VOL. 12 NO. 4
WINTER 2023

POWDER METALLURGY REVIEW

HARDMETALS AT EURO PM2023
IPERIONX: A PM ROUTE TO LOWER-COST TI
SINTERING IRON-COPPER-CARBON ALLOYS

Published by Inovar Communications Ltd


For a sustainable tomorrow

Publisher & Editorial Offices
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Subscriptions
PM Review is published on a quarterly basis. It is available as a free electronic publication or as a paid print subscription. The annual subscription charge is £150.00 including shipping.

Design and production
Inovar Communications Ltd.

ISSN: 2050-9693 (PRINT)
ISSN: 2050-9707 (ONLINE)

POWDER METALLURGY REVIEW

Metal powders and the green revolution

The Powder Metallurgy industry has considered itself 'green' for decades. Whilst many aspects of PM processing are energy intensive, the net shape (or near net shape) capabilities of press and sinter PM, Metal Injection Moulding (MIM) and Additive Manufacturing (AM) mean that very high levels of material utilisation offer a clear 'environment friendly' advantage.

Sustainability is, however, now rising up the agenda in the supply chain at an unprecedented speed, with every aspect of the PM, MIM and AM processes coming under renewed scrutiny.

The depth and intensity of the conversations around this topic at this year's Euro PM2023 Congress and Exhibition and, shortly after, Formnext 2023, highlighted that the implementation of green initiatives is now a top priority for many part makers and powder suppliers.

It is metal powder suppliers in particular who appear to have a real opportunity to differentiate themselves. We are in a market that is becoming ever more competitive as the CO_2 footprint of the powders used in PM, MIM and AM processes becomes a central part of component Life Cycle Assessment calculations.

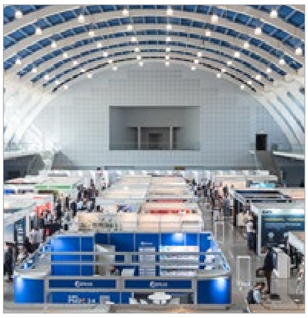
What are the energy costs associated with atomisation at a company? Is the energy source renewable? Is the starting material scrap or from a virgin source? These are just a few of the more obvious questions that are being asked.

In an age of high energy costs, there is, of course, a commercial upside to all of these developments. It will, however, be interesting to observe what pressures are placed on the PM supply chain, and how far we can go towards a truly green industry.

Nick Williams
Managing Director, *PM Review*

Cover image
Recycled titanium metal powders produced by IperionX from 100% scrap feedstock using Hydrogen Assisted Metallothermic Reduction (HAMR) technology (Courtesy IperionX)

POWDER METALLURGY REVIEW



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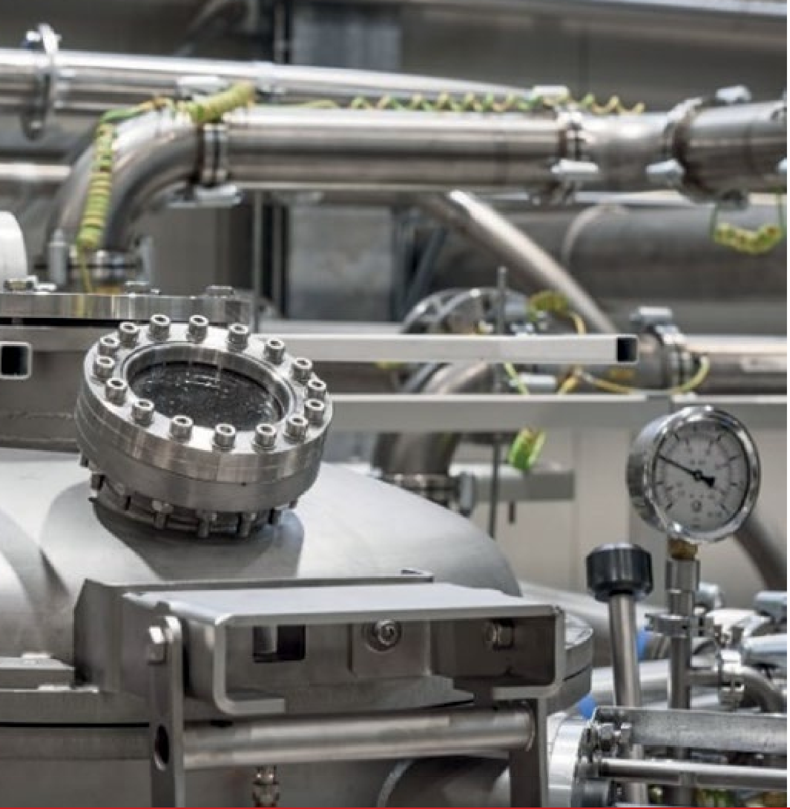

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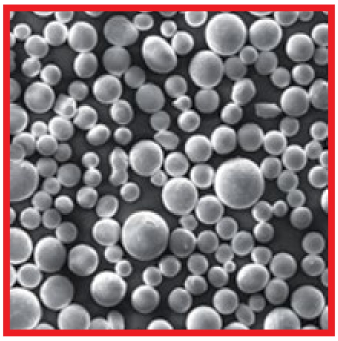

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industry news

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

ArcelorMittal to produce steel powders for Additive Manufacturing industry

ArcelorMittal SA, one of the world's largest steel companies, has announced it is building an industrial-scale atomiser at its facility in Aviles, Spain, to produce steel powders. It was added that a new company, ArcelorMittal Powders, has been established to commercialise its metal powders, targeting users of multiple Additive Manufacturing technologies.

The inert gas atomiser, which is expected to start production in January 2024, will have a batch-size production capability ranging from 200 kg to 3 tonnes, and an initial annual capacity of 1,000 tonnes. In line with ArcelorMittal's commitment to sustainability and decarbonisation, the atomiser will produce powders from scrap steel, using renewable electricity, atomising with industrial gases produced by renewable energy, and using recycled and recyclable packaging solutions.

The steel powders will be offered in size ranges suitable for all existing powder-based metal AM technologies. They can also be used in the latest technological developments such as the brake disc coatings being developed to help automotive OEMs and Tier Ones comply with the EU7 regulation on particle emissions. Here, a layer of powder deposited on the brake disc provides wear and corrosion resistance, significantly reducing the particulate emissions when braking, explains ArcelorMittal.

"Additive Manufacturing is an area we have been investing in and building our capabilities for several years, and we are now ready to scale up our production and offer our customers and partners a reliable and competitive source of high-quality steel powders," stated Colin Hautz, CEO of ArcelorMittal Powders. "From our facility in Spain, we will offer a range of steel powders tailored to our customers' needs. A technology as innovative and disruptive as Additive Manufacturing not only allows us to think about changes in the design and manufacturing process of many parts and components we use today, but also exploit one of the inherently sustainable characteristics of steel – its recyclability."

Marketed under the AdamIQ™ brand name, ArcelorMittal's product portfolio will include stainless steels (316L, 430L, 17-4PH), tool steels (H11, H13, M300) and low-alloy steels (a dual-phase alloy; 4140 equivalent). Drawing on its metallurgical experience, ArcelorMittal's research and development team, dedicated to AM technologies and steel powder production, intends to add further steel powder products for customers to test in 2024.

The company is now looking to scale up its participation in the AM market and intends to scale its steel powders offering in collaboration with customers and industrial partners, through co-design and co-engineering projects.

ArcelorMittal has been producing steel powders in a pilot atomiser at its AM lab in Aviles since 2018. With its dedicated research and development facilities and over fifty full-time researchers, ArcelorMittal reports it has developed a detailed understanding of the interactions between steel alloy design, atomisation parameters, AM process parameters and the final properties of the parts.

powders.arcelormittal.com

ArcelorMittal is building an industrial-scale metal powder atomiser in Spain to target multiple Additive Manufacturing technologies (Courtesy ArcelorMittal)

CNPC Powder raises $13.6 million for expansion of metal powder production

CNPC Powder, with its head office in Vancouver, Canada, reports that it has successfully raised nearly $13.6 million in Series A funding. The company plans to expand its atomisation capacity by adding over forty production lines. Its product portfolio includes titanium alloys, aluminium alloys, iron alloys, copper alloys, nickel alloys, and a wide array of high-temperature and custom alloys. Currently, the annual output from its China-based production facility surpasses 3,500 tons.

CNPC Powder's Series A funding round was led by Shunwei Capital and included participation from Dunhong Asset. Jupiter Capital provided long-term exclusive financial advisory and strategic support. As well as expanding production lines, the raised funds will be vital for advancing technical research and development and recruiting top talent, among other key initiatives.

CNPC plans to expand its atomisation capacity by adding over forty production lines (Courtesy CNPC Powder)

Since the inception of its AM Campus in 2017, CNPC Powder has been committed to the research and production of metal powder materials for advanced manufacturing. Its products, which the company states are supplied to over forty countries, primarily serve the metal Additive Manufacturing industry, but are also used in Powder Metallurgy, Metal Injection Moulding, electronic materials, and various other fields.

The company is certified to ISO9001, ISO41001, ISO45001, ISO13485, and IATF16949 standards, and states that these certifications demonstrate its technological expertise, which is shown through its proprietary process systems, capabilities in alloy research and development, production capacity, and control of powder size and sphericity.

www.cnpcpowder.com

6K Additive acquires Global Metal Powders to expand sustainable Ti and refractory powder production

6K Additive, a division of 6K Inc based in Andover, Massachusetts, USA, has acquired Global Metal Powders (GMP), New Castle, Pennsylvania, USA, for an undisclosed sum. The acquisition will lead to additional proprietary manufacturing and recycling capabilities, used in preparing material before 6K Additive's UniMelt® spheroidising powder production process.

Leveraging GMP's proprietary technology, 6K Additive will produce custom powders from a variety of revert streams across numerous metal alloys, including titanium, chromium, molybdenum, niobium, tantalum, and tungsten. Manufacturing will continue at GMP's facility in New Castle and, combined with the plans announced last year to double 6K's manufacturing capacity, the acquisition will further strengthen 6K Additive's standing as a provider of sustainable titanium and refractory metal powders.

"As the market for sustainably produced powder continues to grow, 6K Additive is the only one equipped to meet the manufacturing volumes demanded by customers for sustainably produced powder, specifically titanium and refractory metals," shared Frank Roberts, president of 6K Additive. "Adding the operational horsepower that GMP brings to our organisation allows us to not only keep pace with the high demand we are seeing for our powders but also further close the recycling loop, which leads to a lower overall part cost for our customers. We welcome the team at GMP to our organisation, and we are excited to add their technology and expertise to help advance our leadership position."

Henry Brougham, founder and Principal of GMP, added, "We are excited to be part of the 6K Additive team and look forward to helping expand their market-leading organisation with our proprietary technology. Combining both organisations' years of experience and knowledge, we will propel the company's processing and feedstock preparation to new heights. When you add our expertise in the refractory market, we are confident GMP will enhance 6K Additive's leadership position within this rapidly growing market."

www.6kinc.com/6k-additive
www.gmpowders.com

Sandvik to acquire US tungsten powder manufacturer Buffalo Tungsten

Sweden's Sandvik AB has announced it is to acquire Buffalo Tungsten, Inc. (BTI), a manufacturer of tungsten metal powder and tungsten carbide powder headquartered in Depew, New York, USA.

Sandvik will be able to produce tungsten powder in the USA (Courtesy Buffalo Tungsten)

The acquisition of BTI is set to expand Sandvik's presence in the North American market and strengthen its regional capabilities in the component manufacturing value chain. The integration of BTI provides the opportunity to optimise material sourcing and boost the local production of tungsten metal powder at its Depew facility. The acquisition will complement Sandvik's existing production of similar products at its Wolfram facility in St Martin, Austria.

"With the acquisition of BTI we take an important step in our strategic ambition to strengthen our presence in the North American market. BTI will enhance our regional capacity to produce tungsten powder locally in US, which will improve our competitive position," shared Stefan Widing, President and CEO of Sandvik.

BTI was founded in 1987 and currently employs forty-eight people. In 2022, the company generated approximately SEK 333 million in revenue. The impact on Sandvik's EBITA margin is anticipated to be minimal, but the earnings per share are expected to increase.

"With BTI we will be able to better meet the customer demand which will give us great opportunities in North America. BTI's contract for clean hydropower from the Niagara Power Project will also enable us to manufacture tungsten in a more sustainable way," added Nadine Crauwels, President of Sandvik Machining Solutions.

Both parties have agreed not to disclose the purchase price, with the transaction expected to close in Q4 2023. Once completed, Buffalo Tungsten will be reported within the business area segment Sandvik Machining Solutions.

www.home.sandvik
www.buffalotungsten.com

Elmet Technologies has acquired HC Starck Solutions Americas

Elmet Technologies, headquartered in Lewiston, Maine, USA, has acquired HC Starck Solutions Americas, a leading manufacturer and supplier of tungsten and molybdenum metals and related alloys. With over 100 years of experience in manufacturing refractory metals, HC Starck Solutions has facilities located in Coldwater, Michigan, and Euclid, Ohio.

Elmet is part of the Anania & Associates Investment Company LLC (AAI) portfolio, and the only fully-integrated, US-owned and -operated tungsten and molybdenum manufacturer. The combined business will offer a broadened portfolio for customers and result in a company with nearly 400 employees across its three facilities.

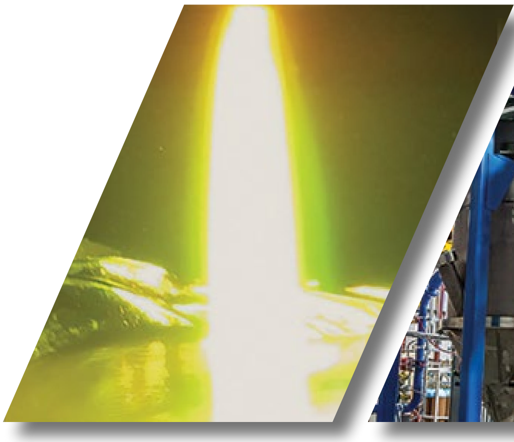
"Unifying our companies provides customers in defence, aerospace, medical, industrial furnaces, semiconductor, and other industries access to a single supplier with a more comprehensive portfolio of products and capabilities," stated Peter Anania, chairman at AAI and Elmet CEO.

"Our organisation now provides a combined 200 years of best practices for increased supply chain efficiency and resilience as well as improved quality, capability, and innovation. The consolidated portfolio now includes a breadth of capabilities and products from the largest extrusion press for refractory metals to fine wire thinner than a human hair. Combined rolling mill capabilities will increase overall capacity and provide shorter lead times to support the largest and most complex projects," added Anania.

The merged company will continue to produce and supply all its existing product lines, including foil, sheet, plate, rod, blocks, bars, powder, cubes, and spheres. There are plans to broaden this product range even further, through investment in both the workforce and manufacturing capabilities throughout the company.

"We also anticipate the development of new advanced materials, components, and solutions as a result of our increased capabilities and combined resources and expertise," said Scott Knoll, partner at AAI and Head of M&A and Strategy at Elmet. "To support that innovation, we have immediate plans to invest in expanding our workforce and manufacturing capabilities at all three of our US sites. These future investments will build on the success of recent investments across the group including state-of-the-art tungsten sphere and cube fragmentation production in Lewiston, spray dried and plasma densified Additive Manufacturing powders and 3D processing in Coldwater and new tungsten plate rolling technology in Euclid, Ohio. This combination will help ensure 'Made in the USA' continues in the tungsten and molybdenum sector for the next 100 years and beyond."

www.elmettechnologies.com
www.hcstarcksolutions.com

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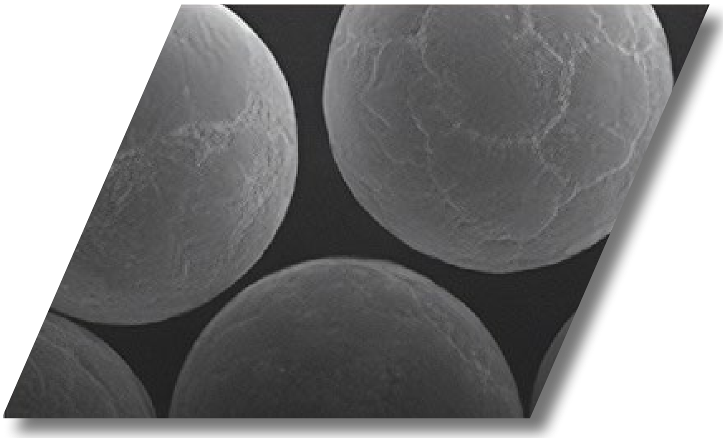

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KDF Fluid Treatment acquisition brings Kymera into the water treatment market

Kymera International, a speciality materials company headquartered in Raleigh, North Carolina, USA, has acquired KDF Fluid Treatment Inc, Three Rivers, Michigan, USA, a provider of sustainable media for water and industrial treatment systems. KDF's media consists of high purity copper-zinc formulations and is used in pretreatment, primary treatment, and industrial applications.

"Kymera's acquisition of KDF aligns with our strategic initiatives and positions us in a growing market segment, where our complementary product capabilities in brass powder media will support significant expansion," stated Barton White, Kymera CEO. "KDF's unique industrial and commercial water treatment systems provide supplemental or replacement technology to dramatically extend product cycle life, decrease maintenance, reduce heavy metals, microorganisms and scale, and lower total costs. Their technology, history and strong customer partnerships have secured them a prominent presence in the industry, and we are excited to add their capabilities to Kymera's portfolio, capitalising on growth and expansion into new markets."

Adam Shebitz, a partner at Palladium Equity Partners, added, "The momentum at Kymera is accelerating, as the company continues to expand its value-added capabilities through organic and inorganic growth. The acquisition of KDF is an ideal fit for Kymera as it represents a specialty materials solution with seamless vertical integration opportunities in a specialised and growing end-market."

Issa Al-Kharusy, CEO of KDF, stated, "We are thrilled to continue expanding the KDF technology. We believe that our businesses are a natural fit and we are confident that the partnership will result in exceptional service for our current and ever-growing customer base. Kymera's focus on specialty materials aligns with our unique offering and their track record of commercial and operational success positions KDF to support the growing water filtration market."

www.kymerainternational.com
www.kdfft.com

DSB adopts Desktop Metal's complete X-series metal Binder Jetting lineup

DSB Technologies, a manufacturer of Powder Metallurgy and Metal Injection Moulding components headquartered in Janesville, Wisconsin, USA, has adopted Desktop Metal's complete X-Series metal Binder Jetting (BJT) product lineup, including Live Sinter simulation and correction software. DSB currently uses Desktop Metal's InnoventX, X25Pro, and X160Pro machines to deliver customer parts made from a variety of metals, including 316L and 17-4PH stainless steels, 4140, and M2 Tool Steel. The company also intends to utilise Desktop Metal BJT technology for aluminium in the future.

"Binder Jetting really is a forming technology that gives us unlimited design potential," stated Paul Hauck, Chief Operating Officer at DSB Technologies. "We can go from a very simple shape to very complex things you can't produce in hard tooling, taking complexity beyond what's possible with Metal Injection Moulding. Binder Jetting creates applications never produced before, and we want to be a leader in that."

DSB is home to over thirty high-temperature continuous sintering furnaces, which is believed to be the largest installed capacity in North America. Out of the 3,630 tonnes of metal powder processed by DSB annually, approximately 90% are grades of stainless steel. Currently, DSB serves markets including aerospace, automotive, defence, electronics, industrial and medical.

"The exciting part about Binder Jetting is the path from concept to part is all digital," Hauck added. "You're not sending a CAD file over to a tool shop that then creates a reverse image. So, you're taking as few as eight weeks, and maybe as many as sixteen or twenty weeks, out of that process."

DSB has gradually implemented Desktop Metal's BJT technology over the past few years. The InnoventX lab-sized machine, first installed in 2021, is used for material development and testing initial sintering parameters. The X25Pro, installed in 2022, allows the team to scale those successful tests up to application development in a mid-size machine that is also capable of bridge production. The X160Pro, installed in 2023, offers the largest build volume for taking applications to serial production.

Hauck added that the Live Sinter software is highly effective in reducing iterations and saving time. "We now have very useful scientific analytical tools that enable successful outcomes. It's helping us solve application problems, get successful outcomes, and get there faster."

www.dsbtech.com

www.desktopmetal.com

Epson Atmix begins construction of its new metals recycling facility

Epson Atmix Corporation, a group company of Seiko Epson Corporation, located in Aomori, Japan, has announced it will invest almost $37 million (5.5 billion Yen) in a new facility to recycle metal waste in order to produce the raw material for metal powder production.

Epson Atmix has begun construction of its new recycling facility (Courtesy Epson Atmix)

The company aims to recycle unwanted metals from various sources, including out-of-specification metal powders used in manufacturing processes at Atmix, metal scraps generated within Atmix, and metal end cuts and used moulds discarded by the Epson Group.

The planned facility is also seen as a step towards achieving Epson's goal of becoming completely resource-free by 2050, as outlined in its Environmental Vision 2050.

Atmix produces a range of metal powders for a variety of manufacturing processes, including Metal Injection Moulding and Additive Manufacturing. The company also produces magnetic powders for use in power supply circuits, as coils for IT equipment such as smartphones, and for hybrid cars and electric vehicles.

The new factory will be equipped with a high-frequency induction furnace for melting metals, an AOD refining furnace for removing impurities from metals, and a casting machine for forming ingots.

A groundbreaking ceremony for the new facility was held on October 12 at the Hachinohe Kita Inter Industrial Park, with construction scheduled to start shortly after. The factory is expected to begin operations in June 2025.

www.atmix.co.jp

IperionX receives US DoD grant and finance offer to grow its titanium powder production

IperionX Limited, based in Charlotte, North Carolina, USA, reports it has been awarded a $12.7 million grant from the United States Department of Defense (DoD). The company also announced in a further press release it had received a Letter of Interest (LOI) from the Export-Import Bank of the United States (EXIM) for a provisional finance sum of $11.5 million.

$12.7 million DoD grant to boost titanium powder production for US defence supply chains

Facilitated through the Defense Production Act Investment (DPAI) Program, the $12.7 million award aims to increase titanium powder production for defence supply chains.

"Robust and resilient defense supply chains are critical to the Warfighting capability of the United States," stated Dr Laura Taylor-Kale, Assistant Secretary of Defense for Industrial Base Policy. "Domestic titanium production is a top priority for the DoD's industrial base programs."

The award will fund the expansion of IperionX's facility in Virginia into a demonstration plant, boosting the company's titanium powder production to 125 metric tons annually. Within five years, IperionX aims to produce 10,000 metric tons of titanium metal powder each year. The company manufactures titanium alloys from either titanium minerals or 100% recycled materials.

As of 2023, the DPAI Program has granted twenty-one awards totalling $674 million. DPAI is overseen by the ASD(IBP)'s Manufacturing Capability Expansion and Investment Program (MCEIP), in the Office of the Deputy Assistant Secretary of Defense for Industrial Base Resilience.

$11.5 million finance from EXIM Bank for US titanium production

IperionX received notification from EXIM that its proposed titanium facility may qualify for equipment finance under two programmes: EXIM's 'China and Transformational Exports Program' and 'Make More in America Initiative'. These programmes allow EXIM to extend its medium and long-term loan and loan guarantee programmes to support projects that aim to reduce Chinese dominance in strategic sectors and promote export-oriented domestic projects.

"Titanium for the US manufacturing sector is currently sourced over long distances from foreign nations," stated Anastasios (Taso) Arima, IperionX CEO. "We are pleased to receive the letter of interest for the provisional sum of $11.5 million in equipment finance from US EXIM that will assist IperionX to re-shore a lower-cost, more sustainable and fully integrated US titanium supply chain that is critical both for the manufacturing of advanced goods as well as for America's national security."

The company is looking to acquire key production assets, such as industrial furnaces and comminution equipment, as it develops its titanium production facility in Halifax County, Virginia. While the LOI is non-binding and conditional, IperionX stated that the potential funding support gives a solid foundation as the company explores various competitive funding options.

www.defense.gov
www.iperionx.com

GKN Powder Metallurgy and Schaeffler partner to drive permanent magnet supply chain

GKN Powder Metallurgy has announced that it will supply Schaeffler AG, a motion technology company based in Herzogenaurach, Germany, with permanent magnets. The companies have entered into a Memorandum of Understanding (MoU), committing to advancing the development of the permanent magnet industry for electric motors in Europe and North America, with the objective of ensuring a stable, local supply chain.

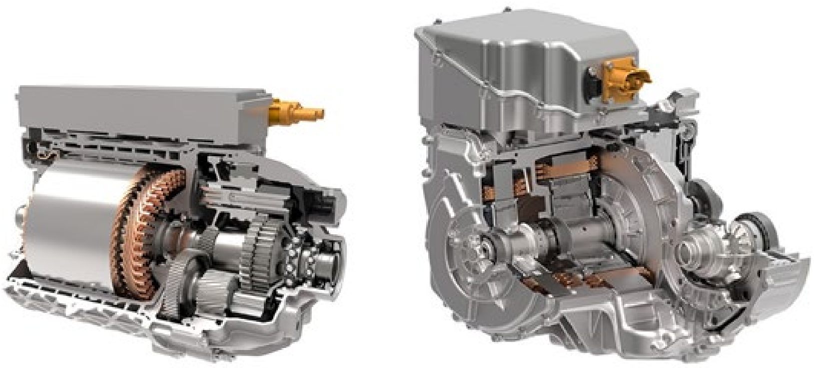
The cooperation is said to reinforce the over-arching sustainability strategies of both companies, and will provide customers with additional security of supply through a local-to-local delivery strategy.

"We are delighted to partner with Schaeffler to jointly provide solutions to the challenges of supplying permanent magnets to the automotive and other industries," stated Diego Laurent, CEO of GKN Powder Metallurgy. "This is a perfect match of two companies with a long-standing relationship and a strong commitment to sustainability and innovation. The development of permanent magnets for EVs is a logical step for GKN Powder Metallurgy, as we continue to expand our capabilities to support the needs for electrification in the industry whilst pursuing ambitious sustainability targets."

In addition to a wide range of components and systems for drivetrains based on internal combustion engines, Schaeffler also offers solutions for hybrid and pure electric vehicles, from 48-Volt hybrids and plug-in hybrids to purely electric vehicles.

Matthias Zink, CEO Automotive Technologies at Schaeffler, added, "Schaeffler has key strengths in passenger car electrification, particularly in electric motors, which are integral components of all electric axles and hybrid modules. To ensure our successful growth in this segment especially in Europe and North America we aim to establish resilient, local, and sustainable supply chains for the relevant components like permanent magnets. This cooperation with GKN Powder Metallurgy is an important step towards this goal."

www.gknpm.com
www.schaeffler.com



Schaeffler's solutions for hybrid and electrified systems include this electric axle system (left) and hybrid transmission (right) (Courtesy Schaeffler)

Kennametal releases report highlighting environmental, social and governance in 2023

Kennametal Inc, headquartered in Pittsburgh, Pennsylvania, USA, has published its 2023 Environmental, Social and Governance (ESG) report, highlighting in detail the progress made over the past year.

"Our ESG report reflects our commitment to accountability and transparency to key stakeholders and details the progress we have made over the last twelve months," said Christopher Rossi, president & CEO. "I am proud of the work our employees have demonstrated in executing our ESG strategy globally, and I look forward to continuing our ESG journey."

The report highlights reductions in Scope 1 and Scope 2 greenhouse gas emissions, energy consumption, water consumption and increase in waste recycling. It also reports on the enhancements to the EHS Management System in order to streamline the collection and analysis of key metrics which help improve safety programmes.

Kennametal's recordable incident rate is said to continue at a world-class level, around 148% better than the US industry standard. There are also advancements in the development and engagement of employees reported with initiatives to advance diversity and inclusion.

Topics included in the ESG assessment are reported to align with GRI Standards Topics, the SASB Industrial Machinery & Goods 2018 Sustainability Accounting Standards and other ESG topics of interest to investors and other key stakeholders. Kennametal also recognises the importance of the United Nation's Sustainability Development Goals (SDG) and has aligned the SDGs with priority topics.

www.kennametal.com

Global aerospace company orders coarse cut Ti powder from PyroGenesis

PyroGenesis Canada Inc., Montreal, Quebec, Canada, has received an order from an unnamed global aerospace firm for plasma atomised titanium metal powders for Additive Manufacturing. Intended for one of the client's ongoing internal R&D programmes, the order is for coarse cut metal powder in the range of 45-150 µm, a size typically seen as a by-product of fine powder production.

The client is the large global aerospace original equipment manufacturer (OEM) in the United States with whom the company has previously disclosed its ongoing qualification process. The order was produced using the Company's NexGen™ plasma atomisation system.

"The significance of this order is the particle size distribution (PSD) that has been requested," stated Mr P Peter Pascali, CEO and president of PyroGenesis. "The PSD for this order is for our coarse cut titanium powder, in this case for powder between 45-150 µm (microns), which we have been producing over the past year as we build our stock inventory. This contract recognises what we believe is the superior overall quality of our powder and establishes a market for a larger percentage of our powder output, thereby improving our overall returns substantially."

Massimo Dattilo, VP PyroGenesis Additive, added, "With some traditional metal powder production processes, as the powder is created then filtered and separated into different cut sizes, the fine cut powder is removed for sale, with the remaining coarsest cut often considered of limited use, or even discarded as unsuitable. However, with PyroGenesis Additive's NexGen plasma atomisation system, the coarsest cut component of the production batch remains of such a high quality, that we store these powders as inventory for future sales. By selling both the fine and coarse cut of each powder production run, the company's yield percentage from raw material is greatly enhanced, which is in line with the company's broader mandate for commodity security and optimisation."

PyroGenesis' development of titanium metal powders is part of its three-tiered solution ecosystem that aligns with economic drivers that are key to global heavy industry. Metal powders are part of the company's Commodity Security & Optimisation tier, where the recovery of viable metals, and the optimisation of production to increase output, helps to maximise raw materials and improve the availability of critical minerals.

"Over the past several years, we designed, then readied, our new NexGen plasma atomisation process with a goal to produce the highest quality metal powders in the Additive Manufacturing industry," added Pascali. "PyroGenesis Additive has taken a cautious, methodical approach to its new production process. Now, with the full-scale commercialisation underway, and a steady influx of initial orders, the long-term conservative strategy that has brought us to this point today is bearing fruit."

In the Autumn 2023 issue of *PM Review* magazine, available to read for free online, we reported on the company's history in metal powders, its vision for the future, and how a combination of the rise of Additive Manufacturing, a drive towards decarbonisation by heavy industry, and concerns around commodity security have fundamentally changed the landscape for metal powder producers.

www.pyrogenesis.com

Nitrex and Linde expand international marketing agreement for heat-treating services

Nitrex, based in Montreal, Canada, and Linde, headquartered in Woking, United Kingdom, have renewed and expanded their joint marketing agreement focused on heat-treating. What began as a local agreement between Nitrex and Linda (formerly known as UPC-Marathon and Praxair, respectively) some thirteen years ago, has now grown into an international marketing agreement that covers Europe and North America.

To date, Nitrex and Linde have collaborated on more than thirty projects. By leveraging their complementary offerings, they have upgraded crucial equipment and assisted customers in achieving high-quality results. Nitrex offers Linde customers equipment and analyses to regulate gas atmospheres. This is made possible by Nitrex's experience in the heat treatment and electrical fields, as well as its technical solutions, support, and gas panels.

"Our competencies complement each other," shared Roman Grosman, National Director of Business Development for Linde in the US. "In the event that Linde's heat treatment clients require equipment that we do not offer, Nitrex can meet this need."

Paul Oleszkiewicz, president, CPO & CSO of UPC-Marathon, a Nitrex company, stated, "This continues to be a win-win relationship. We can supply Linde gas customers with process controls, and in turn, Linde offers a reliable gas supply network. We are both aiming for the highest quality, efficiency, performance, and a greener tomorrow and providing optimal service for our customers."

www.linde.com
www.nitrex.com

Ceratizit acquires US aerospace solid carbide tool maker Xceliron

Ceratizit USA, a subsidiary of the Plansee Group based in Schaumburg, Illinois, USA, has announced the acquisition of all shares of Xceliron Corp, located in Chatsworth, California. Xceliron specialises in manufacturing special solid carbide tools for the aerospace and automotive industries in the United States.

"With its high-quality specialty products, the Xceliron portfolio is an ideal complement to the standard products from our Sacramento site and an important building block for our global growth strategy," stated Mirko Merlo, President – Americas at Ceratizit.

This acquisition is expected to provide Ceratizit with the opportunity to tap into new customer groups and opportunities within the aerospace market, whilst also providing its North American customers with a more comprehensive range of tooling solutions.

The integration will reportedly be actively supported by the company's founders. "We are very pleased that Randy Jones and Ric DiOrio will continue in the successful management of Xceliron and act as Co-Managing Directors," commented Andreas Lackner, Executive Board Spokesman, Ceratizit.

In a joint statement, DiOrio and Jones shared, "We are thrilled to have found the right partner in Ceratizit to take Xceliron to the next level and build on our heritage. Long-term thinking and creativity are two of the values that have also been at the core of our business over the past thirty-three years."

Xceliron has over thirty-three years of history as a precision cutting tool manufacturer and solution provider, offering solutions for difficult-to-machine parts in the aerospace industry, as well as commercial and automotive sectors.

"This acquisition strategically positions us to better serve our customers with an expanded range of custom tooling solutions, specifically tailored to meet the demanding requirements of the aerospace industry. We are eager to leverage Xceliron's expertise and reputation for precision to drive innovation and excellence in round tools," Merlo added.

Both sides have agreed not to disclose the financial details of the transaction.

www.cuttingtools.ceratizit.com
www.xceliron.com

GKN Powder Metallurgy reveals largest ever order for sintered metal gears used in EV differentials

GKN Powder Metallurgy has reported a 'satisfactory' first half of 2023, with improving trends throughout the period and adjusted revenues of £545 million, some 2% higher than 2022 on a constant currency basis. This reflected 1% lower year-on-year volumes, which were reportedly impacted by the accelerating EV transition, operational issues in the US, exiting poor margin business, and the closure of a facility in 2022. However, these were reportedly more than offset by price increases to recover from inflation.

In the period, adjusted operating profit was £50 million, with a margin of 9.2%, compared to 10.5% for the same period in 2022. The decline in margin is attributed, in part, to a positive one-off cost related to equipment downtime at a US plant, which has now been resolved.

From a commercial perspective, GKN Powder Metallurgy had a successful first half of the year, securing new business wins and forecasting a 36% year-on-year increase in peak annual revenues. These wins were valued at approximately 75% of propulsion-agnostic product groups, a significant increase from the prior year. This confirms that the new products developed over recent years are gaining commercial traction.

New contracts for EV products & permanent magnets

GKN reports it has made significant progress in transitioning to its EV portfolio. It has secured new contracts to supply several EV-specific products, including iron powder for use in lithium ferrophosphate (LFP) batteries and sintered metal gears for differentials used in EVs. The differential gears award represents the single largest order ever won by GKN Powder Metallurgy, with production due to commence in 2025.

During 2023, Powder Metallurgy gained momentum in the development of permanent magnets for electric motors. The company achieved a significant milestone during this period by reaching its first commercial agreement for the production of magnets with a global Tier 1 automotive customer. As a key component of its strategy to manage the transition to electric vehicles, Powder Metallurgy's growth in magnets continues to attract significant interest from multiple customers, including both OEMs and tier suppliers, who wish to de-risk their supply chains for these critical components. The company is currently in commercial negotiations and advanced technical qualifications with several other potential customers.

The company has established innovation centres for permanent magnets in both Europe and North America, with both facilities currently producing sample quantities of magnets for customers.

www.gknpm.com

Bodycote acquires Stack Metallurgical Group and Lake City Heat Treating in $145 million deal

Bodycote plc, headquartered in Macclesfield, Cheshire, UK, has announced the acquisition of two US-based heat treatment and Hot Isostatic Pressing (HIP) focused businesses for $145 million, along with plans for the opening of a new HIP facility in Southern California.

The companies purchased are Lake City Heat Treating, based in Warsaw, Indiana, and Stack Metallurgical Group headquartered in Portland, Oregon. Lake City HT, is reported to be a leading medical market HIP and vacuum heat treatment business primarily supplying the orthopaedic implant market as well as civil aerospace customers. Stack Metallurgical Group is a key provider of HIP, heat treatment and metal finishing services primarily for the civil aerospace, defence and energy markets.

Bodycote has purchased Stack Metallurgical Group (above) and Lake City HT as part of its expansion plans in the US (Courtesy Lake City HT)

The two businesses are said to be highly complementary to Bodycote's existing operations and will both expand its geographic footprint in North America and provide additional customer reach.

"These investments are an important and exciting enabler of our strategy to further enhance and grow our Specialist Technologies businesses," stated Stephen Harris, Group Chief Executive of Bodycote plc. "In addition, they will also expand our footprint in aerospace and medical heat treatment on the West Coast and in Indiana in the US. The acquisitions will enhance group margins, are accretive to earnings per share and allow us to further capitalise on the structural growth opportunities in the space, civil aerospace and medical markets."

New HIP facility in greater Los Angeles

Additionally, Bodycote announced plans to open a new HIP plant utilising one of the group's existing sites in greater Los Angeles, California. This capacity is expected to become operational during 2024 and will support the rapid growth in space and civil aviation markets in the Los Angeles area.

Harris added, "The proposed new HIP plant in greater Los Angeles will allow Bodycote to take advantage of the burgeoning HIP market in space and civil aerospace in the region. It will require only modest investment as it utilises an existing Bodycote site and existing HIP vessels that are immediately available for installation."

"Optimal allocation of capital to drive shareholder value remains a top priority for the Group and these investments reflect this," he concluded.

www.stackmet.com
www.lakecityheattreating.com
www.bodycote.com

Equispheres materials recognised as enabling technology by US Army

Equispheres, Inc, Ottawa, Ontario, Canada, reports it has been identified by the US Army as a key enabler to fill critical manufacturing and sustainment capabilities for the Department of Defense.

Equispheres is one of the five winners in the annual xTechInternational competition seeking advanced manufacturing and materials solutions. This event is intended to recognise innovations with immediate value for American industries.

"We are honoured that DOD has recognised the capabilities of our materials technology for defence applications. Our unique production technology allows us to tailor materials properties to specific end-uses, making metal Additive Manufacturing faster, safer, and more accessible to the defence supply chain," said Kevin Nicholds, CEO of Equispheres.

Equispheres was one of a handful of global suppliers chosen from a pool of over 130 candidates as having technology with the potential to significantly impact DoD's near-term and future capabilities.

"US Army collaboration with our international allies and partners is essential to developing state-of-the-art capabilities that benefit the US soldier," added Jessica Stillman, the deputy programme manager for the Army xTech Program. "The latest xTechInternational competition focused on advanced manufacturing and materials and had the largest award amount to date for this series, leading to record participation. We see this trend continuing as we move forward to future international competitions."

Evan Butler-Jones, VP – Product & Strategy for Equispheres, says the recognition is "an excellent opportunity to support the needs of the US DOD for advancing metal Additive Manufacturing, and important recognition of the role that materials technology can play in accelerating its adoption within North America."

www.equispheres.com

Carbon footprint of Höganäs AB's Somaloy 5P SMC powder verified

Sweden's Höganäs AB reports that its Somaloy 5P has become the company's first metal powder to undergo a carbon footprint assessment by an independent third party. The qualification is said to be in accordance with the ISO 14067:2018 standard for measuring the carbon footprint of products.

"The external verification assures Höganäs' credibility when it comes to providing carbon footprint studies for specific products," stated Sofia Poulikidou, LCA specialist at Group Sustainability in Höganäs.

Somaloy is a Soft Magnetic Composite powder, said to offer unique 3D flux properties and developed for component manufacturing of electromagnetic applications, providing high performance and low losses.

Höganäs is now actively working to provide carbon footprint studies for a wide range of products. "This will support our customers in their sustainability and climate ambitions," added Poulikidou. "Customers can use the results to further improve product and production development processes to decrease their climate impact."

www.hoganas.com

VBN Components offers high-performance materials development as a service

VBN Components, Uppsala, Sweden, has introduced its High-Performance Materials Development as a Service at this year's Formnext. The new option enables customers to explore the development of traditionally-challenging materials such as high-carbon steels, hard metals, or refractories such as tungsten or niobium.

"We frequently receive requests from clients for custom materials development," stated Magnus Bergman, CEO of VBN Components. "We believe the time is right to showcase our expertise in this domain to the world and are thrilled to now offer this as a dedicated service. Our participation at Formnext 2023 marks a significant step towards fulfilling the evolving needs of the industry."

Led by material experts, the service will leverage cutting-edge Additive Manufacturing technologies to develop materials with exceptional wear resistance and unique capabilities.

"Our high-performance materials development service is designed to push the boundaries of what's possible in metal Additive Manufacturing, contributing to enhanced performance and longer lifespan of components," added Ulrik Beste, PhD, CTO and co-founder of VBN Components.

www.vbncomponents.se

Globus Metal Powders launched following acquisition of Liberty Powder Metals

Liberty Powder Metals Ltd, based in Middlesbrough, UK, has been acquired by a group of private investors and, following the takeover, has now been renamed Globus Metal Powders Ltd. The company produces a range of metal powders for both Additive Manufacturing and Powder Metallurgy technologies.

"Globus Metal Powders and our team of professionals will continue to drive excellence and growth, we are very proud of the successes achieved and are excited about the new opportunities the acquisition will offer in terms of growth, productivity, and profitability," the company stated.

As the company's new website is still in progress, Global Metal Powders asks those interested to contact Becky Ambrazaitis, Sales Manager, for any further information.

Becky.a@globusmetalpowders.com

Velta plans fully-integrated titanium operation with production of titanium medical implants

Velta plans to start commercial production of finished titanium products (Courtesy Velta)

Velta LLC, located in Dnipro, Ukraine, plans to start commercial production and distribution of finished titanium medical implants by the end of the year. The implants will be additively manufactured and made using Velta's titanium powder, resulting in a fully integrated commercial operation encompassing titanium ore mining, powder processing, and part manufacturing.

"This enormous milestone is the result of many years of intensive work behind the scenes to scale our powder technology at a pace that the global titanium market demands," Velta CEO Andriy Brodsky stated. "By bringing these titanium medical implants to market, we've proven our concept and are eager to stake out our position as the first and leading integrated titanium operation."

In 2017, Velta launched a research and development centre with the aim of innovating a new method for producing titanium powder as an alternative to titanium sponge. This patented approach to titanium powder is significantly faster and more environmentally responsible than existing titanium sponge. As a result, the company has successfully scaled up semi-industrial production of metallic titanium powder at its R&D Centre in Dnipro.

Additionally, Velta has established two new divisions to enhance its vertical integration efforts and strengthen its market position. These divisions are Velta Additive Technology, which specialises in producing finished titanium products using Additive Manufacturing techniques, and Velta Medical, which focuses on manufacturing custom titanium medical implants in Ukraine.

As the company continues to expand its commercial and R&D operations in Ukraine, it is currently undergoing a thorough site selection process to establish a state-of-the-art titanium powder production facility in the United States. Last month, Velta announced its partnership with global engineering consultancy Hatch to develop the new facility, which will initially have an annual production capacity of 1,000 tonnes of titanium powder.

velta.us

6K Additive and Metal Powder Works partner to produce pure copper, copper alloys and bronze AM powder

6K Additive, a division of 6K Inc headquartered in Andover, Massachusetts, USA, and Metal Powder Works (MPW), based in Clinton, Pennsylvania, USA, announced at Formnext the signing of a Memorandum of Understanding and strategic partnership to produce pure copper and copper alloys, leading to strategically important powders such as copper nickel, and bronze alloy powders for Additive Manufacturing.

From left, Nick Pflugh, 6K Chief Commercial Officer; John Barnes, MPW founder & CEO, and Frank Roberts, 6K President (Courtesy Metal Powder Works)

The high-yield production achievable with both Metal Powder Works' DirectPowder™ Process and 6K Additive's UniMelt® microwave plasma is expected to bring economical advantages, faster time to market, and sustainable production from both feedstock creation and the production of these materials.

"The market for copper is eager for a scalable solution that can not only deliver high-quality material, but one that can bring simplicity to the supply chain with a very sustainable process," stated Frank Roberts, president of 6K Additive.

"The synergies between our two organisations ensures customers can source their material sustainably, reliably and with the quality that meets their stringent specifications."

Metal Powder Works CEO and founder John Barnes added, "This partnership is a win for both 6K Additive and Metal Powder Works, but most importantly for our customers who are looking to utilise copper for a variety of applications such as heat sinks, battery components, particularly for the rapidly growing EV market, and critical parts for the marine industry. This alliance with 6K opens up MPW's technology to provide more materials to the AM market."

MPW's DirectPowder process can reportedly produce powders tailored to the manufacturing process through its patented process that provides consistent powder size and shape. Its current powder portfolio includes high-strength aluminium and highly conductive copper powders. The company stated that new alloys are actively in development through its recently-launched MPW Developer Network.

Barnes told *PM Review* magazine that the partnership will both support the production of hard-to-atomise materials as well as offer a faster path to increasing the availability in powder. There are more than 2,000 approved AMS specifications for barstock, as opposed to sixteen AMS specifications for powder.

6K Additive produces its AM powder from sustainable sources and offers a full suite of premium powder, including nickel, titanium, copper, stainless steel, aluminium alloys, and refractory metals such as tungsten, niobium, and rhenium. The UniMelt is a microwave production-scale plasma system reported to offer a highly uniform and precise plasma zone, zero contamination and high throughput production capabilities.

www.metalpowderworks.com
www.6kinc.com/6k-additive

MPIF names Michael Stucky as its new president

The Metal Powder Industries Federation (MPIF) reports it has elected Michael Stucky as the trade association's 32nd president. Stucky's two-year term began upon the conclusion of the MPIF's annual Business Meeting, held on October 30, 2023.

Stucky, currently Business Unit Director at Norwood Medical, Bellbrook, Ohio, has been with Norwood for the past eleven years. Previously, he worked another eleven years at NetShape Technologies and ten at PCC Airfoils.

Being very active within the MPIF and the Metal Injection Molding Association, Stucky has served on the MPIF Board of Governors as MIMA President. He is currently chairman of the MIMA Standards Committee, a MIMA representative on the MPIF Technical Board and was a co-chair of the PowderMet2023, MIM2017 and MIM2018 conferences. Over the years he also served on the PowderMet and AMPM Technical Program Committees. Stucky received the Distinguished Service to Powder Metallurgy Award in 2019.

The MPIF also announced that Timothy Hackett has also been named president of the Metal Powder Producers Association (MPPA) and Stefan Joens as president of the Metal Injection Molding Association (MIMA). Nicola Gismondi and Christopher Adam, have retained their positions as a presidents of Powder Metallurgy Parts Association (PMPA) and Association for Metal Additive Manufacturing (AMAM), respectively.

www.mpif.org

SMS group

Integrated Lifecycle Partnership

To be the leading partner for our customers in this transformation, SMS group bundles all competencies from electrics/automation, digitalization, and technical service. Our goal is to maintain and expand the performance of our customers' plants throughout their entire lifecycle. Together with our customers, we develop integrated solutions specifically geared to the customer's use case. In doing so, we focus on crucial KPIs such as plant availability, product quality, productivity, or delivery reliability but also on increasingly relevant topics such as sustainability and safety.

Additive Manufacturing drives spherical powder development at PSI

PSI Ltd, based in Hailsham, UK, is probably best known for the design, manufacture and installation of gas atomisers to make spherical powder for use in metal AM and other Powder Metallurgy applications. Atomisers supplied range from small laboratory ones, processing a few kilograms, up to large, sophisticated plant delivering multi-thousand tonnes per year.

What is less well known, however, is that PSI is also active in designing plant for a diverse range of other processing technologies. In the field of advanced engineering materials, these are often complementary to its PM activities. These technologies include Chemical Vapour Deposition (CVD) used to coat powders, Electron Beam Physical Deposition (EBPVD) to coat aerospace turbine hot section components, melt spinning and strip casting to make high performance (FeNdB) magnetic powders and several other processing technologies. These all relate to gas atomisation in that they have rapid solidification science in common, generating high performing material microstructures.

For the R&D team at PSI, metal Additive Manufacturing has, over the past five to ten years, thrown up a whole series of interesting and unique challenges. PSI has developed process equipment with specific advantages to the AM industry.

Fluidised Bed Reactors (FBR)

PSI has been working in fluidised bed technology for several years in the processing of metal and ceramic powders. In very simple terms an FBR is a container of powder, generally housed in a pressure or vacuum containment vessel. At the base of the container is a porous plate through which a whole variety of heated gases may be passed, inert or reactive in nature. The gas flow energises the powder (fluidises) so it appears to the naked eye as a pot of boiling liquid.

Heat treatment of metal powders and FBRs is not new but, in combination, offer a range of intriguing opportunities to produce powders with sophisticated properties targeted at the advantages and challenges that metal AM presents. These include the use of coatings that can modify the behaviour of powders when being introduced to the build zone, during melting and the mechanical, electrical and magnetic properties of the finished part. Another use is for the heat treatment of powders to modify their rheological properties to suit a range of different AM powder flow requirements.

The recycling of AM powders is another advantage of FBR. Regardless of the particular AM technique employed, powder not incorporated in the build may become oxidised or experience modified flow properties. FBR's can be used to regenerate such powders.

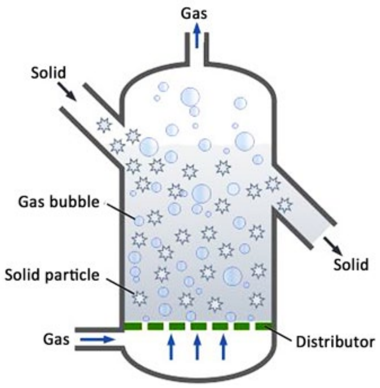
PSI has also used its in-house FBR to process aluminium powders for a non-AM process, namely Cold Spray, where a novel alloy composition and heat treatment resulted in enhanced properties in the coatings. Here, the powders were extracted from the FBR and subjected to a cold gas quench, thus retaining the soft, solution heat treated structure. The advantage of FBRs in contrast to other powder modification processes is that the fluidising action gives a very uniform process environment in terms of temperature and process gas distribution. Furthermore the process can be scaled up to tonnage quantities and can be operated on either a batch or continuous basis.

Gas classification

Generally, after atomisation, metal powders are graded into the familiar size fractions depending on the requirements of each particular AM technique, whether it be PBF-LB, typically at 15–45 µm or 15–60 µm or coarser cuts for PBF-EB use.

Sieving at the finer cuts (in the size range 10–25 µm) can be particularly problematic. It is true that advances in sieve technology have

A fluidised bed reactor at PSI's facility in Hailsham (Courtesy PSI)



A fluidised bed reactor is a container of powder housed in a pressure or vacuum containment vessel (Courtesy PSI) occurred over past years leading to shorter process times and reduced incidence of sieve 'blinding'. However, the problem still essentially remains. A solution exists in the use of gas classifiers to remove the undesirable fines in a powder distribution whilst retaining a sieving operation at the coarser cuts where sieving rates are much faster.

Gas classifiers are cyclone-like devices which are a standard method of separating a powder distribution into coarse and fine fractions. However, simple cyclones are not capable of precisely dividing atomised metal powder into the desired fractions and rejecting oversize and undersize. Gas classifiers generally have a slotted rotor inside a cyclone body which rotates at high speed and greatly enhances the sharpness of cut and, therefore, improves the economics of production and allows powder producers to meet the exacting specifications of those using the powder.

Such classifiers are generally fed with very large volumes of air to suspend and transport the powder. This has no cost consequence if a powder is not sensitive to oxidisation in the air flow. However, when reactive and possibly explosive powders are required to be processed, argon is the process gas of choice. Here, the cost can become prohibitive.

To solve this, PSI has extended its range of gas classifiers from lab scale to those handling hundreds of kilograms per hour under inert gas. Operating in a highly automated way, these efficiently recycle the argon, limiting the cost to just the initial charging of the system.

External melt delivery

As with most manufacturing processes, when efficient use of feedstock within the process is approaching optimum, the focus turns to reducing the input cost of metal.

PSI has introduced the Contipour atomising process whereby two furnaces alternatively feed molten metal to a central holding tundish that supplies the atomising zone. Rather than using the pouring furnaces to melt the desired alloy, those furnaces merely act as holding devices into which metal from an external source is fed. These are adjacent to, but operationally separate from, the atomisation unit which then operates on a continuous basis. Thus when one furnace is empty it is removed and refilled while the other furnace continues to top up the tundish.

This technology is particularly attractive to operators of large melting equipment, such as steel electric arc furnaces, where existing use of melt is currently directed towards other processes such as casting. Here, having already invested in the melting plant, the operator may wish to divert part of the output to the relatively high value adding operation of metal powder production.

Analysis of the CAPEX and OPEX components of powder cost when liquid metal is introduced in this way shows a disruptive reduction in those costs.

www.psiltd.co.uk

Ultra Fine Specialty Products announces line of metal powders specifically for Additive Manufacturing

Ultra Fine Specialty Products, LLC, an affiliate of Novamet Specialty Products Corporation based in Woonsocket, Rhode Island, USA, has announced a new line of metal powders developed specifically for Additive Manufacturing.

With vacuum melt and inert gas atomisation at its purpose-built facility, Ultra Fine has been producing fine, highly spherical metal powders since 1990. These have been widely used by the largest and most successful Metal Injection Moulding companies in the world.

"We are excited to announce that we are now introducing a new line of metal powder products based on our research and development of powders specifically for AM," stated Jeffrey Peterson, president and CEO of Ultra Fine and Novamet Specialty Products.

"We have been working with a number of customers for the past few years, optimising a number of powders specifically for improving the quality and speed of printing for our AM customers, particularly in Binder Jet and PBF-LB [Laser Beam Powder Bed Fusion], but also for CSAM [Cold Spray Additive Manufacturing], PBF-EB [Electron Beam Powder Bed Fusion] and DED [Directed Energy Deposition] technologies."

Dr John Johnson, PhD, COO and CTO of Ultra Fine, added, "These products work because of our unique atomisation process and our ability to put the optimal cuts of powder into a blend for an application."

As well as its atomisation process, Ultra Fine also has the ability to offer smaller (down to 125 kg) or larger (over 1,500 kg) heats to provide small batches for customers testing new materials or for those using high-value, low-volume alloys. These specific powders are said to show increased flowability and density, and have been reported by numerous customers to offer superior additive manufacturability and final product performance.

"We can offer these grades, and even further refine the powder through additional proprietary processes, and still keep the powder at prices far below what others offer for similar or lower-performing powders," added Peterson.

www.ultrafinepowder.com

CMI Hub project takes key step to process rare earth metals in US

The Critical Materials Innovation Hub (CMI Hub), a US Department of Energy Innovation Hub led by the US Department of Energy's Ames National Laboratory and supported by the Office of Energy Efficiency and Renewable Energy's Advanced Materials and Manufacturing Technologies Office (AMMTO), reports it has worked with industry to take a key step in how to process rare earth metals in the United States.

The capability to produce these metals will help the US create a supply chain for neodymium iron boron permanent magnets.

For the past two years, the CMI Hub Open Innovation Projects (OIP) brought together Terves LLC and Powdermet Inc, both based in Euclid, Ohio; Worcester Polytechnic Institute (WPI), Massachusetts; and Ames National Laboratory to produce and refine rare earth metals.

"OIP projects are led by industry to help the next generation of CMI Hub research and development align with industry needs. This project helps enable a domestic supply of rare earth metals for strong permanent magnets," stated Thomas Lograsso, CMI Hub Director, Ames National Laboratory. "These metals are used in multiple industries. They had relied on midstream processing overseas. Through this work, we made ways to bring that to the United States."

Andrew Sherman, Terves Chief Executive Officer, served as project lead. The research group started in spring 2021. Their two-year goal focused on using heat and metal to make high purity neodymium, necessary to enable a US supply chain for neodymium iron boron permanent magnets. The key was creating low-temperature, scalable processes using the Terves foundry to speed up the establishment of rare earth metal manufacturing in the United States.

Terves worked with the Materials Preparation Center (MPC) at Ames National Laboratory on purification. MPC is a specialised research centre recognised for its unique capabilities in purification, preparation and characterisation of rare earths, metals, alloys, and single crystals.

Matt Besser, MPC director, says capabilities within the MPC were repurposed. High-purity calcium metal is usually added to rare earth fluorides to reduce into rare earth metals. High-purity calcium is made by MPC using vacuum distillation where calcium is vaporised and collected, leaving behind impurities that are detrimental in reduction processing. Here, they used the same process to separate volatiles like magnesium and calcium from neodymium from the rare earth metal that was reduced by Terves.

The CMI Hub developed a process to refine powder and reduce it to a metal (Courtesy CMI Hub)

"The desired metals are left behind and the rest can be reused in the process," Besser said. "We used arc melting to remove other unwanted bits. This left us with pure rare earth metals."

Sherman noted that the team's success meets the goal to refine neodymium at a small scale and increase to pilot scale. They worked with MPC to produce high purity neodymium. And, they showed how to make a new metal from recycled raw materials for making magnets. This made a mixed metal of neodymium, praseodymium and dysprosium, which theoretically can be used directly in the production of neodymium iron boron permanent magnets.

WPI made models that helped refine the process. The Terves team then used the models to refine metal and salt compositions at lower processing temperatures. This paves the way to use less energy and lower the cost of supplies. The team also worked with Purdue University who developed a techno-economic analysis and a pricing model.

www.energy.gov
www.ameslab.gov
www.tervesinc.com
www.powdermetinc.com

The metal was further refined through distillation, with the resulting metal remelted and formed into a specific shape (Courtesy CMI Hub)

Pensana receives Innovate UK grant for low-carbon rare earth supply chain

Rare earth metals company Pensana Plc, headquartered in London, UK – working in partnership with Polestar, Route2 and the Universities of Leeds and Hull – has been awarded £316,643 in grant funding by Innovate UK under its CLIMATES programme. This partnership is intended to develop a low-carbon, sustainable supply chain for rare earth magnets.

The project is backed by the UK Government and will use Pensana and Polestar's low-carbon and ESG engineering to review and measure impacts, and identify opportunities, to further reduce carbon in the rare earth products vital for the energy transition.

"Rare earth elements underpin so much of what makes everyday life and work possible, from the cars we drive to the phones we use," stated Nusrat Ghani, Minister of State at the Department for Business and Trade, who highlighted the importance of rare earths in the CLIMATES programme. "It is essential that, in a rapidly changing world, we do all we can to ensure resilient supplies of these and other critical minerals. We are laser-focused on securing robust supply chains, for the growing, green industries that will deliver jobs and prosperity across the UK in the decades to come."

Danny McNeice, Pensana's Sustainability Manager commented, "We believe Polestar 0 will set the standard for the electric vehicle industry, while Route2 has developed market-leading socio-economic impact measurement software and we are delighted to work with them and our academic partners at Leeds and Hull Universities to establish a UK-centric sustainable rare-earth supply chain from 'mine to magnets.'"

The CLIMATES programme is a £15 million fund set up to develop critical mineral supply chains within the UK. Strand 1 was launched in February 2023 with a focus on stimulating research on rare earths across the UK.

Pensana has already developed a strong approach to sustainability through the publishing of its Blueprint for Sustainable Rare Earths and has, through market-leading low-carbon design and renewable power agreements at both its UK and Angolan projects, placed itself well on the way to achieving its strategic goal to be a leading low-embedded-carbon supplier of rare earth products on the market.

www.pensana.co.uk
www.polestar.com

Gevorkyan plans US and Mexican factories following €30 million IPO

Powder Metallurgy parts producer Gevorkyan a.s., headquartered in Vlkanová, Slovakia, reports that following last year's successful IPO and listing on the Start Market, part of the Prague stock exchange, the company is planning further expansion and aims to establish new facilities in both the USA and Mexico in the coming months. Gevorkyan is also seeking listing on the other stock exchanges.

In June 2022, the Start Market IPO raised some €30 million for the company and these funds have been used to expand the facility in Vlkanová. This has included acquiring buildings adjacent to its existing site, increasing production capacity and purchasing new presses, furnaces, machining equipment and industrial robots. The facility now also boasts its own nitrogen and hydrogen generation.

Gevorkyan was established in Slovakia in 1996 by Artur Gevorkyan. Having moved from his native Armenia in the early 1990s, he first founded a Powder Metallurgy magnet plant in Ukraine before leaving the region and moving to Slovakia in 1996. The new business expanded its focus to a wider variety of PM, and today includes MIM, HIP and metal AM production. Over the years, Gevorkyan has gained a growing list of customers which now includes companies such as Linde, Komatsu and Siemens. It also cooperates with fashion brands such as Yves Saint Laurent and Versace, for which it produces metal clasps and decorations.

Artur Gevorkyan, Owner & general manager of Gevorkyan a.s. (right) welcomed the Prime Minister of the Slovak Republic, Ľudovít Ódor, during a recent visit to the Gevorkyan facility (Courtesy Gevorkyan)

In 2022, Gevorkyan was the first foreign company to join the Czech Start Market. The exchange is aimed at small- and medium-sized companies, and after a year of trading Gevorkyan is now looking to enter the main floor of the Prague Stock Exchange. The company is also in parallel negotiations with the Bratislava Stock Exchange.

www.gevorkyan.sk

6K Additive awarded five-year powder purchase agreement with US Army

6K Additive, a division of 6K Inc. headquartered in Andover, Massachusetts, USA, has been awarded a five-year Blanket Purchase Agreement (BPA) from the Army Contracting Command. The BPA is said to support the US Army Development Command (DEVCOM) in their strategic high-performance metal powders initiatives. It enables increased research and development efforts for weapons at DEVCOM, as well as meeting the additional demand for manufacturing prototypes and small quantities for Low-Rate Initial Production (LRIP). The BPA can be utilised by entities at various locations, including Picatinny Arsenal in New Jersey, Benet Labs in New York, Adelphi Laboratories in Maryland, and Rock Island Arsenal in Illinois.

To produce the metal powders, 6K Additive will utilise its proprietary UniMelt microwave plasma platform. This technology allows 6K to source, process, and reclaim scrap components, shop scrap, and used powders, converting them into aerospace-grade metal powders.

Lieutenant Colonel Peter J Stambersky, USA Retired and Director of Federal Affairs at 6K, shared, "My experience from my service in the Army provides me with a unique perspective of the agreement. Clearly, there is a benefit to 6K Additive, but, more importantly, this agreement provides the US Army DEVCOM with access to critical materials for Additive Manufacturing derived from feedstock right here in the US. This agreement further establishes 6K Additive as a trusted partner for powder and as a source for reprocessing retired components from military aircraft and vehicles delivering a closed-loop, secure material supply to the US Army."

The 6K UniMelt plasma production system is capable of converting various forms of high-value metal scrap into high-performance metal powders. These powders are used in Additive Manufacturing, Metal Injection Moulding, and other Powder Metallurgy production techniques. This system provides access to domestically-sourced strategic metals and alloys like nickel and titanium, as well as refractory powders like C103 and tungsten, which are crucial for modern military missions.

The patented 6K Additive process cleans, prepares, and spheroidises scrap alloys, producing high-quality powders that are said to outperform atomisation technologies. This process ensures a secure and traceable domestic supply of critical materials for the US military, while eliminating performance issues that may arise from questionable foreign sourcing.

www.6kinc.com

Researchers develop Powder Metallurgy titanium alloy for dental applications

Researchers from the Banaras Hindu University, Varanasi, India, have published an article in the *Journal of Biomedical Materials Research Part B: Applied Biomaterials* which describes the development of a potential biomaterial (Ti-10Nb) using Powder Metallurgy. The paper, 'Development of Ti10-Nb alloy by Powder Metallurgy processing route for dental application,' examines the microstructural, physical, mechanical, electrochemical, biological and tribological behaviour of the material under various situations.

The alloys were fabricated using four different compaction pressures (600, 650, 700, and 750 MPa) and sintered in a vacuum atmosphere at 1,000°C for 1.5 hours. The density of the samples was measured using Archimedes' principle. X-ray diffraction and scanning electron microscopy equipped with energy dispersive spectroscopy were utilised to investigate the phase composition and microstructure.

Additionally, a profilometer was employed to examine the surface roughness of the various samples. Hardness was evaluated using a Vickers hardness tester, while compression testing was conducted using a universal testing machine. Corrosion and wear behaviour were examined using a potentiostat and a Bio-Tribometer, respectively.

The study discovered that the samples compacted at 750 MPa exhibited the highest hardness, yield strength, compressive strength, and elastic modulus, measuring 450 ± 29.72 HV, 718.22 ± 16.37 MPa, 1543.59 ± 24.37 MPa, and 41.27 ± 3.29 GPa, respectively. Additionally, these samples demonstrated the highest corrosion and wear resistance, with the lowest icorr value of 0.3954 ± 0.008 $\mu A/cm^2$ and wear volume of $(31.25 \pm 0.206) \times 10^{-3}\ mm^3$.

The findings suggest that the developed alloys possess a range of desirable properties, such as high hardness, sufficient compressive strength, good corrosion and wear resistance, apatite-forming capability, and a low elastic modulus, which is advantageous for preventing stress shielding. The authors have stated that these attributes mean the materials should be considered as potential materials for dental implants.

www.bhu.ac.in

MPIF releases Standard 35 for PM Structural Parts

The Metal Powder Industries Federation (MPIF) has published the 2024 edition of *MPIF Standard 35-SP – Materials Standards for PM Structural Parts*. The document provides design and materials engineers with the latest engineering property data and information available to specify materials for structural parts made using the Powder Metallurgy process.

The MPIF explains that each user-friendly section of the standard is clearly distinguished by easy-to-read data tables (inch-pound and SI units) and provides explanatory information for each material listed.

Revised and expanded, this standard was developed by the Powder Metallurgy commercial parts manufacturing industry and includes new material data on prealloyed steel FL-5008 and stainless steel SS-409L-25, revised typical values for iron-copper and copper steel FC-0205-HT & FC-0208-HT, revised typical TRS values for iron-nickel and nickel steel FN-2025-HT & FN-0208-HT, and updated processing conditions for soft magnetic alloys.

The MPIF stated that this standard does not apply to materials for PM self-lubricating bearings (SLB), powder forged (PF) or Metal Injection Moulding (MIM) products that are covered in separate editions of MPIF Standard 35. It was added that publication of the 2024 Edition of this standard now renders the 2020 Edition (and prior editions) obsolete.

www.mpif.org

Fehrmann and Armstrong to use AI for aluminium-based solutions

Fehrmann Tech Group, based in Hamburg, Germany, and the Armstrong Group, headquartered in Singapore, have signed a Memorandum of Understanding (MoU) which establishes the framework for their joint efforts in developing aluminium-based solutions, as well as those for foam and elastomer. Utilising Artificial Intelligence, the partners aim to enhance performance and efficiency in various fields, including automotive and aviation, along with other diverse applications.

"As pioneers in aluminium technology, this strategic collaboration allows us to further expand our innovations into new sectors, particularly in the Asia-Pacific market," stated Henning Fehrmann, chairman and CEO of Fehrmann Tech Group. "We are excited to work with Armstrong Asia, leveraging their extensive experience across industries, to create groundbreaking solutions for foam and elastomer materials."

Armstrong Group added, "Our cross-industry operational expertise across seven countries and sixteen factories will be invaluable in this partnership, as we aim to deliver more innovative and customer-centric solutions in advancing today's noise, vibration, heat and safety management. With fifty of experience, Armstrong Asia serves a broad array of industries including automotive, consumer, office and retail productivity as well as healthcare & medical. Partnering with Fehrmann Tech Group amplifies our commitment to driving innovation and serving our global customer base more effectively."

Fehrmann Materials X, a specialised branch of Fehrmann Tech Group, houses a diverse team of experts with experience in simulation, AI, and data science. The team specialise in materials informatics and the rapid digital development of high-performance materials, working closely with aluminium and metal foundry specialists at Fehrmann Materials to craft sustainable custom alloys suitable for industrial applications in the automotive, aerospace, energy, and electronics sectors. The expertise of Fehrmann Materials X in digital alloy development and materials informatics is intended to significantly enhance the joint venture, boosting the potential for creating cutting-edge material solutions.

www.materialsx.ai
www.armstrongasia.com

"Scrap is the new gold" and other hardmetal and hard materials insights from Euro PM2023 Congress, Lisbon

The European Powder Metallurgy Association's annual congress has always featured technical presentations that reflect the diverse nature of the region's PM industry, from structural parts to hardmetals and other hard materials, Metal Injection Moulding and, more recently, metal Additive Manufacturing. This year's event, however, was of particular interest for the hardmetals community as it fell on the 100th anniversary of the Schröter patent application for metal-bonded WC. Here, Bernard North reports on the latest developments in the industry, as well as the story of its early beginnings.

During the COVID-19 pandemic in 2020 and 2021, the European Powder Metallurgy Association (EPMA) conducted its main annual conferences online. However, in 2022, the situation returned to normal. Its main conference, which also served as a World Congress that year, had approximately 1,070 attendees. This year, the Euro PM2023 Congress and Exhibition was held from October 1-4 in Lisbon, Portugal, at the Lisboa Congress Centre (CCL). The venue was in an impressive location by the Tagus River near the longest suspension bridge in Europe, the 25th of April Bridge, the name of which celebrates the Carnation Revolution of 1974 (Fig. 1).

This review is primarily directed at the forty or so cemented carbides and related hard materials contributions given at the conference. However, general aspects of the conference and the plenary session will also be covered.

The plenary session

Dr Lionel Aboussouan, the EPMA's Executive Director, welcomed the participants and announced that the attendance was around 800, which is slightly lower than in pre-COVID times but still highly commendable. The associated exhibition was well supported, with around seventy organisations having booths.

Aboussouan (Fig. 2) briefly summarised the EPMA's history since its founding in 1989; it has just ten employees and is able to do much of its work through an extensive

Fig. 1 The Euro PM2023 Congress and Exhibition was held at the Lisboa Congress Centre (CCL), set in an impressive location by the Tagus River, near the longest suspension bridge in Europe (Courtesy EPMA)

Fig. 2 Dr Lionel Aboussouan delivers his plenary speech (Courtesy EPMA)

Fig. 3 Antonio Homem, Head of Engineering at Durit, speaking in the Euro PM2023 plenary session (Courtesy EPMA)

Fig. 4 EPMA president Ralf Carlström gave an overview on the status of the European PM industry (Courtesy EPMA)

volunteer network from industry and academia, chairing or presenting at conferences, seminars, and summer schools. EPMA publishes statistics on PM, represents the industry in the EU, and runs and secures funding for joint projects.

Antonio Homem, Head of Engineering at Durit (Fig. 3), spoke for himself and his fellow Congress Chair Prof Teresa Vieira, of the University of Coimbra, on the PM industry in Portugal. Its roots lay in the need for tools for a major fertiliser operation in the south of Portugal, which initially resulted in the import of tools. Domestic manufacturing began with the company Palbit in 1916. SAPEC acquired the business in 1941 and, since then, the product range has expanded to include a broad range of mining, construction, wear parts, and metalcutting tools.

The other main player is Durit, which was founded in 1991 and which also offers a wide range of predominantly cemented carbide products and services. Overall, the PM industry in Portugal directly employs around 1,000 people. It predominantly operates in the hardmetals sector and exports approximately 90% of its production.

EPMA President Ralf Carlström (Fig. 4) gave an overview of the European PM industry. In 2022, tonnages reduced by about 8% to 177,000 tonnes, with ferrous powder declining more than non-ferrous. COVID-19, the war in Ukraine, and inflation all played a role in both the decline and uncertainty for 2023 and beyond. However, the 2022 industry sales value in Europe was an impressive €13 billion, about 30% of which was hardmetals.

PM volumes in automotive declined 3% in 2022 despite an 8.4% growth in vehicle sales, reflecting an ongoing shift towards battery electric vehicles and hybrids. In contrast, both hardmetals and diamond tools showed 4% and 8% growth respectively, in part due to price inflation.

Metal Injection Moulding sales grew by 2.3% in 2022, but there are indicators that this is slowing, with 2023 results expected to show a challenging year for the industry. Hot Isostatic Pressing (HIP) had 7.1% growth reflecting strength in the oil and gas sector. Additive Manufacturing continued to show impressive growth (25% by tonnage, 11% by revenue). Carlström also discussed promising areas for PM in H_2 production, storage, and use, and small modular nuclear reactors. Sales to the aerospace industry sales also showed a recovery.

Matthias Schmidt-Lehr, AMPOWER, gave a talk on industry surveys predicting the growth of AM, and specifically ones conducted in the 2015-17 time frame, which had grossly overestimated AM industry growth rates; he attributed this to primarily polling suppliers, who have an in-built bias toward optimism, and tend to underestimate the users' issues of return on investment, implementation, and customer acceptance. Typically, he stated, it takes five to ten years for R&D efforts in new fields to progress to commercialisation.

It was stated that, by polling both suppliers and users for forecasts with a large (~300 calls per year) sample size, more reasonable and accurate surveys can result. Schmidt-Lehr stated that Laser Beam Powder Bed Fusion (PBF-LB) has most of the market, but is now maturing, and more rapid growth is seen in Directed Energy Deposition (DED) and Binder Jetting (BJT) – especially the latter – while Material Extrusion (MEX) methods, such as Fused Filament Fabrication (FFF), seem to be slowing. That said, he admitted that by only polling current industry players, the surveys still had some bias. Overall, he expects about 20% annual growth in AM, with key drivers being more capable machines, more materials becoming qualified for AM, and the sustainability intents of users.

Individual awards

A number of individual awards were presented in Lisbon. The 2023 EPMA Fellowship Award was presented to Prof Marco Grande, the University of Turin, and the 2023 Distinguished Service Award was awarded to Adeline Riou, Aubert and Duval.

Hilti sponsored the EPMA's annual Thesis Awards, with the Masters Award being presented to Florian Häslich, Fraunhofer IFAM, Dresden, and the Doctorate Award presented to Dr Jianghu Dong of Delft University.

Keynote Paper Awards were given to six presentations, including one by Prof Raquel de Oro Calderon, Technical University Vienna, for her paper on Ru-doped cemented carbides, and the Peter Brewin Poster Award went to Bianca Luna Checa Fernandez, CEIT, San Sebastian, for her poster on Nd-Fe-B materials.

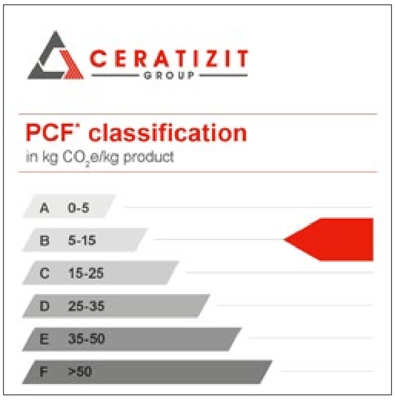


Fig. 5 Ceratizit PCF Classification
** The Product Carbon Footprint (PCF) is the specific carbon footprint in kg CO_2e/kg product. It includes all Scope 1 and Scope 2 emissions as well as those upstream Scope 3 emissions which can be allocated to the product (Courtesy Ceratizit)*

EPMA Sustainability Awards

Ten companies had entered the challenge for EPMA's Sustainability Award. Three winners were announced and acceptance speeches made by company representatives. All three stressed conformance to the Science Based Targets Initiative (SBTi) [1] which, through the Carbon Disclosure Project (CDP), in turn conforms to ISO 14064 to standardise the calculation and reporting of anthropogenic CO_2 on an organisation basis.

Gold Award: Höganäs AB

Andres Jähnke accepted the award, stressing the company's goal of net-zero CO_2 emissions internally by 2030, and by 2037 including its supply chain. He stressed the importance of understanding customer needs – including sustainability ones – to shorten product development lead times.

Silver Award: Ceratizit

Teemu Karhumaa accepted the award and gave a detailed speech, stressing the company's €1.5 billion sales, 8,000 employees and current number four position in the cemented carbide industry, with a near term ambition to be number three. It aims to be the sustainability leader in the industry, being CO_2 neutral by 2025 and net zero (including supply chain) by 2040.

Since a very large portion of the CO_2 production in cemented carbides is in raw materials mining and initial processing [2], it is essential to increase the proportion of scrap reprocessing; he mentioned Ceratizit's goal of 95% material coming from scrap and made the statement, "Scrap is the new gold". He also described Ceratizit's recently-announced PCF classification [3] (Fig. 5) with six openly published levels of kg CO_2 per kg of product, estimated according to the ISO 14067:2018 standard, and stated his belief that customers concerned about reducing their own supply chain CO_2 emissions would find such a standard helpful.

Bronze Award: Sandvik Additive Materials

Dr Paul Davies accepted the award and mentioned some environmentally favourable products – 316 stainless steel being 75% recycled today, Co-free maraging steels, and PBF-LB additively manufactured Ti alloy metalcutting toolholders. Net zero is targeted by 2050.

The Euro PM2023 exhibition

The Euro PM2023 exhibition (Fig. 6) comprised over 70 booths covering a broad range of materials, products, analytical equipment, and services and was well-attended, especially during breaks between technical sessions and during receptions.

Fig. 6 View of the Euro PM2023 exhibition hall (Courtesy EPMA)

Technical programme

Multiple channels were used to communicate technical information to attendees, with approximately 210 technical presentations given in up to six concurrent sessions, supported by around thirty posters on display adjacent to the trade exhibit. Five 'campfires' – informal specific subject presentations with discussion – were organised, along with eight 'Industry Corners' – specific subject presentations adjacent to the trade exhibit. There were also three Young Engineers Day sessions – short talks by students on their thesis studies, and six EPMA Sectoral Group meetings.

It was good to see the EPMA and its industry and academia associates encouraging students in the PM and related fields through the Young Engineers lecture sessions as well as trade show and other activity participation, aided by a low registration fee for students.

Fig. 7 Krupp Chemical Labs, Essen 1926 - the first carbide production site [5] (Source: H. van den Berg, WORLD PM2016)

Approximately forty hard materials presentations or posters were given, mostly in the specified hard materials sessions. Two of them were designated as Special Interest Sessions in recognition of 2023 being the 100th anniversary of the Karl Schröter patent application, which first publicly disclosed such materials [4].

Hard Materials – 100 years of development Special Interest Sessions

Dr Igor Konyashin and Bernd Ries, De Beers, presented a history of patent activity in cemented carbides and synthetic diamond [5]. WC and W_2C were first identified by Henri Moissan in the 1890s, but industrial activity did not develop until the 1920s, driven by the need for wire drawing dies for tungsten filaments for light bulbs.

The Osram Berlin group under Skaupy worked on metal-bonded WC culminating in the 1923 Schröter patent application, followed by Krupp's scale up and commercialisation (Fig. 7) in 1926/7 as 'Widia'. However, the patent was only successfully filed in Germany, the UK, and the US and, in the early 1930s, Fagersta Bruk AB (brand name Seco) launched product in Sweden, as did Moscow Electroplant in Russia (Podebit).

Regarding synthetic diamond, Leypunsky, in 1939, predicted conditions necessary to produce diamond, but without experimental verification. ASEA in Sweden was first to actually make diamond in 1953, but kept it a secret for a decade. GE in the USA first made diamond in 1954 and filed a patent application and published in 1955 in *Nature*, but a Defense Dept. secrecy order prevented the processing of the patent.

The publication, however, spurred DeBeers in South Africa to develop a process, which succeeded in 1958, after which patents were applied for in forty-eight countries.

In the USSR work started in the early 1950s and was successful in producing diamond in 1961, leading to production in Poltava, Ukraine. The 1939 publication was successfully used as a demonstration of prior art in a legal case.

Regarding low pressure Plasma Activated Chemical Vapour Deposition (PACVD) of diamond, this was first achieved in the USSR in 1956, but was kept secret until 1980; Union Carbide filed a patent in 1958. In summary, these case studies demonstrate the extreme importance of both patenting and technical publication to establish prior art.

Prof Walter Lengauer, now retired but formerly of the Technical University of Vienna, summarised the history of TiC and TiCN-based cermets, which are closely related to cemented carbides and overlap in some applications, especially metalcutting and woodworking [6]. Typical modern metalcutting cermets are complex compositions, for example (by wt.%) 40Ti 6W 7Mo 6Ta 1Nb 1Cr 8C 5N 18Ni 8Co.

Compared to cemented carbides, they are less dense and hard, and have better oxidation and diffusion wear resistance, but have low thermal conductivity and high expansion coefficient and hence poor thermal shock resistance and are primarily used for finishing cuts. The rock salt ordered fcc TiCN phase can incorporate many metals including W, Mo, Nb, and Ta. In 1931, eight years after the Schröter cemented WC patent, a patent was filed for Titanit S (metal bonded TiC plus other carbides) and N-free materials were the norm until the 1970s.

Then, starting with Technical University Vienna work, several developments took place, including the spinodal decomposition of TiMoCN compositions, Al-containing cermets, and adding other carbide-forming elements. In practice, modern cermets usually contain cored grains with two rock salt structured phases – TiN-rich cores and Mo, W, other metals, C-rich rims.

Modern analytical techniques have increased our understanding of sintering mechanisms and microstructures and alternative, often high entropy, hard phases and binders

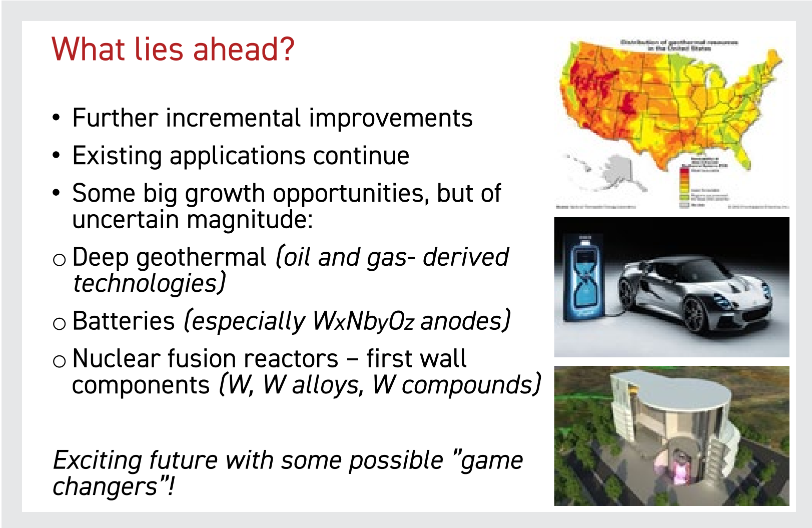


Fig. 8 Possible growth areas for tungsten and cemented carbides [7] (Courtesy Bernard North)

have been investigated. However, the basic constraints of moderate toughness and thermal fatigue resistance continue to limit substitution of cemented WC-based materials despite cermet's advantage in terms of critical raw materials.

Bernard North, North Technical Management, LLC, overviewed the basic technical, practical, and business reasons for cemented carbides having become a €20 billion annual sales business globally, directly employing around 100,000 people and making products which are directly used by millions more, affecting the lives of most people on Earth [7].

Basic technical reasons include very high hardness/toughness combinations, thermal expansion, conductivity, and high temperature properties leading to excellent thermal fatigue resistance, extensive ability to vary and control grain size, carbide grain type and morphology, and metal binder composition, properties and amount to 'trade' properties, as well as the ability to form several types of functional gradient materials (surface metal enhancement or depletion, hard coatings, surface compressive residual stresses), and to form strong bonds with tough underlayers (e.g., brazing to steel) or hard overlayers (diamond, cubic boron nitride, ceramic coatings).

From a practicality viewpoint, cemented carbides benefit by being processable by standard ceramic or PM milling, powder granulation, green part formation, sintering, and grinding etc. techniques all of which are readily scalable, and which in turn help reduce product development cycle times. Applications are spread globally across multiple industries and, in many cases, the hardmetal component comprises only a small percent of total system cost, but its quality, performance, and associated technical support has a disproportionate effect on total costs, hence driving both profitability and ready adoption of new and improved products.

The 'three Rs' of sustainability are exemplified by Reduce (use less material by improving performance and/or lightweighting the part), Reuse (reconditioning of lightly -used tools), and Recycle (estimate about 60% globally of materials are recycled from grinding sludge, scrap, used components etc.) North suggested three major areas to watch, which could have big effects on the hardmetals industry in terms of both materials demand and technical developments, summarised in Fig. 8.

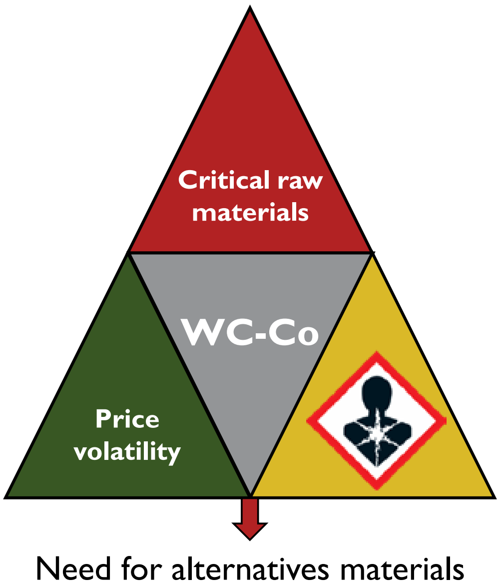


Fig. 9 Raw materials cost and availability, plus potential health concerns, driving alternative materials [12] (Courtesy Jazmina Cuadrado/CEIT)

> *"Schleinkofer emphasised that ease of recycling was now a prime requirement for any product development, and expressed concern that some developments, while they may be technically successful, would be a problem for commercialisation if they presented a recycling challenge."*

Sandvik's Dr Martina Lattermann discussed several applications of computational science in the hardmetals industry [8]. Thermodynamic modelling had improved understanding of phase diagrams and, for example, explained the effect of Cr additions in reducing the Co solid solution melting point and widening its 'C window' on the low C side. Another example is kinetic and thermodynamic modelling of functional gradient materials, both the Co-enriched surface zone commonly used for CVD-coated metalcutting inserts, and Co-depleted surfaces used for some rock drilling applications.

Finite element analysis (FEA) of metalcutting predicts temperature profiles in the 'nose' of the insert and can be combined with advanced analytical techniques to show how, in crater wear, C and Co diffuse into the metal chip resulting in metallic W between WC grains, and consequent embrittlement.

Ceratizit's Dr Uwe Schleinkofer described some technical highlights from his thirty or so years working in hardmetals [9], starting with his PhD thesis at University Erlangen-Nurnberg, where he conducted high temperature mechanical fatigue tests and, through TEM analysis, determined the Co solid solution fcc to hcp phase transformation occurring at the fatigue crack tip. Later developments allowed for the simulation of fatigue crack propagation. He went on to summarise the development of TiB_2 and TiAlN coatings by Chemical Vapour Deposition (CVD) rather than the Physical Vapour Deposition (PVD) method commonly used.

In the field of AM, he described the use of the filament-based Material Extrusion (MEX) process, also known as Fused Filament Fabrication (FFF), a sinter-based AM process, using a 0.4 mm nozzle with 0.1 mm layer thickness to produce, on commercial desktop printers, a variety of shapes, including in multiple grades. The result was an 80% reduction in materials usage, and he amused the audience with his description of the 'skipping rope' test to check filament strength and flexibility!

However, he laid the greatest emphasis on the sustainability drive and reinforced the message made earlier in the conference by his colleague, Teemu Karhumaa, about Ceratizit's initiatives in this area, including reductions in landfill needs, water usage, and, above all, CO_2 generation.

Given the dominant role of reclaim use to reduce CO_2 per kg of hardmetal [2], Ceratizit has introduced an all-reclaim (mixture of chemical reprocessed and 'zinc reclaim') grade 'Upgrade' as well as the PCF classification convention. Schleinkofer emphasised that ease of recycling was now a prime requirement for any product development and expressed concern that some developments, while they may be technically successful, would be a problem for commercialisation if they presented a recycling challenge.

Hard materials – cermets

Prof E Carreno-Morelli, from the University of Western Switzerland, authored a poster [10] demonstrating the use of laser edge preparation of TICNMoNo cermet inserts prior to PVD coating.

Prof Walter Lengauer, formerly Technical University of Vienna, described analytical work [11] on commercial TiCN-based cermets used for saw blades for cutting stainless steel. Optical and scanning electron microscopy, XRD, and Vickers (HV10) hardness and indentation K_{IC} measurements were made. The materials showed similar behaviour to cemented carbides, with higher metal binder level and/or coarser grain size increasing toughness at the expense of hardness. The metallic binder had an fcc phase structure while the hard phase had an ordered fcc rock salt structure. Some of the hard phase elements dissolved in the metal binder.

Lengauer proceeded to describe work sintering TiCN and TiCN/WTiC -based cermets, with varied C levels, using either a N_2 or Ar atmosphere, and showed how N_2 reduced the degree of solubility of metals coming from the hard phase in the metallic binder.

Jazmina Cuadrado, CEIT San Sebastian, summarised driving forces to alternatives to cemented carbides (Fig. 9) and described [12] sintering (1,450°C, 1 h) studies of tough Fe-bonded TiC-Cr3C_2-Mo/Mo_2C cermets. Use of high vacuum encourages surface oxide reduction (Mo_2C instead of Mo is also helpful), but also resulted in significant metal evaporation, while injecting 1.2 bar Ar at 1,300°C to suppress the same resulted in high porosity. A 950°C anneal followed by air quench raised hardness significantly.

Hard materials – superhards

A poster [13] from Prof Ana Senos, the University Aveiro, described attempts to make a cubic BN-reinforced cermet (43 wt.% BN, 57 wt.% TiCN, with 5 or 15 vol.% Ni binder) using Spark Plasma Sintering (SPS) in the 1,600-1,800°C range; the BN transformed to the soft hexagonal form and the work will be continued at lower temperatures.

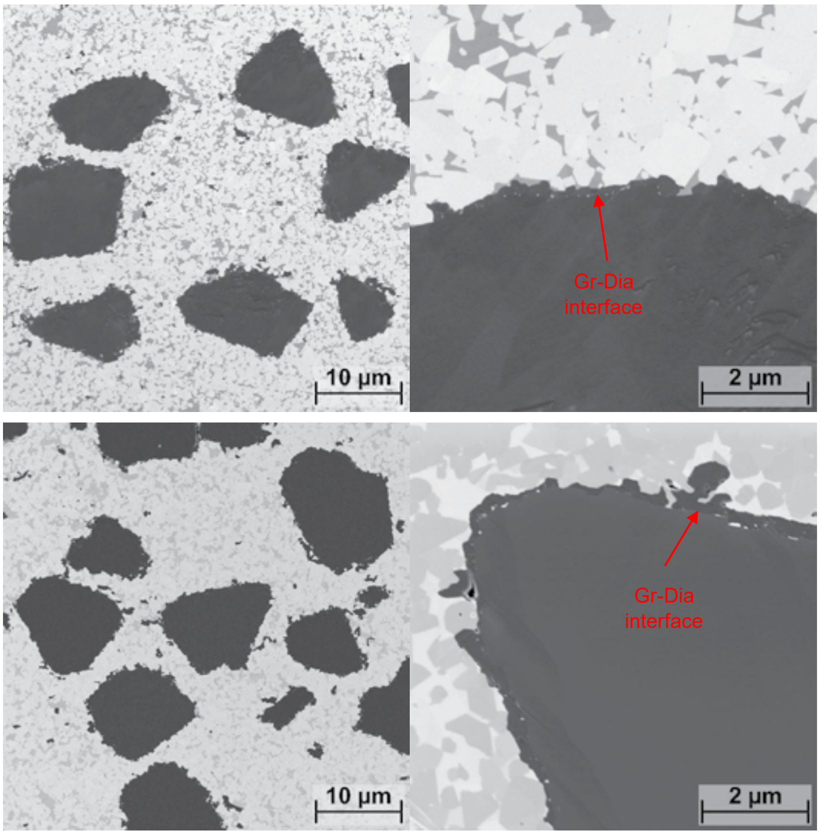


Fig. 10 Diamond grit dispersed in WC-Ni matrix [14] (Courtesy Matthias von Spalden/Fraunhofer IKTS)

Matthias von Spalden, Fraunhofer IKTS, Dresden, described work to produce metal-bonded WC + diamond composites with a targeted vol.% mixture of 14 metal, 56 WC, 30 diamond [14]. To minimise diamond graphitisation, SPS was used to reduce temperature and time at temperature, Ni was used instead of Co, and reactive sintering of W+C was used to form WC in-situ. Densities up to ~97% theoretical were achieved with good diamond retention, albeit with graphite interfaces (Fig. 10). Further work with coated diamond grit and optimised SPS cycles is planned.

Greg Kalfayan, the University of Bourgogne, described SPS at 1,200°C of titanium powder with up to 15 wt.% nanodiamond grit added. Fully dense materials with up to 1190 kg/mm^2 Vickers hardness (5 kg load) were obtained. Analysis indicated significant diamond reaction to form TiC [15].

Matteo Zanon, Kymera International, described detailed studies [16] to qualify a proprietary FeCuNi alloy as an alternative to traditional Co-based compositions for bonding diamond grit for applications such as concrete sawing. The results are very positive, with strong diamond retention and bonding.

Hard materials – Additive Manufacturing

Christian Berger, Fraunhofer IKTS, after initially screening twenty-two commercial spray powders, selected three WC-12Co AM (coarse grain) or HVOF (fine grain) hardfacing powders plus a WC-11Ni hardfacing powder, all averaging about 20 µm in size, for BJT Additive Manufacturing studies [17]. Hollow cubes and test pieces were produced using vacuum sintering in the range of 1,350–1,500°C.

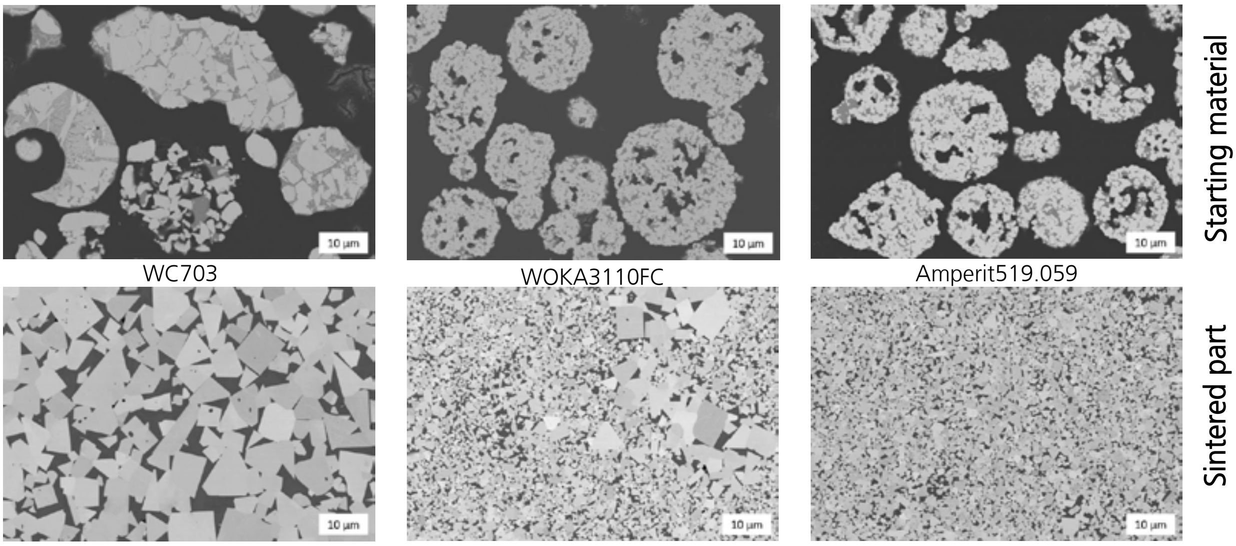


Fig. 11 Comparison of WC-Co starting powders to final sintered microstructures [17] (Courtesy Christian Berger/ Fraunhofer IKTS)

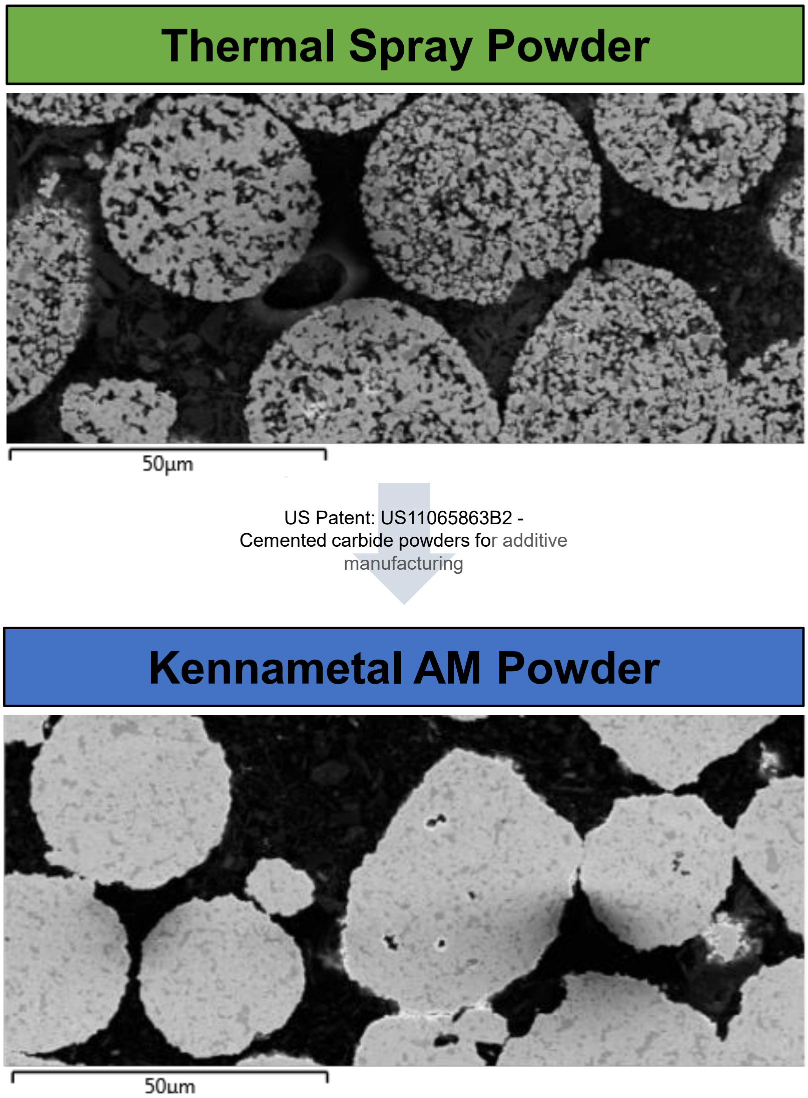


Fig. 12 Porosity reduction in WC-Co BJT processing [18] (Courtesy Dr Dominick Schmidt/Kennametal)

The coarse grain powder designed for AM showed by far the highest green density and lowest shrinkage and had a uniform grain structure, while the fine grain WC-12Co grades showed discontinuous grain growth at temperatures necessary for full densification (Fig. 11). The WC-Ni material needed a higher (1,600°C) sintering temperature to achieve full density, but also experienced significant grain growth.

Dr Dominick Schmidt, Kennametal, discussed the use of AM for metal-cutting tooling, both the PBF-LB H13 steel toolholders and BJT cemented carbide (10% Co submicron) consumable tools [18]. For the former, the geometrical freedom has been manifested in lightweighted 'natural-like' structures with integral cooling and favourable damping characteristics, and he showed an example of AM-enabled fluidics whereby coolant flow can oscillate, thus aiding chip control.

For cemented carbides, Schmidt described how minimising porosity sources through powder (Fig. 12) and sintering cycle optimisation had increased green density, green strength, and microstructural quality, manifested in a transverse rupture strength improvement from 2 GPa in 2019 to 3.7 GPa in 2022, and demonstrated in a severe end milling test.

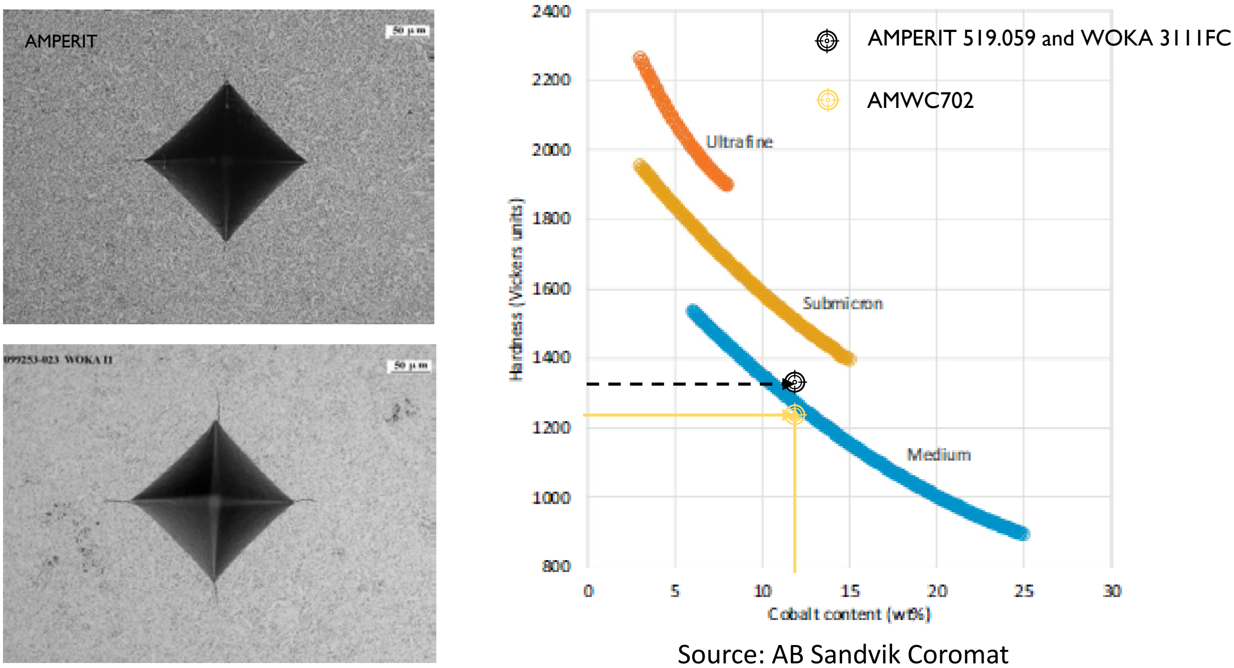


Fig. 13 BJT WC-12%Co materials achieve typical hardness levels (Courtesy Naiara Azurmendi/Tecnalia)

Naiara Azurmendi, Tecnalia, San Sebastian, analysed basic properties of two WC-12%Co thermal spray powders and tested their suitability for the BJT process [19]. Both powders had ~17 µm mean particle size, but one had a broader distribution as well as lower internal porosity, which in turn enabled higher green (38% vs 32% theoretical) density and lower (~25% vs. ~29%) and more uniform shrinkage in pressure sintering (1,455°C, 1 h, 30 bar). Both materials were fully dense with a HV30 hardness ~1,320 kg/mm^2 (Fig. 13) reflecting some grain growth. Plasma spherodised powder had higher green density and lower ~24% sintering shrinkage, but also a less homogeneous microstructure.

Dr Daniel Rodrigues, BRATS Filters presented a poster [20] comparing the microstructures and properties of WC-30 wt.% Co, Ni, or Co/Ni made by conventional press & sinter as well as by PBF-LB. The latter method produced inhomogeneous, non-traditional microstructures, and indentation hardness testing introduced cracks (Fig. 14).

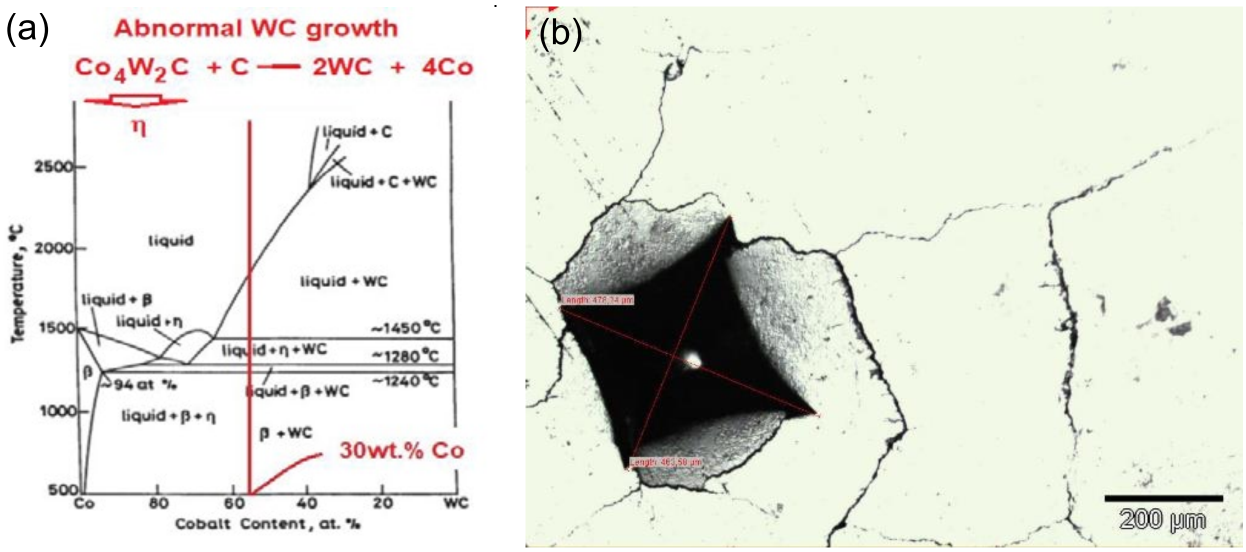


Fig. 14 WC-Co phase diagram section, and cracks around a hardness indent [20] (Courtesy Dr Daniel Rodrigues/BRATS Filters)

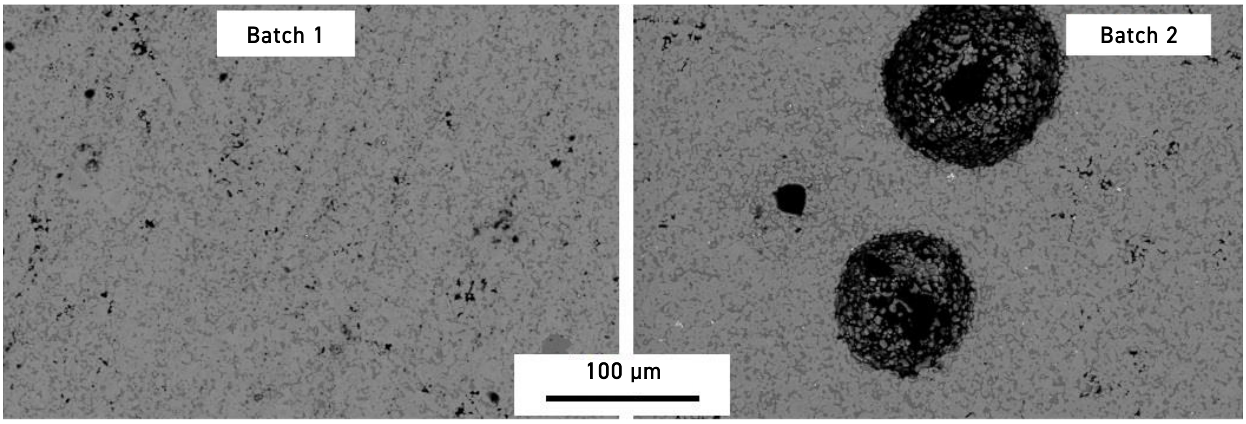


Fig. 15 SEM micros showing elimination of large voids in gel cast NbC 4%WC 20%Ni by using anti-foaming agent [21] (Courtesy Dr Daniel Rodrigues/BRATS Filters)

Dr Daniel Rodrigues also gave a paper [21] on an aqueous gel casting extrusion AM process for NbC-4 wt.% WC-20 wt.% Ni, a material used for guide rolls in steel mills. A 50 vol.% solids gel resulted in sintered densities ~91% and ~98% of pressed and sintered controls, with the higher density arising from the addition of an anti-foaming agent to the gel, which suppressed porosity (Fig. 15). Rodriguez expressed confidence that the process can be refined further to achieve full density and equivalent properties to the conventional route.

Goncarlo Oliveira, University of Coimbra, described [22] exploratory filament based MEX of 50% by volume WC-Co cemented carbide and TICN/WC-Co,Ni cermet into cylindrical shapes with and without cylindrical holes, with a view to establishing a route to internal coolant channel metalcutting tools. Thinner cross-section thicknesses were shown to be helpful in avoiding bloating defects associated with debinding.

Hard materials – reclaim

Alexandre Megret, the University Mons, compared the microstructures of ball milled fine grain (~1.1 µm) WC-7.5%Co (with some Cr_3C_2 dopant) made with 0, 20, 40, 60, 80, and 100% reclaim, sintered using conventional vacuum sintering (1 h at 1,400°C or 1,500°C, sintered thus plus HIPped at 1,200°C, and SPS at 1,200°C for 10 minutes under 50 MPa pressure [23]. The vacuum sintered only materials had significant porosity, while the HIPped or SPS materials had very low porosity levels; as reclaim increased the materials tended toward finer grain size.

Dr Gian Pietro de Gaudenzi of FILMS (OMCD Group) described a corrosion study exercise [24], which suggested a possible route for reclaiming sintered cemented carbides by an electrochemical leaching route through a galvanostatic approach using square wave cycling in 0.2M Na_2SO_4 aqueous solution, tungsten oxides could be separated from the metallic binder. Electrical power needs were in the range 1.3 to 1.6 Wh per g of material.

> ***"The vacuum sintered only materials had significant porosity, while the HIPped or SPS materials had very low porosity levels and as reclaim increased the materials tended toward finer grain size."***

Hard materials – alternative elements and high entropy alloys

Dr Juha Lagerbom, VTT Technical Research Centre of Finland, reviewed the background on high entropy alloys [25], being generally defined as alloys with at least five elements each present between 5 and 35 atomic %, and, in cemented carbide-type materials, the principle can be applied to the hard carbide phase(s) (where C is a sixth element).

Their potential advantages lie in improved thermodynamic stability, refractoriness, and high temperature deformation resistance, albeit at the price of reduced thermal conductivity. He described work on five high entropy carbide (HEC) phases drawn from the metallic elements W, V, Nb, Ta, Ti, Mo, and Cr, made by mechanical alloying of the elemental powders with C followed by pressing to form pellets and then sintering at 2,000°C. XRD, SEM, EDS and micro

XRF studies confirmed formation of an ordered fcc, or rock salt, crystal structure albeit with a second phase in one sample (Fig. 16). Very high microhardnesses of 26 GPa were observed; W and Mo tend to increase ductility while reducing hardness.

Zahid Answer, University Leuven, dry Turbula-mixed C with oxide powders of Ni, Ti, V, Nb, Ta, W and Mo, and then vacuum sintered pressed compacts at 1,420°C [26]. Proportions were chosen to result in a 10 wt.% Ni sintered product. The sintered materials had uniform microstructures with ~5 µm grain size. Thermogravimetric (TGA), differential thermal analysis (DTA), and dilatometry confirmed significant weight loss starting at ~900°C where oxide reduction commenced, shrinkage starting at about 1,290°C, and ~40% linear shrinkage. Partial substitution of W by Mo aided sintering and avoided a WC phase. VPN/K_{IC} combinations were quite good at 12 GPa/10 MPa $m^{1/2}$ and 17 GPa/7.5 MPa $m^{1/2}$, despite some Ni evaporation and porosity, and could be improved by pressure sintering.

Laura Cabezas Penalva, Polytechnic University of Catalonia, performed (Berkovitch tip) nanoindentation mapping on high entropy carbides (grain size ~1.6 µm) bonded with either Co or Ni [27]. Plotting the resulting hardness and Young's modulus data on a 'map' showed distinct regimes for the carbide phase (high hardness and Young's modulus) and metal phase (both lower).

Dr Tim Gestrich, Fraunhofer IKTS Dresden, described detailed thermal analysis and property measurements [28] on five 16 vol.% metal binder hard materials: a WC-Co control, WC-75Ni20Fe5Mn, WC-FeMnNiCoCu, NbC-Co, and WVNbTiTaC-Co. The WC had a 2.7 µm mean particle size.

Conventional sintering yielded fully dense materials with 'typical' microstructures (WC angular, NbC and HEC more rounded grains); dilatometry and DTA showed that the more complex materials initiated sintering at lower temperatures than the control. TGA showed evaporation of paraffin binder and subsequent outgassing due to oxide layer reduction initiating above 600°C (NbC and high entropy carbide at higher temperatures). Thermal diffusivity and conductivity data showed the expected advantage for WC-Co, but the other materials show promise and sintering conditions can be optimised.

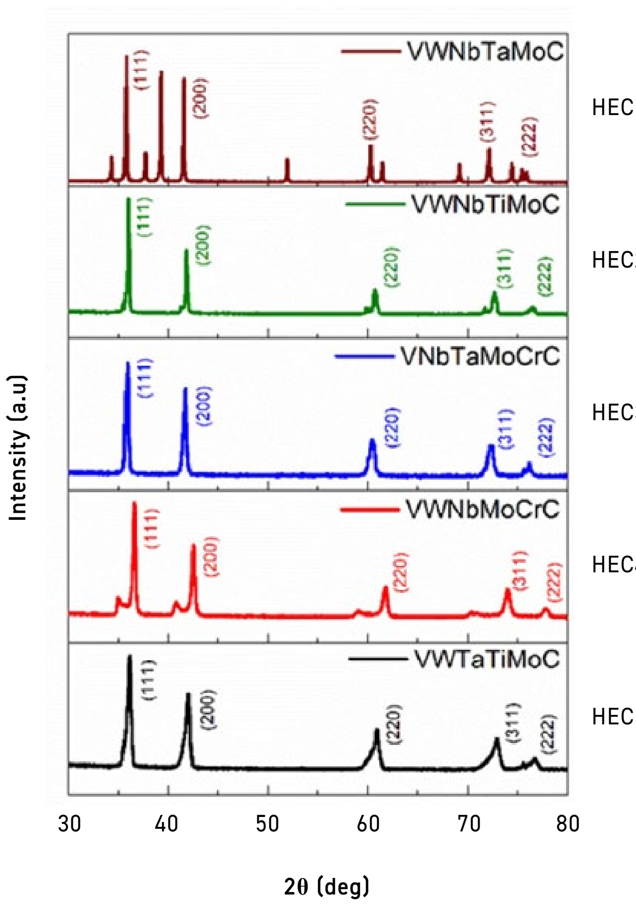


Fig. 16 High entropy carbide compositions forming single phase (except HEC1) ordered fcc 'rock salt' phase [25] (Courtesy Dr Juha Lagerbom/VTT Technical Research Centre of Finland)

Sibel Yöyler, Tallinn University of Technology, described the use of plasma transfer arc to deposit hardfacing layers of 316L stainless steel and TiC (from in-situ reaction of TiO_2 and graphite powders) on to a low carbon steel substrate [29]. Analysis proved that TiC was formed, and the layers had Vickers hardness levels (5 kg load) in the range 500-700 kg/mm^2. AISI G65 dry sand abrasion testing indicated promising deposition parameters for the best wear resistance.

Hard materials – detailed analysis

Prof Raquel de Oro Calderon, Technical University Vienna, reviewed the role of Ru in cemented carbides [30]. Due to its very high cost (currently €16,000/kg), Ru is rarely used except in specialised grades for superalloy and titanium machining. Calderon summarised Ru's role as stabilising the hcp (rather than fcc) Co solid solution phase while also allowing more W to dissolve in it before forming M_6C or $M_{12}C$ eta phases, and hence improving high temperature hardness and fatigue resistance.

Saghar Fooladi Mahani, Polytechnic University of Catalonia, used Vickers pyramidal, conical and Herzian (spherical) indenters at different loads on polished surfaces of three submicron grades

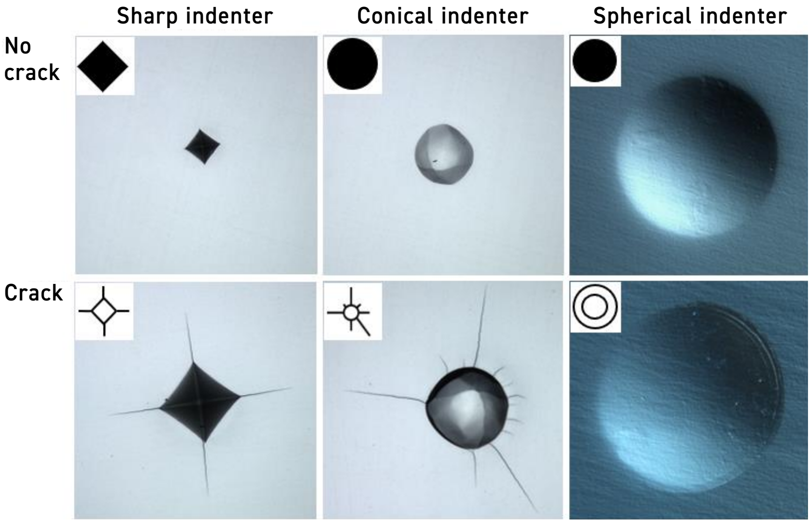


Fig. 17 Typical damage features from pyramidal, conical, and spherical indentors on cemented carbide [31] (Courtesy Saghar Fooladi Mahani/Polytechnic University of Catalonia)

Measurement series	Fe content in µg/g	Ni content in µg/g
1	332 + 12	50 + 5
2	351 + 14	64 + 4
3	348 + 17	66 + 7
4	321 + 10	57 + 5
5	365 + 13	65 + 4
Mean values	343 + 15	61 + 6

Table 1 Ni and Fe levels in large W grain in 92.5W-Ni-Fe heavy alloy [32] (Courtesy Prof Herbert Danninger, Technical University Vienna)

of different Co levels [31]. Using microscopy of the polished indented surfaces (Fig. 17), sequential polishing, and transverse rupture strength testing of indented bars, it was clear that the 'sharper' indenters and lower Co grades encouraged crack initiation and growth.

Prof Herbert Danninger, Technical University Vienna (retired), discussed the use of laser ablation induction coupled plasma mass spectrometry to measure how much Ni and Fe dissolve in the W phase of tungsten 'heavy metals' [32]. The results (Table 1) showed very low solubility of both binder elements, much lower than literature values derived from energy dispersive X-ray spectrometry.

Marc Serra Fanals, Polytechnic University of Catalonia, discussed work [33] using the Dixon and Mood model to determine fatigue strength with reduced testing. A very strong (4 point bend transverse rupture 3,540+/-420 MPa, Weibull modulus 18.5) WC-10%Co-0.5% Cr_3C_2 ultrafine grade was determined to have a 200k cycle fatigue strength of 2,300+/-330 MPa.

Dr Martin Bezuidenhout, Stellenbosch University, [34] discussed handheld power sawing of structural steel with cemented carbide tips with, and without, a PVD TiAlN coating. Progressive measurements of wear dimensions and fractography provided insight on the failure mechanisms at work.

Alexander Medina Peschiutta, University of Luxembourg, discussed several different methods to determine the binder volume concentration in 'green' pastes used for conventional extrusion, the Material Extrusion AM or Metal Injection Moulding of cemented carbides [35]. The results indicate the need for careful interpretation of the results according to the method employed.

Marvin Just, also of the University of Luxembourg, discussed detailed mass flow rate measurements on different size fractions of a spray dried WC-9%Co hardmetal grade [36]. For this powder the maximum flow occurred at a granule size ~130 µm, but the authors stress that the optimum could be affected by other factors such as WC grain size.

Hard materials – corrosion and oxidation

Dr Nuria Cinca, Hyperion, summarised the known effects of grain size, alloying elements, and binder metal level and type on corrosion resistance in acidic and alkaline environments [37]. In brief the metal binder dissolves in acid and WC grains in alkali, and higher W solid solution in Co is generally beneficial despite the concomitant increase in binder metal volume. Cr, Ti, Ta, and Ni are all beneficial due to their formation of protective oxide layers.

Experimental work with three different grain size WC-12%Co grades in acidic, alkaline, and sodium chloride solutions indicated that a finer grain size was beneficial in acidic but detrimental in alkaline solutions.

Angel Biedma, University of Madrid, summarised [38] work looking at $Fe_{15}Ni_{10}Cr_{10}$ (wt.%) bonded WC and TiCN, in each case targeting 20 vol.% metal binder, in terms of their mechanical properties, room temperature reciprocating ball wear profiles, and oxidation resistance between 500-1,000°C. Cr-free variants were also included as was a conventional WC-Co.

Summarising the results: WC materials outperformed TiCN materials in wear resistance, partly due to WC's lower friction coefficient. All of the materials performed well in 500°C oxidation but at higher temperatures the WC materials performed poorly due to WO_3 oxide volatilisation. The WC-based materials, including those with novel binders, were superior in terms of their combination of hardness, toughness, and strength.

Prabin A, Kennametal, studied corrosion of a WC-6% Co fine grain grade [39] varying several parameters: surface roughness, pH of the aqueous medium, temperature, and corrosion inhibitor chemicals. In general, a rougher surface, lower pH, higher temperature, and the absence of an inhibitor lead to more rapid corrosion rates (Fig. 18), but several interactions were noted. In addition, the work indicated that more environmentally acceptable inhibitors did not suppress corrosion as effectively as a triazole derivative chemistry.

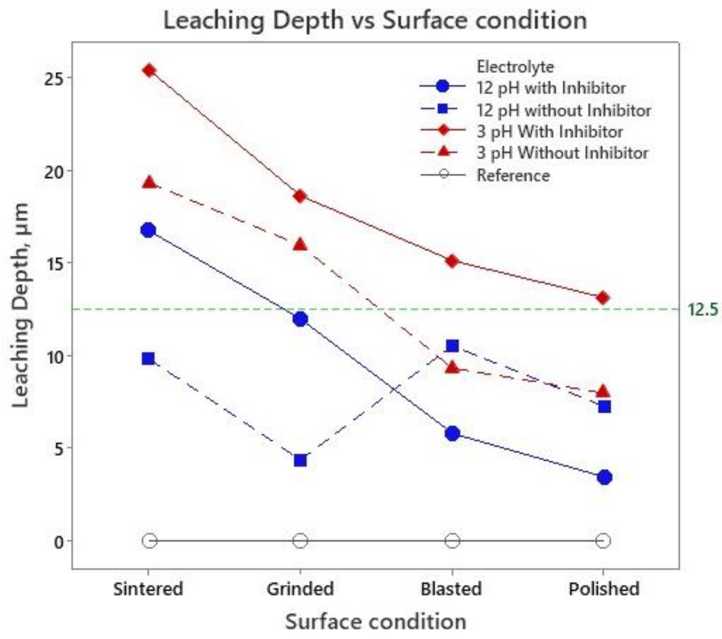


Fig. 18 Cobalt leaching depth as a function of surface condition, aqueous medium pH, and corrosion inhibitor [39] (Courtesy Prabin A/Kennametal)

Hard Materials - other

A poster authored by Bruno Guimares, the University of Aveiro, described the use of green carbide turning insert laser surface texturing [40], which, after sintering, edge preparation, and PVD TiAlSiN coating, gave shorter chips and reduced depth of cut notch wear (the usual end of life criterion) in machining 316L stainless steel.

Yongkwan Lee, Korea Institute of Industrial Technology, described a non-conventional approach [41] to making ultrafine (200 nm) WC by direct Self-Propagating High Temperature Synthesis (SHS) reduction of WO_3 yellow oxide by Mg powder in the presence of C black thus: WO_3 + 3Mg + C > WC +3MgO. The MgO is removed by acid washing.

Dr Suresh Srinivasan, Warwick University, presented an overview [42] on tungsten-based materials for nuclear shielding, both for radiological applications and the extremely demanding needs of the first wall of fusion reactors. A wide range of materials – metallic, metal-bonded, and ceramic-type (e.g., WC, WTiC, W_xB_y) – are all in active consideration and development.

EPMA Hard Materials sectoral group meeting

The EPMA's Kenan Boz reviewed the 2023 and 2024 EPMA event calendars as well as the EPMA sectoral groups and club projects – currently two are active and four more may commence in 2024. Prof Luis Llanes, Polytechnic University of Catalonia, reviewed the 2022 and 2023 EPMA main conferences from a hard materials standpoint – each of them had seven technical sessions and two Special Interest Sessions – as well as the 2023 webinar (156 registrants, eighty-nine attendees) and 2022 WINTEREV at RWTH, Aachen and the planned 2024 WINTEREV at NPL, London.

"Dr Suresh Srinivasan, Warwick University, presented an overview on tungsten-based materials for nuclear shielding, both for radiological applications and the extremely demanding needs of the first wall of fusion reactors. A wide range of materials - metallic, metal-bonded, and ceramic-type (e.g., WC, WTiC, W_xB_y) – are all in active consideration and development."

Fig. 19 Technical sessions at Euro PM2023 were well attended and the event attracted positive levels of attendance from the hard materials community, including from academia and industry (Courtesy EPMA)

Hilti's Dr Steve Moseley summarised pre-competitive project work, noting that there had been several since 2010, but that the trend was unfavourable. Current projects are one comparing three AM processes (six partners and four contractors with a €70k budget), while three contractors were lined up for a project looking into simulated fatigue crack growth.

Projects are currently in the planning phase to investigate binder alloying 'Alloyhard', and one on recycling 'Resqtool' with twelve participants currently expected to look at optimising the current hydrometallurgical and zinc reclaim processes, as well as investigating agro-industrial waste, bio-derived chemicals.

Prof Ana Senos, University of Aveiro, gave an overview [43] of the universities and companies active in hard materials in both the Iberian Peninsula and Latin America, with which the former shares close ties of history and language. In summary, ten universities are active in Latin America (seven in Brazil, two in Argentina, and one in Cuba), plus another seven in Spain and two in Portugal. Senos presented quite detailed summaries of their activities, which cover a broad range of basic materials science studies, processing innovations, and applications of cemented carbides, cermets, and superhard materials. Three companies in Spain (FMD Carbide, Hyperion, and TEMSA) and two in Portugal (Palbit and Durit) manufacture sintered cemented carbide products.

Closing comments

After three full days of the Euro PM2023 event, and drafting a summary of its hard materials contributions, it is clear to this author that significant additions and enhancements to the understanding of hard materials' compositional possibilities, processing and properties were made by the forty or so relevant presentations or posters. Attendance by the hard materials community was good; the hard materials technical sessions typically had about 40 people present.

It is heartening to see the continued active involvement of several European universities in hard materials science, and their close collaboration with industrial companies, government bodies, and with much of it actively facilitated by the EPMA. Besides the direct benefits to research and development, this also creates a stream of rigorously technically trained professionals to lead the industry in the future.

The emphasis on increasing reclaim use is clearly a major development which is no 'flash in the pan', and it will be fascinating to see how it affects technical and business trends in the years ahead. The use of scrap and reclaim materials is not new, and it has long formed an important and increasing part in materials supply and cost reduction. What has become evident in the past year or so is it becoming a 'public virtue' linked to sustainability concerns, and hence becoming a marketing tool.

Several questions come to mind, for example:

- With scrap demand increasing, will it lose its cost reduction benefits, and how much will this favour the more vertically integrated, and generally larger, cemented carbide companies?
- Some product areas, most notably metalcutting, are more amenable to a very high % of material recovery than others (for example, road construction). How will this play out in the years ahead?
- Reclaim methods are, generally, robust to significant variations in composition and grain size, but the bigger issue is scrap sorting and the number of 'flavours' that can be economically and practically maintained and used. Will this tend to suppress some technical developments, and how might proprietary patented compositions affect the outcomes?
- Currently (to this author's knowledge) no distinction is made for raw materials coming from the chemical route in terms of whether they originated as ores or scrap, since they have identical properties. Publishing reclaim % levels will, however, mean such a distinction will have to be made in the future. How easy will that be in practice?
- Looking further ahead, if tokamak fusion reactors employing W-based first wall components of finite life, indeed become technically and commercially practical in the 2030s, how much of the total global W production will they consume, and will the used materials be readily recyclable?

The industry is celebrating its first 100 years, and the confluence of societal and industrial trends with ever improving and broadening technologies bodes well for the next 100!

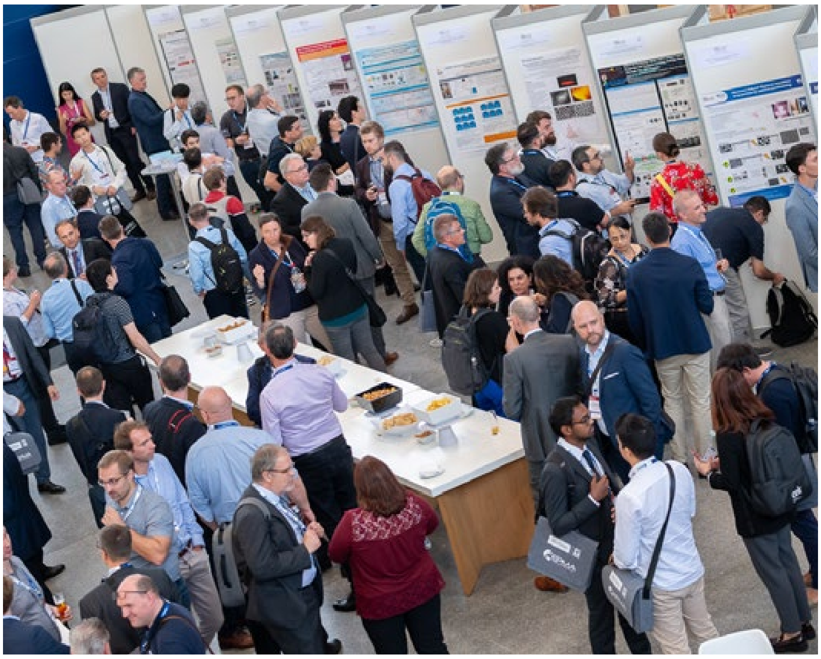


Fig. 20 A well-attended poster session at Euro PM2023 (Courtesy EPMA)

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Proceedings and the Euro PM2024

The conference proceeding can be accessed through the EPMA website, www.epma.com. EPMA's 2024 Congress and Exhibition will be held in Malmö, Sweden, from September 30 - October 2, 2024.

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IperionX: A Powder Metallurgy route to lower-cost recycled titanium plate, billet, bar and preforms with reduced CO_2 emissions

Titanium offers unique strength, lightweighting, and corrosion-resistant properties, making it a highly sought-after metal for various applications. However, its high cost and complex manufacturing process have limited its use. To combat this, IperionX has developed technologies that offer a cost-effective and sustainable solution for titanium production. Its Hydrogen Assisted Metallothermic Reduction (HAMR) and Hydrogen-Sintering and Phase Transformation (HSPT) processes promise a more affordable and environmentally friendly approach that reduces dependency on the import of critical materials.

Titanium is unique among metals. Across numerous applications, titanium alloys are preferable to the more common structural stainless steel and aluminium alloys. Titanium is strong, lightweight, and highly corrosion resistant, making it the ideal metal for broad applications in defence, aerospace, space exploration, transportation and electric vehicles, unmanned vehicles, and many other advanced manufacturing applications.

Unlike stainless steel and aluminium, however, titanium is expensive and difficult to process. This means titanium is typically reserved for only the highest value applications: jet engines, naval applications, medical implants, and, of course, Iron Man's suit.

There is, however, a reliance in many countries on imported titanium feedstock. As an example, the US is a leading consumer of titanium metal for aerospace and defence applications, but has no upstream domestic production capacity, leaving it highly reliant, leaving it highly reliant on titanium feedstock imports to service these multi-billion-dollar industries. Primary titanium metal manufacturing capacity is now almost non-existent in the US, with the last domestic plant shutting down in 2020. As of 2023, the current US titanium metal demand from the aerospace, medical, space and defence sectors is heavily reliant on international supply chains, which are dominated by production from China and Russia. Not only are these supply chains environmentally and societally unsustainable, but this

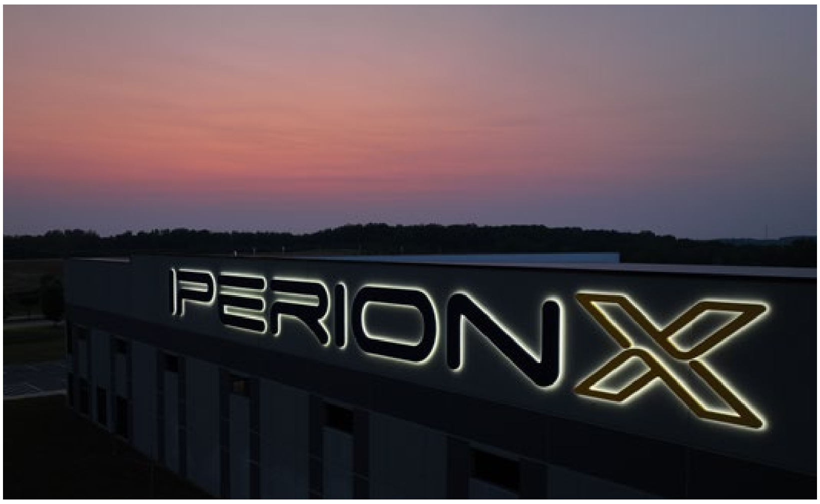


Fig. 1 Headquartered in Charlotte, North Carolina, with a pilot facility in Salt Lake City Utah and a soon-to-be-commissioned commercial production in South Boston, Virginia, IperionX's mission is to be a leading developer of US-based sustainable critical mineral and critical material supply chains, starting with titanium, to facilitate the global transition towards low carbon, resource efficient manufacturing (Courtesy IperionX)

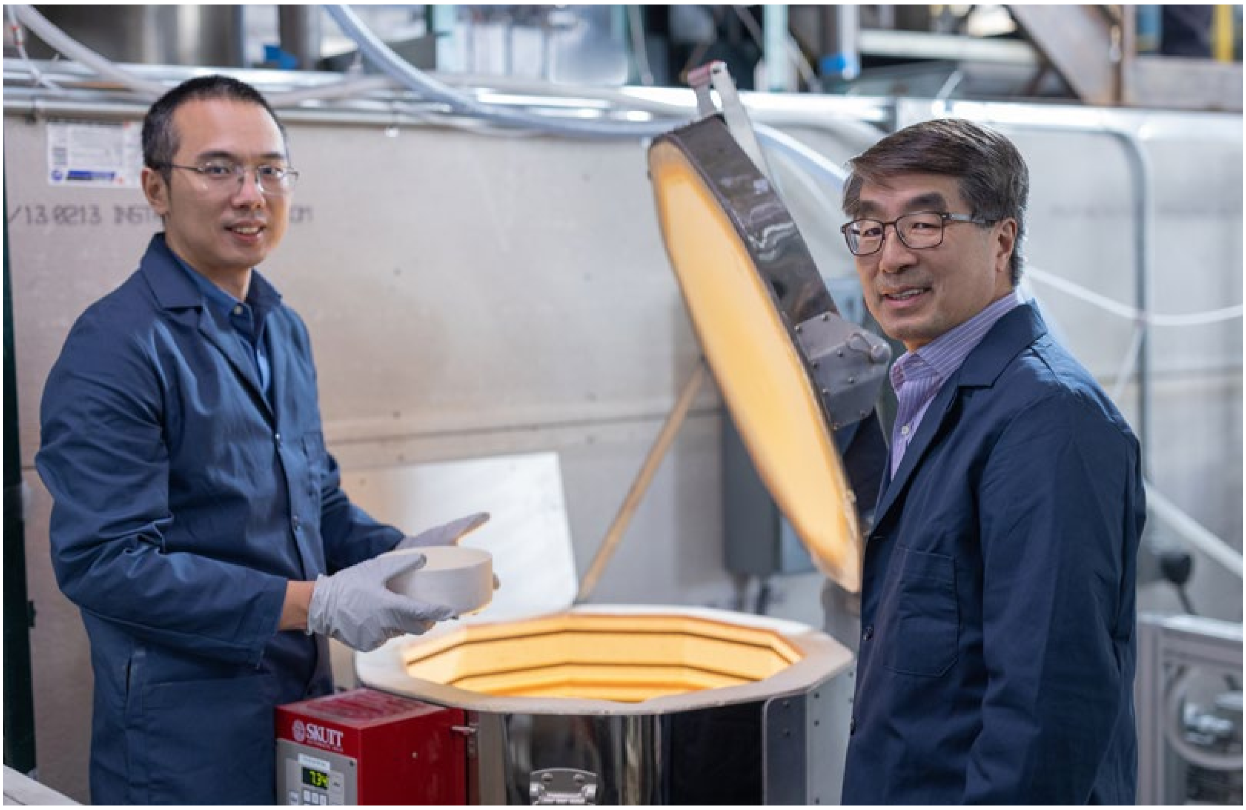


Fig. 2 Prof Zak Fang (right) with Prof Pei Sun (left), both of the University of Utah. Fang is the inventor of a series of Ti process technologies from mineral to products, including the HAMR process for making primary Ti metal directly from ore concentrate in the form of Ti powder, the Granulation Sintering Deoxygenation (GSD) process for making spherical Ti alloy powder for Additive Manufacturing, and the HSPT process to achieve wrought like microstructures and mechanical properties without resorting to wrought processes (Courtesy IperionX)

import reliance represents a serious threat to national security given the widespread use of titanium feedstocks within defence sectors.

In addition to the more commonly known aerospace and defence applications, titanium can be used in place of stainless steel and aluminium to lightweight electric cars and other vehicles, thereby reducing lifetime emissions. It is also a key material used in electrolysis equipment for hydrogen and hydrogen fuel cells — both important components of the rapidly growing green hydrogen economy. Titanium, therefore, has a significant role to play in the global transition to a green economy, but first a more cost-effective, sustainable processing and manufacturing solution must be developed. IperionX's technologies seek to provide that solution.

> ***"IperionX's technologies were developed over more than a decade of research and development at the University of Utah, with partial funding from the Department of Energy's ARPA-E programme, and provide a proven means of producing titanium powders and products for a fraction of the cost and energy consumption of the industry standard Kroll process."***

Innovative PM-based technology

IperionX's technologies were developed over more than a decade of research and development at the University of Utah, with partial funding from the Department of Energy's ARPA-E programme, and provide a proven means of producing titanium metal powders and products for a fraction of the cost and energy consumption of the industry standard Kroll process.

Hydrogen Assisted Metallothermic Reduction
Included in IperionX's technology suite is a process for making low-cost, recycled titanium metal powders from 100% scrap feed-stock, called Hydrogen Assisted Metallothermic Reduction (HAMR) technology. This patented technology is a low-cost, low-energy consumption titanium powder production process that utilises hydrogen to destabilise Ti-O, making it possible to turn the use of Mg to remove oxygen from TiO_2 or high-oxygen scrap from thermodynamically impossible to thermodynamically favoured. This allows TiO_2 to be reduced and deoxygenated directly by magnesium to form TiH_2, with low oxygen levels that can meet the needs of industry. TiH_2 can be further processed to titanium metal through standard industry methods.

Hydrogen-Sintering and Phase Transformation
Subsequently, IperionX's Hydrogen-Sintering and Phase Transformation (HSPT) technology provides a highly efficient means of turning HAMR powder into titanium mill products (billet, bar, plate, etc.) as well as near-net-shape preforms (parts that are closer to the end-use part dimensions) with wrought-quality microstructure. HSPT provides a powder metallurgical pathway to manufacture traditional mill product feedstock and near-net-shape preforms at far lower cost and with lower carbon emissions than current industry methodologies are capable of achieving.

Fig. 3 Titanium scrap used in IperionX's circular, low carbon titanium manufacturing process (Courtesy IperionX)

> *"HSPT provides a powder metallurgical pathway to manufacture traditional mill product feedstock and near-net-shape preforms at far lower cost and with lower carbon emissions than current industry methodologies are capable of achieving."*

The Kroll process

Primary titanium metal – known as titanium sponge – is produced via a complicated, expensive, and inefficient method known as the Kroll Process. The Kroll Process, which was first developed in the 1940s, relies on multiple processing steps that first refine the mineral to a pure TiO_2, then convert this to a $Ti\text{-}Cl_4$ intermediate that can be reduced in a Kroll reactor to form a sponge-like (hence the name) pure titanium metal. This sponge must then be further processed and blended either with master alloy material or with high-grade alloy scrap.

It is then fed through multiple time-consuming and energy-intensive vacuum arc remelt steps to produce large titanium alloy ingots. These are then forged into semi-finished goods which are then further processed to create final products (wire, plate, bar, sheet). Significant yield losses are incurred at multiple stages of this processing journey, which is, by its nature, highly energy-intensive and produces significant greenhouse gas emissions.

Titanium usage and status as critical material

While the US was the first nation to commercialise titanium sponge production in the 1950s, by 2020 it did not have a single large-scale

Fig. 4 Recycled titanium metal powders produced by IperionX from 100% scrap feedstock using Hydrogen Assisted Metallothermic Reduction (HAMR) technology and used in the HSPT process (Courtesy IperionX)

operational titanium sponge plant. Today, there is a minimal commercial titanium sponge production capacity for this critical material used in many US defence systems, including military aircraft, with newer aircraft using increasing amounts of titanium, often in excess of 20% by weight.

Currently only Japan, Russia, and Kazakhstan have titanium sponge plants certified to produce aerospace rotating-quality sponge that can be used for engine parts and other sensitive aerospace applications. Data from the US Geological Survey shows that, in 2018, Russian and Chinese titanium sponge producers controlled 61% of the world's titanium sponge production, an increase on their combined 55% share in 2008 and 37% share in 1998. Today, China and Russia control roughly 70% of global titanium sponge capacity.

Without domestic titanium sponge production capacity, the US is dependent on imports of titanium sponge and scrap and may lack the surge capacity required to support defence and critical infrastructure needs in an extended national emergency. IperionX's proposed closed-loop titanium supply route has the potential to reduce this national security vulnerability and to supply additional markets that IperionX believes will be important for a transition to a net-zero economy. Titanium's historical high-cost and high-CO_2 profile have precluded its use in a variety of other industries, including consumer electronics and medical technology – IperionX's potential low-cost, climate-friendly titanium supply opens up an array of potential new applications.

The commercial case for HSPT

Traditionally, titanium alloys with satisfactory mechanical properties can only be produced via energy-intensive, costly wrought and cast processes, while titanium alloys produced using low-cost Powder Metallurgy methods consistently result in inferior mechanical properties, especially low fatigue strength.

Wrought processing is the methodology of choice for producing semi-finished and near-net-shape product in today's titanium industry, particularly across critical applications such as aerospace and defence, where superior mechanical properties are required. However, wrought titanium is expensive, complex to produce, and leaves much to be desired when it comes to sustainability and greenhouse gas emissions. These disadvantages are driven by the multi-step, inefficient, and energy-intensive thermomechanical processing routes (forging, rolling, etc.) used by industry incumbents.

Furthermore, many titanium components start their lives as raw ingot, bar, or billet feedstock, and are subtractively machined down into their final parts, generating enormous amounts of scrap mate-

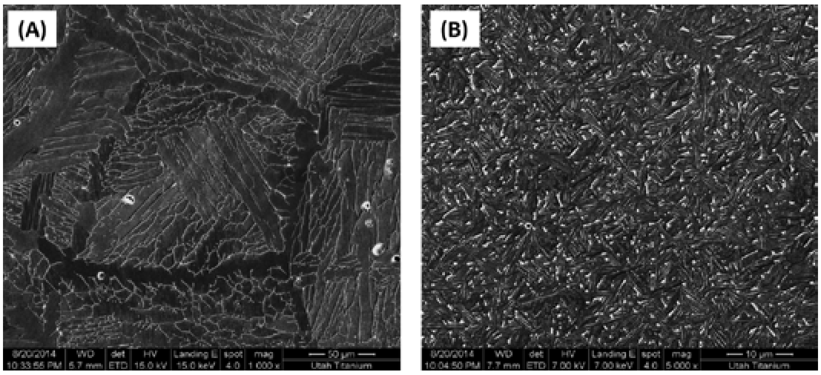


Fig. 5 SEM imagery of titanium Ti-6Al-4V alloy microstructures, comparing vacuum sintering in (A) and HSPT in (B). Note that A has a 50 µm scale and B has a 10 µm scale (Courtesy IperionX)

rial. For example, it is common for an aerospace company to need to purchase 10-20 kg of titanium alloy mill product in order to get a 1 kg final part. This represents what is called a 10:1 or a 20:1 buy-to-fly ratio, with the remaining 9-19 kg of generated scrap needing to be either landfilled, downcycled to the ferro-titanium market, or, in some cases, cleaned and re-certified to go back to the inefficient melting and thermo-mechanical working steps again.

Powder Metallurgy has long presented a potential new and improved means of producing near-net-shape parts, bypassing most of the inefficient, unsustainable, and expensive thermomechanical process steps. However, until recently, products produced via PM titanium production methods yielded relatively poor mechanical properties.

Additional process steps, such as pre-alloying powder feedstock or high-pressure sintering, can be combined with traditional PM methods to improve mechanical properties. However, these additions typically come at significant cost, nullifying any potential economic benefits associated with PM over thermomechanical production.

Therefore, in order for PM-based alternatives to replace wrought processing, they must produce titanium alloy products with wrought-like material characteristics, but at traditional PM costs or better.

It is here that there is an opportunity for IperionX's Hydrogen Sintering and Phase Transformation process – a novel technology that represents a new microstructural engineering approach for producing low-cost titanium and titanium alloys with high density, very fine microstructure, and exceptional fatigue strength.

The high fatigue strength is achieved by creating refined microstructures without resorting to the thermomechanical processing. This is accomplished by generating an ultrafine-grained as-sintered microstructure through hydrogen-enabled phase transformations, facilitating the subsequent creation of fatigue-resistant microstructures via traditional heat treatments. The exceptional strength, ductility, and fatigue performance made possible using HSPT are a breakthrough in the field of low-cost titanium processing, with significant implications for the broader titanium mill product industry.

How it works

HSPT builds on the well-understood benefits of using hydrogenated titanium powder as the feedstock. The core of HSPT's innovation is the utilisation of a dynamically controlled hydrogen environment during the sintering process step. Hydrogen serves as a temporary alloying agent, which helps to refine the microstructure of titanium alloy via phase transformations. HSPT can be simplified into three primary steps:

1. A β-phase sintering step in the presence of hydrogen to densify the solid form;
2. A phase transformation step at a lower temperature in the presence of hydrogen, during which homogeneous precipitation of ultra-fine-grain (UFG) phases are achieved; and
3. Dehydrogenation, during which the remaining hydrogen is removed by annealing under inert gas or vacuum, leaving a fine and uniformly distributed α+β microstructure.

A key point here is that, unlike traditional PM methods, whereby titanium alloy powders are typically sintered in high-vacuum environments, HSPT sinters titanium powder in a hydrogen-enriched atmosphere. The hydrogen partial pressure is strategically-controlled during the β-phase sintering step to improve the self-diffusion characteristics of the β-titanium—leading to improved densification. During the lower-temperature precipitation step, hydrogen is again used as a temporary alloying agent to induce the desired phase transformations, resulting in the formation of an ultra-fine-grain microstructure in the as-sintered state. The net result is that, instead of forming a coarse lamellar structure with widths in excess of 10 µm that is typically achieved with conventional vacuum sintering of titanium alloys, the HSPT process instead produces ultra-fine lamellae with widths in the submicron range.

Fig. 5 shows the differences between a conventional Ti-6Al-4V alloy microstructure achieved by vacuum sintering vs a Ti-6Al-4V alloy microstructure achieved by HSPT. The HSPT microstructure is finer and more uniform in distribution, which results in superior mechanical properties.

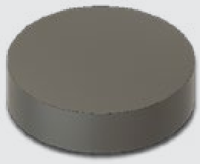
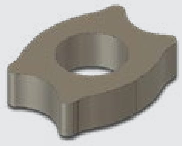
Final Product – Watch Case	Traditional Subtractive Manufacturing Feedstock – Titanium Bar		IperionX HSPT-enabled Near-Net-Shape Product - Titanium Ovoid	
				
Targeted Final Product Weight: <30 g	Feedstock Weight	Machining Scrap Generated	Feedstock Weight	Machining Scrap Generated
	>300 g	>270 g	~150 g	~120 g
	× Feedstock cost to customer exceeds 14 X the material value in final product × Majority of feedstock lost to scrap × Scrap cannot be fully recycled by Kroll-based industry		✓ Feedstock cost to customer reduced by 50% using IperionX's HSPT Tech. ✓ Machining scrap reduced by >50% ✓ Machining scrap can be recycled by IperionX to produce more product	

Table 1 Indicative watch-blank example of potential material savings with HSPT (Courtesy IperionX)

Fig. 6 AM-grade Ti-6Al-4V powder in a flow meter at IperionX (Courtesy IperionX)

Applications

As previously explored, conventional routes for producing titanium mill products (bar, sheet, plate, etc.) and then machining away the unwanted material leads to stubbornly high scrap-to-final-product ratios. HSPT technology enables angular powders to be manufactured into a wide variety of semi-finished and near-net shape titanium products, mitigating the inefficient and costly subtractive manufacturing methods used by conventional Kroll-based producers.

Furthermore, because HSPT manufacturing produces near-net-shape products, IperionX customers experience significant savings on the cost associated with material losses during subtractive manufacturing. While exact unit pricing depends on the application, an indicative example of the cost and material savings associated with HSPT is shown in Table 1.

In this example, based on IperionX's experience with watchmaker Panerai, savings of approximately 50% can be achieved via HSPT compared to traditional subtractive manufacturing. IperionX would not be selling angular powders directly, but rather a near-net-shape ovoid manufactured from angular powder. Ongoing commercial dialogues with a variety of potential customers demonstrate a significant interest in HSPT products made from angular powders. However, due to the highly disruptive nature of these technologies, existing markets and applications are limited.

In August, IperionX announced a collaboration with Lockheed Martin, whereby it would produce titanium plate components for testing by Lockheed. In September, a similar announcement was made for titanium plate production for US Army testing, with another collaboration around titanium plate with GKN Aerospace announced in October.

Details of the collaborations remain confidential. However, the announcement of collaborations with both the US Army, who are interested in titanium armour

Fig. 7 Spherical Ti-6Al-4V powders are also being developed by IperionX for use in AM and other processes (Courtesy IperionX)

plating for ground combat vehicles, and Lockheed Martin and GKN Aerospace, who are interested in titanium components across their high-performance aerospace products, is a strong validation of the HSPT technology's prospects.

In addition, the US Department of Defense (DoD) announced in late October that IperionX will receive a $12.7 million funding grant award under the Defense Production Act Title III Program. The funding will go directly towards IperionX's commercial titanium recycling facility in Virginia. IperionX concurrently complected an oversubscribed $16.7 million financing to existing shareholders. DoD's award, and the company's oversubscribed financing, further validate the titanium technologies and strongly position IperionX to achieve its goals of re-shoring a sustainable titanium supply chain to the US.

In the future, as IperionX advances the technology from laboratory and pilot scale to full commercial scale, the company believes that the Powder Metallurgy-based HSPT technology can enable the mass proliferation of titanium by rendering it cost competitive with comparable stainless steel and aluminium products.

Concluding thoughts

Although the aerospace industry is the largest consumer of titanium in the US today, titanium doesn't need to be limited to high-cost applications in the defence and aerospace sectors. Cost-effective and sustainably produced titanium products have the potential to be manufactured, thanks to IperionX's titanium processing technologies. HSPT technology, combined with IperionX's HAMR technology for producing low-cost, sustainable titanium, has the potential to further broaden the use of titanium products, just like Henry Bessemer's technology did for steel and Charles Hall and Paul Heroult did for aluminium in the 19th century.

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A review of the sintering of iron-copper-carbon alloys for structural Powder Metallurgy applications

Iron-copper-carbon alloys have been used for structural Powder Metallurgy applications for more than half a century and, to this day, they remain popular for the production of automotive components. In this comprehensive review, Prof Randall German covers the sintering of iron-copper-carbon alloys, with a focus on the popular composition FC-0208. The review, which is intended to help producers to optimise properties as well as identify future research needs, explores powder characteristics, processing conditions, and the response parameters of mechanical properties and dimensional control.

This review focuses on the sintering of an important Powder Metallurgy alloy based on iron, copper, and carbon. A popular composition consists of iron with 2% copper and 0.8% carbon, designated FC-0208. The alloy has been used for structural applications since at least the 1950s and remains a favourite for fabrication of automotive components. Detailed here are linkages between powder characteristics, processing conditions, and response parameters of mechanical properties and size change (dimensional control). The sequence of events during sintering are detailed in terms of copper melting, spreading, and grain boundary penetration along with carbon diffusion. The balance between these aspects, as well as post-sintering heat treatment, determines the mechanical properties. This compilation is intended to help optimise properties and identify needed research.

Powder Metallurgy consists of several forming alternatives that rely on high sintering temperatures to bond the particles. In the case of the iron-copper-carbon alloys, sintering is at a temperature greater than the copper melting point (1,083°C). The melting of copper leads to significant strengthening as required for structural applications.

There is no equivalent composition in cast or forged metallurgy. This is because castings would undergo deleterious copper segregation to boundaries and interfaces during cooling, leading to a problem known as hot shortness. Thus,

Fig. 1 An FC-0208 copper steel drive pulley produced by Capstan Atlantic. It is used in the power steering of an electric autonomous vehicle and won an Award of Distinction in the Automotive – Electric Vehicle category for conventional PM components in the Metal Powder Industries Federation's 2023 Design Excellence awards (Courtesy MPIF)

Allowed Copper Content Range	1.5 to 3.9	%
Allowed Carbon Content Range	0.60 to 0.91	%
Theoretical Density at 2% Cu and 0.8% C	7.733	g/cm^3
Typical Porosity	12	%
Typical Density	6.8	g/cm^3
Major Application	Automotive	70% of sales
Yield Strength at 6.8 g/cm^3	410	MPa
Tensile Strength at 6.8 g/cm^3	475	MPa
Tensile Strength at 7.2 g/cm^3	595	MPa
Tensile Strength at 7.7 g/cm^3	645	MPa
Fracture Elongation at 6.8 g/cm^3	2	%
Thermal Expansion Coefficient at 6.8 g/cm^3	6.8	ppm/°C
Elastic Modulus at 6.8 g/cm^3	125	GPa
Bulk Hardness at 6.8 g/cm^3	74	HRB
Unnotched Charpy Impact Energy at 6.8 g/cm^3	8	J/cm^2
Transverse Rupture Strength at 6.8 g/cm^3	890	MPa
Fracture Toughness at 6.8 g/cm^3	20	$MPa\sqrt{m}$
Heat Treated Tensile Strength at 6.8 g/cm^3	755	MPa
Jominy Hardenability, Depth of 50 HRC	< 3	mm
Green Density at 400 MPa Compaction Pressure	6.77	g/cm^3
Green Density at 800 MPa Compaction Pressure	7.45	g/cm^3
Green Strength at 400 MPa Compaction Pressure	21	MPa
Green Strength at 800 MPa Compaction Pressure	50	MPa
Fatigue Endurance Strength at 6.8 g/cm^3	185	MPa
Grain Growth Rate Parameter	0.7	$\mu m^3/s$
Iron-Iron Average Grain Boundary Energy	0.74	J/m^2
Iron-Liquid Copper Average Interface Energy	0.38	J/m^2
Iron-Liquid Copper Mean Contact Angle	10	degrees
Iron-Liquid Copper Mean Dihedral Angle	0 to 10	degrees
Iron+Carbon-Liquid Copper Mean Dihedral Angle	30 to 45	degrees
Liquid Copper Self-Diffusion Rate at 1,150°C	$4.7 \cdot 10^{-9}$	m^2/s
Iron Self-Diffusion Rate at 1,150°C (Gamma-Iron)	$4.5 \cdot 10^{-15}$	m^2/s
Iron Diffusivity in Liquid Copper at 1,150°C	$4.6 \cdot 10^{-9}$	m^2/s
Carbon Diffusion Rate in Gamma-Iron at 1,150°C	$5.0 \cdot 10^{-11}$	m^2/s

Table 1 Nominal properties for FC-0208 in the as-sintered condition

copper is often avoided in steels. The exceptions are precipitation hardening stainless steels and weathering or corrosion-resistant construction steels.

Powder Metallurgy avoids strength degradation by sintering at a temperature where the copper is liquid, but the steel is solid. Contacting steel grains sinter bond to form a rigid three-dimensional network responsible for strength, with copper partially filling inter-grain pores. After sintering, even with 12% porosity (6.8 g/cm^3), iron-copper-carbon steels reach a tensile strength of 475 MPa or more. For comparison, pure iron at 6.8 g/cm^3 density only reaches a tensile strength of 205 MPa. If residual pores cause a problem with fluid containment, the pores can be filled by infiltration using liquid copper, bronze, or other alloys, or impregnation with a cross-linking polymer. Consequently, the iron-copper-carbon alloys are used for applications in automobile engines, transmissions, brakes, and shock absorbers, as well as lawn mowers, hand tools, appliances, hardware, and hydraulic systems [1]. By way of introduction, summary data on the FC-0208 alloy are given in Table 1.

The diffusion data illustrates how liquid copper accelerates sintering. At 1,150°C, iron transport in liquid copper is a million times faster than iron solid-state self-diffusion. This is the reason liquid copper significantly enhances sintering. Sintering below the copper melting temperature lowers the tensile strength [2], so production sintering occurs with peak temperatures from 1,120°C to 1,150°C.

Solubility

The iron-copper and iron-carbon phase diagrams are well established. Carbon is essentially insoluble in copper. Carbon is soluble in gamma iron (γ-Fe), and that solubility increases with temperature from 727°C to 1,153°C. At 1,150°C, copper solubility in gamma iron (γ-Fe) is about 8% while the solubility of iron in liquid copper is about 3%. Both solubilities increase with temperature.

Carbon increases the stability of γ-Fe while slightly reducing the solubility for copper: At 1% C the copper solubility in iron drops to about 7% [3]. Likewise, carbon increases the dihedral angle of liquid copper with respect to iron grain boundaries, reducing liquid penetration and component swelling. Carbon diffusion into γ-Fe is fast since carbon diffuses as an interstitial, with a diffusivity during sintering of $5 \cdot 10^{-11}$ m^2/s.

Copper is soluble in γ-Fe. As copper diffuses into iron there is a progressive loss of liquid, but this is a slow process. At a typical sintering temperature, the self-diffusion rate for γ-Fe during sintering ranges from $1 \cdot 10^{-15}$ m^2/s to $6 \cdot 10^{-15}$ m^2/s. Copper has the same crystal structure as γ-Fe, so it undergoes substitutional diffusion. The process is slow compared to the diffusion rate of iron in liquid copper. While it takes hours for copper to fully absorb into iron, the saturation of iron in liquid copper occurs in minutes. On cooling, the dissolved copper precipitates due to a significant solubility decrease when γ-Fe transforms into α-Fe [4].

> *"Liquid copper penetration of iron grain boundaries occurs in the first few minutes after melt formation. The rate slows as the carbon content increases. The dissolved copper subsequently diffuses from the grain boundaries into neighbouring iron."*

Sintering mechanism

The PM iron-copper-carbon compositions start with mixed elemental powders. Copper melting initiates liquid phase sintering. The Fe-Cu system behaviour is treated by classic models in terms of melting and spreading, diffusion, coarsening, and densification [5-10]. The process consists of overlapping events:

- During heating, graphite dissolves into solid iron after it transforms into γ-Fe
- Copper melts and flows between iron particles, pushing them apart over the first few minutes after melt formation
- Molten copper dissolves iron and penetrates iron grain boundaries, leading to further grain separation over the next few minutes. Some iron dissolves into molten copper
- Liquid copper on the grain surfaces and grain boundaries diffuses into the surrounding iron (it is soluble up to about 8% in the absence of carbon but saturates at a lower concentration if carbon is present). This is a slow step requiring hours at temperature
- Iron dissolved into the melt diffuses and precipitates to induce grain bonding and coarsening where the number of iron grains slowly decline as the overall grain size enlarges
- During cooling, the reduced solubility of copper in iron causes copper precipitation, while carbon also precipitates as cementite (Fe_3C) in α-Fe

Liquid spreading and wetting

During heating, prior to liquid formation, copper spreads on iron by surface diffusion. Then copper melts and collects in the regions between iron grains. Pores can form where the copper particles existed prior to melting because capillarity wicks liquid copper into the smaller intergrain regions. Copper spreading requires that the iron be free of oxides. Thus, sintering is in a reducing atmosphere, containing hydrogen or carbon monoxide, to provide oxide reduction during heating. Without removal of surface oxides, copper spreading is inhibited [10]. Thus, most FC-0208 sintering involves a reducing atmosphere.

If the green density is low, a burst of iron grain rearrangement is possible when copper melts, especially at high liquid contents (40 vol.%). More typically, the particles are compacted and unable to rearrange due to sinter bonding. Instead, copper spreads in the pores between the iron grains. It also dissolves surface asperities. This wetting of copper on iron is a precursor to liquid copper penetration of iron grain boundaries. Both are influenced by carbon as evident in terms of the dihedral angle [11]; Fe-Cu at 1,180°C has a dihedral angle of 0-20° while Fe-Cu-C has a dihedral angle closer to 45°, which means less intergrain penetration.

Liquid copper penetration of iron grain boundaries occurs in the first few minutes after melt formation. The rate slows as the carbon content increases. The dissolved copper subsequently diffuses from the grain boundaries into neighbouring iron. These events start after the liquid forms and continue over the remainder of the sintering run as long as γ-Fe exists.

The fast transport of iron dissolved in liquid copper enables bond formation between neighbouring iron grains, forming a rigid skeletal structure. The bond size is determined by the relative crystal misorientation of neighbouring grains as well as the carbon content. Since steel (iron with dissolved carbon) is stronger than copper, intergrain bond growth is a primary contributor to strengthening.

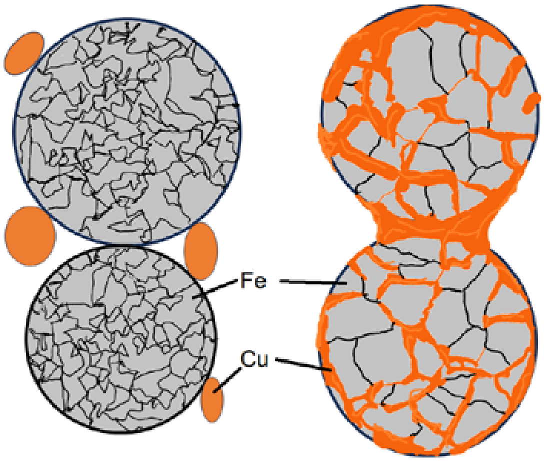

Fig. 2 Cross section sketches: On the left two contacting iron particles prior to liquid formation, and on the right the dilated copper penetrated structure after reaching the liquid phase sintering temperature

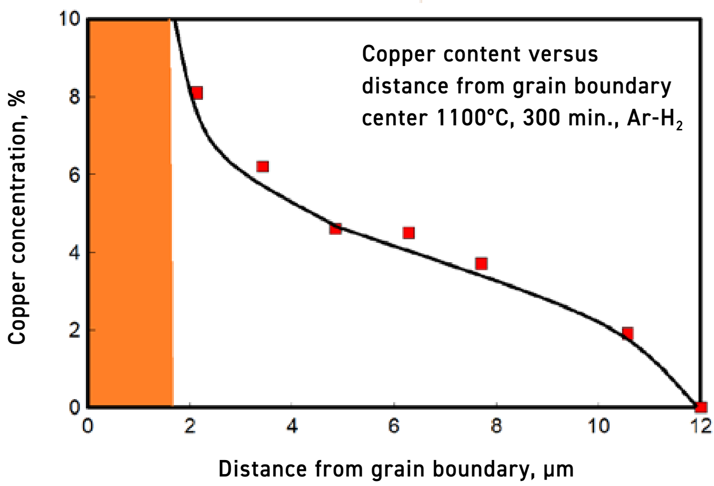

Fig. 3 Plot of the copper concentration from the centre of a copper-filled iron grain boundary after 300 min hold at 1,100°C

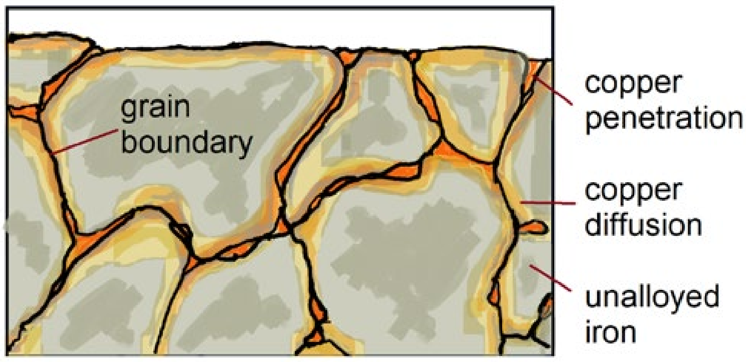

Fig. 4. A sketch of the copper distribution within a powder compact after liquid penetration of intergrain regions and grain boundaries, with subsequent diffusion into the gamma iron perpendicular to the boundaries

Grain boundary penetration

When copper melts, liquid penetrates between the iron particles and grain boundaries, leading to swelling [7, 9, 12]. The process is illustrated in Fig. 2. Initially, liquid copper wets contacting iron particles, causing a dimensional dilation as the particles are pushed apart. This is followed by a slower step of liquid penetration of grain boundaries in the iron grains. The penetration rate ranges from 1-10 µm/min [7, 13], depending on relative crystal misorientation of the contacting grains.

Grain size enlarges with the α-Fe to γ-Fe phase transformation (body-centred cubic to face-centred cubic) near 912°C. The grain size after penetration is larger than the grain size in the starting powder. However, not all grain boundaries are penetrated, due to differences in grain orientation and grain boundary energy.

A slower step is solid-state diffusion of copper into iron, perpendicular to the grain boundaries. Reflecting the diffusion profile, the copper concentration declines with distance from the grain boundary, as plotted in Fig. 3. Even after 300 min at 1,100°C, liquid copper is still present as a grain boundary layer, reportedly a few micrometres in width. Grain boundary penetration accounts for 85% of the observed swelling [14].

Fig. 4 sketches the idea of grain boundary penetration and solid-state diffusion of copper into γ-Fe grains. The latter results in a copper concentration gradient near the grain boundary. For example, after sintering sponge iron with 2% Cu (1,150°C, 60 min) the copper concentration averaged 1.9%, but varied from 0.3 to 3.4% with location [15].

Microscopic pores in the iron particles provide a capillary attraction for liquid copper. For this reason, the surface area of the powder is a predictor of how much copper wicking and dimensional change takes place [16, 17]. Surface connected pores in the iron powder wick molten copper, reducing the amount of grain boundary penetration. Thus, iron powder with a high surface area cuts swelling. Process atmosphere plays a role since oxide reduction during heating forms small surface-connected pores into which molten copper flows by capillary action [18]. For this reason, cyclic oxidation and reduction is a means to increase surface area for improved liquid copper wetting and penetration.

Carbon effect on copper grain boundary penetration

Dimensional expansion is larger as the copper content increases, but carbon has the opposite effect [19]. For Fe-2Cu, pressed to 7.0 g/cm^3 and sintered at 1,120°C for 30 min, the net compact swelling is about 0.2%, increasing to 0.5% at 3% Cu [20]. But as shown in Fig. 5, the swelling declines as the carbon content increases. This data is for 3% Cu with a green density ranging from 6.5 - 7.0 g/cm^3 [22]. A higher green density increases swelling [21].

Swelling is due to the low dihedral angle that induces copper to wet iron grain boundaries. The dihedral angle is distributed, because at the grain level crystallographic orientations are randomly distributed: The dihedral angle varies with the relative misorientation of contacting grains. Carbon reduces wetting and swelling. As illustrated for Fe-10Cu in Table 2, alloying with graphite increases the dihedral angle and reduces copper dissolution into iron. These experiments with 10% Cu show the dihedral angle and amount of undissolved copper increase in proportion to the graphite content [23].

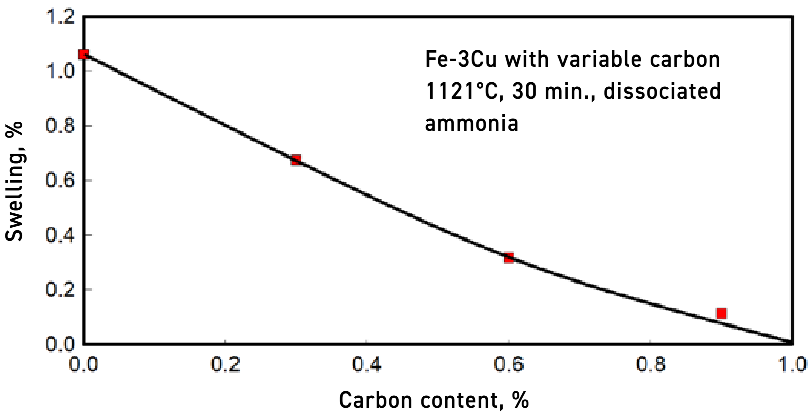

Fig. 5 Data for Fe-3Cu with varying carbon level, giving the swelling versus carbon content for 6.5-7.0 g/cm^3, sintered at 1,121°C for 30 min in dissociated ammonia

Graphite Content %	Dihedral Angle Deg.	Undissolved Copper %
0	5	2.4
0.5	10	3.9
1.0	30	6.2

Table 2 Graphite effect on wetting and dissolution in Fe-10Cu at 1,140°C

Solution: Reprecipitation

After grain wetting and grain boundary penetration, the iron-copper-carbon structure coarsens and densifies, giving shrinkage, especially notable at high liquid contents. Iron grains in contact with liquid copper undergo solution-reprecipitation where the average grain size enlarges over time. Solution-reprecipitation is the main growth mechanism, although grain coalescence and grain boundary migration are observed in iron-copper systems. In coalescence, grains contacting with a low crystal misorientation rotate to align crystal planes (eliminating any grain boundary), and fuse into a single grain.

If there is a chemical difference between contacting grains, then the composition gradient produces grain boundary motion [24]. The pure grain dissolves into the grain boundary liquid and condenses as a saturated iron-copper alloy. The net effect is migration of the liquid-filled boundary. This is termed 'diffusion induced grain boundary migration.' Over time, the average iron grain size enlarges as the number of solid grains declines. A rigid structure forms due to the intergrain bonding that effectively halts densification. Further, the grains undergo shape changes to better fit together, giving a higher packing density to

"If there is a chemical difference between contacting grains, then the composition gradient produces grain boundary motion. The pure grain dissolves into the grain boundary liquid and condenses as a saturated iron-copper alloy. The net effect is migration of the liquid-filled boundary."

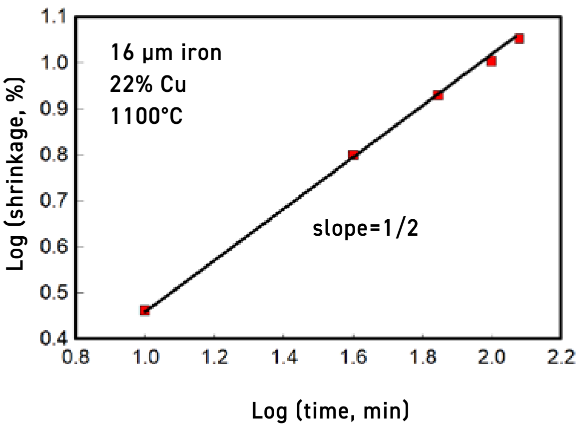

Fig. 6 A plot of the sintering shrinkage versus time on a log-log basis. The shrinkage kinetics agree with the square-root of time behaviour expected for solution-reprecipitation

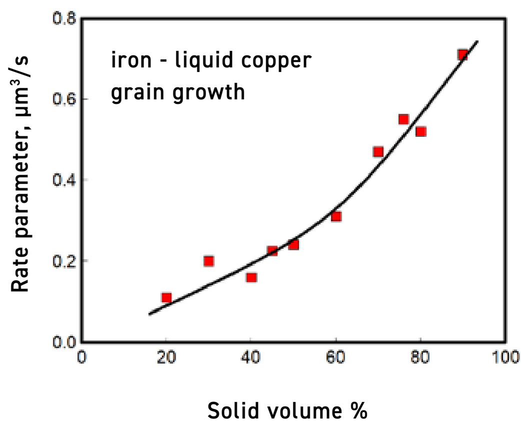

Fig. 7 A plot of the experimental grain growth rate parameter versus the solid volume percentage for Fe-Cu alloys

help eliminate pores. This requires long hold times. Grain growth is not significant during a normal 20-30 min sintering cycle. For example, a 75 µm starting grain size enlarges to 76 µm in 30 min at 1,150°C. Of the three coarsening mechanisms, coalescence, diffusion induced boundary migration, solution-reprecipitation, the latter is dominant in the iron-copper system.

Densification is evident after melt forms. The solid grains (largely iron with dissolved copper) bond, coarsen, and undergo grain shape accommodation to allow repositioning and shrinkage [13, 25]. Asperities and small grains supply iron into the copper-rich liquid, allowing fast diffusion in the liquid, followed by alloy precipitation on large grains. The change in dimension ΔL normalised to the starting dimension L_O gives the shrinkage $\Delta L/L_O$ as a function of time and temperature as follows [6]:

$$\left(\frac{\Delta L}{L_O}\right)^M = \frac{B}{T}\, t\, exp\left[-\frac{Q}{R\,T}\right]$$

where B is a collection of material and geometric constants (atomic volume, atomic vibrational frequency, surface energy, gas constant, and iron grain size), t is the hold time at absolute temperature T, Q is the activation energy, and R is the gas constant. The exponent M reflects the time dependence. This behaviour is plotted on a log-log basis in Fig. 6. The slope M in the above equation is 1/2, some studies report a slope of 1/3.

As full density is attained, shrinkage halts. However, grain growth continues with the median grain size G_{50} enlarging with time as follows:

$$(G_{50})^3 = (G_O)^3 + \frac{K}{T}\, t\, exp\left[-\frac{Q_G}{R\,T}\right]$$

where G_O is the grain size when liquid first forms; K (the grain growth rate parameter) is similar to the term B with the inclusion of a liquid volume fraction term $V_L^{2/3}$ in the denominator, and Q_G is an activation energy applicable to grain growth. The variation with solid content is plotted in Fig. 7. These experiments show 90Fe-10Cu coarsens at the rate of 0.7-0.8 µm³/s at 1,150°C. The coarsening rate increases as the solid content increases since the grains are closer together [25].

While densification by grain rearrangement is fast, it only operates when liquid first forms, if there is sufficient liquid. On the other hand, solution-reprecipitation is relatively slow, but continues as long as the liquid remains [26].

Microstructure

Besides composition and density, copper steel mechanical properties are influenced by microstructure. Three microstructure changes occur during sintering – grain enlargement, grain shape accommodation, and neck growth. The most significant is neck growth, since it is responsible for bonding between the iron grains. This bonding results in the rigid three-dimensional skeleton that provides strength.

Another important microstructure feature is the pore structure – both pore size and pore shape. Large pores are a problem with respect to dynamic properties, such as impact toughness and fatigue strength. The fatigue endurance strength depends on the pore structure [27]. Large copper particle sizes create large pores that lower fatigue endurance strength [28]. Additives such as halides (chlorine and fluorine) and boron in trace amounts promote pore rounding. While pores with angular shapes are typical, rounded pores provide more resistance to crack propagation.

Grain growth

Several factors influence sintered grain size. The α-γ phase transformation on heating nucleates a coarse grain structure. Other factors are alloying, impurities, and heat treatment. Both strength and hardness decrease as the grain size enlarges. The primary grain growth mechanism is iron dissolution, diffusion in liquid copper, and precipitation onto larger iron grains. Small grains disappear as large grains grow, a process sometimes termed 'Ostwald ripening.' Another mechanism is directional motion of the liquid-filled regions, dissolving iron on one side and precipitating iron alloyed with copper on the opposite side [13]. In cases where the neighbouring iron grains align with similar crystal orientations, no grain boundary exists and coalescence results in the larger grain absorbing the smaller grain.

Typical for liquid phase sintering, grain size is larger as the liquid (copper) content decreases [15]. This is illustrated in Fig. 8 for five copper concentrations. The cause is a shorter diffusion distance for low liquid contents.

The grain size distribution conforms to a Weibull model [6]:

$$F(g) = 1 - exp\left[\ln\left(\frac{1}{2}\right) g^{P}\right]$$

Where *F(g)* is the cumulative fraction of grains of size g or smaller, with $g = G/G_{50}$, and *P* is close to 3, but it depends on how grain size is measured. Usually, the random grain intercept technique is applied and *P* = 2. So, while the median grain size enlarges, the distribution in sizes remains self-similar. Only the median size is needed since the size distribution follows the above expression. In the FC-0208 system considerable grain growth occurs at the α-Fe to γ-Fe transformation, prior to reaching the sintering temperature.

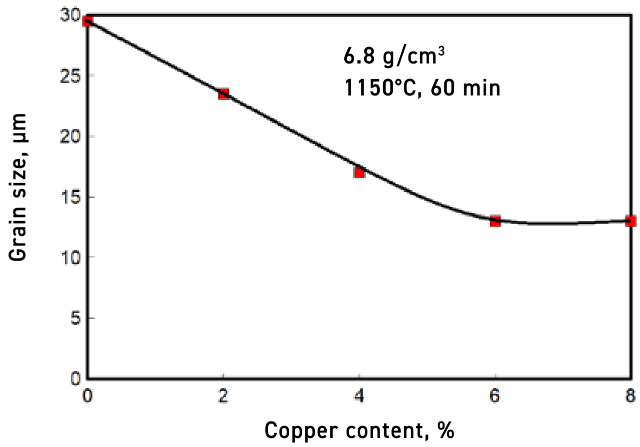

Fig. 8 Sintered grain size as a function of copper content for liquid phase sintered Fe-Cu alloys

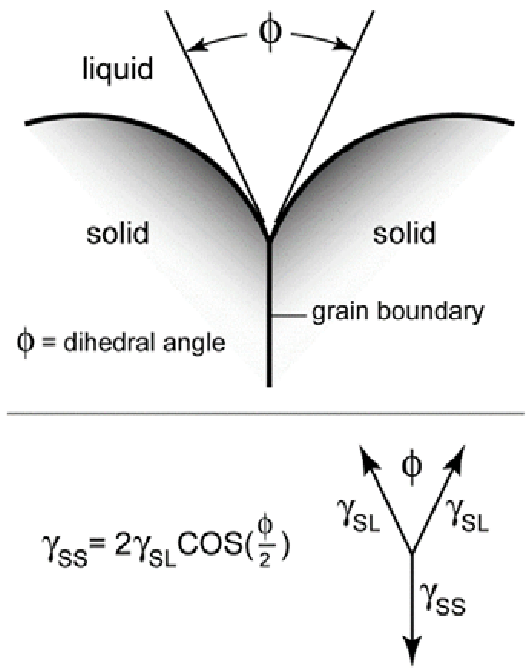

Fig. 9 The dihedral angle is defined by the vector equilibrium between the grain boundary ($_{SS}$) and the wetted solid-liquid ($_{SL}$) surfaces

Dihedral angle

The dihedral angle ϕ represents a balance of the solid-solid grain boundary energy γ_{SS} with the solid-liquid surface energy γ_{SL},

$$\gamma_{SS} = 2\,\gamma_{SL}\cos\left[\frac{\phi}{2}\right]$$

The basis for this equation is shown in Fig. 9. The degree of iron grain-grain bonding is directly related to the dihedral angle. A low dihedral angle induces more liquid penetration and more swelling when copper melts. Since the iron-iron bond size is small with a low dihedral angle (the bond size depends on sin(ϕ/2)), the material has a low sintered strength [29].

Depending on the carbon level, the Fe-Cu dihedral angle ranges

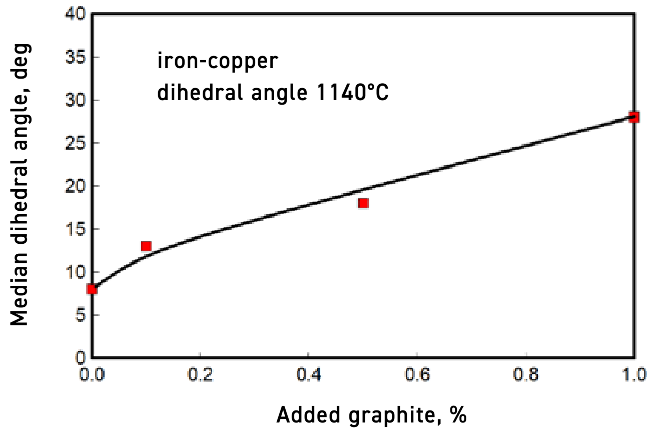

Fig. 10 The median dihedral angle after 1,140°C for 60 min for liquid copper and iron grain boundaries for Fe-10Cu with varying graphite contents

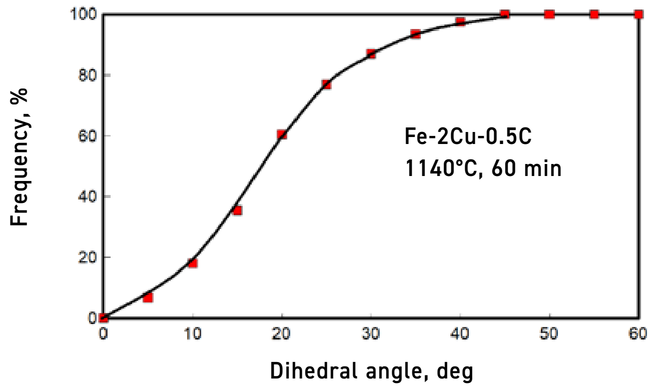

Fig. 11 A plot of the cumulative dihedral angle distribution for Fe-2Cu-0.5C measured after 60 min at 1,140°C. The median is 18°

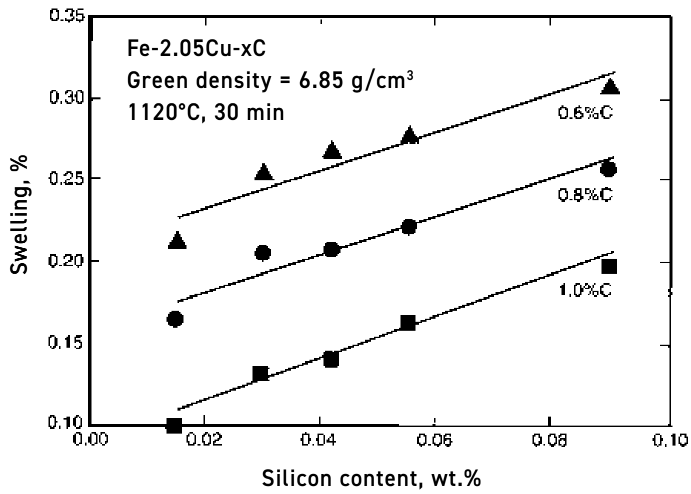

Fig. 12 Data for Fe-2Cu alloys and three carbon levels, illustrating the dimensional change associated with silicon. A higher silicon level getters more oxygen, allowing increased liquid copper penetration of iron grain boundaries, resulting in more swelling

from a few degrees in the absence of carbon to 30° or more with 1% carbon, as plotted in Fig. 10.

A high dihedral angle (from carbon alloying) reduces grain boundary penetration and enlarges the iron-iron sinter bond size. Both carbon alloying and bond size enlargement combine to increase sintered strength. Sintering in a reducing atmosphere removes oxides, lowers the dihedral angle, and improves wetting and grain boundary penetration by liquid copper [10, 23]. Carbon in the alloy results in a higher dihedral angle and less swelling [3].

The dihedral angle is distributed since grain boundary energy varies with crystal misorientation. In a sintered body, a wide variety of misorientations form due to random grain contact, leading to variation in the dihedral angle as plotted in Fig. 11 for Fe-2Cu-0.5C, giving a median of 18° [23].

As the dihedral angle increases there is less swelling. Both oxygen and carbon play a role, where the former is removed using a reducing atmosphere. It is possible to add a strong oxide former to influence wetting. For example, a higher silicon level getters more oxygen, allowing for increased liquid copper spreading on the iron grain boundaries [30, 31], resulting in more swelling. This is demonstrated in Fig. 12 for Fe-2Cu with 0.6, 0.8, and 1.0% carbon.

Pore structure

Uniaxial die compaction is used to form most PM green bodies, giving an inhomogeneous pore structure [32]. Die wall friction causes a change in pressure and green density with distance from the top and bottom punch surfaces. Fig. 13 illustrates this, using micrographs taken of polished faces at the top, side, and bottom. The porosity (black) and pore spacing are obvious differences. Quantitative microscopy of 7.0 g/cm³ FC-0208 in the green condition shows that pore size varies with respect to the compaction direction. In turn, pore orientation is a factor in dimensional change since

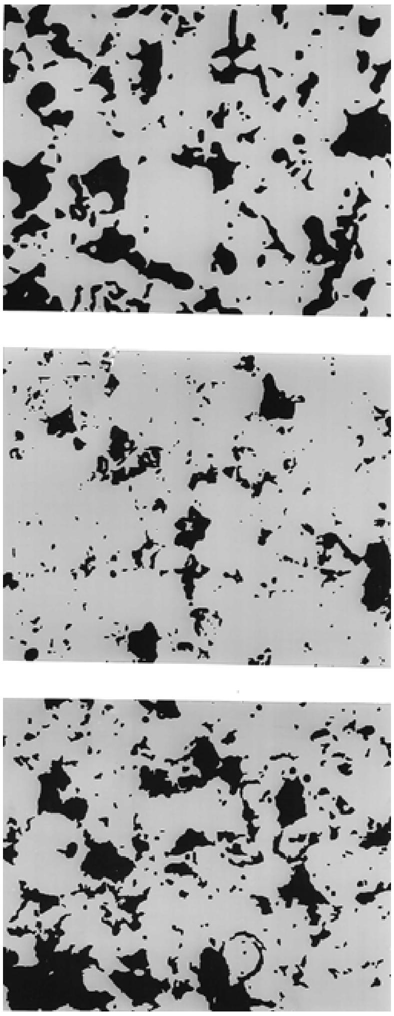

Fig. 13 Comparative polished sections taken from the top, side, and bottom of an FC-0208 after sintering. The pores are black

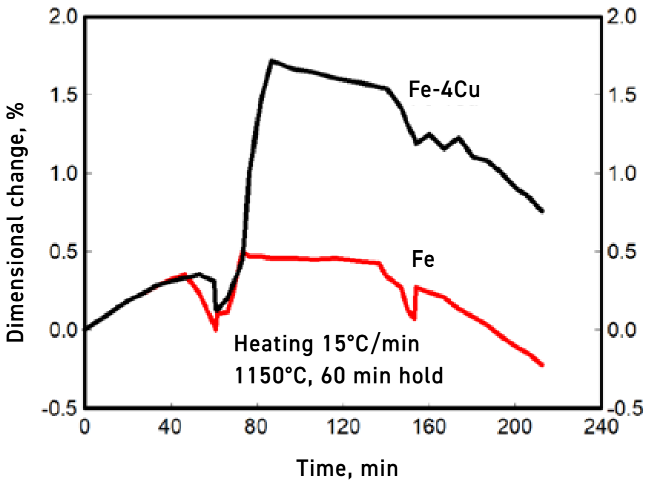

Fig. 14 Comparative dilatometry traces for water atomised iron and the same powder mixed with 4% Cu, heated at 15°C/min to 1,150°C for one hour. Pure iron gives net shrinkage while Fe-4Cu shows net expansion

pores attempt to spheroidise during sintering. For an anisotropic pore shape, this causes a different dimensional change in the axial versus lateral direction.

Dimensional change

Dilatometry measures compact size *in situ* to identify the events responsible for dimensional change [11, 16, 33-35]. Thermal expansion initially causes swelling, then the iron phase transforms from α-Fe to γ-Fe and gives a distinctive dimensional change: contraction during heating and expansion during cooling. The face-centred cubic γ-Fe phase is higher in density, so the phase change near 912°C produces contraction. Carbon dissolution into γ-Fe contributes a small shrinkage, while copper melting provides a more noticeable change. Molten copper induces expansion by penetrating between iron particles and diffusing into γ-Fe. Carbon dissolution in γ-Fe suppresses copper induced swelling by increasing the dihedral angle. This allows more iron-iron bonding. The dimensional behaviour is illustrated by the dilatometry curves in Fig. 14 [36]. After reaching the sintering temperature, the isothermal hold allows for sintering densification. On cooling, the events reverse with residual copper solidifying (shrinkage event), iron reverting to α-Fe (body-centred cubic) in a swelling event, with copper and cementite precipitating out of solution.

For iron-copper-carbon alloys, dilatometry curves are assembled from the individual phases – pure iron, iron + copper, iron + carbon, and iron + copper + carbon. The shrinkage and size change data are collected versus changes in powders, green density, heating or cooling rate, peak temperature, and hold time, as well as atmosphere and additives. Fig. 15 is an example of the FC-0208 dimensional oscillations during sintering. Three replications are shown, reaching 1,120°C for 30 min, with heating and cooling at 10°C/min in a $80N_2$-$20H_2$ atmosphere. Notations on Fig. 15 provide identification of the main events. These are

"For iron-copper-carbon alloys, dilatometry curves are assembled from the individual phases – pure iron, iron + copper, iron + carbon, and iron + copper + carbon. The shrinkage and size change data are collected versus changes in powders, green density, heating or cooling rate, peak temperature, and hold time, as well as atmosphere and additives."

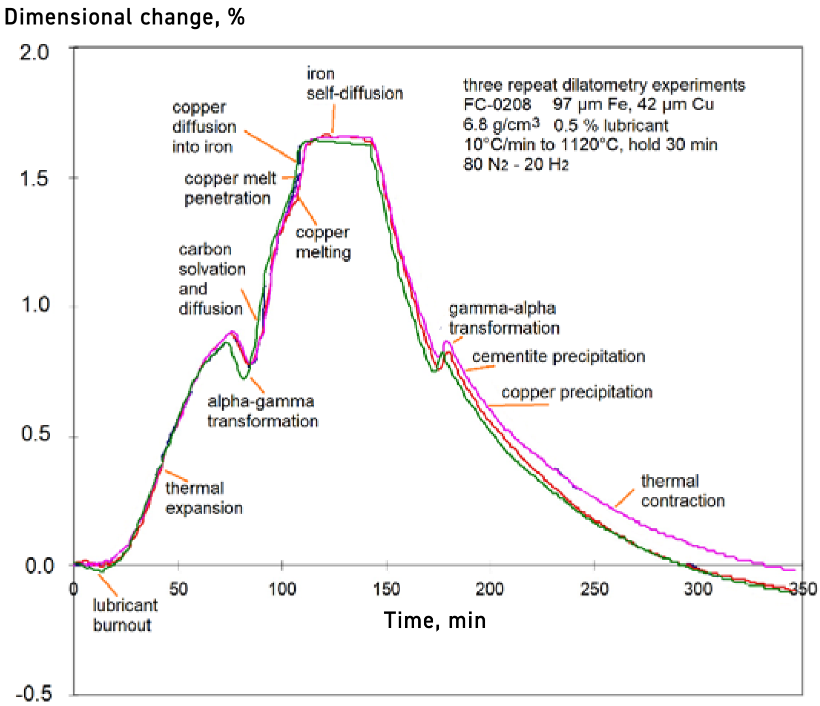

Fig. 15 Three replica dilatometry experiments on FC-0208 with notations on the individual events

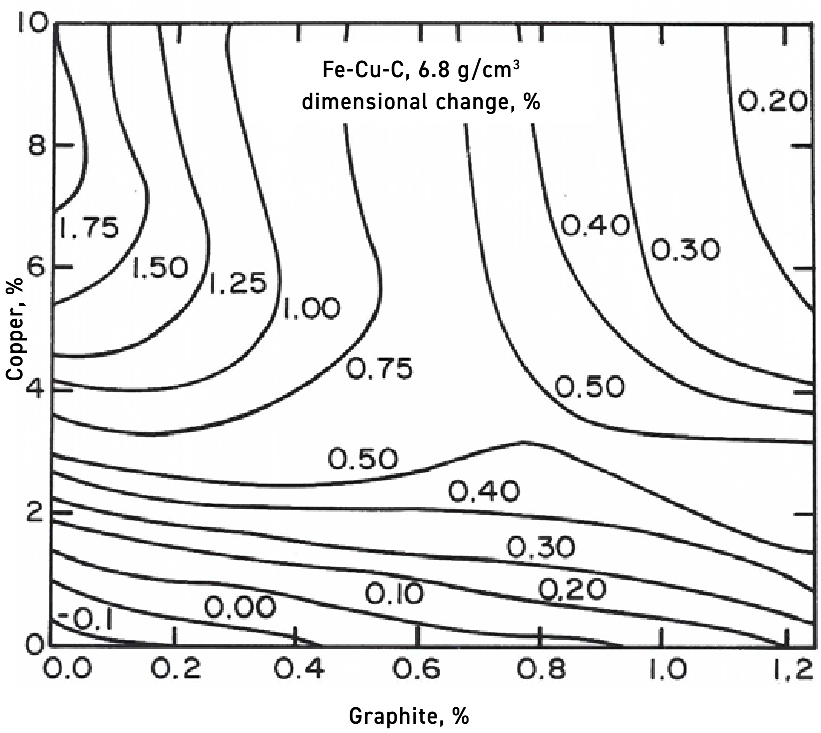

Fig. 16 Dimensional change contours versus copper (vertical) and graphite (horizontal) contents for compacts of 6.8 g/cm³ green density, sintered at 1,120°C in dissociated ammonia

identified in individual runs, starting with pure iron and repeating with increasing complexity. Additional insight comes by plotting the first derivative of the shrinkage, termed the shrinkage rate, versus time or temperature. Without dilatometry, the option is to record only the final dimensions, thereby missing the complex combination of expansion and contraction events during heating.

Note dimensional change for FC-0208 is relatively small after sintering. The combination of compaction springback and sintering dilations result in a component size slightly larger than the tool size [19, 28, 37]. Due to compaction gradients, there is a difference in dimensional change in the pressing direction versus the perpendicular lateral direction. In some combinations, one direction swells while the perpendicular direction shrinks. It is common to correct sintered dimensions by machining, or repressing. Repressing provides a slight boost to the density, hardness, and strength.

Component size control is complicated by several interactions during sintering. These include atmosphere effects on carbon loss or gain, heating and cooling rate effects on phase change and precipitation, hold time and peak temperature effects on copper diffusion, and particle sizes roles for the iron, copper and graphite. Composition and green density factors are dominant. The composition behaviour is illustrated by Fig. 16 for a 6.8 g/cm³ green density using one set of powders. Note that in this case at 2% Cu the swelling increases as the carbon content increases.

Other reports with coarser iron powder give a different behaviour [21]. For 7.1 g/cm³ sintered density the swelling $\Delta L/L_0 = 0.75 - 0.5$ (%C), where %C is the carbon content. At lower densities the swelling is smaller. In practice, swelling is kept small via proprietary combinations of powder types, composition, and processing parameters.

Powder	Compaction [MPa]	Density [g/cm³]	Sintering [°C, min, atmosphere]	Dimensional Change [%]
80 µm Fe	414	6.8	1,120, 30, H_2-N_2	0.13 to 0.16
85 µm Fe	580	6.9	1,120, 25 min, N_2	0.12 to -0.30
74 µm Fe	490	6.9	1,130, 20, Endothermic	0.00
80 µm Fe	463	---	1,150, ---, N_2-H_2	0.20
---	560	---	1,120, 25, Endothermic	0.44
80 µm, 40 µm Cu	690	7.15	1,120, ---, N_2-H_2	0.35
---	800	7.34	1,120, 20, N_2-H_2	0.07
97 µm Fe, 42 µm Cu	---	6.80	1,120, 30, N_2-H_2	0.05
68 µm Fe, 16 µm Cu	640	7.1	1,150, 30, N_2-H_2	0.00
--- *	750	7.1	1,120, 60, Endothermic	0.00
80 µm Fe	500	7.0	1,135, 60, H_2	0.00
80 µm Fe**	700	7.11	1,120, 60, Ar	0.00
<150 µm Fe	550	6.89	1,130, 20	0.25
*Fe-4Ni-1.5Cu-0.5Mo-0.6C **Fe-4Cu-1.2C-0.45P				

Table 3 Examples of FC-0208 processing cycles and dimensional change (--- indicates missing data)

Example dimensional change data
Sintering is associated with strengthening and elimination of surface area, often with the annihilation of pores. The iron-copper-carbon alloys exhibit combinations of sintering shrinkage and swelling. Net component dimensional change depends on several processing details [3, 19, 21, 22, 28, 35, 38-43]. Some example reports for FC-0208 are collected in Table 3.

Dimensional change depends on the composition, powder characteristics, and processing details. Based on thirty-eight reports for FC-0208 with an average 6.9 g/cm³ green density, the mean swelling is 0.26% when sintering is between 1,120-1,150°C for 20-30 min. Higher temperatures (up to 1,260°C) or longer times (up to 180 min) reduce the swelling. By contrast, injection moulded FC-0208 using carbonyl iron powder exhibits 14.5% shrinkage [44]. This is a case where the low initial density enables sintering densification. Such behaviour is illustrated in Table 4 showing the green density influence on dimensional change for Fe-10Cu sintered at 1,165°C for 20 min [45].

Dimensional change for FC-0208 is adjustable via additives, powder selection, and processing parameters such as hold time, peak temperature, and green density [28]. Smaller particle sizes and lower green densities favour shrinkage. Alloying with Mo or Ni improves the heat-treated strength, but dimensional change increases [40, 42]. For example, adding 0.85% Mo to Fe-2Cu-0.8C increases swelling to 0.38% (1,120°C, nitrogen-based atmosphere). Additions of MnS, P, Si, or Ni also increase swelling.

Zero dimensional change alloys
The FC-0208 alloy is attractive because of several factors, including low cost, high strength, and little dimensional change. Sintering at 1,120-1,150°C is compatible with a stainless steel belt furnace. Such units are the *de facto* standard for the industry. Cost escalates as sintering temperature increases since the furnace belt must be fabricated from refractory metals or ceramics. The near zero dimensional change evident for some entries in Table 3 allows for easier tooling design.

Green density [g/cm³]	Sintered density [g/cm³]	Dimensional change [%]
4.8	5.2	- 3.2
6.4	6.4	0.0
7.2	6.8	1.5
7.4	6.9	2.2

Table 4 Green and sintered density for Fe-10Cu compositions at 1,165°C after 20 min in hydrogen (negative entry indicates net shrinkage)

Processing variables

As knowledge accumulates on sintering, it is evident that essentially every parameter has an influence on final dimensions and properties. It is unfortunate that many reports on sintering FC-0208 fail to provide full documentation. In spite of the missing details, the available data allows for an assessment of how each variable influences the sintered characteristics.

Iron powder characteristics
Useful iron powders are produced using water atomisation. A typical water atomised iron powder has a median particle size near 100 μm and contains about 0.1% oxygen and 0.05% carbon. An apparent density near 2.9 g/cm^3 is typical. Compaction pressure determines the green density. For example, one powder grade responds as follows:

- 355 MPa → 6.6 g/cm^3
- 490 MPa → 6.9 g/cm^3
- 660 MPa → 7.2 g/cm^3

A standard test involves reporting the green density using 414 MPa compaction pressure. The corresponding green density and green strength is 6.82 g/cm^3 and 13 MPa. Admixed lubricants increase the green density by about 1.6%. Besides lubrication, other factors improve green density, such as increased dwell time during compaction, cyclic pressurisation, and powder heating.

Copper powder characteristics
On heating, the melt forms at the copper particles. That melt wicks into the surrounding iron structure, first along the grain surface, then into grain boundaries, and finally dissolves into the iron since it is soluble in the gamma phase. Because of the flow of copper into iron, the sites formerly consisting of large copper particles form pores. For this reason, it is appropriate to select smaller copper particle sizes to reduce the pore size at those sites. A typical copper powder has a median size of 26 μm and apparent density of 2.3 g/cm^3. One option is to coat the iron powder with copper. This is possible by electroplating or dusting copper powder onto the iron followed by sinter bonding, resulting in partially prealloyed powder.

Graphite powder characteristics
Carbon is a potent strengthening agent in ferrous systems. For example, Fe-2Cu has a strength of just 250 MPa after vacuum sintering at 1,100°C for 30 min, while Fe-2Cu-0.8C under equivalent processing reaches 550 MPa [46]. The graphite particle size and graphite type influence dimensional change [39, 41]. Graphite is available from natural sources or is man made. Further it is either crystalline or amorphous, and available over a wide size range from 0.5-60 μm. When milled, graphite forms a flake due to its anisotropic hexagonal close-packed crystal structure. During sintering some graphite is lost due to reaction with residual oxygen in the powders, so the final carbon content is lower than the added graphite quantity [46, 47].

Smaller graphite particles, with more surface area, dissolve into γ-Fe more rapidly during heating. This results in reduced copper swelling and a higher sintered density. Small graphite particles, nominally below 15 μm, favour shrinkage, while larger graphite particles favour swelling. When compacted at 490 MPa with a sintering cycle of 20°C/min to 1,130°C and 20 min hold, zero dimensional change occurs with 20-25 μm graphite particles [39].

Mixing
The normal protocol is to mix the iron, copper, graphite, and lubricant powders prior to compaction. Separation because of differing particle characteristics occurs with transport or vibration, so mixing is performed at the compaction site. An added polymer helps avoid segregation and dusting. Mixing is performed in a rotating container. Mixing for 10-30 min in a partially-filled mixer is sufficient for homogenisation without work hardening the iron powder. Conceptually it is possible to prealloy the iron with copper [36].

Coated iron powder option
Coated powders are a means to ensure copper homogeneity in the compacted powder, avoiding segregation in powder handling [14, 44]. An electrolytic coating provides homogeneity to the copper distribution, giving net sintering shrinkage. Early sintering occurs at the copper-copper contacts and on liquid formation there is a burst of shrinkage. This is the opposite from the situation with mixed powders, which exhibit swelling on melt formation. For coated small iron particles, the greater the copper content, the more sintering shrinkage when the copper melts.

In the cases where carbonyl iron powder is coated with copper, the small particle size induces sintering densification at temperatures as low as 845°C. When heated to 1,200°C for 60 min in hydrogen the density exceeds 95% of theoretical. For 2% copper, the sintered strength is 420 MPa with 18% elongation (72 HRB). Although the starting powder contained 0.78% carbon, a high initial oxygen level resulted in a final

"Carbon is a potent strengthening agent in ferrous systems. For example, Fe-2Cu has a strength of just 250 MPa after vacuum sintering at 1100°C for 30 min, while Fe-2Cu-0.8C under equivalent processing reaches 550 MPa."

0.01% carbon, resulting in the high ductility. The combination of small particle size and copper coating allowed for a high sintered density.

Lubricant

Lubricants are added to the powders to improve compaction tool life and to minimise stress during ejection after pressing. Several lubricants are in use. Common choices are 0.2 to 1.0% of zinc stearate, ethylene-bis-stearamid, stearic acid, or mixtures of these [38,48]. If a halide (chlorine or fluorine) is in the lubricant, then there is increased activity during burnout that improves sintered strength.

Ideally lubricants burn-off cleanly during heating to the sintering temperature. No large lubricant role is detected with respect to strength, although a lower lubricant content (0.5%) is typical. Die wall lubrication is an option where the tooling is sprayed with a lubricant mist prior to powder fill. This slows the compaction press and encounters some geometry limitations.

Compaction pressure and green density

As noted above, iron powders increase green density as the compaction pressure increases. At low green densities the particles rearrange and densify when copper melts, but for high green densities, the opposite occurs with swelling. As a demonstration of higher compaction pressures leading to higher green densities, Table 5 gives data from a 15 µm carbonyl iron powder and 68 µm water atomised iron powder.

Compaction Pressure [MPa]	Carbonyl Green Density [g/cm^3]	Water Atomised Green Density [g/cm^3]
280	6.30	6.70
420	6.60	6.95
560	6.85	7.20
700	7.08	7.33

Table 5 Compaction pressure effect on green density for carbonyl and water atomised iron powder

Most iron-copper-carbon PM components are formed by high pressure uniaxial die compaction. The green density slightly improves with higher pressures, but the gain is small. This is because the iron powder work hardens to resist further densification. Compaction is generally at pressures higher than 500 MPa, and in some cases ranges up to 740 MPa. After ejection, elastic stress in the component relaxes, leading to dimensional expansion known as springback, often 0.2% or more. Dimensional change during sintering is determined from either the tool size to sintered size or the green size to sintered size [19]. The springback strain is the difference. The net change depends on the iron powder characteristics, but typically falls in the range from 0.1 to 0.3% swelling from the die size.

"Most iron-copper-carbon PM components are formed by high pressure uniaxial die compaction. The green density slightly improves with higher pressures, but the gain is small. This is because the iron powder work hardens to resist further densification."

Alloying changes the theoretical density of the iron-copper-carbon alloys. Wrought iron has a theoretical density of 7.86 g/cm^3. Due to impurities, such as oxygen, iron powder is slightly lower in theoretical density, about 7.7 g/cm^3. The FC-0205 alloy has a theoretical density of 7.77-7.78 g/cm^3 while the FC-0208 alloy is slightly lower at 7.73-7.74 g/cm^3 [27, 49]. If a lubricant is added to prolong tool life, then it reduces the theoretical density.

One option for improved green density is to heat the compaction tooling [43]. Heating to 60°C increases the green density by about 0.1 g/cm^3. This is effective for thinner sections and small components. Otherwise, it is necessary to preheat the powder and the tool, a technology termed warm compaction. Temperatures up to 120°C and pressures up to 800 MPa result in 7.48 g/cm^3 green density with 40 MPa green strength. When a cellulose binder and lubricant are added, the theoretical density drops to 7.34 g/cm^3. When sintered at 1,120°C for 20 min in nitrogen-hydrogen, the dimensions are tightly clustered with a coefficient variation of 0.067%.

In die compaction, dimensional change differs between the vertical (axial) and lateral directions [43]. High pressure compaction along the vertical axis flattens the pores and particles. In sintering, the natural tendency is for the pores to spheroidise while the residual strains relax. The anisotropic pore shape results in a difference in dimensional change depending on orientation with respect to the compaction direction [14, 15, 37]. The actual behaviour depends on the processing factors, including the iron particle size, surface area, green density, and copper particle size. As an example, with a standard water atomised iron powder the role of the copper particle size is as follows (sintering for 30 min at 1,120°C):

- 0.17% vertical swelling using 15 µm copper powder;
- 0.54% vertical swelling using 26 µm copper powder.

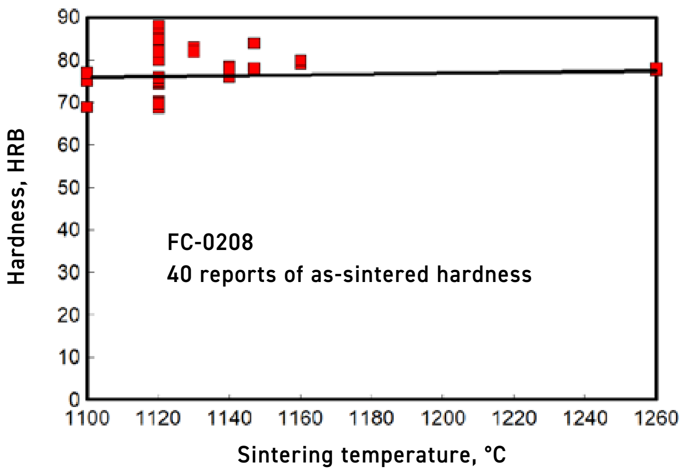

Fig. 17 Plot of as-sintered hardness versus sintering temperature for nominally 20-30 min hold

> *"Carbon control can be a challenge since both oxygen in the powder as well as the process atmosphere remove carbon, lowering the sintered strength. Heat treatments are less effective when carbon is lost."*

Atmosphere effect

For FC-0208 the sintering atmosphere is predominantly nitrogen-hydrogen blends, with 5% or more hydrogen [50]. The atmosphere plays a role in dimensional change, although other factors tend to be more important [34]. Endothermic atmospheres are based on natural gas. Since the molecular makeup of natural gas varies over time, the resulting atmosphere composition also varies. An endothermic atmosphere consists of nitrogen with hydrogen and carbon monoxide as reducing agents, and traces of carbon dioxide and methane (for example, 42% N_2, 33% H_2, 24% CO, and less than 0.4% of CO_2 and CH_4) [51]. If the hydrogen level is low, sooting occurs as the hydrocarbons are not fully combusting and form graphite flakes instead of volatile species.

Carbon control can be a challenge since both oxygen in the powder as well as the process atmosphere remove carbon, lowering the sintered strength. Heat treatments are less effective when carbon is lost. In an extreme case, starting with 0.78% C in a carbonyl iron powder, the sintered carbon level drops to nearly zero due to decarburisation during heating [44]. One of the reaction products is carbon monoxide, especially for smaller iron powders. The reaction product stabilises residual pores and prevents full densification. An excess of carbon is effective in removing oxygen during heating prior to pore closure. Atmosphere adjustments to control final carbon rely on knowing the starting carbon level, peak temperature, heating rate, moisture, and methane content [35].

Other atmospheres used for FC-0208 include pure hydrogen, argon-hydrogen, vacuum, and dissociated ammonia ($25N_2$-$75H_2$). The dewpoint of the sintering atmosphere determines decarburisation and in turn that influences dimensional change, with a further influence depending on the furnace and its performance.

Heating rate

Production heating rates average 15-17°C/min. Improved sintered properties are possible with heating rate linked to the rate of sintering, with slow heating during periods of rapid shrinkage and fast heating as the shrinkage rate declines [52]. The concept is called rate controlled sintering but is rarely employed in practice.

Sintering temperature

A first requirement for the sintering temperature is to melt the copper. Sintering then takes place at a temperature between 1,120-1,150°C. The average temperature employed in 85 reports is 1,136°C. Higher temperatures cause slightly more sintering shrinkage or less swelling, but from a statistical standpoint the mechanical properties are not sensitive to sintering temperature in the 1,120-1,150°C range. This is evident in Fig. 17 for sintering 20-30 min at temperatures ranging from 1,100-1,260°C. The sintered hardness has no correlation to temperature over this range (correlation coefficient = 0.003). A similar lack of correlation is evident with the tensile strength, reflecting a dominant role from factors such as carbon content and final density.

Sintering time

Sintering time is generally in the range from 20-60 min. The average over forty-seven reports is 40 min at temperature. Dimensional change is small as a function of hold time. For Fe-5Cu, compacted at 300 MPa formed from water atomised powder of approximately 100 µm particle size, the sintering shrinkage versus hold time is almost unchanged from

5-60 min at 1,150°C [53]. A majority of the dimensional change occurs during heating to the sintering temperature, as evident in Fig. 18. This data was gathered using a dunk heating cycle, reaching the peak temperature in about 2 minutes. Note the consistent difference in axial and radial shrinkage and the small dimensional change after the heating transient.

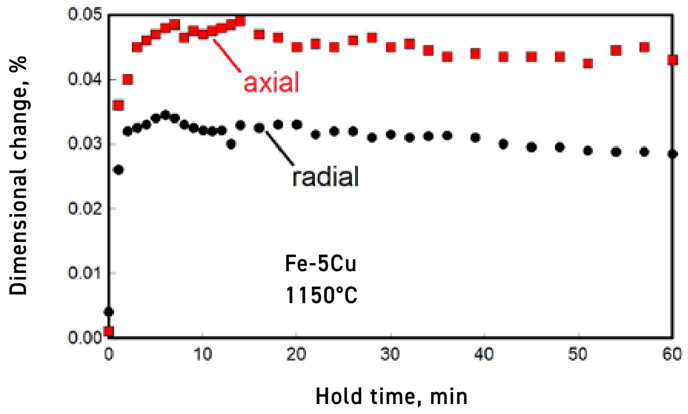

Fig. 18 Showing the dimensional change for Fe-5Cu at 1,150°C in an atmosphere of Ar-7H_2. The bulk of the change occurs on heating to the peak temperature in the first minute of sintering.

Cooling Rate

Cooling rate controls the carbide and copper precipitation events important to strength and hardness. Furnace cooling is typical, but some furnaces allow for rapid cooling to increase hardness and strength; faster cooling gives smaller precipitates to improve strength. The critical temperature range for precipitation control is from 850-600°C.

In trials with FC-0208, heated to 1,120°C for 25 min in nitrogen, a net shrinkage of 0.3 to 0.6% is observed, but more swelling occurs with rapid cooling; about 0.17% for 0.4°C/min to 0.20% for 1.5°C/min [19]. Hardness increased about 10% with the faster cooling rate.

Microwave heating

Microwave heating was applied to ceramics in the early 1980s. About fifteen years later, the idea spread to metal powder components embedded in a ceramic powder [54, 55]. Microwave heating is fast. To avoid graphitisation of lubricants and binders, a preliminary slow heating cycle is required prior to microwave heating. To ensure uniform heating, the components are embedded in a susceptor, often granular silicon carbide.

As noted, sintered strength and hardness are improved by fast cooling and this is one advantage from small batch microwave heating. The same benefit is realised in traditional lab furnaces using dunk cycles. Shorter hold times help retain carbon, adding to the strength, but lower ductility [56]. Table 6 gives FC-0208 density data for 20 min hold in a microwave. The carbon level is reduced due to oxygen from the starting powders. The microwave sintering results are typical to the alloy; for example, FC-0208 with conventional sintering to 7.0 - 7.1 g/cm^3 exhibits an average hardness of 80 HRB.

For iron, microwave sintering shrinkage is lower after the alpha-gamma phase transformation. By about 1,250°C the shrinkage is about the same as with conventional sintering, especially after a 60 min hold. For a green density of 7.0 g/cm^3, extending the microwave sintering to 1,300°C for 20 min increases the sintered density to 7.33 g/cm^3 [57]. Carbon loss can be a problem, resulting in low hardness [58]. For example, FC-0208 using water atomised 78 µm iron with 31 µm copper and 9 µm graphite, sintering to 6.8 g/cm^3 at 1,120°C for 30 min in a 95% nitrogen atmosphere resulted in a hardness of 32 HRB because the carbon level dropped to 0.4%.

A susceptor is required to convert the microwave radiation into heat. Silicon carbide rods or granules are selected for this role. This is effectively the same as traditional sintering where electric current is passed through silicon carbide heaters. When directly compared, conventional and microwave sintering results are comparable.

Surface area effect on dimensional change

Dilatometry shows the sintering shrinkage and swelling events compensate for each other with respect to dimensional change. Surface pores in sponge iron powder are one means to control swelling [8]. Effectively, the surface area of the iron powder provides an interface for copper spreading without a dimensional change [16, 17]. Based on surface area, different iron powders can be mixed to supply essentially zero dimensional change.

Peak Temperature [°C]	Green Density [g/cm³]	Sintered Density [g/cm³]	Hardness [HRB]
1100	7.07	7.06	77
1140	7.00	7.00	78
1260	7.05	7.07	78

Table 6 Density data from FC-0208 microwave sintered for 20 min

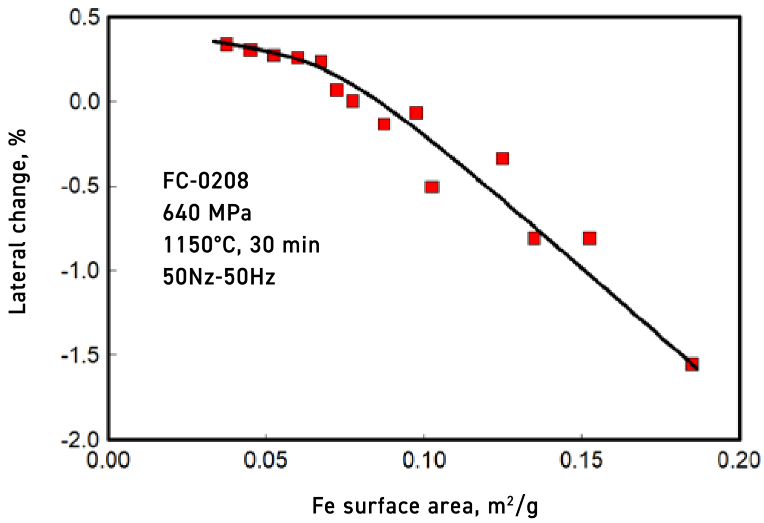

Fig. 19 Plot showing the lateral dimensional change (negative is shrinkage) versus iron powder surface area for FC-0208 compacted at 640 MPa and sintered at 1,150°C for 30 min in a hydrogen-nitrogen atmosphere

For example, sintering 80 µm Fe, 16 µm Cu, and 8 µm graphite compacted to 7.0 g/cm^3 at 640 MPa and heated at 15°C/min to 1,150°C for 30 min in an atmosphere of 50N_2-50H_2 showed the lateral shrinkage behaviour plotted in Fig. 19 [59]. The behaviour fits a polynomial:

$$\Delta L/L_O = 0.45 - 0.65\,S - 50.6\,S^2 + 10.4\,S^3$$

where S is the iron powder specific surface area in m^2/g, giving zero dimensional change at 0.09 m^2/g (about 85 µm Fe). This curve is sensitive to the green density [60].

Mechanical properties

Several reports provided data on the mechanical properties using the processing parameter combi-

Parameter	Mean	Median	Range	Standard Deviation
Fe Particle Size, µm	65	72	5 to 117	28
Cu Particle Size, µm	51	22	11 to 200	67
Compaction Pressure, MPa	541	608	269 to 740	158
Lubricant Amount,%	0.75	0.75	0 to 1	0.3
Lubricant Type*	EBS	EBS	Die wall to ZnSt	
Sinter Density, g/cm^3	6.96	7.10	5.80 to 7.71	0.30
Heating Rate, °C/min	17	20	2 to 75	15
Peak Temperature, °C	1,135	1,120	1,050 to 1,330	41
Hold Time, min	36	30	5 to 180	35
Atmosphere,% N_2	68	89	0 to 95**	36
Cooling Rate, °C/min	114	20	13 to 1,100	317
Additives, if used	MnS, B, Ni, Mo, P			
Elastic Modulus, GPa	122	126	85 to 155	29
Yield Strength, MPa	418	410	220 to 965	126
Tensile Strength, MPa	532	505	250 to 1,034	170
Hardness, HRB	80	80	32 to 108	14
Fracture Elongation,%	2.6	2.4	0 to 9	1.8
Fatigue Endurance Limit, MPa	200	202	52 to 345	58
Unnotched Impact Energy, J	12	8	2 to 61	14
Transverse Strength, MPa	1,050	1,029	410 to 1,378	220

*EBS = ethylene-bis-stearamid, ZnSt = zinc stearate **typically hydrogen or endothermic gas as the balance

Table 7 Summary of processing variables related to mechanical property reports on iron-copper-carbon alloys nations mentioned earlier. Maps show sintered strength versus copper and carbon contents for otherwise fixed processing [41]. For this study, a total of eighty-two reports were gathered, but some were incomplete with respect to processing details. Table 7 provides a sense of the sintered properties based on several studies [1, 2, 20-22, 27-30, 38, 41, 44, 46, 47, 56-77]. This analysis uses the mean (average) and median (centroid) to focus the discussion. Dispersion about these is reflected in the range and standard deviation. The range shows the lowest and highest reported and the standard deviation reflects the dispersion about the mean. For example, the mean or average iron particle size is 65 µm, with half of the studies relying on 72 µm or smaller powder. The size range varied from 5-117 µm and the distribution showed a 28 µm standard deviation (68% of the reports were from 37-93 µm).

Tensile strength and hardness are reported in most studies, while parameters such as the copper particle size are reported less frequently. About one-third of the reports involve additives, with 0.3% to 0.5% MnS or 0.45 to 0.60% P (as a compound) being the most common. Other additives include boron, nickel, and molybdenum.

Predominantly the mechanical property data are collected in the context of press-sinter processing. The highest strengths came from controlled cooling rates or heat treatments. Some specific combinations are summarised in the Appendix.

Hardness

Composition (alloying), density, and heat treatment are dominant influences on the sintered hardness. Statistical analysis of the as-sintered hardness shows density is the most significant factor, followed by carbon content, then copper content. Based on 52 reports in the 6.9-7.0 g/cm^3 sintered density range the mean hardness is 80.4 HRB. Hardness falls if carbon is lost. The hardness exceeds 100 HRB for high sintered densities or post-sintering heat treatments [61, 63, 66, 72, 73]. With respect to microstructure, the hardness is proportional to the inverse square-root of the grain size [77]. In the as-sintered condition, a grain size of 1 µm corresponds to about 92 HRB, while a more typical 100 µm grain size gives 75-85 HRB.

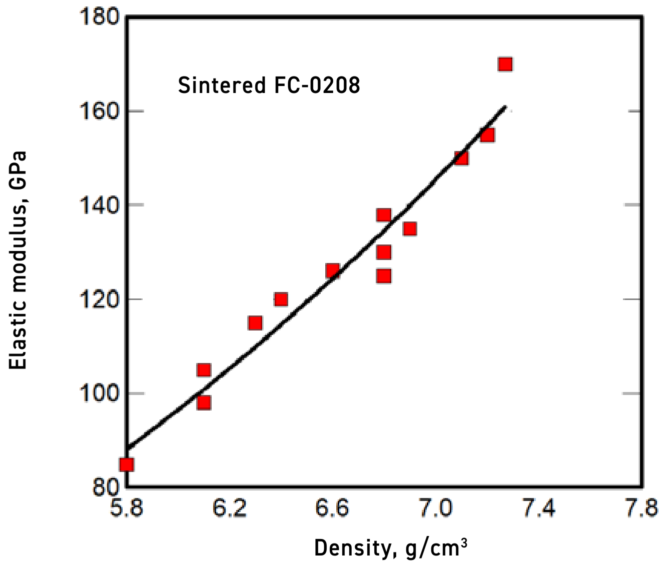

Fig. 20 Plot of the elastic modulus versus density for FC-0208. The power law dependence on fractional density is shown by the black line

Elastic properties

Full density ferrous PM alloys exhibit an elastic modulus close to 200 GPa [78]. Porosity lowers the elastic modulus. For FC-0208, elastic modulus ranges from 85 GPa (5.8 g/cm^3) to 170 GPa (7.3 g/cm^3). Because the copper content is low in FC-0208, the elastic modulus is little changed from that of pure iron. Further, there is no significant change reported with heat treatment. The linkage between elastic modulus E and fractional density f takes the form of a power law relation [49],

$$E = E_O \, [f]^N$$

where E_O is the full density elastic modulus (190-210 GPa), and N is a factor that depends on microstructure parameters such as pore shape. Using 200 GPa for the full density E_O gives N = 2.7, with a correlation coefficient of 0.954 and standard error of the estimate at 18 GPa. Fig. 20 shows this equation fit to the experimental data.

"Composition (alloying), density, and heat treatment are dominant influences on the sintered hardness. Statistical analysis of the as-sintered hardness shows density is the most significant factor, followed by carbon content, then copper content."

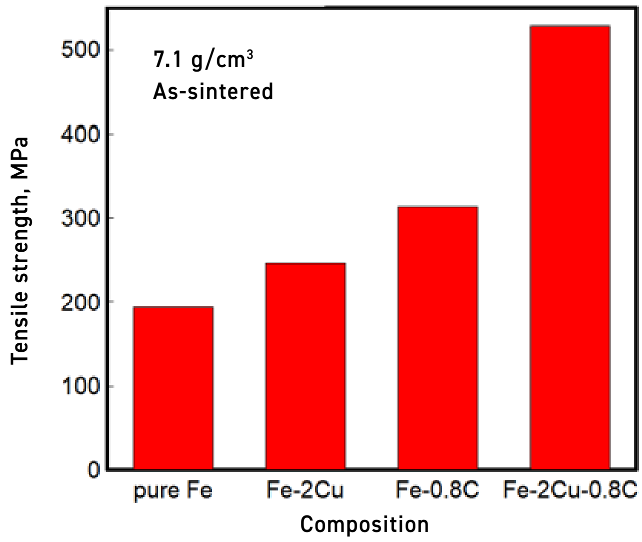

Fig. 21 Tensile strength for sintered iron, Fe-2Cu, Fe-0.8C, and FC-0208 at approximately 7.1 g/cm³. Fabrication involved compaction at 640 MPa and sintering at 1,150°C for 30 min in 50N$_2$-50H$_2$

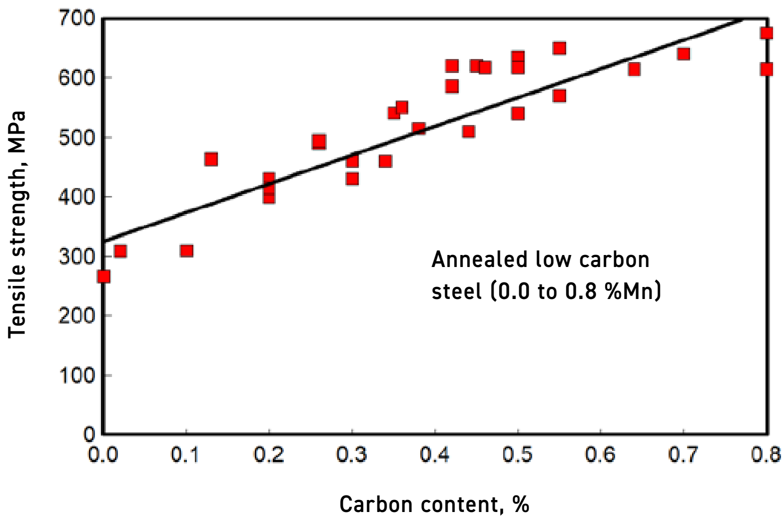

Fig. 22 The tensile strength of various full-density Fe-C compositions in the annealed state (Mn contents vary from 0-0.8%)

Alloying effects on strength and ductility

Pure iron is soft and requires alloying to promote strength, hardness, wear resistance, and other desirable properties. One form of pure iron is Armco Iron (named after the American Rolling Mill Company, now AK Steel International). At full density it has a yield strength of 133 MPa, tensile strength of 266 MPa, and 45% elongation. Porosity reduces these values.

To understand the relative alloying contribution to mechanical properties, Fig. 21 plots the tensile strength at 7.1 g/cm³ density (92%) [59]. The tensile strength for sintered pure iron is 194 MPa or 73% of wrought. Effectively, 8% porosity causes a 27% strength reduction. Likewise, fracture elongation is 45% for wrought iron, but drops to 21% for 7.1 g/cm³ sintered iron; 8% porosity reduces ductility 53%. The addition of copper or carbon increases strength, and the effect is additive for FC-0208.

Carbon is a potent strengthening agent. This is evident for full density iron in the annealed condition, as plotted in Fig. 22 [78]. The annealed condition effectively reflects the situation after sintering.

For an annealed full-density carbon steel, the linear trend for the ultimate tensile strength σ_U as a function of carbon content is as follows:

$$\sigma_U = 330 + 470\ (\%C)$$

where %C is the final carbon content. The above relation predicts 706 MPa as the peak tensile strength for annealed, full density for Fe-0.8C. Copper is less potent as a strengthening agent [47].

Nitrogen also increases strength and hardness, while reducing ductility. In sintered ferrous alloys, carbon is from added graphite while nitrogen is from the sintering atmosphere. Unfortunately, the final nitrogen content is rarely reported.

For the FC-0208 composition, alloying effects are approximately linear. In one example, with 0.67% C (sintered to 7.1 g/cm³ at 1,120°C in 90N$_2$-10H$_2$) the hardness (*HRB*), yield strength in MPa (σ_Y), and ultimate tensile strength in MPa (σ_U) follows the below relations with respect to the copper content (*%Cu*) [47]:

$$HRB = 73.9 + 6.38\ (\%Cu)$$
$$\sigma_Y = 206 + 131\ (\%Cu)$$
$$\sigma_U = 176 + 197\ (\%Cu)$$

Besides alloying and density, other factors influencing the mechanical properties are heat treatment and post-sintering infiltration. Example combinations are summarised in the Appendix.

The impact toughness shows considerable range due to a high sensitivity to density, oxidation, test temperature, and sample surface finish. The impact toughness is higher (14-16 J/cm² at 6.9 g/cm³) for oxide-free samples [21, 79], but just 2 J/cm² at 5.8 g/cm³ [76].

Properties such as the impact energy and fatigue strength are sensitive to porosity, pore shape, and the testing method. These in turn point to a role of powder characteristics and sintering temperature. At 75% density (5.8 g/cm³), the fatigue endurance limit is 90 MPa, and it increases to 230 MPa at 93% density (7.2 g/cm³). In wrought ferrous alloys the fatigue strength is usually about half the tensile strength. For FC-0208 the ratio is lower, at 39%, over the 6.7-7.2 g/cm³ density range [2, 21, 70].

Composition and fractional density are dominant factors in the as-sintered strength data. In the industry, the FC-0208 composition is allowed to vary in copper and carbon contents (1.5-3.9% Cu and 0.60-0.91% C). Assuming yield and tensile strength are linear functions of copper or carbon contents, then the FC-0208 test data fits a function of composition (*%Cu* and *%C*) and fractional density *f* as follows:

$$\sigma = f^N [a + b\%Cu + c\%C]$$

where *a*, *b*, *c*, and *N* are given in Table 8 for both the yield and tensile strength. The value of *a* is defined by the strength of unalloyed full-density iron at 133 MPa yield and 266 MPa tensile.

The predicted tensile strength using this equation at 6.8 g/cm³ (88% dense) is 495 MPa, slightly lower than the 505 MPa median strength reported in Table 7. If FC-0208 is taken to full density, then the expected yield strength is 751 MPa and expected tensile strength is 807 MPa.

The mechanical properties decline with heating, what is termed thermal softening. Example data is given in Table 9 for a density of 6.62 g/cm³ [68]. An elevated temperature increases the ability of dislocations to cross-slip, contributing to dynamic recovery and less work hardening, corresponding to lower strength.

	a	b	c	N
Yield Strength (MPa)	133	175	335	5.7
Tensile Strength (MPa)	266	140	300	3.5

Table 8 Coefficients and exponential factors for predicting FC-0208 strength data

	Test Temperature		
Property	20°C	150°C	300°C
Elastic Modulus, GPa	126	112	96
Yield Strength, MPa	418	345	233
Tensile Strength, MPa	510	450	350
Elongation,%	1.4	1.5	2.1

Table 9 Thermal softening data for FC-0208 at 6.62 g/cm³

"Several modifications to the Fe-Cu-C system are in use. Additives include boron, phosphorus, nickel, bronze, tin, molybdenum, chromium, manganese, manganese sulphide, and various carbides."

The FC-0208 strength is sometimes reported using a three point bending test adopted from ceramics, where it is called the modulus of rupture. In PM, it is called the transverse rupture strength (TRS). Underlying this test is the assumption of brittle fracture. The maximum surface tensile stress when fracture occurs is reported as the TRS strength, but sample deflection must be less than 0.4% strain prior to fracture. For FC-0208, the TRS fracture stress far exceeds the yield strength, hence plastic flow occurs prior to fracture. For an elongation of 2-3%, the deflection prior to fracture induces work hardening, thereby providing the high apparent strength. When the test is applied to FC-0208, the average TRS is 1065 MPa or nearly twice the tensile strength [70]. Most PM alloys exhibit a TRS that is about 1.6x the ultimate tensile strength [31]. For transverse rupture strength, the measurement scatter is reported as 86 MPa.

Modified alloys

Several modifications to the Fe-Cu-C system are in use. Additives include boron, phosphorus, nickel, bronze, tin, molybdenum, chromium, manganese, manganese sulphide, and various carbides [19, 30, 46, 62, 63, 69, 80, 81]. Additives are used to promote specific processing or property changes. For example, phosphorus in low concentrations (≈ 0.5%) increases the sintered density and hardness at the expense of the ductility. Dimensional change is close to zero using 700 MPa compaction pressure, and sintering at 1,120°C for 60 min.

Manganese sulphide (MnS) is added to sintered steels to extend

Alloy	Density [g/cm³]	Processing	Yield Strength [MPa]	Tensile Strength [MPa]	Elongation [%]
Fe-2Cu-1Ni-0.45P	6.8	691 MPa, 1200°C, 30 min, H_2	220	470	7
Fe-2Cu-1Mo-0.45P	6.8	691 MPa, 1200°C, 30 min, H_2	255	480	7
Fe-1Ni-1Mo-0.5C-0.4B	7.7	1,200°C, 30 min, H_2	---	560	2
Fe-4Cu3P	7.0	1,100°C. 30 min, vac	---	550	3

Table 10 Mechanical properties of some modified alloys

cutting tool life [82]. For FC-0208, the relative tool life extension is nearly eight-fold. Unfortunately, sulphur interferes with carbon dissolution and contributes to increased swelling [83]. For example, with no MnS the dimensional change is 0.05% swelling. But when 3% MnS is added swelling increases to 2.63% (sintered at 1,130°C for 20 min in a mixture of endothermic gas and hydrogen). Other sulphur sources, such as FeS, also induce expansion. Such data presents a dilemma between minimised dimensional change and improved cutting tool life after sintering.

Nickel and molybdenum are used to reach higher strengths via post-sintering heat treatments. Sinter hardening is an approach where cooling from the sintering temperature is intentionally controlled to tailor properties [19]. Higher strength arises by sintering at 1,250°C to promote a homogeneous distribution of the alloying additions. While copper assists sintering by forming a liquid phase, additives such as Ni and Mo remain solid and are slow to homogenise.

Tin and bronze allow for a lower sintering temperature [80, 84, 85]. However, the compacts are formed without carbon, resulting in low sintered properties. For example, Fe-3Cu-2Sn sintered at 950°C for 60 min in hydrogen delivers a tensile strength of 200-260 MPa depending on the tin particle size. High copper contents, such as 15Cu-2Sn, fail to reach 300 MPa tensile strength. When carbon is added, the tensile strength declines. In spite of many demonstrations using tin or bronze additions, the sintered properties are inferior compared to FC-0208.

One option is to use partially prealloyed iron powder. These powders bond the alloying elements directly to the iron powder, thereby resisting separation during handling. As expected, the mechanical properties are sensitive to the carbon content. A few examples of modified alloys are given in Table 10 [46, 69, 86]. In some instances, the interaction of the alloying elements with carbon leads to lower strength or lower ductility. Another difficulty is the dimensional change; swelling up to 0.7-1% is possible.

Boron is a potent additive to iron-copper-carbon alloys [62, 63, 86]. At low concentrations it aids densification because it segregates to the iron grain boundaries to form a liquid phase. Higher sintering temperatures are needed since the Fe-B eutectic temperature is 1,174°C. The microstructure change is shown in Fig. 23. The liquid phase rounds and shrinks the pores, leading to densification, but ductility remains low.

Boron treated Fe-Cu alloys reach up to 1200 MPa tensile strength when forged. However, carbon contents are kept low to avoid embrittlement. One option is to source the carbon from an admixed carbide, such as Cr_3C_2. This keeps the carbon out of solution at lower temperatures. During sintering the carbide decomposes

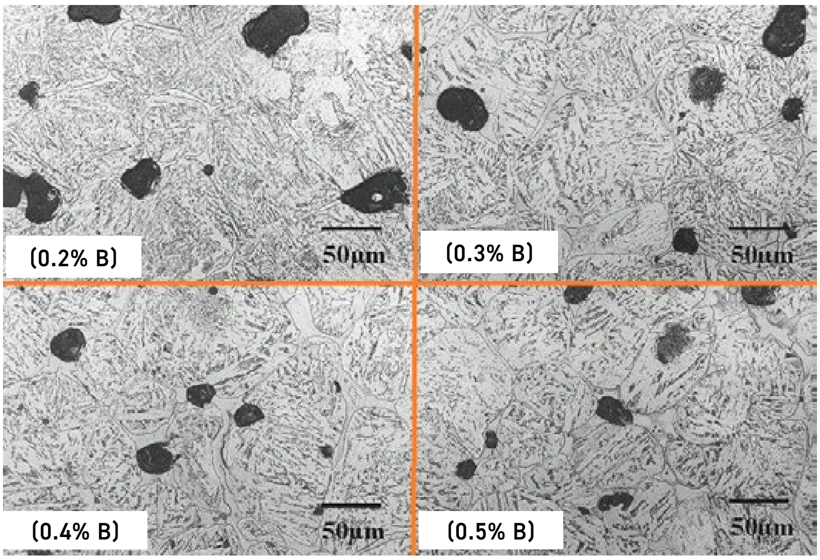

Fig. 23 These four images illustrate the pore rounding and densification associated with boron additions to FC-0208

to release carbon. The sintered strength ranges from 370 MPa (8% Cu, 0.15% B, no carbon) to 900 MPa (5% Cu, 0.15% B, 0.2% C).

Sintered density and residual porosity effects

Porosity is a key factor in determining mechanical properties. Sintered iron without alloying shows a 27% reduction in tensile strength between full density and 7.1 g/cm^3 (decreasing from 266 MPa to 194 MPa with 9% porosity). Likewise, FC-0208 strength and ductility decline with porosity. Full density as-sintered tensile strength is nominally 800 MPa, and heat treatments reach to 1000 MPa.

For FC-0208, the data relating strength to fractional density is confounded by other factors, such as composition and additives (MnS, P, Ni), as well as microstructure. Heat treatment is another factor. Analysis of the mechanical properties shows the ultimate tensile strength σ_U changes with composition and fractional density as presented above in Equation 9. Table 11 gives the average tensile strength and average elongation for FC-0208 by density level from 6.7-7.2 g/cm^3. The number of reports for each density ranges from 2-8. Adjustments to these values are possible via additives, heat treatments, or rapid cooling from the sintering temperature.

Ductility is generally low and scattered. For sintered ferrous alloys, relative elongation ε fits the following relation [87]:

$$\varepsilon = \varepsilon_0 \frac{f^{3/2}}{[1 + C\,(1-f)^2]^{1/2}}$$

where f is the fractional density and ε_0 is the full density elongation. The parameter C reflects the sensitivity to porosity and is equal to 1600 for ferrous alloys. Assuming full density ductility for FC-0208 at 15% results in a prediction of 2.5% elongation at 6.8 g/cm^3 while the measured average is 1.5%.

Density [g/cm^3]	Average Tensile Strength [MPa]	Average Elongation [%]
6.7	400	1.3
6.8	475	1.5
6.9	494	2.1
7.0	500	2.8
7.1	595	4.0
7.2	595	2.3

Table 11 Average tensile strength for various density levels

Heat treatments, cooling rate effect

Copper steels are responsive to post-sintering heat treatments. Example cycles are summarised in Table 12. On cooling from the sintering temperature, the copper solubility drops when iron transforms from γ-Fe to α-Fe. Accordingly, the copper precipitates in a dispersion of submicrometre (25-125 nm) inclusions [15, 88]. However, the typical short sintering cycle restricts copper diffusion into the iron, so full hardening is not seen in the grain interior. The time-temperature-transformation diagram for Fe-4Cu (no carbon) suggests precipitation starts at 750°C in 1 s. The onset occurs at longer times as the hold temperature declines. Simultaneously carbon precipitates as cementite (Fe_3C). Accordingly, various combinations of time, temperature, and cooling rate are used to increase strength after sintering.

Sinter hardening is an option where cooling from the sintering temperature is controlled to induce the desired heat treatment. Cooling rates of 2-5°C/s are possible with properly designed furnaces. Depending on alloying, the tensile strength can exceed 1100 MPa, but ductility is limited to about 2% elongation.

A few efforts to examine hardenability using the Jominy end-quench test find FC-0208 is essentially not responsive [74, 89].

Toughness testing issues

Impact testing shows a low fracture energy for FC-0208. For FC-0208, the median impact energy is 8 J/ cm^2, but the test variation is high. The impact toughness increases with sintered density, ranging from 8 J/ cm^2 at 6.5 g/cm^3 to 12 J/cm^2 at 6.9 g/cm^3. The alloy is notch sensitive, so testing is performed on unnotched samples [75, 90]. One report of 61 J/cm^2 relies on infiltration with a proprietary alloy

Alloy	Solutionise	Hardening Cycle	Hardness
Fe-2Cu	Furnace cool 75°C/min	500°C, 200 min	68 HRB
Fe-1.8Ni-1.8Cu-0.5Mo-0.6C	915°C, oil quench	175°C, 60 min	118 HRB (42 HRC)
Fe-2Cu-0.8C	Sintering	water quench	104 HRB (65 HRC)
Fe-8Cu-0.7C	900°C, 60 min, water quench	500°C, 60 min	---
Fe-8Cu-0.15B-0.1C	900°C, 60 min	180°C, 120 min	---

Table 12 Example heat treatment cycles applied to iron-copper-carbon alloys

Density [g/cm³]	R = -1	R = 0	R = 0.5
5.3	32		
5.8	90		
6.3	75		
6.7	188	133	98
6.7	161*		
6.8	216		
6.9	217	140	107
6.9	128**		
6.9	189*		
7.0	201		
7.0	241	157	121
7.1	229	167	
7.2	230		
7.2	260*		
*average of four reports **axial loading			

Table 13 Density and R (maximum to minimum stress ratio) value influence on fatigue endurance strength in MPa

[66]. Over a temperature range from -80 to 150°C, the impact energy exhibits little change [79]. However, there is sensitivity to the sintering furnace atmosphere, possibly reflecting a nitrogen role.

Fracture toughness varies with alloying but usually K_{Ic} is in the range from 17 to 23 MPa√m. The higher values correspond to higher density (7.2 g/cm³) and more copper (3% Cu) [65]. The general assumption is fracture toughness scales with the yield strength.

Infiltration or hot isostatic pressing (1,050°C, 100 MPa, 30 min) are means to close pores and improve toughness [90].

Fatigue testing and issues

Most of the fatigue strength data are for FC-0208, are taken at ten million cycles. The alloy is sensitive to surface conditions, such as pores, oxides, and even surface marking [75, 79]. Additionally, large and irregular surface pores reduce fatigue life [42]. This sensitivity is used to justify unnotched samples for fatigue and impact testing.

Several fatigue tests are applied to FC-0208. These include axial tension-compression, bending, and rotating loading. Further, the load cycle, reflected by the *R* value or minimum to maximum stress ratio, varies in the data reports. Some reports are for 50% chance of survival, while others are for 99% or 99.9%. The rotating beam test is the most common for sintered steels. In that test *R* = -1 (full compression to full tension on each rotation). Table 13 gives examples of density and *R* value with respect to the endurance strength (average stress for failure in 10 million cycles) [2, 21, 47, 67, 70, 75, 79]. Density is an important factor, while less important are the powder characteristics and sintering conditions.

In the 6.8-7.2 g/cm³ density range the values average 215 MPa. In some applications, the material is subjected to surface treatments such as blackening (oxide formation), shot peening, electroplating, infiltration, or surface marking – which all influence the fatigue behaviour.

Post sintering treatments

After sintering the component size and properties are adjustable via heat treatments, infiltration, machining, densification, sizing, or surface treatments, such as shot peening. Heat treatments are able to improve tensile strength to the 1000 MPa level with 2% elongation [66].

Infiltration

For components that must be leak free, an option is to infiltrate the sintered body with copper or a copper alloy. Bronze is a lower temperature option versus pure copper. Nickel in the infiltrant improves strength but increases distortion [91]. Generally, infiltration with Cu-Ni alloys improves strength due to the elimination of pores. The FC-0208 composition increases tensile strength from 529 MPa (as-sintered 7.07 g/cm³) to a 677 MPa tensile strength with infiltration to 7.81 g/cm³ [59]. Fracture elongation increased from 5 to 6%. Iron is included in the infiltrant alloy at about 2 to 3% to curtail surface erosion on the component caused by iron dissolution on melt formation.

Capillary forces pull the liquid metal into the pores. The depth of infiltration varying with the square-root of time after melt formation [92-94]. The specific rate depends on the pore size, but for example compacts sintered using 120-150 µm iron infiltrate to a depth of 35-40 mm in 4-5 s after the copper melts. Ignoring gravity, the depth of infiltration H varies with the hold time t, pore size d, melt viscosity η (3.9 mPa·s for liquid copper), and copper-iron surface energy γ (0.38 J/m²for liquid copper) as follows:

$$H = \left[\frac{\gamma\ d\ t\cos(\theta)}{\pi^2\ \eta}\right]^{1/2}$$

with θ being the contact angle of liquid on solid; for Fe-Cu it is essentially 0°, so $\cos(\theta)$ is unity. Usually there is only a small mechanical property benefit since the infiltrant is comparatively weak. A typical strength after infiltration is in the 450-500 MPa range.

Hot Isostatic Pressing

Hot Isostatic Pressing (HIP) is rarely applied to the FC-0208 composition, largely due to cost. Hot Isostatic Pressing at 1,050°C and 100 MPa for 30 min closes pores in previously sintered material [90]. This requires encapsulation in a ductile metal or glass that softens prior to reaching the peak temperature. If the sintered density is about 95% of theoretical, then no container is required. Strength improves with hot isostatic pressing, reaching 625 to 650 MPa. The HIP temperature is the most important variable with regard to densification.

Powder forging

Powder forging relies on a press-sinter precursor step to prepare a porous forging blank. That blank is hot compressed to full density using a high strain-rate strike. Even a copper-free low carbon steel, Fe-0.5C, exhibits 600 MPa strength after powder forging [96]. Likewise, forged FC-0208 reaches high strength, as illustrated in Table 14 [76]. Note the fatigue strength increases dramatically with densification, increasing from 168 MPa at 6.65 g/cm^3 to 460 MPa at full density.

Property	As Sintered	Powder Forged	Heat Treated
Density [g/cm^3]	7.0	7.7	7.7
Hardness [HRB]	78	103	109
Yield Strength [MPa]	410	870	625
Tensile Strength, [MPa]	555	980	1,130
Elongation [%]	2	15	10
Impact Energy [J/cm^2]	8	4	4
Fatigue Strength [MPa]	230	470	460

Table 14 Mechanical properties for FC-0208 as-sintered, after powder forging, and after forging followed by heat treatment

Final comments and research suggestions

The field of Powder Metallurgy has rationalised alloys, design features, and production processes around applications. For example, 17-4 PH (AISI 630) stainless steel components are predominantly fabricated by Powder Injection Moulding and sintered to form small, complex shapes. High vanadium carbide content tool steels are predominantly fabricated into ingots by Hot Isostatic Pressing. Tubular porous stainless steel filters rely on cold isostatic pressing and high temperature sintering. Likewise, iron-copper-carbon is fabricated by die compaction and liquid phase sintering to form automotive engine components and other structural devices.

The mechanism for liquid phase sintering FC-0208, and the competing chemical reactions that occur during sintering, are well understood. Unlike other metallic systems, FC-0208 has a low sensitivity to peak temperature and hold time, as long as the copper melts. A high sintered density is beneficial and largely arises from a high green density, since there is little sintering densification. Accordingly, the route to high sintered properties involves attaining a high green density. This is possible using slower compaction strain rates, cyclic pressurisation, high compaction pressures, heated tooling or other warm compaction options, and tailored particle size distributions. In one comparison of four particle size distributions, a bimodal blend gave the highest fatigue strength due to a small and uniform pore size [27]. Along these lines, research is needed to identify particle size distributions that optimise specific mechanical properties such as impact toughness or fatigue strength. One option is to employ computer simulations to link

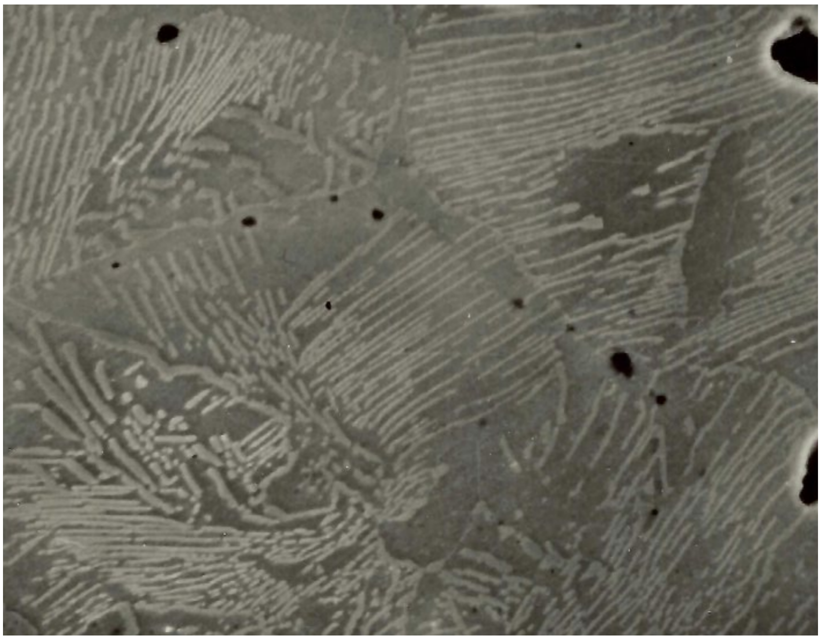

Fig. 24 Scanning electron microscopy image of cross-sectioned FC-0208 doped with 0.01% boron. The system was compacted to 6.8 g/cm^3 and sintered at 1,120°C for 30 min in a nitrogen-hydrogen atmosphere

pore size distribution to particle size distribution and compaction parameters [97]. Discrete element analysis and finite element analysis both would be helpful in such a study. Further, the pore structure is adjustable via processing and composition factors, such as the addition of low concentrations of boron as illustrated in Fig. 24. The resulting mechanical properties are attractive and justify focused research.

Two tests used for FC-0208 need critical evaluation. The impact toughness test using unnotched samples is not correlated to the standard V-notch Charpy test. Likewise, the transverse rupture strength is suspect since the FC-0208 is ductile and undergoes deflection and work hardening prior to fracture. Further, it appears the test results are adjustable via sample preparation details: polishing and bevelling edges improve the test results, but the standard test methods do not specify such conditions.

Automotive applications are often linked to the fatigue behaviour. With respect to fatigue, the property reports are far from uniform. Statistical studies are appropriate with controlled surface conditions to ensure repeatable results. Indeed, testing should replicate the use conditions; if the use condition is as-machined, then fatigue testing should be on samples with the same surface finish.

With iron-copper steels there is concern about copper entry into the steel recycle stream. Yet, copper is intentionally added to other steels for strength or corrosion resistance. Unfortunately, the alternative PM alloys without copper are not compelling substitutes from a cost-performance perspective. Research is needed to understand steel recycling and how it is applied to other steels containing copper, such as precipitation hardened stainless steels (17-4 PH, 15-5 PH) and weathering steels (A242, A588).

The FC-0208 PM alloy has been quite successful and represents a mainstay for Powder Metallurgy. In spite of its long history, there are opportunities for improvement. Without focused research on higher properties, then it is possible that the copper steels might eventually be replaced. Already on a strength per unit mass basis the reinforced polymers are competitive and are starting to replace press-sinter ferrous components.

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Appendix

Example mechanical properties are condensed below. The abbreviations are as follows:

%Cu = percent copper,

%C = percent carbon,

P = compaction pressure in MPa,

T = sintering peak temperature in °C,

t = sintering hold time at peak temperature in min,

g/cm^3 = sintered density in g/cm^3,

σ_Y = yield strength in MPa,

σ_U = tensile strength in MPa,

ε_F = fracture elongation in%,

HRB = Rockwell B hardness.

% Cu	% C	*P*, MPa	*T*, °C	*t*, min	g/cm^3	σ_Y, MPa	σ_U, MPa	ε_F,%	HRB
10.0**	0.8	---	1,120	30	7.3	965	1034	5	105
1.9	0.8	415	1,125	40	6.6	418	510	---	---
2.0	0.0	700	1,120	45	7.1	240	500	6	82
2.0	0.4	---	1,120	60	6.8	---	250	---	32
2.0	0.6	740	1,120	60	7.1	---	575	3	85
2.0	0.6	608	1,260	20	7.3	---	---	4	78
2.0	0.7	640	1,150	30	7.1	---	529	5	---
2.0	0.8	---	1,147	26	6.7	400	485	---	78
2.0	0.8	269	1,120	30	6.7	332	432	3	81
2.0	0.8	---	1,120	30	5.8	240	240	1	50
2.0	0.8	---	1,120	30	6.6	365	425	1	70
2.0	0.8	276	1,120	30	6.7	324	416	1	70
2.0	0.8	324	1,120	30	6.9	358	486	2	75
2.0	0.8	310	1,120	30	6.9	343	515	2	76
2.0	0.8	---	1,120	25	6.7	580*	700*	---	103*
2.0	0.8	660	1,250	60	6.9	510	683	3	100
2.0	0.8	310	1,120	30	6.9	355	484	2	75
2.0	0.8	441	1,120	30	7.2	444	626	3	85
2.0	0.8	317	1,120	30	6.9	342	489	2	75
2.0	0.8	441	1,120	30	7.2	454	647	3	85
2.0	0.8	---	1,120	30	7.2	450	520	1	84
2.0	0.8	441	1,120	30	7.2	429	611	3	85
2.1	0.7	---	1,120	20	7.0	466	573	3	86
2.5	0.7	---	1,120	20	7.0	534	673	3	90
3.0	0.8	---	1,140	30	7.2	450	475	---	---
3.1	0.7	---	1,120	20	7.0	525	664	1	---

**heat treated post sintering | **press-sinter 2% Cu followed by infiltration to total of 10% copper*

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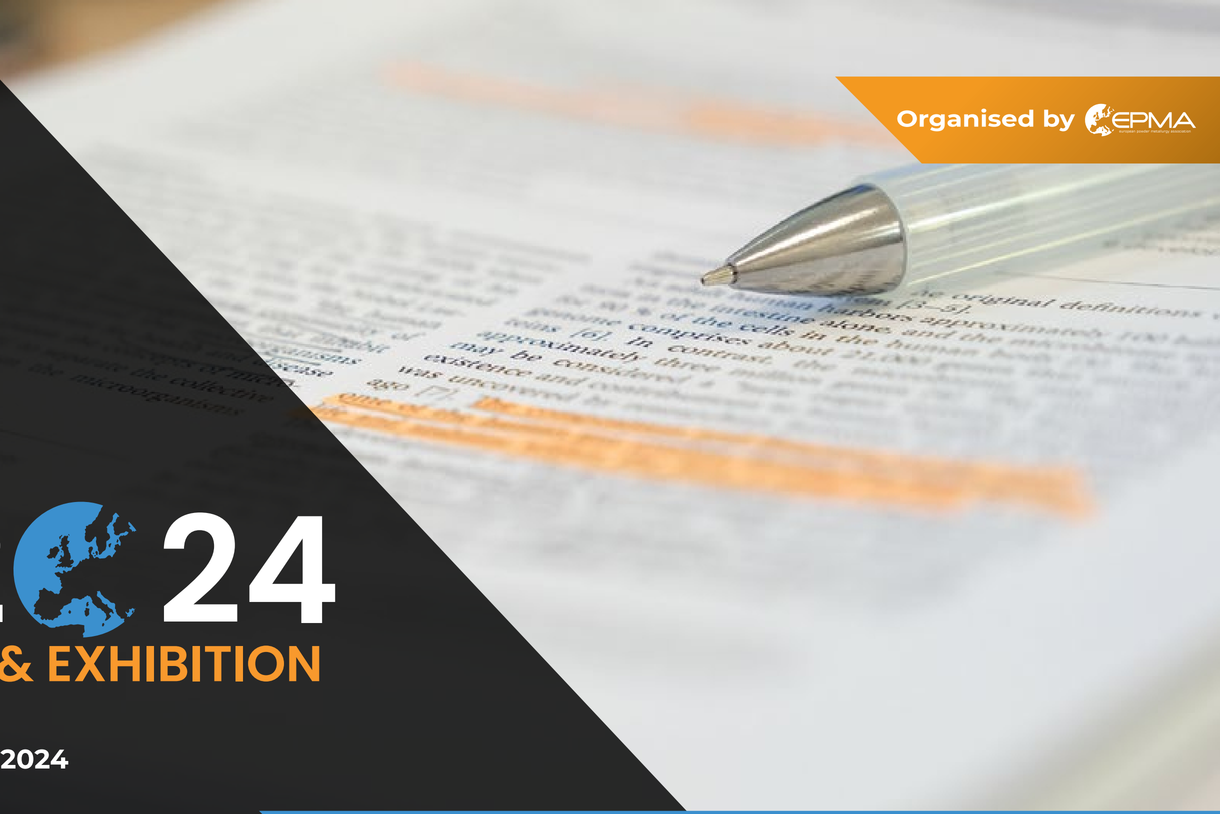

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