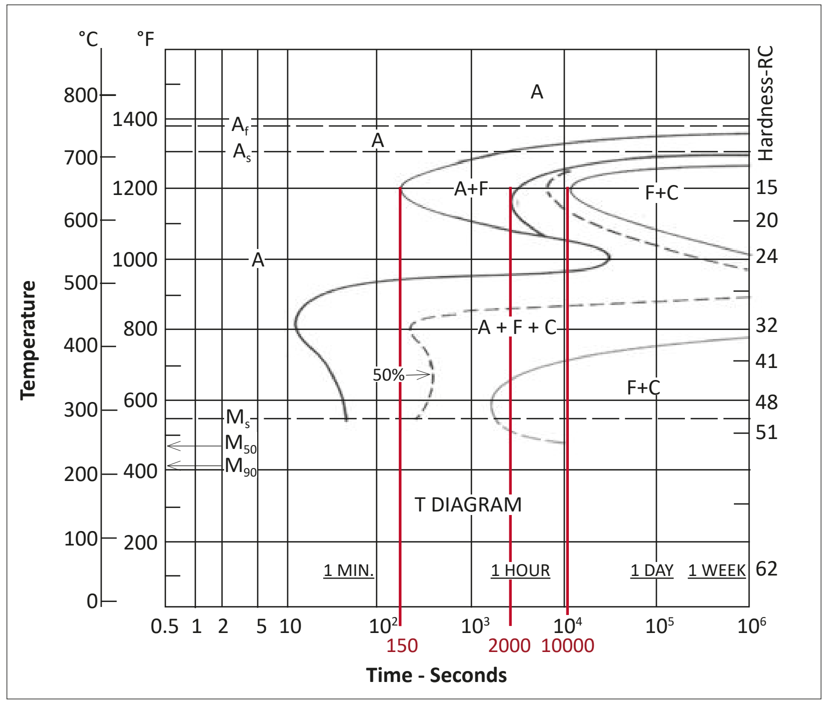

Fig. 8 TTT diagram from the published literature for 4340. 1% ferrite at 150 s, 1% pearlite at 2000 s and 99% pearlite at 10,000 s at 650°C [3]

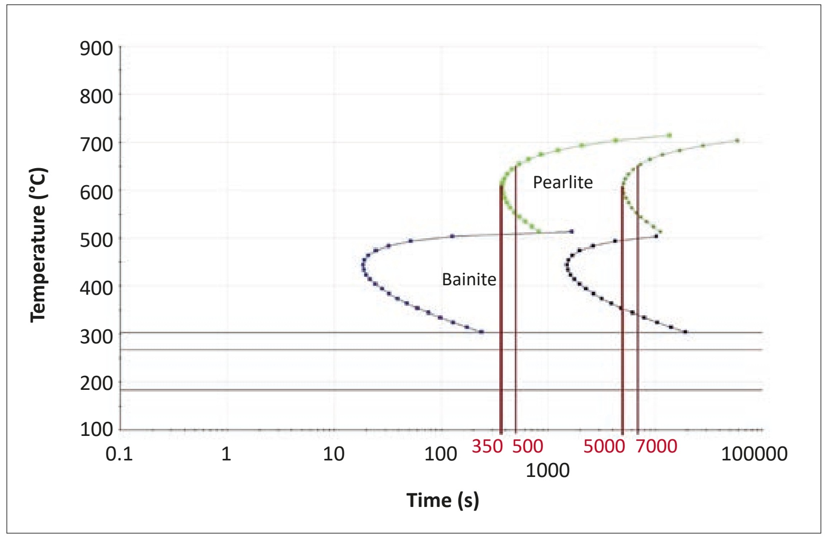

Fig. 9 TTT diagram for 4340 with grain size 15 µm, calculated using JMatPro. 0.1% pearlite at 500 s and 99.9% pearlite at 7000 s at 650°C [3]

Elements (%)	C	Mn	Si	Cr	Ni	Mo	Cu	Fe
4340	0.37	0.74	0.26	1.45	1.50	0.19	0.16	bal.

Table 5 Chemical composition of the 4340 material according to specification [3]

Elements (%)	C	Mo	Fe
Astaloy Mo	0.6	1.5	bal.

Table 6 Chemical composition of the Astaloy Mo material according to specification [3]

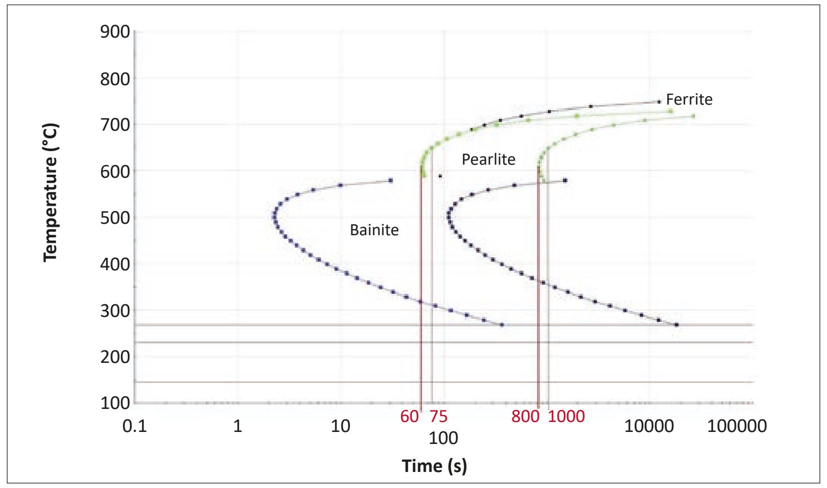

Fig. 10 TTT diagram for Astaloy Mo with grain size 20 µm, calculated using JMatPro. 0.1% pearlite at 75 s and 99.9% pearlite at 1000 s at 650°C [3]

rates of up to 3000°C/min in the gas, i.e. about 45°C/s. Table 7 presents a matrix of all the trials performed during the project.

To be able to use the uniform rapid quenching feature, a lower pressure than the maximum HIP pressure of 2000 bar had to be used as the maximum pressure for the trials. 1700 bar was considered to be sufficient for the use of URQ and also high enough to give relevant results on the influence of HIP pressure on phase transformation. It was decided to also run the low pressure cycles in the HIP, in order to make the high and low pressure cycles as similar as possible. To be able to control the HIP temperature and rapidly quench the material, 100 bar had to be used as the minimum pressure.

Also, it was noted that the Astaloy Mo, 500 s, 650°C isotherms were so short that the temperature only just reached 650°C before quenching. This fact could possibly affect the results. However, the low and high pressure cycles looked similar in this respect and therefore it was concluded that the comparison should still be valid.

For the evaluation of phase volume fractions, each specimen was wet ground, polished with diamond paste and, subsequently, electrolytically polished. The phase fraction evaluation was then performed using the grid method on SEM images. Micro Vickers hardness testing was also performed on each specimen using a hardness tester with a load of 1 kg.

Figs. 11 and 12 show the trial results for 4340 in the form of comparisons of phase fraction transformed to pearlite and hardness, at low and high pressure, respectively. The equivalent results for Astaloy Mo are shown in Figs. 13 and 14.

The phase fraction evaluations unequivocally supported the thesis

Sample	Austenitisation temperature [°C]	Austenitisation time [min]	Isotherm [°C]	Pressure [bar]	Hold time [s]
4340					
4340 1000s	850	15	650	100	1000
4340 1000s P	850	15	650	1600	1000
4340 1500s	850	15	650	100	1500
4340 1500s P	850	15	650	1700	1500
4340 2000s	850	15	650	100	2000
4340 2000s P	850	15	650	1700	2000
4340 3000s	850	15	650	100	3000
4340 3000s P	850	15	650	1700	3000
4340 6000s	850	15	650	100	6000
4340 6000s P	850	15	650	1800	6000
Astaloy Mo, 0.6% C					
AstMo 500s	850	15	650	100	500
AstMo 500s P	850	15	650	1700	500
AstMo 1000s	850	15	650	100	1000
AstMo 1000s P	850	15	650	1600	1000

Table 7 Matrix of the trials performed within the project [3]

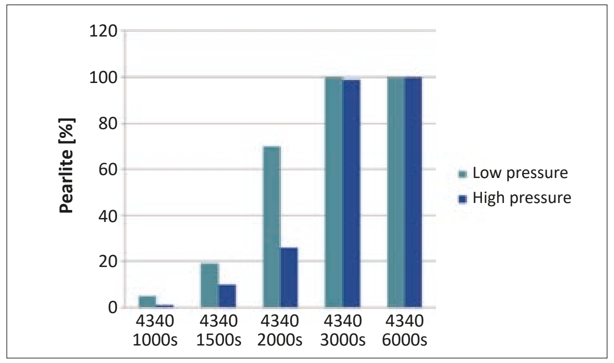

Fig. 11 Comparison of phase fraction transformed to pearlite, at low/high pressure, for each of the 4340 samples [3]

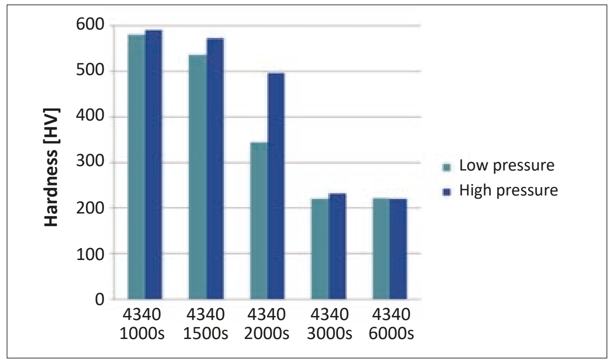

Fig. 12 Comparisons of hardness, at low/high pressure, for each of the 4340 samples [3]

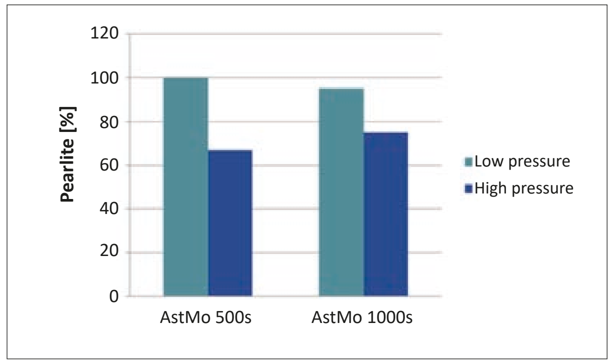

Fig. 13 Comparisons of phase fraction transformed to pearlite, at low/high pressure, for both Astaloy Mo [3]

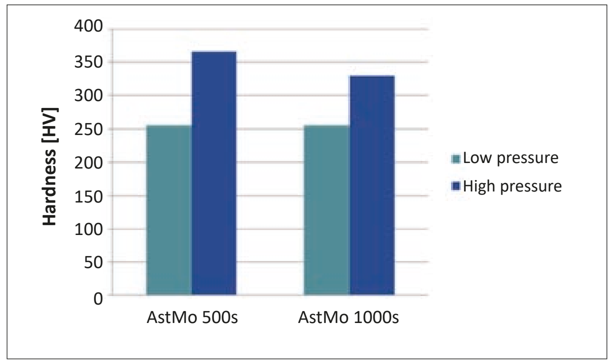

Fig. 14 Comparisons of hardness, at low/high pressure, for both Astaloy Mo samples [3]

that HIP pressure influences the phase transformation from austenite to pearlite by pushing it towards longer times. For 4340, the 1500 s and 2000 s samples provided the clearest evidence, where the 2000 s sample subjected to low pressure contained almost three times as much pearlite as the sample subjected to high pressure. Hardness testing also supported this conclusion for all samples except for the 6000 s sample, where a difference in 2 HV lay within the margin of error. The hardness values were higher for the high pressure samples, because of a lower level of pearlite phase transformation, i.e. higher amounts of martensite. For Astaloy Mo, the phase transformation evaluation and the hardness measurements for both HIP cycles, 500 s and 1000 s, also showed that HIP pressure increases hardenability.

4340 HIP cycle logs, employing embedded thermocouples, displayed an increase in the load centre temperature during the isotherm at 650°C, although the gas temperature stayed constant, and it was considered most likely that this observation represented the exothermic austenite to pearlite phase transformation. In the HIP cycle logs, the bump appeared both larger and earlier in time at low pressure than at high pressure. Not only did this observation strengthen the thesis that HIP pressure pushes the austenite–pearlite phase transformation towards longer times, but it also suggested that the pressure suppresses the rate of the phase transformation.

References

[1] Reaching full density of 100Cr6 PM steel by capsule free HIP of high-velocity compacted material, H Magnusson *et al*, as presented at the World PM2016 Congress, Hamburg, October 11-14 2016, and published in the Proceedings by EPMA, UK.

[2] Near net shape fabrication of an impeller by Hot Isostatic Pressing, T Geneves *et al*, as presented at the World PM2016 Congress, Hamburg, October 11-14 2016, and published in the Proceedings by EPMA, UK.

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World PM2016: Developments in hardmetal processes and applications

WORLDPM2016 congress and exhibition

Developments in the processing and application of hardmetals were the subject of a number of papers at the World PM2016 Congress, held in Hamburg, Germany, 9-13 October 2016. In this review Dr David Whittaker highlights two presentations that discuss the use of Electric Resistance Sintering and Spark Plasma Sintering to process hardmetals. A further two presentations look at the development of a novel binder phase and the influence of numerous factors on the wear and fracture mechanisms of drill bits during drilling of reinforced concrete.

Development of the electric resistance sintering process for the fabrication of hardmetal parts

The first of these papers, from M A Lagos and I Agote (TECNALIA Research & Innovation, Spain), J M Gallardo and J M Montes (University of Seville, Spain), T Schubert and T Weissgaerber (Fraunhofer IFAM, Dresden, Germany), L Prakash (Kyocera Unimerco Tooling AS, Denmark), C Andreouli and V Oikonomou (MERTEC, Greece) and D Lopez and J A Calero (AMES, Spain), reported on the development of the ERS (Electric Resistance Sintering) processing of WC-Co hardmetal within the EC Seventh Framework project, EFFIPRO.

Cemented carbides are usually produced by a sintering process that involves the participation of a liquid cobalt phase. However, the presence of this phase during sintering stimulates the growth of the WC grains. Thus, the control of grain growth of the carbide phase during liquid phase sintering is an important objective. In general, decreasing WC particle size increases mechanical properties such as hardness, wear resistance and transverse rupture strength. It is known that fracture toughness decreases with increasing hardness in conventionally processed hardmetals, whereas the increase of hardness in nano-structured composites does not further reduce their bulk fracture toughness.

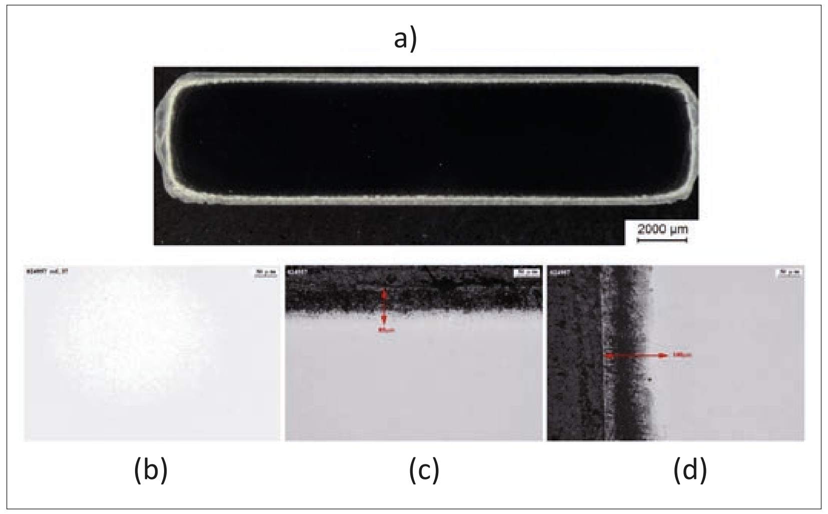

Fig. 1 Cross section of a blank produced by ERS, distribution of the porosity: (a) Whole blank, (b) Core of the material (scale bar 50 microns), (c) External porosity, contact with the punches (scale bar 50 microns), (d) external porosity, contact with the ceramic die (scale bar 50 microns) [1]

Very fast sintering processes offer an opportunity to avoid the liquid phase sintering and thus limit WC grain growth. Electric Current Assisted Sintering (ECAS) encompasses a family of consolidation methods in which mechanical pressure is combined with electric and thermal fields to enhance particle bonding and densification. ECAS techniques can be classified with respect to the processing time. In conventional ECAS processes, such as SPS (Spark Plasma Sintering), processing time is in the range of minutes and, for this reason, a controlled atmosphere is mandatory. However, in very fast processes such as EDS (Electro Discharge Sintering) and ERS (Electric Resistance Sintering), processing time is a few seconds and the process can be performed in air. This is a very important advantage from an economic point of view. In ERS, the current is produced by a low-voltage transformer (around 10 V) and therefore, in this process, it is easier to control the current applied to the sample with a consequent improvement in the homogeneity of products.

Within the EFFIPRO project, the scale up and development of ERS technology for the fabrication of cutting tools was carried out. A fully automated pilot plant equipment was developed, comprising an electrical press of 15 tonne capacity in combination with low voltage transformers. The initial objective was to produce hardmetal inserts and small drills for automotive and aerospace applications. The work presented in this paper covered the development of hardmetal cutting tools by ERS. WC-Co powders (submicron size) were used with Co contents being at 6 wt.% and 10 wt.%. Granulated powders from a commercial source were procured with organic wax and a pre-treatment was performed in order to eliminate organic components. These particles were agglomerated into spherical granules with a size between 100 and 200 microns. The apparent density of the powder was 3.7 g/cm^3.

In the ERS processing, the powder was filled in a ceramic die between two copper electrodes. Tests were performed in air. In order to fabricate different cutting tools, blanks of different sizes were obtained. Typical samples had 22 mm diameter with a length of 10-16 mm. The maximum applied current density was between 50 and 60 kA/mm^2 with a holding time of 500 ms. The maximum applied pressure was 100 MPa.

As a first stage in the development of the technology for cutting tool manufacture, blanks with the following dimensions were obtained: triangular inserts, with 16 mm side and 5 mm thickness, and small drills of 7 mm diameter and 16 mm length.

The ERS samples typically consisted of a dense core surrounded by a porous surface layer. External porosity at the surface in contact with the metallic punches was smaller than that in the contact with the ceramic die (Fig. 1). Porosity at the core is very low, as can be observed in Fig. 1. After removing the surface layer, the samples obtained using ERS showed very high density levels within the range typically required for hardmetal tools.

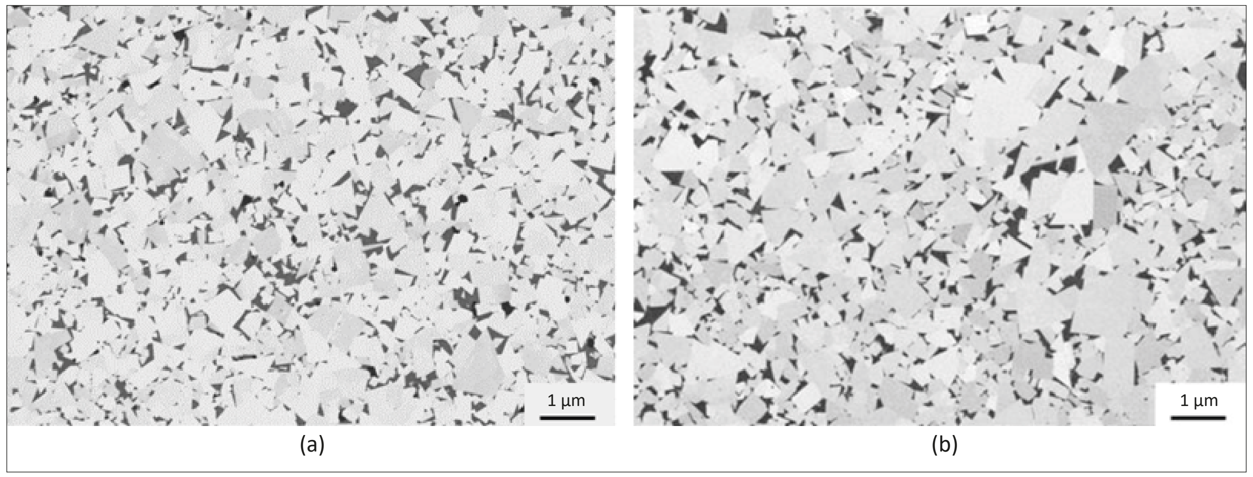

Fig. 2 HRSEM images of polished and ion-etched cross sections of ERS/WC6Co (a) and Sinter HIP material (b) [1]

Composition	Process	Density (g/cm^3)	Hardness HV30	Fracture toughness K_{1c} (MPa√m)	H_c (Oe)	M_s (10^{-7} T*m^3*kg^{-1})
WC6Co	ERS	14.7	1960±15	9.6±0.5	470	101
	Sinter-HIP	14.8	1860±15	9.5±0.5	385	103
WC10Co	ERS	14.3	1750±20	10.3±0.5	420	150
	Sinter-HIP	14.4	1620±15	10.2±0.5	320	154

Table 1 Summary of the mechanical properties obtained [1]

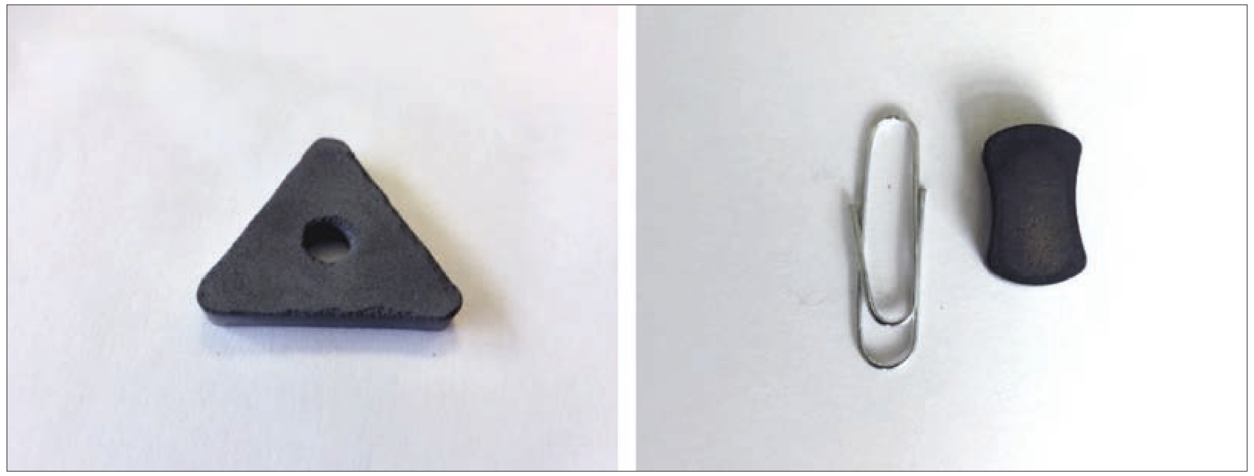

Fig. 3 Blanks produced by ERS technology [1]

In the ERS process, electrical current crosses the material and heating is produced by the Joule effect. Higher temperatures are produced at the core of the material due to the fact that temperature is higher where the electrical resistance of the material is also higher. Resistance of the powder mainly depends on the green density, which is lower at the core. External porosity is produced by the fast cooling of the material in contact with the copper punches and ceramic die. It is possible to improve this phenomenon by using ceramic dies with lower thermal conductivity. Using these blanks, final tools are being produced and coated for machining trials. The characterisation of these tools is currently in progress.

The microstructure obtained in the processed materials is shown in Fig. 2. In order to compare the ERS process with conventional processing, the microstructure of a material processed by Sinter-HIP is also presented in this figure. In both cases, the microstructure consisted of polygonal WC grains surrounded by a Co metallic matrix. Measured mean linear intercepts indicated a WC-grain size of about 274 nm for the ERS-processed material and 326 nm for the conventional process. The grain size obtained was very similar to values reported in the literature for SPS of nanocrystalline powders. It is important to note that, in the reported work, submicron powders were used rather than nanoscale ones. The very low grain growth observed during processing opens the possibility of nanocrystalline hardmetals. The contiguity of ERS WC6Co was measured at around 67% WC/WC combined with 33% WC/Co boundaries. The corresponding values of contiguity for the Sinter-HIP samples were 56% WC/WC and 44% WC/Co.

The measured mechanical properties for the ERS and Sinter-HIP processed materials, with 6 and 10% Co, are compared in Table 1. The ERS materials showed higher hardness compared to conventional materials processed by Sinter-HIP and similar fracture toughness (K_{1c}).

In ERS materials, a small variation in hardness was observed between the core and surface of the samples, giving a small increase in hardness at the surface. The cause of this phenomenon is not completely clear at the moment. The authors proposed that a possible explanation could be linked to the slightly coarser grain size observed at the core of the samples. In ERS, samples are heated by the Joule effect and the temperature at the core of the samples is slightly higher than at the surface. This longer time at high temperature could produce a very small increase in grain size. Another possible explanation is the migration of Co from the core of the samples. This migration can generate small microporosity and thus a local decrease of the hardness. However, no significant variations in Co content were in fact observed.

Microstructural characterisation of WC–Co hardmetals is often performed using magnetic measurements. By measuring the magnetic saturation M_s or the coercivity H_c, it is possible to obtain an approximation of the WC grain size, cobalt content and even determine the presence of additional phases. Generally, an increase of coercivity is observed with a decrease in WC grain size. The comparison of the measured H_c values of ERS with Sinter-HIP samples confirms the observed reduction in grain size.

The paper concluded with a couple of practical considerations regarding the ERS technology. Firstly, one of the most interesting advantages of this process route is the possibility of obtaining near net shape products. For this reason, the production of complex shapes was also studied. Fig. 3 shows blanks with complex shapes obtained by ERS. In addition to the external shape of the blanks, the possibility of internal holes could be achieved by the integration of small inserts within the ceramic dies. Another important aspect of the technology is the energy consumption compared with conventional processing. In the reported work, it was possible to calculate the energy consumption of the pilot plant equipment for the production of small parts (inserts and small drills). For the prototypes produced, energy consumption was around 1-3 kWh/kg, depending on the geometry of the blank. It is difficult to compare the energy consumption of a pilot plant equipment to an industrial scale plant such as conventional Sinter-HIP, but, taking into account the very short processing time and the low currents, it seems that the new process is very interesting from an energy efficiency point of view. The pilot plant equipment developed within the EFFIPRO project is fully automated and enables the fabrication of around 120-200 blanks per hour.

Novel WC Hardmetal with Cr/Fe Binder Alloy Sintered by SPS

A paper by A Garcia-Junceda (IMDEA Materials Institute, Spain) and I Sáez and J M Torralba (Universidad Carlos III Madrid, Spain), addressed the processing of a novel WC hardmetal with a Cr/Fe binder alloy in a route involving mechanical alloying of the feedstock material and sintering by SPS. Tungsten carbide (WC) is the most commonly used compound in the production of hardmetals. Also, Co has been used for many decades as the optimal binder for a wide range of applications, including drilling, construction and milling tools. Recent health and safety concerns, however, have driven interest in the partial or total substitution of Co or Ni by other more economic and less toxic binders, due to the carcinogenic character of these metals.

Although detailed studies of chromium solubility in WC are not available, the C-Cr-W phase diagram suggests that a certain solubility should exist. A number of studies of the fabrication of WC-Fe hardmetals have been published and, on the basis of these, it seems that there is difficulty in achieving a combination of high hardness and toughness. In addition, Fe dissolves a much lower quantity of WC than Co, at their respective eutectic temperatures. Within the context of this background, the authors therefore chose to study a new WC-CrFe hardmetal, processed by a PM route including mechanical alloying of the two raw powders and Spark Plasma Sintering (SPS).

The composition of the Cr/Fe binder alloy powder used in the investigation is given in Table 2. This Cr/Fe powder had an irregular morphology, whereas the WC powder used exhibited a polygonal shape. The particle size of the WC powder was coarser than that of the Cr/Fe powder, as shown in the particle size distributions in Fig. 4 and in the corresponding values given in Table 3. The volume fraction designed for the WC reinforcement in this hardmetal was 30 vol.%. The mechanically alloyed WC-CrFe powders were produced by high-energy ball milling in a planetary ball mill. The milling parameters selected were: 1) charge ratio: 10/1 wt.%, 2) ball diameter, 10 mm, 3) ball material, WC-Co, 4) speed, 350 rpm, 5) selected time, 10 h, and 6) argon atmosphere. In addition, 0.5 wt.% of stearic acid was chosen as the process control agent. To analyse the evolution of the mechanically alloyed powder structure, samples were taken after each two hours of milling time.

The evolution of the morphology, microstructure and composition of the particles is shown in the electron micrographs in Fig. 5, where the grey areas correspond to the Cr/Fe phase and the white regions to the WC. As expected for a ductile-brittle system, the mechanical alloying had just started after two hours of milling with clear large grey areas of ductile Cr/Fe phase, whereas the initial WC brittle powder had been fractured into small particles with only a few carbides having been incorporated into the Cr/Fe phase, Fig. 5(a). After four hours of milling, it was possible to distinguish some laminar regions of Cr/Fe and WC, Fig. 5(b). When increasing the milling time, fine WC particles were randomly dispersed in the powder and the morphology of the powders tended to be more rounded, smaller and with a more homogeneous microstructure, Figs. 5(c), (d) and (e). The particle size distribution showing the evolution of the MA powders is shown in Table 4. After these analyses, the ten hour-MA powders were selected as suitable powders for the subsequent consolidation by SPS.

The prepared WC-CrFe powder was consolidated by SPS. The powder was poured into a cylindrical graphite die (20 mm in diameter) and heated in a vacuum chamber (10^{-3} Pa). The temperature of consolidation was 1250°C

Powder	Cr	Fe	Si	C	N	P	S
CrFe	71.4	Balance	0.4	0.04	0.03	0.02	0.004

Table 2 Chemical composition of the Cr/Fe powder used as metallic binder (in wt.%) [2]

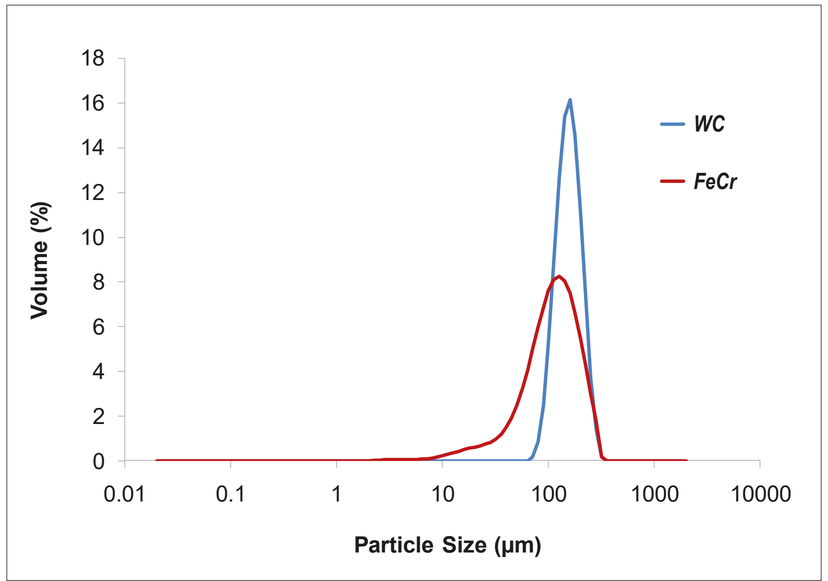

Fig. 4 Particle size distribution of the raw powders [2]

	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
Cr/Fe	45.4	117.6	220.0
WC	114.8	163.9	232.2

Table 3 Particle size distribution of the raw powders [2]

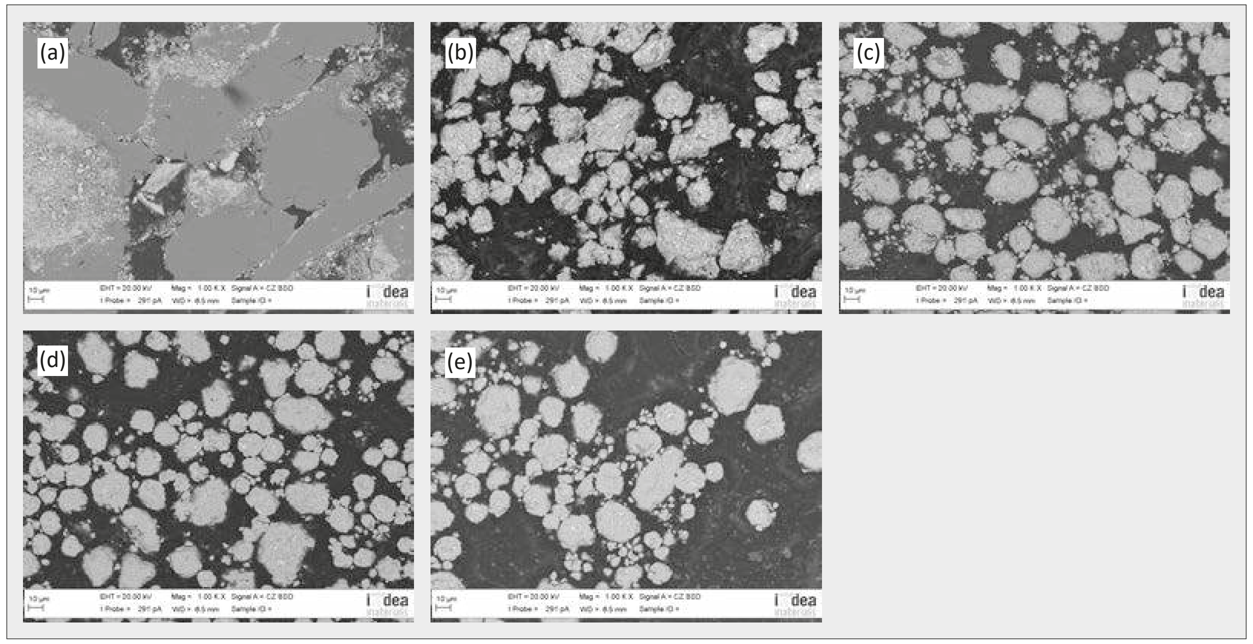

Fig. 5 Morphological and microstructural evolution in the WC-CrFe particles after: (a) 2 h, (b) 4 h, (c) 6 h, (d) 8 h, (e) 10 h of milling [2]

MA Time (h)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
2	1.4	9.0	116.7
4	5.5	26.4	77.2
6	4.0	14.8	51.9
8	4.1	14.6	31.6
10	4.9	14.4	42.6

Table 4 Particle size distributions of the MA WC-CrFe powders [2]

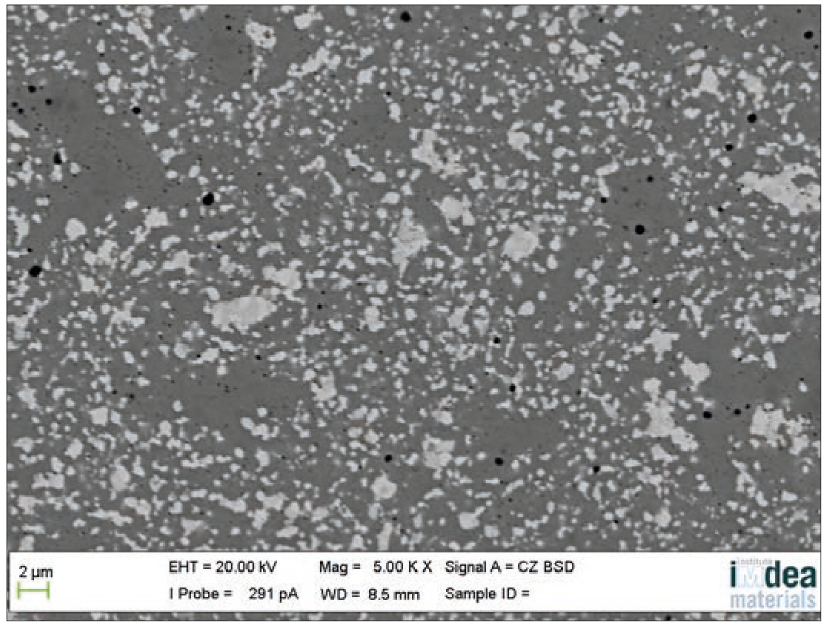

Fig. 6 SEM micrograph of the SPS WC-CrFe hardmetal [2]

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and the heating rate was 100°C/min, with a holding time of ten minutes followed by furnace cooling. The pressure during the consolidation was 80 MPa. A micrograph showing the WC-CrFe hardmetal obtained after the consolidation by SPS at 1250°C is shown in Fig. 6. In this SEM image, it is possible to observe that the WC reinforcement (white phase) had a different size and shape along the matrix, being coarser than in the MA powder obtained after 10 hours, Fig. 5(e). Moreover, there were some Cr/Fe areas (grey phase) free of reinforcement. In addition, some small and equiaxed pores were apparent in the microstructure, since the densification achieved was 95%. Finally, the Vickers hardness measured in this WC-CrFe hardmetal was 1758 HV1, a high value in comparison with other hardmetals where the volume fraction of reinforcement is higher than 30%.

Although more studies need to be carried out, these preliminary results highlight the viability of sintering this new hardmetal, formed by a Cr/Fe metallic binder and a WC reinforcement, using a field-assisted sintering technique such as SPS.

Phase transformations in iron-based alternative binders for hardmetals

The next paper also focused on the development of a novel binder phase and was provided by Lisa Toller (Uppsala University, Sweden) and Susanne Norgren (Sandvik Coromant R&D and Uppsala University, Sweden).

The concerns over the potential carcinogenic effects of cobalt were a strong motivating factor, in the reported work, for the development of a binder material that reduced, rather than totally eliminated, the cobalt content in hardmetals.

Previously reported studies had shown that it is possible to produce hardmetals with transformation toughening iron-rich binders. Hardmetals with transformation toughening binders have similar hardness but increased toughness compared with hardmetals with cobalt binder and are therefore interesting materials for replacing hardmetals with cobalt binder. There is a current lack of in-depth knowledge on where and how deformation-induced martensite is formed in this type of hardmetal. The reported work therefore investigated the possibility of using a combination of ion polishing, indentation and Electron Backscattering Diffraction (EBSD) to gain a better understanding.

The majority of the hardmetal used in metal cutting applications is coated with hard ceramic coatings. A common coating procedure is Chemical Vapour Deposition (CVD). During the CVD coating procedure, the hardmetal is heated to ~800 or 1000°C and held for several hours at temperature. It might be expected that this heat treatment could influence the crystal structure of the binder phase and, therefore, it was deemed to be important to also study the binder in coated samples.

The hardmetal samples in the reported study were manufactured to contain 20 vol.% binder phase and 80 vol.% WC. Tungsten carbide raw material powder was used for the hard phase and Fe/Ni powder for the binder phase, adjusted with commercially pure Fe, Ni and Co to reach a binder phase composition of 50 Fe - 25 Ni - 25 Co (in wt.%). The powder was milled, along with polyethylene glycol and ethanol, for eight hours and pan dried in a nitrogen atmosphere. The samples were

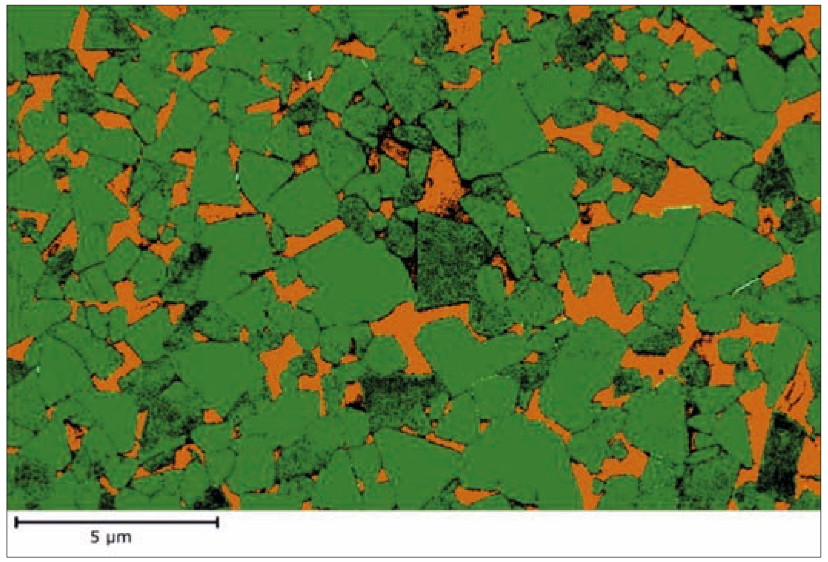

Fig. 7 EBSD map of the hardmetal after ion polishing. The binder phase is mainly austenitic with only minor areas indexed for martensite. WC in green, austenite in orange and martensite in the lightest grey, unindexed pixels are displayed in black. The increased amount of un-indexed pixels in the centre of the map is due to contamination [3]

Fig. 8 Secondary electron image of the hardmetal in the bottom of the 100 N indentation. The WC grains show a large amount of slip lines [3]

uniaxially pressed into square inserts and vacuum sintered at 1410°C for one hour. One sample was coated by chemical vapour deposition. Prior to coating, the sample was cleaned by the standard procedures used for cobalt-based turning inserts. The coating was a commercial multi-layer coating, including one step at 860°C and one step at 1000°C.

To study the microstructure of the hardmetal and, in particular, the binder phase, polished cross sections were prepared. To minimise the amount of defects at the surface and avoid any deformation-induced martensite formed during polishing, the surface was ion polished. After ion polishing, the binder phase was found to be mainly austenitic (Fig. 7). Small traces of martensite could be found in the grain boundaries between the WC and the binder phase and in WC-WC grain boundaries. The authors postulated that this martensite could have been a result of increased strain in grain boundaries that had caused a phase transformation. However, this could also be misindexing and further investigation is required on the issue.

To study deformation-induced martensite, indentation and scratch tests were performed on the polished surface. Indentation was done with a Rockwell C indenter with a 200 µm radius at 10 and 100 N load. Imaging and phase characterisation with EBSD was performed with a scanning electron microscope, equipped with an EBSD detector. After indentation, the tungsten carbide grains within the indentation were found to contain large amounts of slip lines (Fig. 8). EBSD maps made to study the binder phase did not, however, show any significant amounts of martensite in either of the indentations made with the different loads. Overall, the amount of deformation in the binder phase during indentation was less than expected and scratch tests were made to increase the amount of deformation. The scratch tests gave a large increase in the amount of deformation. Secondary electron images showed large amounts of deformation in the 100 N scratch with severe crushing of the WC grains

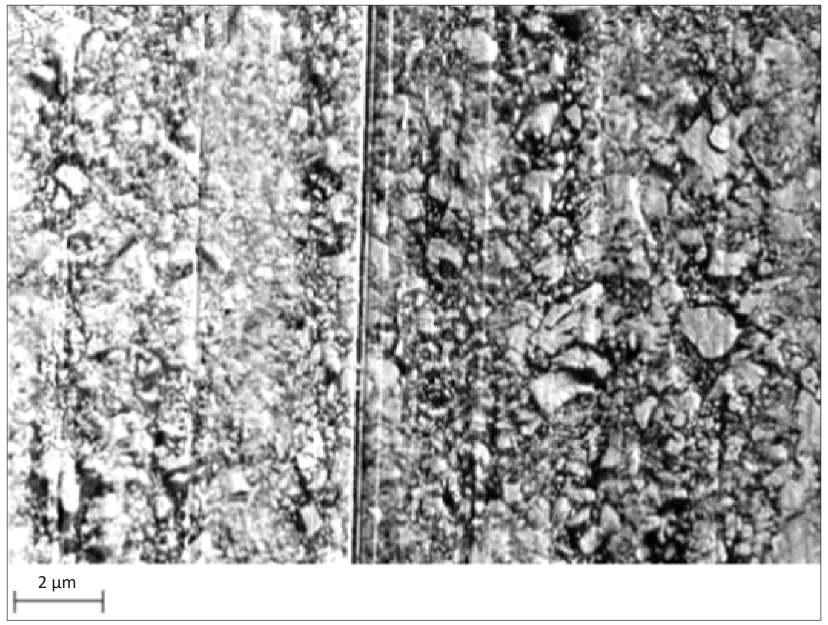

Fig. 9 Secondary electron image of the bottom of the 100 N scratch: the hardmetal in the bottom of the scratch, showing the crushed WC grains [3]

(Fig. 9). Due to the severe surface damage in this scratch, no attempts were made to carry out EBSD studies on this surface. In the 10 N scratch, the tungsten carbide grains had retained their shape, but EBSD showed very poor indexing of the binder phase, indicating a deformed binder. In the areas that were indexed, some showed a martensitic structure. These areas were found also in the centre of the binder phase areas and not only in the grain boundaries as for the as-polished surface in Fig. 7. This led to the conclusion that EBSD could detect that a deformation-induced phase transformation had occurred in the scratch and opens up the possibility that this experimental technique can be used to study smaller deformations and individual binder phase grains in terms of how they behave during deformation.

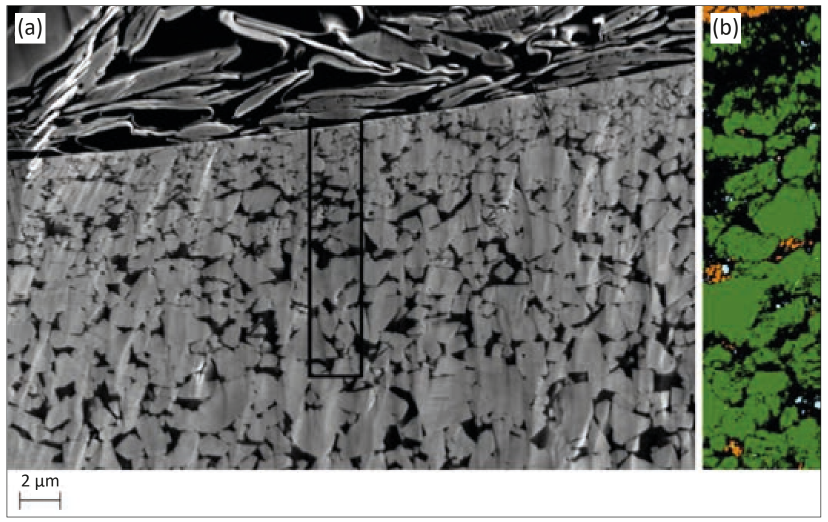

Fig. 10 (a) Secondary electron image of a part of the cross section through the 100 N scratch, showing crushed WC grains down to approximately 10 µm depth, (b) EBSD map of area marked in (a) showing the heavily deformed area close to the surface and deformation-induced martensite further away from the surface [3]

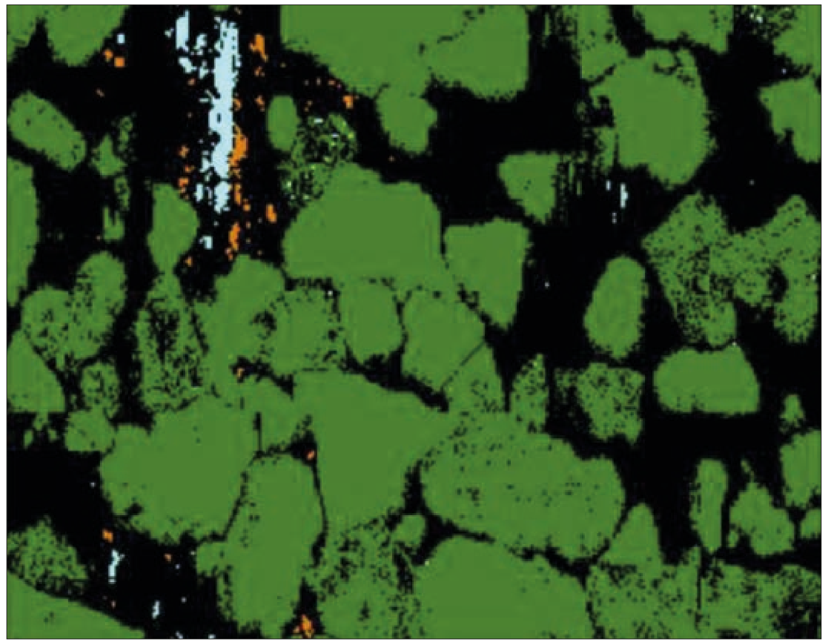

Fig. 11 Bottom of the 10 N scratch at the coated sample: EBSD map of an area in the bottom of the scratch showing WC in green, austenite in orange and martensite in the lightest grey, unindexed pixels are displayed in black. The overall impression is very similar to the uncoated sample [3]

Since it was not possible to study the 100 N scratch from the surface, a cross section was made through the scratch to study the underlying material. Imaging in the SEM showed a zone with crushed WC grains stretching approximately 10 µm into the hardmetal (Fig. 10a). EBSD mapping revealed martensite in the binder down to 10 µm depth (Fig. 10b). Closer to the surface, the indexing was rather poor in both the binder and the WC and this could be explained by a heavily deformed material. These results show that deformation-induced martensite is not only formed at the surface.

To obtain a first indication as to whether the coating procedure affects the phases and phase transformations in the binder, a sample of the hardmetal bulk from a CVD-coated insert was polished and subjected to a 10 N scratch. EBSD showed that the binder phase at the as-polished surface was also mainly austenitic after coating, similar to the uncoated sample in Fig. 7. When looking within the scratch, the binder again looked very similar to the uncoated case (Fig. 11). It appears that the heat treatment in the CVD coating does not affect the main characteristic of the binder, but this remains to be investigated.

The overall conclusion was that the combination of ion polishing, scratch testing and EBSD was found to be useful in studying deformation-induced martensite and could be a good complement to XRD and etching. Its benefits are mainly the possibility to obtain local crystallographic information, in addition to its being largely non-invasive. However, more work needs to be done on optimising the test conditions.

The influence of loading spectrum, workpiece material, hardmetal grade and drill bit design on the wear and fracture mechanisms of drill bits during rotary-percussive drilling of reinforced concrete: Experimental studies and FEM

Finally, an application-related paper, by Steven Moseley, Siavash Momeni, Carsten Peters, Günter Domani and Jan Allaart (Hilti AG, Liechtenstein) focused on a particularly exacting type of application for hardmetal drill bits, namely the rotaty-percussive drilling of reinforced concrete [3].

The wear and fracture mechanisms in such drill bits in this type of application vary greatly and the reported study was therefore aimed at investigating the active wear mechanisms and intensity as a function of binder content, WC grain size and the properties of different combi-hammer tools. Additionally, Finite Element Modelling (FEM) was employed to visualise the complex stress states within the drill heads during drilling and to compare them with experimental observations of fracture.

The hardmetal grades used in the study are listed in Table 5. Various WC grain sizes from fine to extra-coarse and a cobalt binder content from 6% to 12% by weight were tested in pure concrete. In reinforcement steel drilling tests, only the grade 6M was tested. The drill bit geometries tested are shown in Fig. 12. Hilti TE-YX drill bits with a diameter of 20 mm were used for tests in concrete. These have a solid, single-piece hardmetal head brazed to the steel helix. Wear mechanisms in concrete drilling were investigated at various positions of this drill bit, also indicated in Fig. 12. For tests in reinforcement steel, Hilti TE-CX drill bits with a diameter of 12 mm were used. These drill bits have a solid, single-piece hardmetal head resistance welded to the steel helix. Wear mechanisms were investigated at three of the five locations shown in Fig. 12, namely the axial edge, edge 45° and side.

Hilti combi-hammers with different rotational speeds and single-blow impact energies were used, Hilti TE 30 and Hilti TE 70, as again shown in Fig. 12. Basic specifications for these combi-hammers are listed in Table 6. An abrasive high SiO_2 aggregate concrete of strength class C50/60 (minimum unconfined compressive strength 50 MPa) with 16 mm diameter 520-grade hot formed carbon-steel reinforcement (rebar) was used as the work-piece material (Fig. 12). In concrete-only drilling, wear mechanisms and wear intensity were evaluated after 1.6, 4.8 and 16.2 metres drilled using an automated test rig. In rebar, three centrally drilled holes were made manually and the active wear

Grade	WC grain size	Co-content (Wt. %)	Hardness (HV10)
6M	Medium	6%	1420
12F	Fine	12%	1540
11M	Medium	11%	1300
6E	Extra-coarse	6%	1310
10E	Extra-coarse	10%	1130

Table 5 Hardmetal grades used [4]

Test	Combi-Hammer	Impact energy (J)	Rotational speed (min^{-1})
MP-1	TE 30	3.2	750
MP-2	TE 70	10.5	360
MP-3	TE 70 (mod.)	10.5	720

Table 6 Specifications of the Hilti combi-hammers used [4]

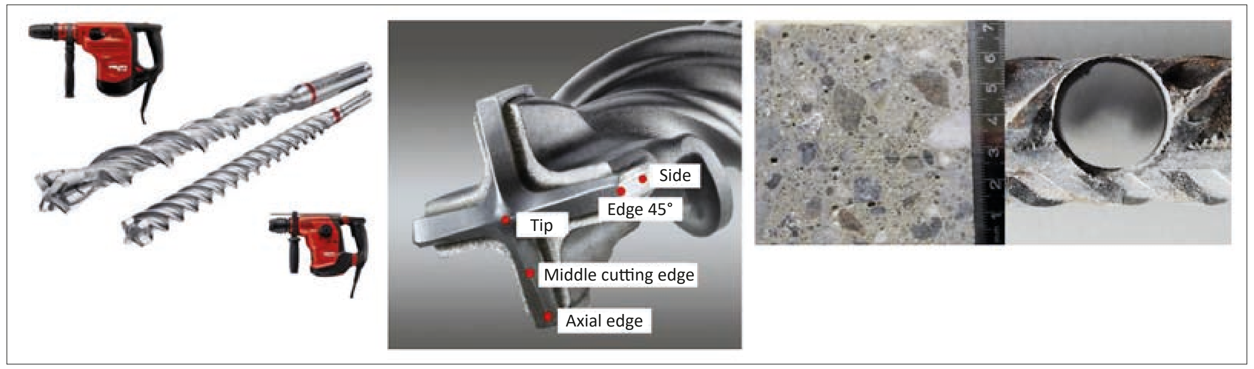

Fig. 12 Experimental set-up. Hilti TE-YX and TE-CX drill bits with TE 30 and TE 70 combi-hammers (left), locations for wear analysis (all five for concrete drilling, three for rebar drilling) (middle), concrete used and a centrally drilled 12 mm hole through a 16 mm rebar (right) [4]

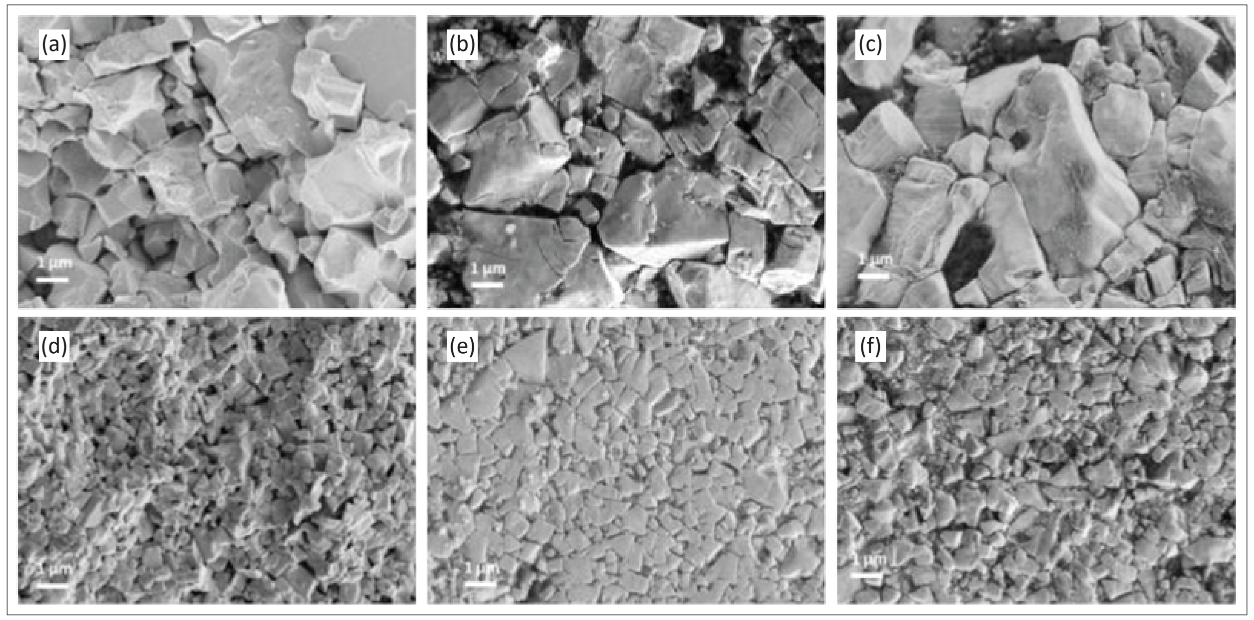

Fig. 13 Wear intensity assessment reference pictures. (a) Grade 6M, new (Co etched away to expose the WC grains) with fatigue/abrasion intensity '0' defined by no damage or wear to the WC, (b) Grade 6M after drilling, fatigue intensity '3', (c) Grade 6M after drilling, fatigue intensity '1', (d) Grade 12F, new (Co etched away to expose the WC grains) with fatigue/abrasion intensity '0', (e) Grade 12F after drilling, abrasion intensity '3', (f) Grade 12F after drilling, abrasion intensity '1.5' [4]

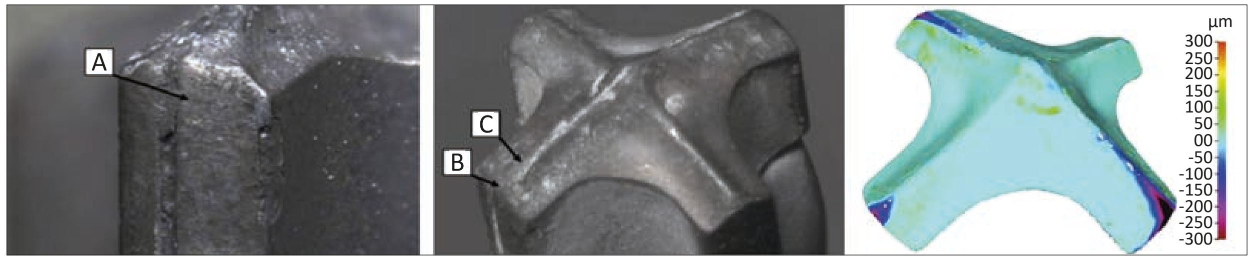

Fig. 14 Left and middle: Locations where wear mechanism analysis was performed (A, B and C, corresponding to 'Side', 'Edge 45°' and 'Axial Edge', respectively). Right: Comparison of surface topography of a used drill bit (3 holes in rebar) with the new condition, indicating height changes. (Note: the scanned drill bit was rotated clockwise through 90° compared to the middle picture) [4]

mechanisms were documented, but no qualitative or quantitative wear intensity assessment was made.

The authors firstly discussed the results obtained in concrete-only drilling. In rotary-percussive drilling of concrete, abrasive wear and fatigue-based wear are predominant. In the reported study, a subjective measurement of wear intensity was developed based on an evaluation of SEM images, providing a rating between 0 and 3 with 0 being no wear and 3 being high intensity wear. Fig. 13 demonstrates how this evaluation was performed, relating the degree of abrasive and fatigue-based wear observed on the WC grains and Co binder to arbitrary wear intensity values.

Testing of two hardmetal grades (6M and 12F) with all three combi-hammer parameters was performed by drilling to 1.6 m. A combination of fatigue-based and abrasive wear was observed at the five positions. The degree of fatigue-based wear at a given location on the drill bit was similar in all combinations of hardmetal grade and combi-hammer type. More fatigue was observed to occur at the tip region than at the gauge, largely due to the difference in peripheral speed (intensity 1.75 vs. 1.25). On the other hand, the degree of abrasive wear was more sensitive to the test variables (impact energy, rotational speed and hardmetal grade) and differed significantly depending on location on the bit. At the side, 12F exhibits a higher degree of abrasion than 6M (intensity 2.5 vs. 1.5).

In the drilling tests for 16.2 m with MP-1, all hardmetal grades exhibited the highest level of fatigue at the tip and the lowest level at the side (intensity 2.25 vs. 1.5). The result was, however, exactly opposite for abrasion where the highest level was observed at the side and the lowest level at the tip (intensity 2.25 vs. 1.25). The intensity of abrasive wear of the medium-grained WC hardmetal grades was slightly higher than the grades with extra-coarse WC. Again, this trend was reversed for fatigue, where the extra-coarse grained materials exhibited a marginally higher intensity of fatigue wear than the medium WC materials.

The wear surfaces of all of the hardmetal grades at all locations were examined, indicating that the wear mechanisms did not change significantly with drilling depth. For fatigue-dominated wear, cracking, plastic deformation and crushing of the WC grains were evident from the beginning to the end of the test. For abrasion-dominated wear, abrasion polished the surface, although some WC grain cracking, fragmentation and loss of WC grains were also evident. After the greater drilling distance, the loss of WC grains was more severe, partly because of the washing-out of the Co binder.

Hardmetal grades with higher binder contents (11E and 11M) exhibited gauge wear (i.e. diameter loss) at a significantly higher wear rate than grades with lower binder contents (6E and 6M). At a given binder content, the grades with extra-coarse WC wore faster than the grades with medium WC, with the effect of WC grain size being more pronounced at higher binder content. Greater wear of the cutting edges results in lower drilling performance and, therefore, more time per hole.

The results from tests of the drilling of reinforcement steel (rebar) were then discussed. These tests involved the drilling of three central holes, using the combi-hammer parameters, MP-1. Testing was performed manually, with total drilling time per operator not exceeding the maximum daily vibration exposure limits. Wear analysis was performed at three locations on the drill bit after three holes drilled, as shown in Fig. 14. A typical hole takes around 2-3 minutes, when the drill bit is new, and up to 10 minutes or more with a worn, blunted drill bit, where drill tip temperatures may consistently achieve values of 700°C in the hardmetal and 300°C many centimetres away from the cutting edge in the steel helix. Simulation estimates above 500°C even after only two consecutive impacts, whereas, in drilling a hole, many thousands of impacts are experienced by the drill bit. These thermal effects have a significant influence on the wear mechanisms, which differ considerably from those active during concrete drilling.

FEM-based simulations were performed, an example being shown in Fig. 15. These metal forming simulations estimate stresses and strains in tool and work-piece as well as the temperature distribution. Modelling always requires experimental verification and the example in Fig. 15 shows good correlation with the actual fracture events observed close to the cutting edge. Other, dynamic impact simulations have also been proven to accurately predict the locations of experimentally observed macroscopic fracture.

Even after only three holes in 16 mm rebar embedded in concrete at a depth of 5 cm, the wear was clearly evident. The 3D topography of the used drill bit was compared with that of its new condition. Positive deviations indicated a build-up of material on the hardmetal surface (concrete dust and steel chips) while negative deviations indicated material removal via wear or fracture events. Most wear occured in the edge (shoulder) region at the outer diameter of the primary cutting edge, with similar though lesser effects seen at the secondary cutter shoulder regions. Occasional micro-fractures were visible on the cutting edge and along the gauge region in the direction of rotation. The groove along the height of the side region (gauge) was partially filled with steel debris from the drilling process that had built up.

Underneath the build-up of steel on the surface, the wear mechanism was one of smooth wear of the WC grains. The exact mechanism is unclear at present, although an initial assessment indicates that it is abrasion possibly enhanced by chemical wear (dissolution of WC into the rebar).

At the worn, rounded shoulder regions, where high temperatures and contact pressures are generated, very different wear mechanisms and phenomena were observed. In this case, binder removal was evident to a depth of around 30-40 µm, presumably through a binder extrusion mechanism. The top few micrometres under the wear surface were completely devoid of binder, with nothing supporting the cracked WC grains. In this sub-surface region, the formerly smooth crystallographic facets of the WC were also roughened by an unexplained phenomenon.

Along the cutting edge close to this shoulder region (~1.5 mm away), other phenomena occurred. Spalling of the surface in a series

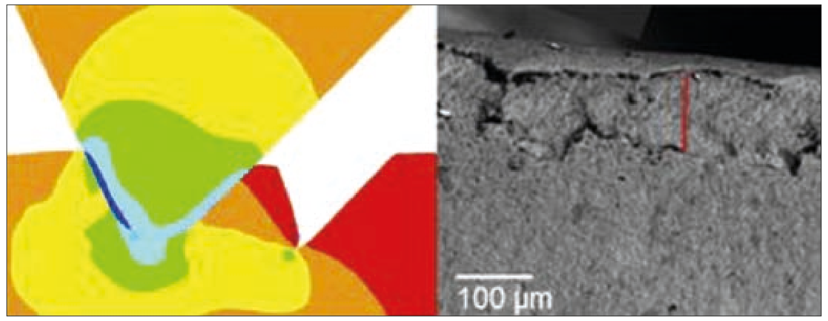

Fig. 15 FEM simulation of the compressive stresses at the drill bit cutting edge and within the rebar. Actual observed fracture events are shown in the right photograph, correlating very well with the region of high compressive stress (dark blue) close to the radius of the cutting edge [4]

of micro-fractures occurred, with these regions often observed to be filled with compacted, pulverised concrete dust, composed primarily of silica. A region extending approximately 10 µm below the surface had a very different microstructure to the bulk. Fractured WC grains were re-embedded into the surface along with drilling debris and the binder regions indicated that melting of the binder had occurred. The originally facetted, contiguous WC grains had become rounded and isolated from one another and a multi-phase solidification structure was evident. Chemical and XRD phase analysis is needed to identify the phases present. New evidence from wear analysis of rock drilling buttons has indicated that $CoSi_2$ forms at the surface when drilling rocks containing high amounts of SiO_2. This phase has a high melting point (1325°C) close to that of the Co binder (approx. 1300°C), so it seems likely that surface flash temperatures in excess of 1300°C are generated during rebar drilling. Further work is ongoing to clarify the phenomena observed.

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