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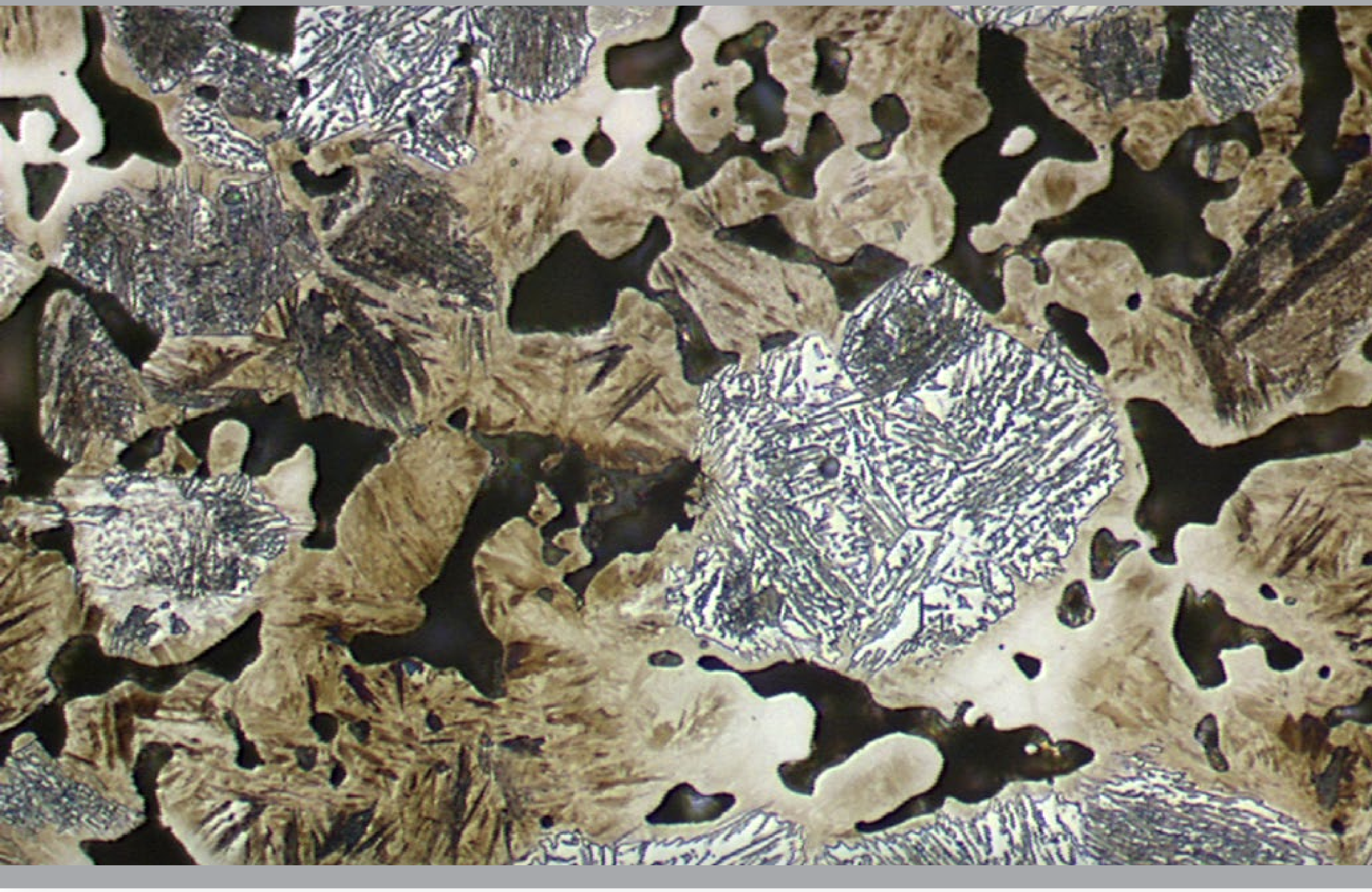
POWDER METALLURGY REVIEW



METALLOGRAPHY FOR PM

COMPANY VISIT: ATOMISING SYSTEMS

PM2014 WORLD CONGRESS

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Publisher & editorial offices
Inovar Communications Ltd
2 The Rural Enterprise Centre
Battlefield Enterprise Park
Shrewsbury SY1 3FE
United Kingdom

Editor & Publishing Director
Paul Whittaker
Tel: +44 (0)1743 454992
Email: paul@inovar-communications.com

Managing Director
Nick Williams
Tel: +44 (0)1743 454991
Email: nick@inovar-communications.com

Consulting Editor
Dr David Whittaker
Consultant, Wolverhampton, UK

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

Please contact Paul Whittaker, Editor
Tel: +44 (0)1743 454992
Email: paul@inovar-communications.com

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Tel: +44 (0) 207 1939 749
Fax: +44 (0) 1743 469 909
Email: jon@inovar-communications.com

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POWDER METALLURGY REVIEW

Controlling properties and understanding performance

It has been a busy year for many in the Powder Metallurgy industry. With a host of events to choose from, including the PM World Congress in Florida, there were numerous opportunities to hear about exciting technical developments that are pushing our industry in new and rewarding directions.

In this issue we review a number of presentations from the PM World Congress (page 61) and the inaugural Additive Manufacturing for Powder Metallurgy conference, AMPM2014, which addressed the challenges of controlling component properties and performance (page 53).

Product quality is a key issue for all PM producers and metallography provides an essential method to analyse materials, helping to ensure quality and understand problems that can arise. Tom Murphy, Hoeganaes Corporation, looks at the importance of correct sample preparation for achieving the best possible results (page 29).

We also report on a visit to Atomising Systems Ltd, a company that has been installing metal atomisers throughout the world for over 20 years. The company also manufactures a range of specialist metal powders (page 39).

Bo Jönsson and Roger Berglund of Sandvik Heating Technology discuss Kanthal APMT, an advanced FeCrAlMo alloy suited to high temperature furnace applications. Their article describes several applications within PM where there is the potential to increase sintering temperatures and extend the lifetime of critical components (page 45).

Paul Whittaker
Editor, *Powder Metallurgy Review*

Cover image
Micrograph of FLNC-4405. The base powder is a prealloyed steel containing 0.85 wt/o Mo with additions of 2.0 wt/o Ni, 1.5 wt/o Cu, and 0.6 wt/o graphite. Sintered at 1120°C with conventional belt furnace cooling (courtesy Hoeganaes Corp.)

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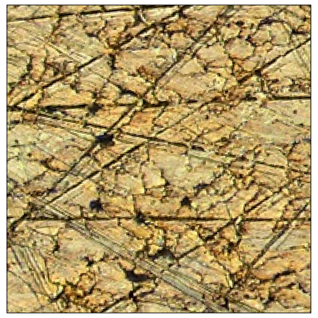
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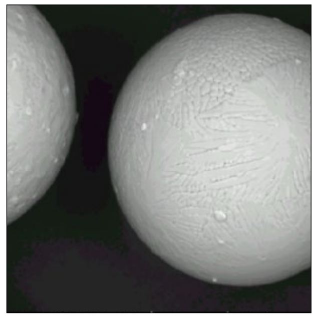
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29 **Introduction to Metallography for PM: Part 1, Sample preparation techniques**

Metallography is the study of the physical structure of metals using microscopy. The process has many advantages as a method to characterise PM products and helps to ensure product quality and understand issues that arise. In part one of our Introduction to Metallography for PM, Thomas F Murphy, Hoeganaes Corporation, USA, looks at how best to prepare samples for the process.

39 **Atomising Systems Ltd: Making the equipment that makes the powder for over 20 years**

Atomising Systems Ltd builds and installs metal atomisers throughout the world for a wide range of applications as well as manufacturing a range of specialist metal powders. Dr David Whittaker reports on a visit to the company.

45 **Kanthal APMT™: Making high temperature sintering more cost competitive**

Kanthal APMT is an advanced FeCrAlMo alloy manufactured by Sandvik Heating Technology that is ideally suited to high temperature furnace applications. Bo Jönsson and Roger Berglund present the alloy's basic properties and describe several applications within PM where there is the potential to increase sintering temperatures and extend the lifetime of critical components.

53 **AMPM2014 Conference Review: Controlling component properties and performance in Additive Manufacturing**

The Additive Manufacturing with Powder Metallurgy (AMPM) conference took place in Orlando, Florida, from May 18-20, 2014. We report on the three presentations that addressed the challenges of controlling component properties and performance.

61 **Advances in PM materials and process developments at the PM2014 World Congress**

The PM2014 World Congress on Powder Metallurgy and Particulate Materials, Orlando, Florida, USA, May 18-22, featured some 92 technical sessions, posters and special interest programmes. Dr David Whittaker reports on a number of key presentations on advances in PM materials and the processing of PM products.

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

Federal-Mogul to purchase TRW's engine valve business

Federal-Mogul Holdings Corporation, Southfield, Michigan, USA, has announced that its Powertrain division has entered into a definitive purchase agreement to acquire TRW's engine valve business.

The TRW engine valve business is a leading developer and supplier of engine valves for passenger car engines, heavy-duty engines and large-bore engines for industrial and marine applications. The business has annual global sales of $610 million and employs 5,400 people in twelve countries. The transaction is expected to close in the first quarter of 2015.

"The TRW acquisition is a sign of our commitment to the long-term development of Federal-Mogul's Powertrain division in order to enable its growth and to create value for the company's customers and shareholders," stated Carl Icahn, Chairman of the Board, Federal-Mogul Holdings Corp.

"We are very pleased to bring TRW's engine valve business into Federal-Mogul Powertrain as it will add a completely new product line to our portfolio, strengthen our market position as a leading developer and supplier of core components for engines, and enhance our ability to support our customers to improve fuel economy and reduce emissions," stated Rainer Jueckstock, Co-CEO, Federal-Mogul Holdings Corp and CEO, Powertrain.

www.federalmogul.com

Plansee expands production in Korea

Austria's Plansee Group has announced plans to build a new plant in South Korea. The Governor of the Korean province Kyung-pil Nam and Bernhard Schretter, member of the Executive Board of Plansee Holding AG, recently signed a memorandum of understanding relating to the purchase of the necessary land and the construction of a building providing 2,500 m² of production and office space.

Plansee is investing €10 million in the project and will merge its existing Korean production and sales sites in the new development, expanding production capacity. "This investment is a vital step in ensuring our continued growth in South Korea," stated Schretter. "With this new construction on the doorstep of our key customers, we are emphasising our long-term commitment as a supplier to the semiconductor and display industry in South Korea."

The new company building is scheduled for completion in 2016. "The Dongtan High-Tech Industrial Complex, where Plansee has decided to invest, is an optimal investment destination since it houses many outstanding electronics companies, R&D centres and human resources," stated Govenor Kyung-pil Nam. "We promise to do our utmost to provide all necessary administrative support so that Plansee can successfully carry out its investment."

www.plansee.com

Hyundai Motor Group builds new iron powder plant

Hyundai Motor Group in Korea has recently completed the construction of a 25,000 tonnes/year water atomisation plant near the Hyundai Steel complex in Dangjin, South Chungcheong, to produce Powder Metallurgy (PM) grades of pure iron powders. Hyundai Motors plans to use the iron powders for existing PM automotive components as well as for new applications in the next generation of automobiles being developed by the company.

Hyundai Steel Co. told *Powder Metallurgy Review* that the entire demand for PM grade iron powders in Korea of around 70,000 tonnes/year has to date been met with imported powders, mainly from the United States and Sweden. With the new iron powder plant in Dangjin, Hyundai Motors will be able to supply much of the group's needs for iron powders for its PM components requirements.

The company also stated that the new plant will help to create a stable iron powder supply situation and will enable it to develop and optimise iron powder grades for specific automotive components. Hyundai Motors hopes in this way to achieve improvements in the quality and performance of its automobiles.

The Dangjin Iron Powder Plant commenced pre-production trials for iron powders in March 2014, and has been in volume production of high quality water atomised iron powders since July. The pure iron powder produced by the new plant has already been applied to a number of automotive PM components. It is expected that production will be increased rapidly next year.

Hyundai Motors also states that it expects to adopt more automotive parts using alloyed iron powders which will help to bring the current Korean average of 6 kg of PM parts per car closer to the levels of the United States (20 kg/car) and Japan (10kg). The company has been developing new technology in conjunction with Hangyang University in Korea, to produce new grades of partially alloyed iron powders having less segregation and better compressibility than some currently available alloyed iron powder grades. It expects to complete this development over the next two or three years.

www.hyundai-steel.com

Hilti reports profit increase

Hilti Group, headquartered in Schaan, Liechtenstein, has reported sales growth of 7.8% (in local currencies) during the first eight months of its current business year. Despite continued negative exchange rate effects, the Group has posted an increase in both its operating result (+23.8%) and net income (+34.3%).

Compared to the corresponding period of the previous year, sales in Swiss Francs grew by 3% to CHF 2945 million (approximately €2442 million). The difference in sales growth expressed in local currencies reflects the continued negative exchange rate effects, in particular in the emerging markets, stated Hilti.

"We are still operating in a very heterogeneous and volatile economic environment and the challenging exchange rate situation, particularly in emerging markets, continues to negatively impact our results," stated Christoph Loos, CEO. "Against this backdrop we are pleased with our development and take important investments to prepare for future growth."

The most dynamic sales increase was recorded in Latin America where sales have grown by 17.3%. Double-digit growth rates were also achieved in Eastern Europe / Middle East / Africa (+14.5%) and Asia/Pacific (+11.8%). In North America, sales were up 7.3% year-on-year. Europe has also posted single-digit sales growth (+4.7%).

www.hilti.com

CVMR and AREVA announce an alliance for metal powder production and refining facilities

Canada's CVMR Corporation (CVMR) and AREVA Federal Services LLC (AFS), a US subsidiary of AREVA, have announced an alliance for the deployment of metal powder production, refining and manufacturing plants in the United States.

It is stated that the plants will position CVMR as the industry leader for powdered metal production based on their proprietary systems and technology. The projects will include research and production facilities.

CVMR's vapour metallurgy processes and technologies have been developed over 28 years and are claimed to offer highly cost effective and environmentally neutral powder production. A large portion of the US plant will be dedicated to the production of metal powders used in Additive Manufacturing, graphite and graphene processing.

AFS will provide engineering, procurement and construction management services under the direction of CVMR. The company will start the industrial production plant conceptual design and cost estimate in 2014 with early completion in first quarter 2015. "The resources of AFS are complementary to the rapid development of our projects and expeditious delivery of advanced materials to industry," stated Kamran M Khozan, Chairman and CEO of CVMR Corporation.

CVMR will develop the plants as part of a global initiative for fully integrated metal extraction, refining and manufacturing operations. The company is also reported to be developing other metal production facilities around the world.

www.cvmr.ca
http://us.areva.com

GKN increased profits for Q3 2014

GKN plc has issued its Interim Management Statement covering the period 1 July to 30 September 2014, with sales for the three months reported as reaching £1,789 million. This, the company states, represented a 3% organic increase which was offset by £119 million (6%) adverse currency translation.

The company reported that third quarter trading profit increased to £160 million, with organic trading profit increasing by £20 million (14%) while the adverse currency translation impact was £11 million and the reduction due to acquisitions/divestments was £1 million. Trading margin increased to 8.9% and group profit before taxation increased 6% to £139 million.

"We have delivered a good performance in the third quarter despite adverse currency translation continuing to impact reported sterling results. Looking forward to the rest of the year, tougher prior year comparators mean that organic growth is likely to be more modest but we expect our market leading positions, advanced technology and extensive global footprint to make 2014 another year of progress," stated Nigel Stein, Chief Executive, GKN plc.

GKN Powder Metallurgy

GKN Powder Metallurgy reported sales of £225 million in Q3 2014, compared with £234 million in Q3 2013. Trading profit was £21 million, compared with £23 million in the same period of 2013. Year-to-date sales amounted to £696 million, with trading profit of £74 million. The company stated that GKN Powder Metallurgy's organic sales increased 3% but adverse currency translation reduced sales by 7% and trading profit by £2 million. GKN stated that trading margin was slightly lower due to a major equipment failure at one plant and softer sales in Brazilian markets.

www.gkn.com

Sandvik reports stable Q3 2014

Sweden's Sandvik AB has reported that its order intake for Q3 2014 amounted to 21.0 billion SEK and invoiced sales to 22.6 billion SEK. Operating profit totalled 2.5 billion SEK, or 10.9% of invoiced sales. Changed metal prices and currency rates contributed positively to Q3 earnings, stated the company.

"The global market situation remained relatively unchanged in the third quarter, albeit with variations between markets and segments. Business conditions remained favourable in N America, most notably for Sandvik Machining Solutions. Demand in Europe fluctuated as the weaker market conditions in Russia indirectly impacted other parts of the continent. Global demand from the mining industry remained stable and was on a par with levels observed earlier in the year," stated Sandvik's President and CEO Olof Faxander.

www.sandvik.com

Difficult start for Carpenter Technology

Carpenter Technology Corporation announced financial results for the quarter ended September 30, 2014, reporting net income of $13.5m, compared to $34.6m in the same quarter last year. Net sales for the first quarter of fiscal year 2015 were $549.8m, and net sales excluding surcharge were $440.1m, an increase of $28.0m (7%) from the same quarter last year, on 11% higher shipments.

Operating income was reported at $22.1m, a decrease of $33.7m from the Q1 of the prior year. Operating income excluding pension earnings, interest and deferrals was $24.5m, a decrease of $37.3m (or 60%) from Q1 of the prior year. The reduction in operating income was stated as being primarily due to the operational issues experienced in the first two months of the quarter and additional Athens depreciation expense.

"After a difficult start to the quarter, performance in September improved. The Latrobe press is now back on line and we saw our Specialty Alloys Operations (SAO) segment mix improve in September. That said, we need to drive further improvements and successfully work through recent challenges at our Reading mill," stated William A Wulfsohn, Carpenter's President and CEO.

"Looking forward, we expect SAO to continue year-over-year volume growth as we expand the number of customer approvals for Athens production. We also anticipate that SAO's sales mix will improve based on recent trends in our backlog. In addition, the Performance Engineered Products (PEP) segment is expected to improve year-over-year. Finally, we remain focused on keeping our selling, general and administrative expenses flat. These are the crucial building blocks in terms of driving profitable growth as we progress through our fiscal year," added Wulfsohn.

www.cartech.com

Mitsubishi Materials to acquire majority share in Hitachi Tool carbide tool

Mitsubishi Materials (MMC) of Japan has announced that it will acquire a 51% shareholding in Hitachi Tool Engineering Ltd, a leading Japanese cemented carbide cutting tool and wear part producer. Hitachi Tool Engineering Ltd is a division of Hitachi Metals Ltd which will retain the remaining 49% of shares. The transaction is scheduled to be completed by April 1, 2015.

Production of carbide tools by Hitachi Tool will become part of MMC's Advanced Materials & Tools Division and the acquisition is expected to help MMC achieve a 10% share or higher of the global carbide tool market. Hitachi Tool specialises in the production of carbide tools for a variety of difficult-to-cut materials and complicated shape products for power generation equipment, aerospace materials and other applications.

It was stated that Hitachi Tool will benefit from MMC's considerable global sales network, and will also be able to take advantage of MMC's raw material procurement, integrated supply chain and recycling network.

The company will continue its tool steel business partnership with Hitachi Metals. Along with conventional type tool steel, there are some high speed tool steel grades made by Powder Metallurgy process (HAP series) having superior wear resistance and toughness because of higher alloy content and uniform fine microstructure.

Hitachi Tools reported net sales of Yen 18,966 billion ($177.8 million) for the financial year 2013/2014.

www.mmc.co.jp

Kennametal announces fiscal first quarter 2015 results

Kennametal Inc. (NYSE: KMT), headquartered in Latrobe, Pennsylvania, USA, has reported results for its fiscal first-quarter 2015, stating sales for the period totalled $695 million, compared with $620 million in the same quarter last year.

Operating income was $61 million, compared with $59 million in the same quarter last year. Operating income also included $7 million of restructuring and related charges, primarily due to equipment relocation and facility expenses. Adjusted operating margin was 9.9%, compared with an adjusted operating margin of 9.7% in the prior year.

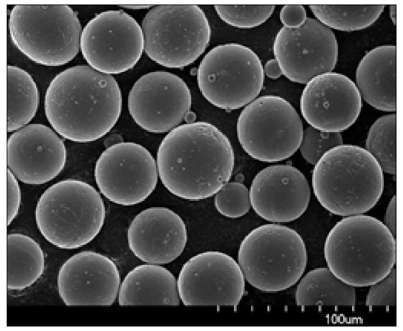
"September quarter sales growth was driven by ongoing demand strength in our Industrial segment; however, our Infrastructure business was challenged by continued weak conditions in underground mining and road construction, partially offset by modest improvement in the oil and gas sector. We are pleased to have made significant progress in integrating the Tungsten Materials Business, and we are accelerating measures to reduce costs and improve efficiencies that should position Kennametal for improved profitability," stated Carlos Cardoso, Kennametal Chairman and Chief Executive Officer.

Kennametal is updating its outlook due to weaker economic conditions anticipated for the remainder of fiscal 2015. Key factors include softer customer demand in the Eurozone, lower drilling activity in the oil and gas sector, as well as a continued decline in underground mining production levels.

The company now expects fiscal 2015 total sales growth in the range of 2 to 4%, with organic sales growth of 1 to 3%. Previously, the company had forecast total sales growth in the range of 5 to 7%, with organic sales growth of 3 to 5%.

www.kennametal.com

AP&C Advanced Powders and Coatings achieves AS9100 certification



SEM image of AP&C powder

AP&C Advanced Powders and Coatings Inc. (AP&C), based in Boisbriand, Canada, has announced that the company's quality management (QM) system is now AS9100 certified. The company stated that with the new certification it is better positioned to service its aerospace customer base as they move from R&D to production of aircraft components with Additive Manufacturing and other Powder Metallurgy processes.

AP&C specialises in the production of high purity spherical powder of titanium and other reactive metals by plasma atomisation. Its powders are used in a range of PM applications including Additive Manufacturing, Metal Injection Moulding (MIM), Hot Isostatic Pressing (HIP) and coatings.

"It's another exciting milestone achievement for our company which is dedicated to producing spherical metal powders from high melting point alloys using its proprietary plasma atomisation systems. We supply our titanium, nickel superalloys and other special products to a growing biomedical and aerospace customer base," stated Jacques Mallette, CEO.

"AP&C is a quality focused organisation that prides itself in consistently enhancing its QM system in order to offer superior quality products and service to its demanding customer base," added Mallette.

www.advancedpowders.com

World's largest metals research consortium to be established with €1 billion funding

The world's largest research consortium in the field of metals research and manufacturing is to be created by European industry in the form of Metallurgy Europe. The R&D programme has recently been selected as a new Eureka Cluster and will bring together over 170 companies and laboratories from across 20 countries. Funding for the project has been stated as €1bn over seven years.

The European Powder Metallurgy Association and a number of other European organisations such as the European Space Agency (ESA), European Synchrotron Radiation Facility, the Institut Laue-Langevin and the Culham Centre for Fusion Energy are reported to be providing their expertise and innovation to this initiative.

A number of Europe's largest engineering and manufacturing companies are participating, including Airbus Group, BP, Siemens, Daimler, Rolls-Royce, BMW, Thales, AvioAero, PSA Group, BAE Systems, Philips, Ruag, Sener, Bombardier, OHB Systems, Linde Group, ESI, Rolex, Richemont, ArcelorMittal, Sandvik, Bruker, SKF, Johnson Matthey, Tata Steel, GKN, Boston Scientific, ThyssenKrupp, Outokumpu, Haldor Topsøe and Fiat. There are also over 60 small and medium-sized companies joining the programme.

"This new programme allows us to enter the high-tech metals age. The top management of industry have come together for the first time on this important topic, and there is a confident feeling that Metallurgy Europe will deliver many unique, exciting and profitable technologies," stated Professor David Jarvis, Chairman of Metallurgy Europe and head of strategic and emerging technologies at ESA.

"The top management of industry have come together for the first time on this important topic, and there is a confident feeling that Metallurgy Europe will deliver many unique, exciting and profitable technologies," added Jarvis.

The team believes that the programme has the potential to create over 100,000 new jobs in the materials, manufacturing and engineering sectors.

www.esa.int

Ivor Jenkins Award presented to Dr Brian James

The UK's Institute of Materials, Minerals and Mining (IOM3) announced that the recipient of its 2014 Ivor Jenkins Medal award was Dr W Brian James CEng FIMMM FAPMI FASTM. The prestigious award is presented to individuals in recognition of a significant contribution that has enhanced the scientific, industrial or technological understanding of materials processing or component production using particulate materials.

In a career spanning over 40 years in both the UK and USA, Dr James has become widely known and highly respected in the PM structural parts and Powder Forged sectors of the industry. He has developed and commercialised new material grades, developed the first ever production PF connecting rod at GKN and has led many material development programmes at Hoeganaes Corporation in the USA.

Dr James has been extremely active in PM standards development with MPIF, ASTM and ISO committees.

www.iom3.org

EPMA Distinguished Service Award presented to Jan Tengzelius

Jan Tengzelius receives his award from Prof Lorenz Sigl

The European Powder Metallurgy Association (EPMA) has presented its 2014 Distinguished Service Award to Jan Tengzelius.

Tengzelius, who began his career in Powder Metallurgy in 1974 when he was employed as development engineer at Höganäs AB, Sweden, was presented the prestigious award at Euro PM2014 International Conference and Exhibition held in Salzburg, Austria, by Congress Co-Chairman Prof Lorenz Sigl.

Following his graduation at Sweden's Royal Institute of Technology in the field of metallurgy, Tengzelius worked at the Institute for Metals Research in Stockholm before starting at Höganäs AB. From 1977 until 1983 he was in charge of product development at Höganäs with focus on high strength sintered steels such as phosphorous alloyed materials and diffusion alloyed powders in the Distaloy family. He was also instrumental in the development of bonded powder mixes and soft magnetic materials.

In 1983 he took over as managing director of the development company Cold Isostatic Pressing Systems. In this position he was responsible for development and technology sales of this forming method primarily for the production of PM cylinder liners. Between the years 1989 to 2006 he was the R&D manager for Höganäs followed by some years as Technical Director for Future Technologies, a department that lies within the Höganäs AB Global Development area with responsibility for development of technologies outside of the company's existing business areas. He is now acting as an advisor to this department.

Over the years he has contributed to the growth of PM especially by developing metal powders and consolidation methods for highly loaded components, working in close partnership with research organisations, customers and users of PM components. Tengzelius has also headed the development of soft magnetic composites, designated Somaloy, especially suited for design of electrical motors and generators having low energy losses and high torque.

Tengzelius is a former member of the EPMA council and board and was also active as chairman of the Powder Metallurgy group of Jernkontoret in Sweden and as a secretary of the ISO TC 119 sub committee on PM.

www.epma.com

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Advances in carbide and diamond tools at IMTS 2014

The 2014 International Manufacturing Technology Show (IMTS 2014) held at McCormick Place, Chicago, September 8-13, was reported to be one of the top four shows in the event's history. More than 114,000 people visited the show's 2035 exhibitors, representing a 14% increase in visitor numbers over IMTS 2012. There was an abundance of innovative manufacturing technology on show including new cutting tool developments. Below is a look at some of the novel developments in cutting tool materials announced by leading carbide and diamond tool producers who exhibited at the show.

Kennametal

Kennametal announced the introduction of a range of new Beyond Drive cutting tool inserts specifically designed for cast irons, steels, and stainless steels. Like all of Kennametal's Beyond line of products, Beyond Drive inserts feature a proprietary post-coat surface treatment that improves edge toughness, reliability, and depth-of-cut notch resistance, and a micro-polished surface to reduce friction and workpiece sticking (BUE). A fine-grained alumina layer allows for increases in cutting speed, improving productivity and reliability at high cutting temperatures.

With Beyond Drive, adding a new titanium oxy-carbonitride outer coating (TiOCN) not only increases wear- and abrasion-resistance, its bronze colour is also an effective wear indicator. Depending on the application, field tests are showing anywhere between 30% and 125% more parts per edge.

New Beyond Drive cutting tool inserts from Kennametal

Late last year, Kennametal finalised its acquisition of the tungsten materials business from Allegheny Technologies Inc., including the full line of Stellram sintered carbide tools (formerly ATI Stellram). High-feed milling inserts are available with proprietary Stellram X-Grade technology which uses ruthenium as a key ingredient, combined with a cobalt binder, for superior thermal-cracking and propagation resistance. The results are metal-removal rates up to three times higher than conventional carbide cutting tools and longer tool life.

www.kennametal.com

NTK Cutting Tools

NTK Cutting Tools, a division of NGK Spark Plugs of Japan, has released a new proprietary group of advanced composite cutting tool materials called Bidemics, which have been specially developed for machining superalloys such as INCO718.

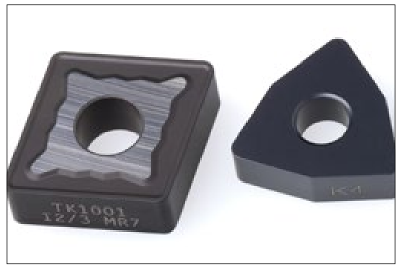
Bidemics have the potential to achieve much higher productivity with up to 1600SFM machining capability compared with whisker ceramic cutting tools. The Bidemics tools are said to excel under the same machining parameters with results in some cases showing two to three times tool life depending upon the cutting conditions.

At IMTS 2014, NTK released two new grades of Bidemics; JX1 for all non-scale machining and JP2 a TiN coated multi-tipped brazed grade for semi-finishing and finishing applications machining high temperature superalloys.

www.ntkcuttingtools.com

Seco Tools, LLC

Seco Tools, LLC unveiled several new milling, turning, threading and tool holding products that were developed with material advancements in mind, including a square shoulder cutter that enhances side-milling operations, a multi-edge system that meets the industry's demand for narrow cutting-edge grooving and parting-off tools, an insert that provides high-performance threads in a single pass, and toolholders with special vibration damping capabilities.



Seco Tools TK grades exhibited at IMTS2014

Seco spotlighted several milling insert grades including the new MS2050 that utilises an advanced coating technique and substrate. The special PVD coating on the MS2050 not only strengthens the insert's wear resistance but also eliminates reaction with the workpiece material. Regarding advanced turning inserts for different materials, Seco exhibited its Duratomic coated TK1001 and TK2001 carbide grades as well as the TP1030 cermet grade.

From its Secomax line, the company highlighted CBN010, an uncoated PCBN grade that has high resistance to edge chipping when cutting hardened steels, and CBN060K for turning case hardened steels. The new Thread Chaser inserts incorporate multi-tooth patterns to allow push and pull threading of OD and ID features with one or two passes.

www.secotools.com

Widia Products

The Widia Products Group, a division of Kennametal, announced new geometries and grades in its Victory family of turning inserts that are said to deliver smooth machining, improved efficiencies and extended tool life for difficult-to-machine materials like Inconel and Rene in complex aerospace applications or cobalt-chromes and high-alloy stainless steels in precise medical applications. The Victory FS is, for example, available in WS10PT, a high-performance PVD coated grade. All inserts in the WS10PT grade undergo a post-coat treatment to increase compressive stress and improve edge toughness. This delays built-up edge and results in longer tool life.

The WIDIA Victory turning portfolio features the Universal Roughing geometry for smooth chip forming

WS10PT is also available in other geometries including the Universal Roughing (UR) geometry designed to increase roughing and medium turning performance in stainless steels and steel workpieces. The UR geometry has a unique chip breaker and rake profile design without inflection points which breaks up stringers without concentrating cutting forces that result in breakage. Its positive rake angle further reduces cutting forces while improving depth of cut notching resistance.

www.widia.com

Norton / Saint-Gobain

Norton / Saint-Gobain, one of the world's largest abrasives and diamond tool manufacturers, recently introduced Norton Paradigm Diamond Wheels featuring a new proprietary patented bond for high grinding performance on carbide round tools and periphery grinding on carbide and cermet inserts. Norton stated the new Paradigm wheels offer a high grain retention and uniform structure to provide a high G-ratio (ratio of material removal rate versus wheel wear) for up to 2.5x longer wheel life and a 30% higher material removal rate compared to other superabrasive wheels when grinding 6% and 12% Co carbides.

Paradigm grinding wheels from Norton

An entirely new abrasives platform, Norton Vitrium3, which features a patent-pending bond technology developed by the Norton / Saint-Gobain R&D team was also launched at IMTS 2014. This revolutionary bond features an exclusive chemistry that promotes excellent grain adhesion, resulting in improved product versatility across a wide range of applications.

www.nortonindustrial.com

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Production ceases at Tasmanian PM plant

ACL Bearings has finally closed its steel-back sintered bearings and PM part facility in Launceston, Tasmania, Australia, after failing to find a purchaser for the business. The company had been in receivership since 2009 and received a multi-million dollar Federal Government bailout. However, as no offers were received for the business as a going concern the decision was taken in July 2013 to close the plant by the summer 2014 with the loss of 136 jobs.

At its peak ACL Bearings had a workforce of 730 and was one of Australia's major PM producers supplying steel-backed sintered engine bearings, PM valve seat inserts, timing pulleys and sprockets, and a range of PM parts for water and oil pumps and brake systems to companies such as Ford, GM Holden, Toyota and Mitsubishi. The company was spun out of the failed Repco group in 1986 when senior managers bought ACL from the new owners of Repco.

ACL relied heavily on the automotive sector and, whilst a considerable portion of its production was exported, the decision by Ford to halt car production in Australia from 2016 combined with disadvantageous exchange rates for the Australian dollar in recent years dealt the final blow. In 2012 ACL received a prestigious design award from the MPIF for a PM part it supplied to Futuris Automotive for use on steering columns. The part involved a sinter-brazed spacer tube used on the Ford Falcon steering assembly since 2008 to provide positive steering adjustment and supporting the tilt adjustment components.

The company also manufactured its own copper-based powders using water atomisation in a plant having 150 tonnes/month capacity. Copper-lead powders were produced for use in bearing strip from which steel-backed engine bearings are formed. The copper-lead powder is bonded to steel by sintering and rolling in a continuous atmosphere furnace. Production of the sintered copper-lead strip was said to be approximately 35,000 metres per month.

www.acl.com.au

This PM sinter-brazed spacer tube used in a steering column tilt/reach assembly won a MPIF Award in 2012 for ACL

RUAG looks to Additive Manufacturing for space satellite applications

Swiss company RUAG Space plans to soon be equipping satellites with components made using Additive Manufacturing (AM), making them lighter and cheaper. As part of a pilot project, specialists at RUAG Space have built an antenna support for an Earth observation satellite.

RUAG previously supplied the European Space Agency (ESA) with a similar support for its Sentinel-1A radar satellite, although the support for ESA was manufactured using conventional methods. Collaborating with experts from the company Altair, RUAG engineers have now completely redesigned this support to optimise it for 3D printing.

The Altair Software made it possible to exploit the freedom of design provided by Additive Manufacturing by optimising the topology of the component in order to use only as much material as necessary. Produced by EOS, the finished component is just half the weight of the previous component and has improved rigidity.

To check that the new support is ready for use in space, it is currently undergoing a range of intensive qualification tests that are scheduled to be completed by the end of the year. "Our goal is to fit Sentinel-1 successors with antenna supports that have been manufactured using a 3D printer," stated Michael Pavloff, Chief Technical Officer at RUAG Space.

Producing this antenna support is by no means a one-off. "3D printing has enormous potential for our business, and we're currently in the process of developing further space applications," added Pavloff.

The design specialists at Altair also benefit from the joint project. "The collaboration with RUAG Space and EOS allows us to deliver even more innovative end-to-end design and optimisation processes to exploit the benefits of Additive Manufacturing," stated Pietro Cervellera, Managing Director of Altair.

www.ruag.com

Additive manufactured antenna bracket for a Sentinel-1 satellite (Image: RUAG)

Mazak introduces new hybrid metal Additive Manufacturing machine

Japan's Mazak Corporation, known for its range of machine tool systems, has unveiled its new INTEGREX i-400AM (Additive Manufacturing) HYBRID Multi-Tasking Machine. As a mix of additive technology and Mazak's advanced multi-tasking capabilities, the machine is claimed to significantly reduce part cycle times while providing high-efficiency processing.

"As a turnkey system, the INTEGREX i-400AM offers manufacturers a new innovative alternative to conventional processing in terms of part design and machining," states the company. The technology is especially well suited for small lot production of very difficult-to-cut materials such as those used in the aerospace, energy and medical industries.

With the additive capability, manufacturers can generate or clad near-net-shape component features then quickly complete them with high-precision finish machining operations, as well as laser mark parts if needed.

In operation, the INTEGREX i-400AM melts metal powder using fibre laser heat. Cladding heads (Additive Manufacturing nozzles) apply the molten material layer by layer, each of which solidifies as the desired shape grows. The system can join different types of metals to one another, a capability beneficial in the efficient repair of existing worn or damaged components such as aerospace turbine blades, the company states.

Mazak offers two types of cladding heads, either high speed or high accuracy. Users select the appropriate head based on the intended process and the particular metal powder to be used.

www.mazak.com

Production of final products now 34.7% of total AM market

Wohlers Associates, Inc. has reported that revenues from the production of parts for final products now represent 34.7% of the entire market for Additive Manufacturing (AM). The Wohlers Report 2014 shows that this market segment has increased from less than 4% in 2003 to more than one-third of total revenues from AM products and services worldwide.

The use of AM for this sector is stated to have grown by 65.4% in 2013 to an estimated $1.065 billion, up from $643.8 million the prior year.

"We are seeing companies push the limits of AM to new levels and apply the technology in entirely new ways," stated Terry Wohlers, President of Wohlers Associates and a principal author of Wohlers Report 2014.

www.wohlersassociates.com

Höganäs expands Digital Metal facility

Höganäs AB, Sweden, has announced that its Digital Metal division has expanded production capacity and staffing levels following increased interest in the company's Additive Manufacturing technology.

The Digital Metal process involves a unique proprietary precision ink-jet technology for making complex metallic components. The system works by laying down powdered metal in thin layers, interspersed with a special ink/glue. Once complete the object is sintered for strength, resulting in a metal component with high resolution and tolerance.

The Digital Metal facility at Höganäs

Two new Digital Metal printers designed in-house at Höganäs will be added to its facility at the end of Q3 2014. Total capacity will then consist of four Digital Metal printer systems with increases in the necessary staff to meet market demand.

"The interest is based on our ability to offer a combination of good tolerances, surface finish and detailed accuracy. These benefits are further enhanced by the ability to offer high productivity. The printing takes place at room temperature without melting, and post treatment is facilitated by eliminating the need of building support," stated Ralf Carlström, Director Digital Metal. "Digital Metal will continue to build more printers based on the evolving market demands."

Digital Metal's components are currently predominantly made from stainless steel, however other materials such as titanium, silver and copper are stated as being close to commercialisation.

www.hoganas.com

Renishaw achieves TÜV certification for its AM250 laser melting machine

Engineering company Renishaw Plc, based in Wotton-under-Edge, Gloucestershire, UK, has obtained a compliance certificate from TÜV SÜD for its AM250 laser melting machine.

The TÜV SÜD certification is an electrical safety standard valid in the United States and Canadian markets, often mandatory for many institutions. The certification attests that Renishaw's AM250 laser melting machine can be implemented in a manufacturing environment without the complications, cost and uncertainty of any additional testing or field inspections.

For core manufacturing industries like automotive, aerospace or electronics, TÜV SÜD certification is essential. It confirms the equipment is a fully-tested, fully-functional structure that can be used 24/7 in industrial environments.

www.renishaw.com

China's Zhuzhou Cemented Carbide celebrates 60 years of hardmetal production

Zhuzhou Cemented Carbide Group Co. Ltd. (ZCC), a wholly owned subsidiary of China Minmetals and China's leading hardmetal producer, has been celebrating the 60th anniversary of its founding with a range of events.

ZCC was initially established in 1954 as one of China's 156 key projects in the nation's first Five Year Plan. In the first thirty years of its existence, the company adopted cemented carbide production technology introduced from the former Soviet Union. In this phase of its development, the company described its main production as hard alloy and this comprised just six brands and around 500 product types.

A step-change came in 1984 with the opening up of Chinese manufacturing to the adoption of Western technologies. At this point, ZCC introduced a modernised and upgraded production line based on plant and technology acquired from Sandvik AB, Sweden. Since then, the company has grown into an internationally competitive cemented carbide producer.

Today, ZCC has developed its business based on six 'Diamond' brand product series comprising metal cutting tools, mine excavation and petroleum drilling tools, hard

Staff at ZCC's headquarters in Zhuzhou during the celebrations marking 60 years of hardmetal production

materials, tungsten and molybdenum products, tantalum and niobium products and rare earth metal products. The company now has over a hundred brands and over 50,000 product types. It operates two product business divisions, twelve manufacturing plants and has five wholly-owned subsidiaries. Annual output has increased from the initial 700 tonnes to over 5,000 tonnes.

Technical innovation

Since the reform of the business in the 1980s, ZCC has invested a cumulative RMB 5 billion Yuan in more than twenty projects, all aimed at technology advances and quality improvements.

The company's Technology and Analysis/Testing Centre is the only national-level key laboratory in the Chinese cemented carbide industry. In running this technology innovation platform, ZCC states that it is continuously refining and improving its R&D methodologies, upgrading the quality of its technical R&D personnel and improving the response times of its scientific research programmes. Sales revenue arising from these innovation projects now exceeds 30% of the company's total annual sales and almost 600 patents are in force.

Technological innovations have also focused on the optimisation of production systems through integration of the industrial production chain. In this context, ZCC has been adhering to a philosophy of strengthening its various business units in order to grow the company. Currently, six industrial business units cover cutting tools, IT processing tools, hard materials, drilling & digging tools and refractory metals.

Global expansion

Since the 1980s, ZCC has been pursuing a 'two markets' strategy, involving competition in the international market place. As part of this globalisation strategy, ZCC has transformed its export business from the supply of cemented carbide semi-finished products into the supply of highly processed and high added value finished products. The export volumes of products such as cutting tools, bar materials, spherical teeth, rollers and micro-drills, have been enjoying double-digit annual growth levels.

While many enterprises were adversely impacted by the global financial crisis in 2008, ZCC states that its products have been winning the favour of foreign customers on the basis of both quality and competitiveness.

The company told *Powder Metallurgy Review*, "These high quality ZCC products continue to change the world's perceptions of the 'Made in China' label and have made Zhuzhou well known to overseas customers."

Looking to the future, ZCC's stated development goal is to grow into an internationally recognised brand that ranks amongst the top four cemented carbide enterprises globally.

"This will be achieved through continuing product development programmes, quality control enhancements, improvements to the sustainability of production and acceleration of developments aimed at improving the agility of production systems," the company commented to *PM Review*.

In June 2014, the long-standing collaboration between ZCC and Sandvik AB was extended and formalised through the signing of a letter of intent to cooperate in the cutting tools field by the setting up of a joint venture. This agreement is seen as an important contributor to ZCC's future growth prospects.

www.chinacarbide.com

Wu Guogen, ZCC's General Manager, at the 60th anniversary celebrations

The 60th anniversary celebrations included a variety of entertainment

Breakthrough in improving properties of partially alloyed iron powders

Researchers in the Department of Metallurgy and Materials Science at Hanyang University-ERICA, Ansan, Korea, and the Powder Technology Team at Hyundai Steel Company's new iron powder plant in Dangjin, have reported a significant breakthrough in reducing segregation in Fe-1.5Cu-1.75Ni-0.5Mo partially (diffusion) alloyed iron powder grades by the use of nanopowder alloying elements. The research is expected to lead to the development of improved partially alloyed iron powders which in turn will enhance the quality and mechanical properties of structural PM parts.

Compared with conventional grades of partially (diffusion) alloyed steel powder, where insufficient diffusion of the separate alloying elements into the surface of iron powder can lead to segregation, a new process using nanopowder alloying elements has been found to show better partial alloying with improvements in powder compressibility, higher green strength and sintered density.

As a result the sintered mechanical properties of the new partially alloyed steel powder were also found to be improved. According to a paper by J.-P. Choi, G.-Y. Lee, J.-I. Song, J.-S. Lee (Hanyang University) and J.-C.Yun (Hyundai Steel), published in *Materials Transactions* (Vol. 55, No. 8, August 2014, 1356-1362), and also a Poster Paper presented at the PM2014 World Congress and published in *Int. Journal of Powder Metallurgy* (Vol. 50, No. 3, 2014, 14), the main aim of the research was to minimise the risk of segregation of the alloying elements (Cu, Ni and Mo) used to produce partially alloyed iron powders. Segregation, they stated, leads to compositional variations of the green compacts causing varying dimensional changes during sintering and also an inhomogeneous microstructure with the obvious degradation of mechanical properties.

To overcome the problem of segregation in an economic and practical way, Professor Jai-Sung Lee and his team studied the use of a mixture of nanoscale oxide powders comprising CuO-NiO-MoO_3 which were co-reduced with iron powder in a hydrogen atmosphere (Fig. 1 (left)). Commercially available water atomised iron powder with an average particle size of 20-180 µm was used as the base iron powder for this study. The nanosized oxide powders with average particle size 100-500 nm were prepared by ball-milling the oxide powders of CuO (99.9%, D50 = 4 µm), NiO (99.9%, D50 = 7 µm), and MoO_3 (99.9%, flake type) to an agglomerate particle size of 5 to 10 µm in a high energy SPEX mill at a speed of 1060 cycles/min for 1 h. The oxide mixture was composed of wt% 1.88 for CuO, 2.23 for NiO and 0.75 for MoO_3 taking into account the stoichiometry and composition of the final product (Fe1.50Cu1.75Ni0.50Mo) after heat treatment in a hydrogen atmosphere.

The ball-milled oxide powders were mixed with an organic binder in an ethyl alcohol medium using a tubular mixer. After the binder mixing, the oxide powder mixture was dried at 60°C for 12 h and sieved down to powder agglomerates smaller than 45 µm (325 mesh), and then blended with iron powder using a tubular mixer for 2 h. The blended powder mixture was finally subjected to a reduction/heat treatment in a hydrogen atmosphere. This provided not only a reduction process for the oxide powders but also stimulated alloying among the individual elements, and their subsequent partial-diffusion into the iron powder. Optimal reduction and alloying of the oxide powders was found to be 700°C for 1 hour.

The authors stated that during the heat treatment process, the organic binder melted and acted as an adhesive interlayer between the fine alloying powders with large surface area, leading to their improved physical bonding with the iron powders. As a result, the approach in this study was found to contribute to improvements in chemical homogeneity without segregation during blending, transport and handling of the powder mixture compared to conventional processes.

Key benefits of the new nanopowder approach are the strong adhesion of the homogeneous alloying powders during hydrogen reduction, and also the morphological change of the partially alloyed iron powders. Whereas in conventional diffusion alloying where the individual alloying elements occur randomly on the surface of the iron powder, in the nanopowder process the fine, agglomerated alloying elements are more uniformly located on the surface and in particular fill in the grooves in the rough surface of the iron powder. (Fig. 2) This prevents lump formation on the iron powder particles, and improves flowability and apparent density of the powder as can be seen in Table 1.

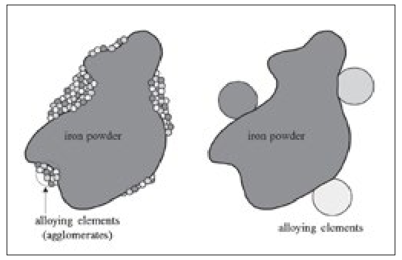

Fig. 1 Design concept of partially alloyed iron powder fabrication: (left) nanopowder process and (right) conventional diffusion alloying [1]

Powder sample	Flow (s/50±0.1g)	Apparent density (kg/m³)
Produced powder	23.8	3.34 x 10^3
Commercial powder	26.2	3.05 x 10^3

Table 1 Flowability of partially alloyed iron powders [1]

	Produced Powder	Commercial powder
Yield Strength (MPa)	357 ± 13.4	335 ± 23.0
UTS (MPa)	515.6 ± 9.2	506.3 ± 24.0
Elongation (%)	2.46 ± 0.45	1.99 ± 0.54
Rockwell hardness (HRB)	88.8 ± 2.5	81.8 ± 3.5

Table 2 Comparison of mechanical properties of sintered partially alloyed Fe-1.5Cu-1.75Ni-0.5Mo powders produced using the nanopowder route and conventional (commercial) route [2]

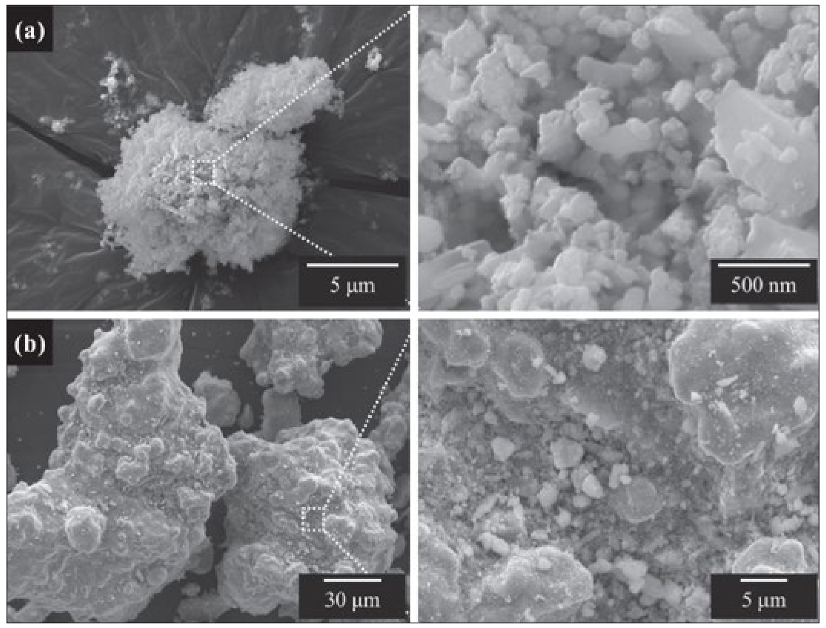

Fig. 2 SEM micrographs of (a) the ball-milled oxide mixture of CuONiOMoO_3 and (b) the iron powder blended with the oxide mixture [1]

Powder properties

In order to ascertain and compare the mechanical properties of the new powder with commercially available powder, samples were compacted at a pressure of 600 MPa and subsequently sintered at 1200°C for 2 h in H_2 atmosphere. Fig. 3 shows the microstructure and pore size distribution of the sintered powder samples. The sintered density for the nanopowder approach was 7.34 x 10^3 kg/m³, corresponding to 93% of theoretical density (TD), whereas the commercial powder had a lower density of 7.27 x 10^3 kg/m³ (91%TD). The difference is attributed to the different green density reached in both powders during compacting.

An important feature of the sintered microstructure using the nanopowder approach is the pore size distribution. The sintered part using the new partially alloyed iron powder was found to have fine, homogeneous pore structure with average pore size of 7.57 µm and with a narrower size distribution than that of the commercial powder (average pore size of 12.07 µm).

The researchers anticipate that the very homogeneous sintered microstructure of the newly developed powder might be influenced by the uniform distribution of the fine alloying elements throughout the matrix, which not only decreases the diffusion path between the particles but also increases the number of grain boundaries acting as high diffusion paths for atomic diffusion during the sintering process. They state in their paper that the uniform distribution of fine copper powders particularly enhances pore elimination by penetration of the Cu melt into the interparticle pores during sintering at 1200°C which is higher than the melting point of Cu.

Mechanical properties of Fe-1.5Cu-1.75Ni-0.5Mo having a sintered density of ~7.10 g/cm³ using the newly developed partially alloyed iron powder and containing 0.6% carbon and 0.5% zinc stearate and sintered at 1130°C for 30 min in Ar-H_2 atmosphere were outlined in the poster paper displayed at PM2014 (Table 2). As can be seen there are improvements in all values.

References:

[1] 'Improvements of Powder Properties and Chemical Homogeneity of Partially Alloyed Iron Powder by a Nanopowder process', Joon-Phil Choi, etal. *Materials Transactions*, Vol. 55, No 8 August 2014, 1356-1362
[2] 'Effect of Chemical Homogeneity on Microstructure and Mechanical Properties of Partially Alloyed Iron Powder Material', Joon-Phil Choi, etal. *International Journal of Powder Metallurgy*, Vol 50, No. 3, Summer 2014, 14.

For more information contact:

jslee@hanyang.ac.kr

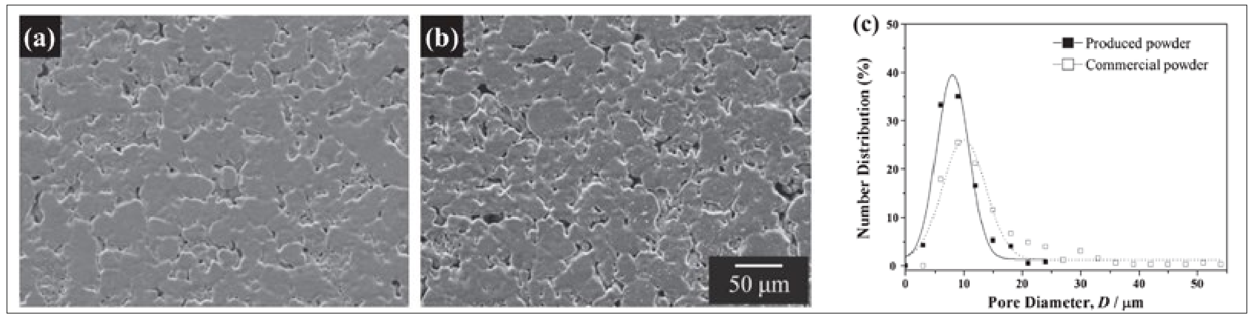

Fig. 3 SEM micrographs of the surface of the sintered parts using (a) produced powder and (b) commercial powder, and (c) pore size distribution corresponding to (a) and (b) [1]

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Introduction to Metallography for Powder Metallurgy: Part 1, Sample preparation techniques

Metallography is the study of the physical structure of metals using microscopy. The process has many advantages as a method to characterise Powder Metallurgy products and helps to ensure product quality and understand issues that arise. The correct preparation of samples involves a number of critical steps and is essential for accurate results. In part one of our Introduction to Metallography for Powder Metallurgy, Thomas F Murphy, Hoeganaes Corporation, USA, one of the PM industry's most recognised metallographers, looks at how best to prepare samples for the process.

As manufacturers and users of Powder Metallurgy (PM) materials, our mission as an industry is to develop and produce products that meet, and where possible, exceed our customers' expectations and requirements. In order to accomplish this goal, we must evaluate our products on a periodic and systematic basis using all the available test techniques. This evaluation requirement also extends to end-users, those in new product development and researchers, where the attributes that influence and determine the properties are defined.

Physical, mechanical, chemical, and metallographic tests are some of the methods used most often to characterise PM products. Of the aforementioned test techniques, metallography is probably the most effective and efficient diagnostic tool considering the amount of relevant information generated compared with investments in both manpower and equipment.

Metallography is primarily a collection of visual and imaging techniques that provide an insight into the history of a material or part and its behaviour. With consolidated PM materials, where the properties are controlled by the density, the combination of chemical composi-

Fig. 1 Metallographic testing underway in the Hoeganaes laboratory

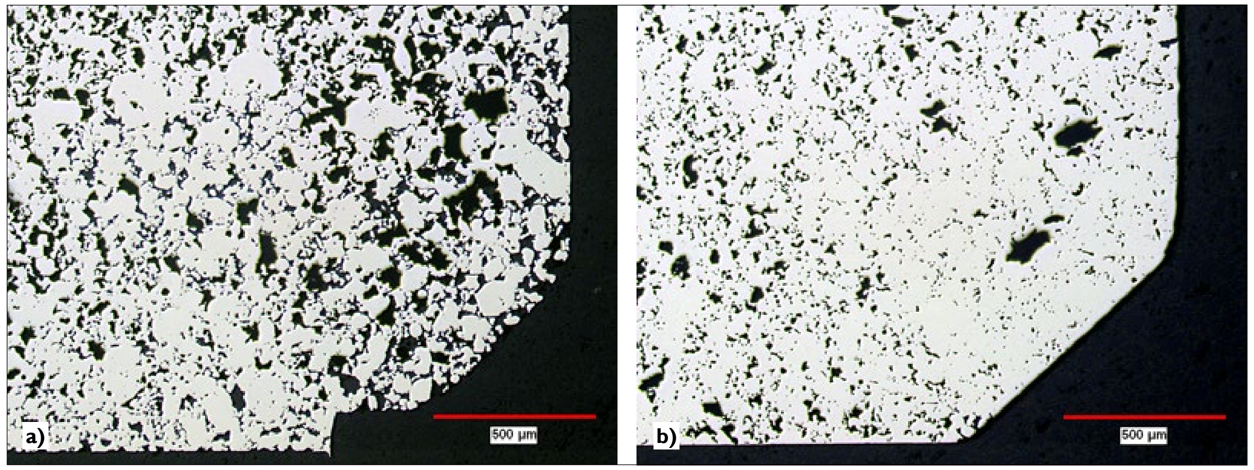

Fig. 2 Density difference as seen after altering the die fill and compaction conditions on the same type of part, unetched

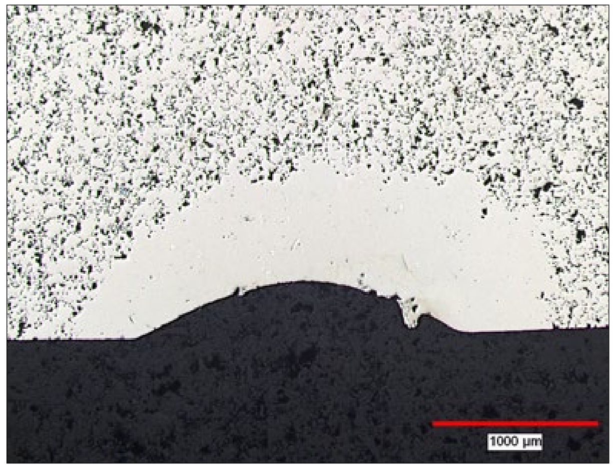

Fig. 3 Cross-section through a surface defect, unetched

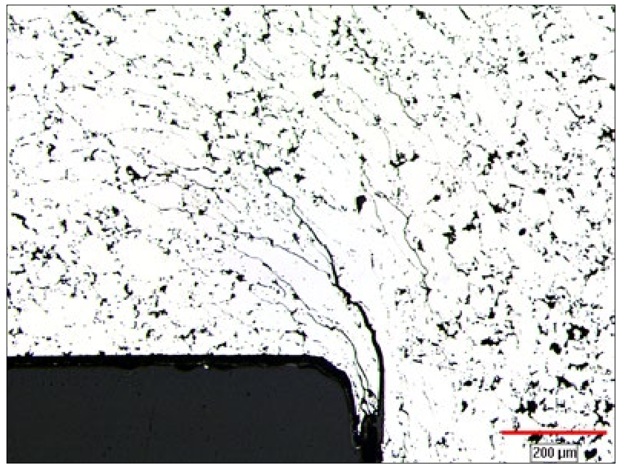

Fig. 4 The site of delaminations and cracks from improper powder and material flow during compaction, unetched

tion and alloying method, and the microstructure, metallography is the only test method capable of providing information on all three contributors. This is accomplished by the examination of properly selected and prepared metallographic specimens.

Sample preparation

Metallic specimens are opaque and, as a result, optical examination must be performed on carefully prepared planar (two-dimensional) surfaces. This preparation sequence is normally separated into several well-defined steps. These are:

1. Sample selection
2. Sectioning
3. Mounting
4. Grinding
5. Polishing
6. Etching and/or coating

Each step in the sequence must be successfully accomplished before progressing to the next. In most cases, errors committed during one step in the progression cannot be corrected later in the process. Adhering to the best possible procedures is the only means to produce consistent, reliable results. Basically, the effectiveness of an examination is ultimately dependent on the quality of the sample preparation.

Sample selection

When designing a test programme, the selection of the sample(s) to be tested is of primary importance. All the skill and care used in subsequent preparation will be wasted if the samples do not contain the desired information.

There are several reasons for selecting specific samples. These include:

- General microstructural analysis: Process control-type test, proportions of microstructural features contained in the cross-section(s) should be representative of what is in the part volume
- Failure analysis: Part failed in service or during testing
- Specific area analysis: Defect area or difficult to manufacture region
- Quantitative analysis: Part and cross-section is used to represent the typical or desired microstructure. Stereological or image analysis testing is usually performed on these specimens.

Figs. 2 and 3 represent cross-sectional areas that could fit into several of the sample selection categories mentioned above. In Fig. 2 (a and b), the results of changes in part design, die fill, and compaction are seen as a large difference in local density in the same location of a redesigned part. In Fig. 3, a cross-section through a surface defect is shown. In both figures, the features shown could be considered the reason for failure, a defect or difficult to manufacture region, or an anomaly found during a process-control test. Additionally, quantitative testing could be performed on the surfaces to provide values of the local density, size of features, specific locations, etc. In both cases, prior knowledge of the parts is a significant factor in the sample selection. With the density distribution issue, this was the location where a higher density was desired. In Fig. 3, the defect was visible from the part surface.

Another defect type is shown in Fig. 4. It is an area showing delaminations and cracks caused by difficulties with powder and material flow during compaction. The location of this condition is probably not visible nor is the location apparent from the part surface. It might have been an isolated region or characteristic of a compaction problem.

Sectioning

Samples selected for metallographic examination in the as-received state are rarely in the condition to be prepared for metallographic examination. Almost all must be reduced in size or have a specific section removed prior to preparation.

> *"...the effectiveness of an examination is ultimately dependent on the quality of the sample preparation"*

Pre-sectioning

In some cases, the parts are too large for the available metallographic sectioning equipment and pre-sectioning is required. This is normally accomplished with a non-precision device such as a hacksaw, band saw, or abrasive cutter. It is vitally important that the preliminary reduction in sample size be performed in an area a significant distance from the regions intended for analysis. Usually, the blade or sample feed, blade pressure on the sample, and/or coolant flow is not sufficient to protect the sample from substantial damage. This may render the sample unusable or require an exaggerated amount of grinding to eliminate the thick layer of surface damage. Fig. 5 (a and b) shows an extreme example of the depth and extent of surface damage from pre-sectioning. The prepared surface shown in these images was sectioned perpendicular to the pre-sectioned cut, where the upper side of the cross-section is the location of the pre-section cut.

The force of the blade cutting into the sample was sufficient to create an artificial increase in density along this surface (Fig. 5a) in addition to the material flow seen in Fig. 5b. The combination of transformation products has also been altered due to overheating, where the coolant flow and cooling capacity was not capable of preventing considerable microstructural damage.

Another example of improper abrasive sectioning can be seen in Fig. 6. The dangers of insufficient cooling when using an abrasive saw are seen as a change in the transformation products. In the lower magnification example, Fig. 6a, three distinct colours can be seen

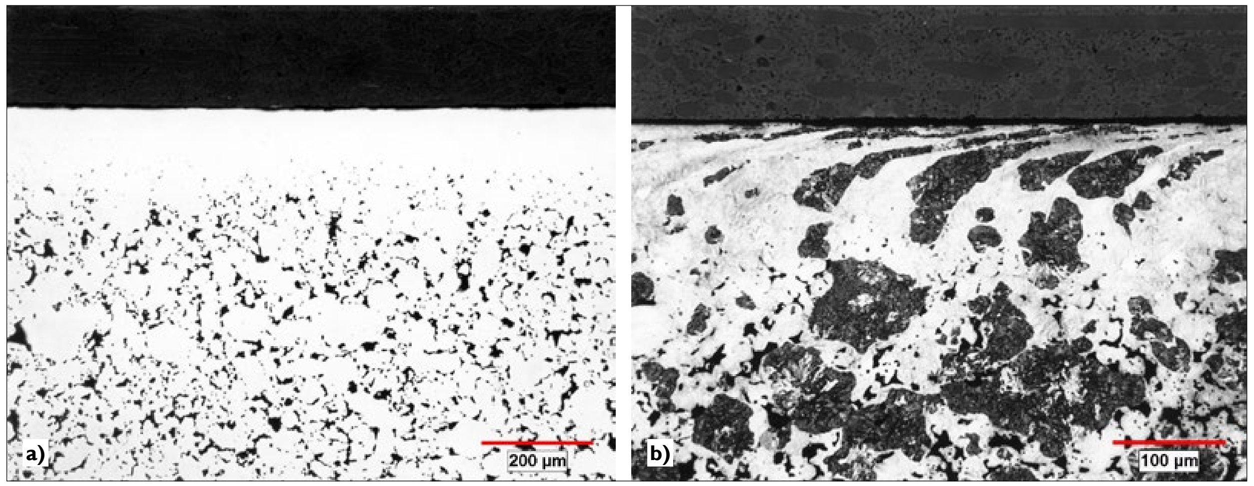

Fig. 5 Surface of a porous part sectioned using excessive pressure and insufficient coolant flow. This resulted in densification and an alteration of the transformation products at the surface. a, unetched; b, etched with 2% nital/4% picral

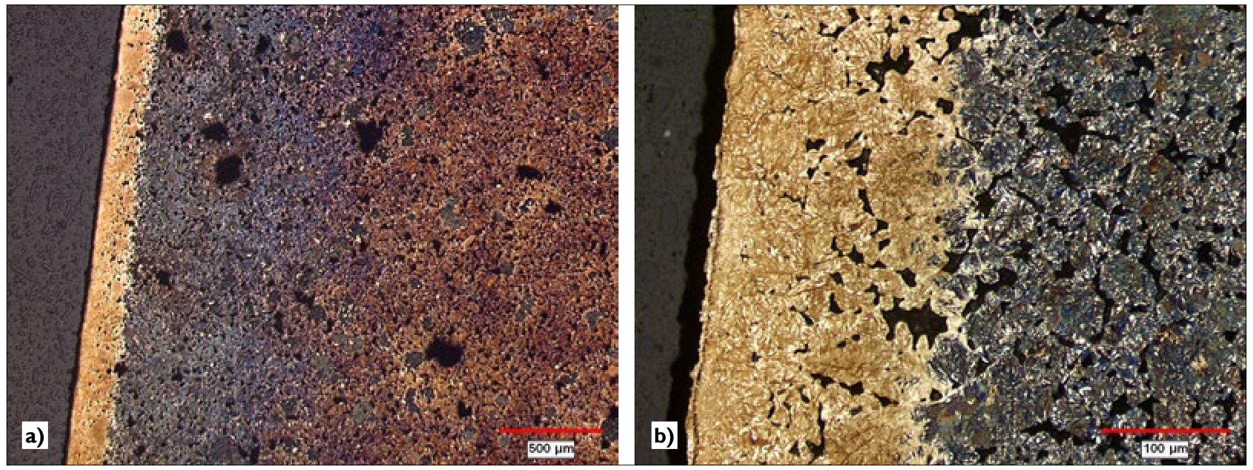

Fig. 6 Surface damage caused by insufficient cooling during abrasive sectioning. The variation in colours indicates a change in the combination in transformation products. a & b, etched 2% nital/4% picral

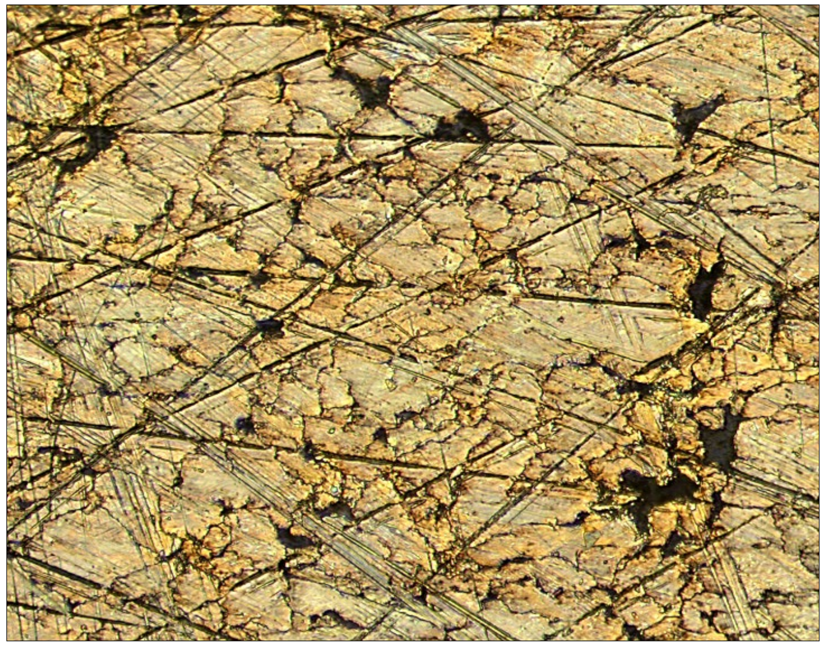

Fig. 7 The as-cut surface of a porous sintered bronze part taken directly from a precision wafering saw

in the cross-section; light tan on the outside (left side), dark blue to dark grey in the middle, and an orange/brown layer on the inside (right side) of the sample section. Each colour represents a different combination of transformation products with only the inside layer toward the right side being correct. The first two layers are seen at a higher magnification in Fig. 6b as martensite and retained austenite along the outside edge and a bainitic layer as the intermediate transformation product.

Precise sectioning

A wafering saw can be used as an alternative sectioning method if precise section location or sample protection is required. This device uses a blade that is substantially thinner than what is used in a typical metallographic cut-off abrasive saw. The blades are composed of SiC, Al_2O_3, or other abrasive materials. In addition, metallic substrate blades with a diamond impregnated rim are sometimes used. Location of the blade on the sample is often made using a micrometer. Consequently, the position of the cut surface is more accurate compared with the other sectioning methods and relatively thin sections can be produced if desired. The amount of surface damage is also substantially less than what would be generated using the other sectioning techniques due to the small amount of material being removed and the light loads on the sample. Fig. 7 shows an as-cut surface from a porous PM sintered bronze part. The scratch pattern from the abrasive contact is apparent, as would be expected, but several of the pores are already visible and have not been filled with the cut metal, even though bronze is relatively soft. This sectioning process has several drawbacks. It is much slower, possibly requiring several minutes to remove a small section, only relatively small samples can be sectioned and the thin blades are easily damaged.

Choosing the cross section

In addition to ensuring the cutting conditions in the abrasive saw are correct to protect the sample from damage, another important consideration is determining where the cross-section should be taken. In many situations, the microstructural features contained in the cross-section must be representative of the microstructure in the material volume. Not all planar sections removed from a sintered part satisfy this condition. The following example illustrates this problem with a simple sample geometry.

The decision to be made is where to remove a section from the cylindrical specimen and have this one section represent the entire microstructure of the part volume. The structure of the cylinder is composed of two distinct compositions, one cylindrical in shape located in the centre, extending from top to bottom, and the other surrounding the first, also extending from top to bottom. The diameter of the inner cylinder is half the diameter of the overall cylinder. This is shown in Fig. 8 with the orange region, A, and the grey region, B, representing the two microstructures. The question is; in which direction should the single section be removed to represent the overall microstructure in the cylinder?

Fig. 8b represents the two sampling directions that were considered. The transverse section is removed across the cylinder circumference and the longitudinal section is taken at the diameter of the circle, from top to bottom. With the diameter of the A composition half the overall diameter, the area occupied by A is remarkably different in the two sections. In the transverse section, A occupies 25% of the overall, A + B area. In comparison, A represents 50% of the A + B area in the longitudinal section. In looking at the individual contributions of A and B to the A + B volume, the transverse section gives the correct proportion of the compositions, 25% A and 75% B. The longitudinal cross-section overestimates the A contribution to the cylinder volume by a factor of 2. It is clear that analysing an unrepresentative cross-section will result in incorrect observations if the overall microstructure is of interest.

Removal of liquids

Another factor to be considered in the pre-mounting of samples is the entrapment of liquids in the pore structure. Oil, rust inhibitor, or other fluids contained in the pore network can result in analysis problems with PM materials. This not only affects metallographic testing, but chemical analysis results may also be affected if the fluids are not removed prior to testing. Chemical extraction of the liquids can be accomplished using a Soxhlet extractor, as pictured in Fig. 9. The apparatus is a condensing unit where a solvent is boiled in the bulb located at the bottom (a), the vapours travel up through the assembly to the condensing area at the top (c), and drip down onto the part located in the centre section (b). The condensed liquid fills the centre section until the syphon tube (d) on the side fills and the collected solvent drains. This process continues for a predetermined time, dissolving the oils in the part. It is crucial that the solvent is capable of dissolving the fluid trapped within the pore network.

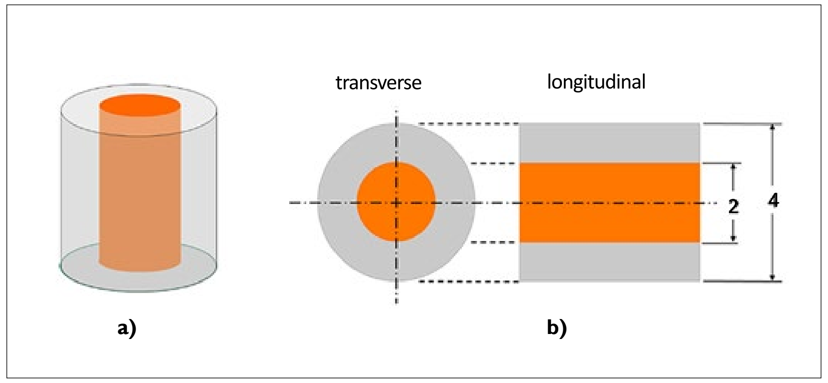

Fig. 8 Sample sectioning problem. The illustration (a) represents the overall cylindrical sample and the drawing (b) showing the compositions with the two sectioning directions. The values 2 and 4 are arbitrary and only intended to show the proportions of areas A and B [1]

Another method of removing the unwanted liquids is to heat the part under vacuum. The oil, etc. will be forced to the surface of the part and/or outgas under vacuum. The heating temperature should be carefully determined to not alter the microstructure in any way.

Mounting

Once the proper cross-section is removed from the part or test piece, it is usually encased in a plastic or polymer carrier mount for further processing. The mounting process offers several preparation advantages over preparing unmounted samples. The size and shape of the mounts makes handling and processing easier and more convenient, in addition to fitting standard configurations

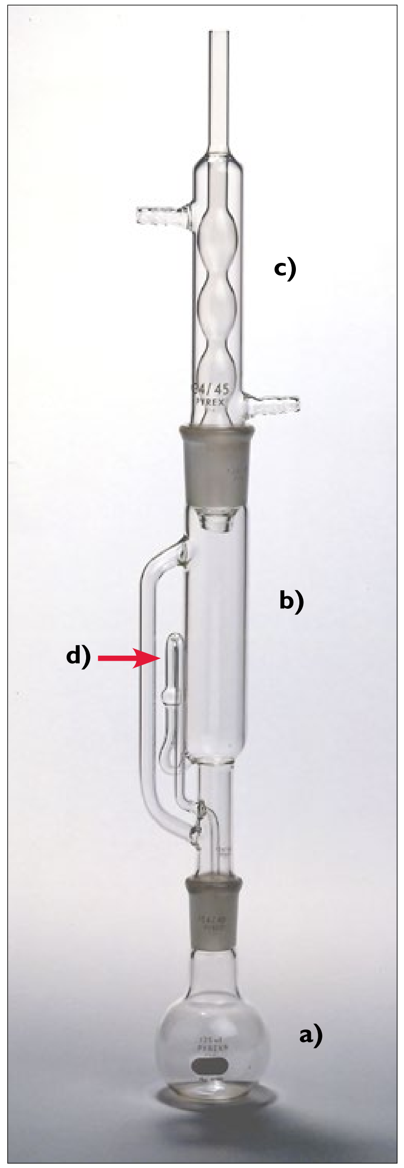

Fig. 9 Soxhlet extractor assembly. (a) Solvent reservoir bulb, (b) sample containment section, (c) condensing area, (d) syphon tube

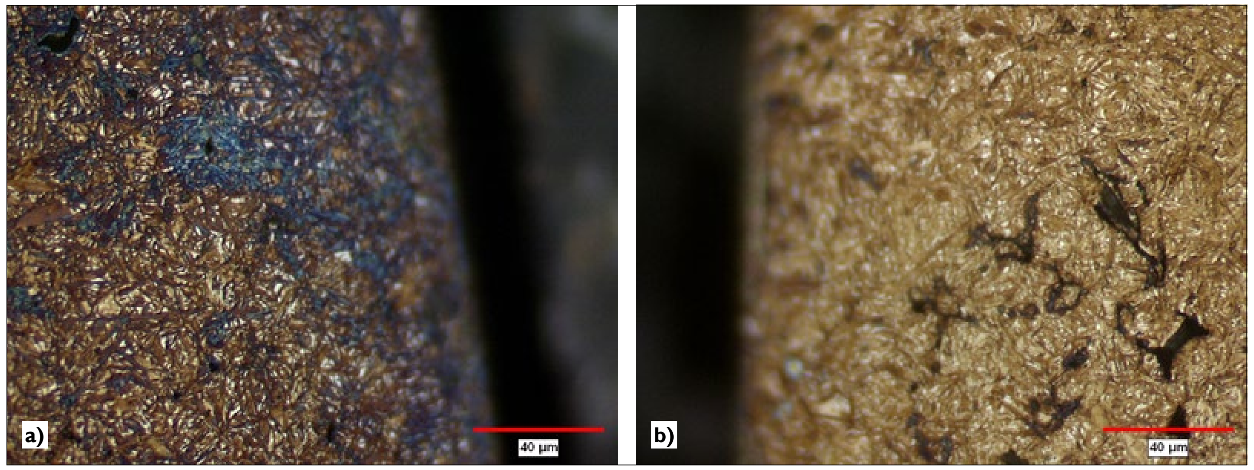

Fig. 10 The edges of two rounded gear teeth. The cause was a gap formed between the sample and the mounting material. The blue staining (a) is bleeding of entrapped liquid onto the sample surface. (a and b) etched with 2% nital/4% picral

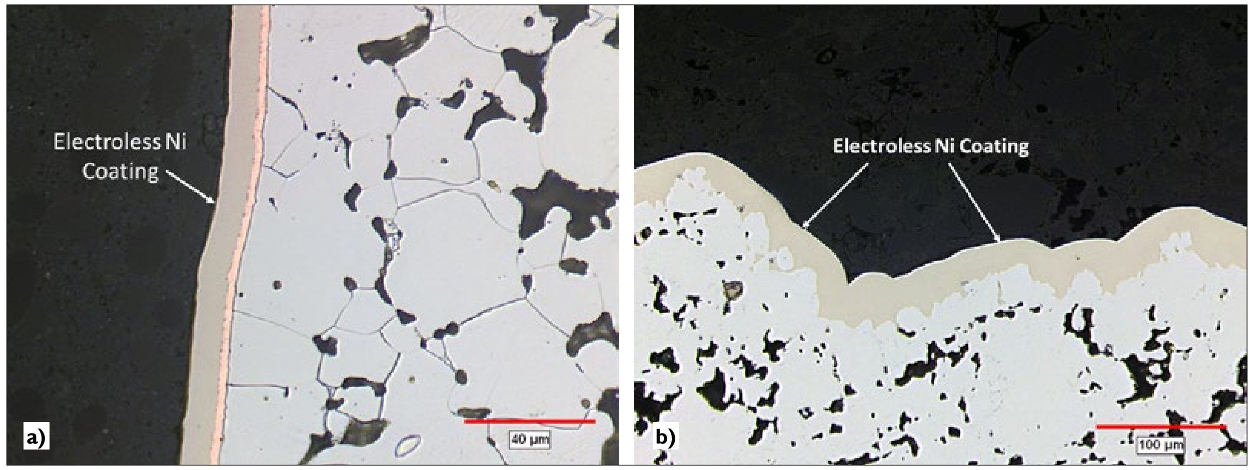

Fig. 11 Use of electroless Ni coatings for edge protection. (a) coating over a thin layer of Cu, (b) protection of a tensile fracture surface (a) etched 2% nital, (b) unetched

for automated or semi-automated preparation systems. Furthermore, mounting helps to maintain the integrity of edges and intricate shapes, permits the preparation of small cross-sections or individual powder particles and strengthens and protects delicate or fragile sections.

Three basic material categories are used for mounting. They are thermosetting, thermoplastic and castable. The thermosetting and thermoplastic groups require both pressure and elevated temperature to create the mount. Each material must be processed in a specific sequence of heating, cooling, and application of pressure. These must be followed to maximise the quality of the metallographic mount. Conversely, the castables are generally liquids that are used at room temperature, although the performance of some castable materials can be improved with elevated temperatures.

The thermosettings are Bakelite (phenolics), epoxy powder, and diallyl phthalate and are available in the form of powders, granules, and preforms. Thermoplastics are acrylics such as polymethyl methacrylate (Lucite), usually available in powder form. Epoxys and some acrylics comprise the castable group and are usually used as two or three part liquid or liquid-solid systems. As a word of caution, since the thermosettings and thermoplastics require heat and pressure to form the mount, the increased temperature and pressure may have adverse effects on some of materials to be prepared. Some microstructures may be changed by the heat required to create the mount and the increased pressures can cause physical distortion to fragile, thin, or delicate samples.

The physical properties of each mounting material type are different and one type may be preferred over another in a given application. For example:

- Thermosetting mounts are generally harder than the other mounting material groups
- Castable epoxy has adhesive qualities that may help retain small samples
- Thermoplastic mounts are generally clear, which may be beneficial where planar location is important.

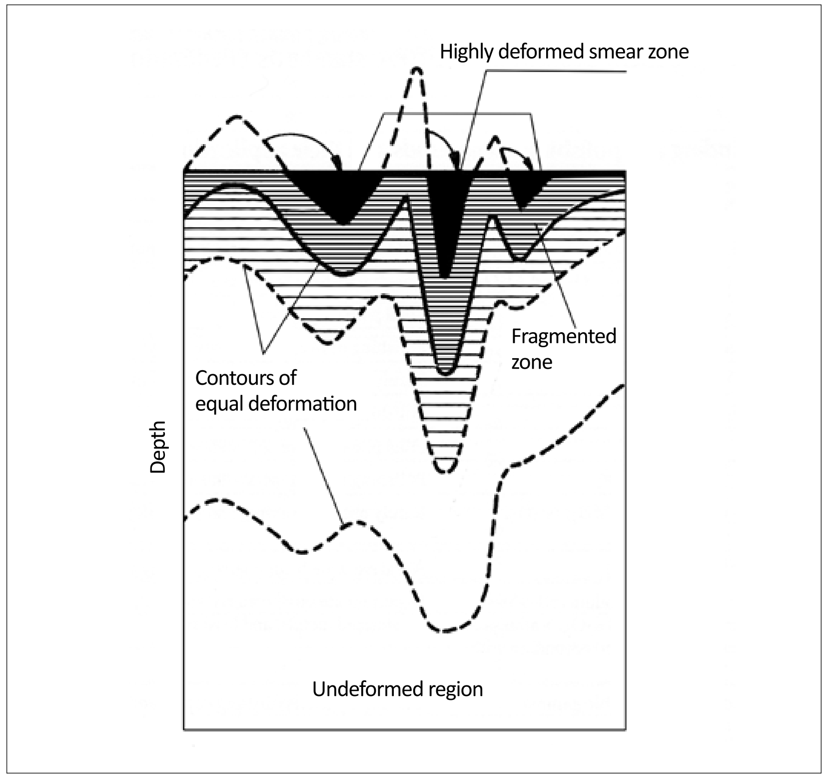

Fig. 12 Schematic drawing of a surface taken 90° to the original abrasive cut edge. The deformation is displayed as deformation contours [2]

Fillers and additives

Particulate fillers are often added to a mounting material to enhance specific mount properties. Hard additives such as glass fibres, ceramic, or metallic particles are sometimes mixed into the base mounting powder or liquid to increase the overall mount hardness. This will help to maintain the planar surface of the mount and minimise the height difference between the mounting material and the sample during preparation.

In addition, electrically conductive powders such as copper and graphite are added to the mounting material to create conductive mounts for use in electron microscopy. The additives can cause difficulties in some situations by contaminating the surface to be analysed. In addition, the metallic additives can sometimes react with etchants or cleaning fluids and change the appearance of the samples. Further, the conductive mounting materials use additives the PM industry routinely uses in alloying ferrous powders.

The possibility of erroneous information coming from the mounting material is real and should be recognised.

Edge protection

A possible problem arising from the use of high shrinkage mounting material, or not allowing the mount to cool under pressure where required, can be a gap created between the sample edge and the mount.

This condition often results in rounding of the sample edge and entrapment of liquids or abrasives in the gap. This is seen in Fig. 10 where the edges of two gear teeth are not in the same plane as the core of the teeth. The dark space at the edge of the teeth is the gap. It is obvious in these photomicrographs that any evaluation of the edges is questionable. The blue areas in Fig. 10a are stains produced by entrapped liquid from in the gap bleeding out onto the sample edge. These stains are not part of the microstructure and tend to disguise or hide the real microstructure.

In situations where the sample edges are of primary interest and importance, surface coatings can be applied as protection from rounding. One of the most effective is the use of electroless Ni coatings. Fig. 11 shows two uses of the electroless Ni coating. Fig. 11a illustrates how the applied Ni coating covers a thin Cu plating, allowing for measurement of the thin inner layer. In Fig. 11b, the electroless Ni coating was applied to the edge of a fracture surface. The delicate nature of the fracture surface is retained by the coating, thus allowing for evaluation of the fine features on the prepared cross-section. In both cases, the coatings were deposited, then the sections removed from the bulk sample using a wafering saw. It is important to mention that the direction of cutting should be into the Ni coating to prevent possible peeling of the coating from the sample.

Removal of the deformed metal layer

Regardless of the method used, abrasive sectioning of the sample produces a layer of material not representative of the true microstructure. The depth of the deformation is dependent on the sectioning technique and the skill of the operator. A schematic drawing of the deformation is shown in Fig. 12. The section illustrated in this figure is taken perpendicular to the original abrasive cut edge, where the most deformation is located at the edge, decreasing in severity with movement away from the edge. In metallographic sample preparation, the deformed and fragmented zones must be removed in order to reach the region representing the true microstructure, the undeformed region.

In porous specimens, the deformation caused by sectioning appears as a distortion of the microstructure and on occasion, as a densified layer, as was demonstrated in Fig. 4. The porosity network also presents

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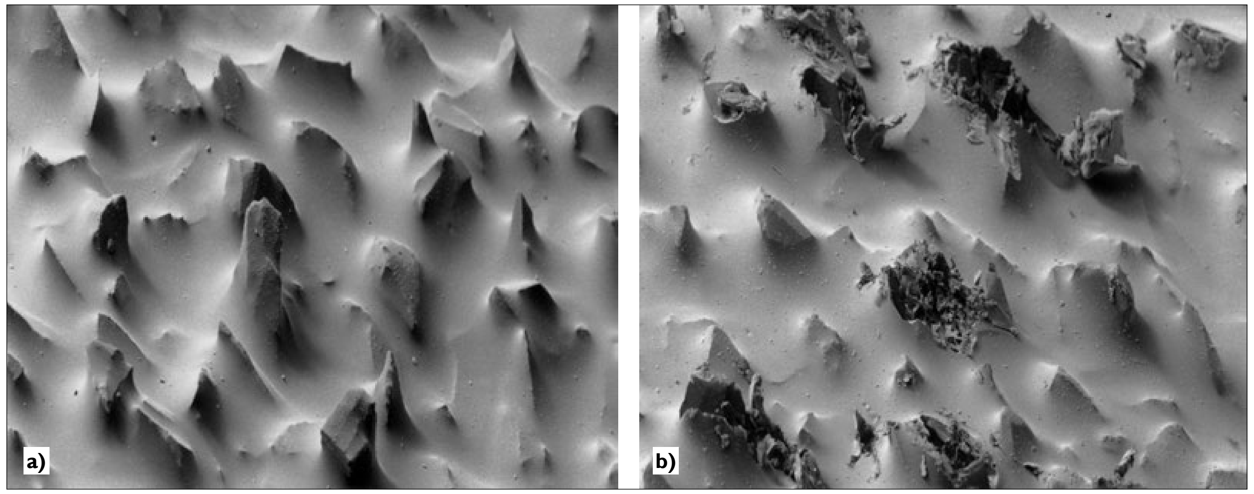

Fig. 13. Appearance of SiC paper in a new (unused) condition and after 30 seconds use

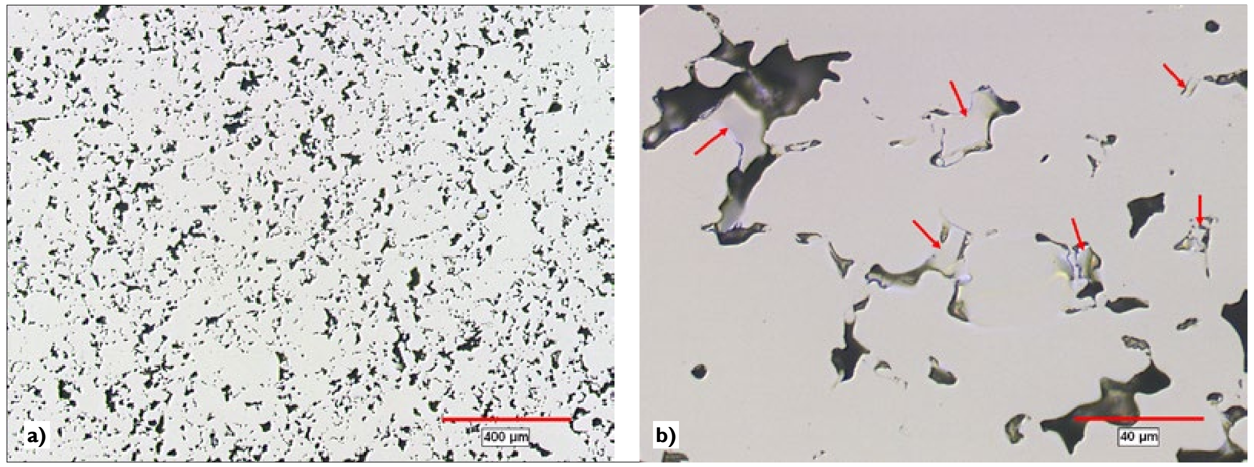

Fig. 14. As-polished surface photographed at two magnifications. The unopened porosity is not as apparent at the lower magnification (a), while in the (b) image, covered or filled pores are obvious, unetched

preparation problems unique to PM, where the pores are filled with material smeared into the void spaces by the force of the blade into the specimen. During sample preparation, the aim of the next two steps in the process, grinding and polishing, is to remove all of the deformation in order to reveal the true microstructure. This is done in a series of abrasive steps where a sequence of finer abrasives is used to remove the existing deformation on the surface, while producing a thinner deformation zone. At the conclusion of the final polishing step, the prepared surface should represent the true microstructure. It is at this point where an accurate evaluation of the microstructure can be performed.

Grinding and polishing

Grinding is often separated into two stages; coarse and fine. Each is basically a machining process where the individual abrasive particles are used as cutting surfaces. These can be silicon carbide (SiC), aluminium oxide (Al_2O_3), diamond, or others in special cases or with specific metals. The use of SiC is restricted only to grinding, while both Al_2O_3 and diamond abrasives are available in the range of particle sizes appropriate for both grinding and polishing. As a grinding medium, the abrasives may be used in the form of continuous belts, pressure sensitive adhesive (PSA) backed papers, rotating wheels, or composite discs and are suitable for both manual and automated preparation procedures.

Polishing abrasives are applied to various cloths or textiles as a paste, slurry, or aerosol. The cloth type is determined by the material being polished as well as by the abrasive composition and its particle size.

Grinding stage

Coarse grinding is used to remove all damage introduced during abrasive sectioning and provide a planar surface for further preparation. It may also be the means of locating a specific feature in a defect analysis, where the sample is ground to the location of a feature of interest. Subsequent grinding steps with finer abrasives are intended to remove the damaged layer produced in the previous step. As processing proceeds and finer abrasives are used, the damaged layer becomes thinner.

In the grinding process, variables such as sample pressure, cutting speeds, abrasive concentration, and grinding times are operator controlled and should be regulated to fit the specific application. In the vast majority of procedures, a coolant/lubricant is used to facilitate grinding and protect the sample from further damage. Alloy type, sample hardness, size, shape, etc. all control the effectiveness of the grinding medium.

During grinding, the abrasive particles embedded in a substrate material will crack, break, or wear. As this happens, the effectiveness of the grinding step is diminished. Abrasive particles may still be present on the substrate, but their abrasion rate will be substantially lower compared with the original paper. Where preparation processes are designed to be predictable and reproducible, overusing or reusing grinding papers is discouraged. In most situations, grinding for a time <30 seconds per paper is recommended. This effect can be seen in Fig. 13, where the cutting surfaces of a new SiC paper are shown in the (a) image and the condition of the same paper after 30 seconds use is shown in (b). Abrasive particles remain in the used paper, but many have been broken and removed and the performance of the paper is unknown.

Polishing

At the conclusion of grinding, a small deformed layer remains. This is removed through coarse and fine polishing. As with grinding, polishing is essentially an additional number of machining steps, but with much finer abrasives. There may be two or three individual steps with the cloth type usually changing with the change in abrasive particle size. The cloth and abrasive type, along with the particle sizes, are dependent on the material being prepared. As stated earlier, diamond and Al_2O_3 are the most frequently used polishing abrasives, although other polishes are used with specific metals and alloys.

Often, a three step procedure is used. Coarse polishing removes the scratches and damage from fine grinding. The intermediate step is usually where the pores are defined and opened, and final polishing removes the finest scratches; those from the second polishing step. Having the pore structure revealed accurately is of critical importance when preparing PM materials.

If the pores are not open, the appearance of the as-polished and etched microstructures cannot be correct and the results from any analysis are questionable. It is sometimes difficult to recognise the closed pores at a low magnification, but at a higher magnification, the condition is apparent. This is shown in Fig. 14 where image (a) is at a low magnification and (b) is a higher magnification view of the same surface. Arrows indicate locations of some of the closed pores in (b).

Recommendations

The following are a few suggestions for the metallographic preparation of PM materials:

- Samples must be cleaned between each grinding and polishing step. This will help prevent the carry-over of a coarser grinding or polishing abrasive to a finer grinding paper or polishing cloth. Washing the samples with soap and warm running water helps keep surfaces clean. An ultrasonic cleaner is also recommended.
- During polishing, using an etchant before the intermediate step can help open the pores. Where the preparation procedure is intended to remove a controlled amount of material using abrasives, the etching process will help by chemically removing material. Do this with caution, overetching at this stage can distort the size and shape of the pores.
- PM materials often retain water and lubricant from cleaning and polishing. Placing the prepared samples in a vacuum chamber can help dry the samples prior to analysis.

After preparation

At the conclusion of sample preparation, cleaning, and drying, the samples are ready for inspection and documentation. With the samples prepared correctly and free of stains, the colours of the as-prepared and etched surfaces should be characteristic of the alloy, phase, transformation product and constituent. White, tan, blue, or orange features should appear the same in photomicrographs.

Any change in appearance from what occurs naturally or in the etched/ stained condition should be investigated. For example, ferrite and retained austenite are naturally white and should be reproduced as white. Any colour shifts from improper microscope or camera adjustments should be corrected to make the reproduction and documentation of the microstructure more accurate.

With the extensive use of digital imaging, there are several possible causes for these colour shifts. They include uneven sample illumination, improper voltage setting of the microscope lamp, colour temperature or white balance setting on the camera, or software shading correction.

All efforts should be made to ensure these variables are controlled, thus producing the best images possible.

Author

Thomas F. Murphy, FAPMI
Scientist, Research & Development
Hoeganaes Corporation
Cinnaminson, NJ USA
Tom.Murphy@hoeganaes.com

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Atomising Systems Ltd: Making the equipment that makes the powder for over 20 years

Atomising Systems Limited, located in Darnall, a suburb of Sheffield, UK, builds and installs metal atomisers throughout the world for a wide range of applications. As well as equipment supply the company also manufactures a range of specialist metal powders. Dr David Whittaker reports for *Powder Metallurgy Review* on a visit to the company during which he met with founder and Chairman Dr John Dunkley, Managing Director Simon Dunkley and Dr Paul Rose, the company's Technical Sales Manager.

Dr John Dunkley, Chairman of Atomising Systems Limited (ASL), has some 40 years' experience in atomising plant technology, dating back to his early career when he started the Davy-McKee operation that supplied and installed such plant from 1974. When Davy-McKee decided to withdraw from this business, Dunkley saw the opportunity to found his own company to exploit the gap in the market created by its withdrawal.

Initially selling atomising plants, the company has grown to become a world-leading designer, manufacturer and supplier of these systems. However, quite early in the company's development it was recognised that the capital equipment business was subject to significant peaks and troughs in demand. In order to establish a more stable business model, the decision was therefore taken to expand into the manufacture and supply of a range of specialist powders.

The original office based operation was established in 1992 in a unit on the Sheffield Science Park. It started with a major project in Beijing to engineer an aluminium-lead alloy powder plant. This involved special safety engineering for the explosible powder.

In 2000, the company moved to a larger, 850 m^2 facility with a 250 kW power supply in Meadowhall, Sheffield, which allowed the installation

Fig. 1 ASL management team, left to right: Mark Wall, Powder Sales, Dr Paul Rose, Technical Sales Dr John Dunkley, Chairman, Simon Dunkley, Managing Director, Pete Taylor, Engineering, Ing. Dirk Aderhold, Technical Director

Fig. 2 Atomising Systems has been at the Darnall, Sheffield, facility since early 2012

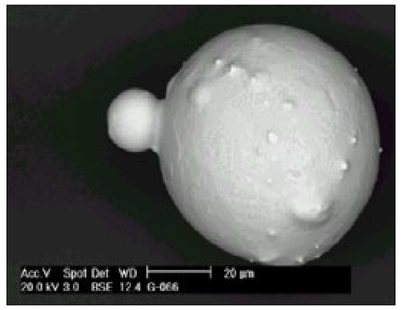

Fig. 3 Powder produced without anti satellite process

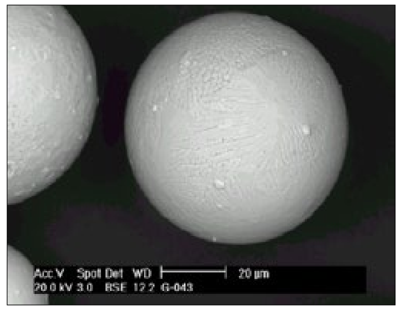

Fig. 4 Powder produced via ASL's anti satellite process

of a gas atomiser on which an anti satellite system was installed in 2003 (Figs. 3 & 4).

With the continuing increase in the powder making element of the company's business model, the need for additional shop floor space was recognised and this motivated the move to the current site, which was accomplished in early 2012. As Dunkley commented "When we moved into the new premises, we thought that we would never fill all of the additional space," but this assumption proved to be unfounded.

The facility, its third home during its 22 year history, has around 3200 m^2 of floor space and, in preparation for the move, the company upgraded the site's transformer rating by 1 MW from the pre-existing 750kW. The huge area came in handy during the months before the move when it was used as an assembly facility for six centrifugal atomisers for a Chinese customer and allowed every unit to be fully pre-assembled and test run before shipment. This large order was shipped in just six months from contract signature, a tribute to ASL's Engineering Manager, Peter Taylor. The atomisers were all PLC controlled and had online classification for automated super-fines removal.

Atomising Systems' recent sales figures are split fairly evenly between the two areas of the business, equipment manufacture and powder production. The company has a current complement of 36 employees across both operations.

The move into powder production

In developing its business, the company has made extensive use of public funding support for R&D. This funding has come from a range of national and European Union sources but, as Dunkley highlighted, the UK Government's SMART scheme (Small firms Merit Award for Research and Technology) has proved to be a particularly useful mechanism.

The company is currently involved in its sixth SMART-funded project. These projects have aided developments both in atomising plant technology and in powder materials. The first of these projects, run by Simon Dunkley, generated a capability for solder powder production. A wholly owned subsidiary company, QQ Solders Limited, was established in 1996 in a separate small industrial unit in Sheffield to manufacture and market these powders.

Through subsequent projects, a wide range of other powder types have been developed and taken to market, with more than 90% being exported. Customers include major multinationals. Prominent examples were cited as:-

- Gas atomised Cuprobraze powders for heat exchanger applications
- Water atomised stainless steel powders for filter applications
- High purity (5N) Si-P alloy powders for cancer treatment
- Gas atomised Ag-28Cu powder for brazing
- Water atomised AgSn powder for dental amalgams
- Gas atomised self fluxing alloys for hard facing
- Gas atomised, fine, low-satellite steel powders for HVOF thermal spraying
- Gas atomised special Cu alloys for diamond tool manufacture.

Recent market demands required a move into large scale water atomisation and prompted the move into larger premises with more power in 2011/2012. In this context, Paul-Rose underlined that the company demonstrated an impressive ability to ramp up capacity rapidly, to meet an increase in powder production levels from around 200 tonnes per year to around 250 tonnes per month within a twelve month period.

This was achieved not only by installing and commissioning new equipment, but also by recruitment and training of additional shop floor personnel to cope with three shift, 24 hours/day production. At the same time the company achieved ISO 9001 quality registration for its powder business and is now working towards environmental standard ISO 14001. To increase sales activity, Mark Wall, formerly with Ecka Granules UK, joined ASL in 2013 as Sales Manager, Powder Division. He has over 30 years experience of metal powder production, QC and sales.

Markets for atomising plants

The company has now built and supplied around 140 plants to customers in 34 countries. These range from major powder suppliers, of whom around half have dealt with ASL, to small in house operations.

Within this global reach, demand has been and continues to be strongest in the Far East. Around fifteen projects have now been completed in China and six in Korea. An increase in enquiries for projects in India has also been observed. Meanwhile recent projects have also gone to Spain, Sweden, South Africa, Germany, Russia, Austria and Ireland.

Although the production of atomising equipment supplied by the company has spanned a wide size range, with capacities varying between one and 250,000 tonnes per annum, there have also been numerous plants supplied for pilot production and pure research, ranging from a 10 kg unit for Madrid University to a 500 kg unit for a major powder producer.

The company estimates that only around 25% of its business in plant supply is for the PM sector, including some eleven iron powder projects. Likewise, the applications for ASL powders embrace a number of non PM applications such as dentistry, brazing, thermal spraying and hard-facing. There is a very broad spread of industrial sectors supplied with atomisers, including 10 units varying from 10 kgs to 6 t capacity for processing of molten smelted alloys for hydrometallurgical refining (Pt, Co, Ni, Ag, Au). Also precious metal alloys (Pt, Pd, Au, Ag, etc) where over

Fig. 5 A silver atomiser installed for a customer in China

Fig. 6 Gas atomiser unit ready for delivery to a customer in Russia

thirty plants have been supplied, including nine for the silver powder used in electrical contacts. A major market has been the supply of more than ten units for the manufacture of electronic grade solder powders using proprietary ultrasonic and, later, centrifugal atomisation technology.

Rose also commented that the company's business in atomising plant does not end with supply of the equipment. The need has also been found to provide a lot of education and training in plant operation and quality control procedures to customers as a support to the equipment supply.

Development of proprietary atomisation technology

Atomising Systems Ltd is essentially a technology supplier and thus recognises the need for continuing renewal of its proprietary technology, as systems are supplied into the market to serve ever more demanding and specialist applications. To this end, it seeks out new ideas all over the world and has cooperated with researchers in Russia, USA, China and Ukraine. It is also developing relationships with UK universities where funding for PM and atomising research seems to be recovering from a rather low level reached in

Fig 7 A four tonne iron powder atomiser

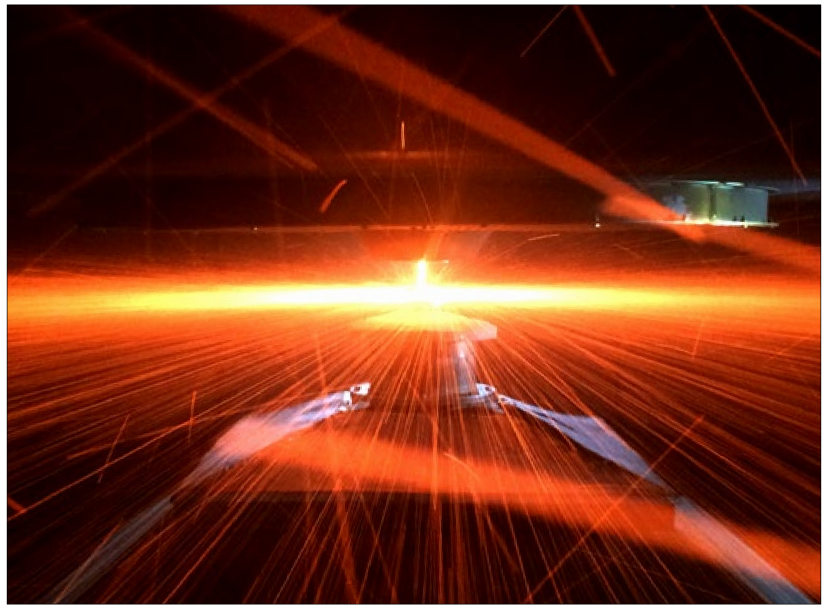

Fig. 8 Centrifugal (spinning cup) atomisation has been supplied for solders and is now under development for higher melting alloys

Significant technology developments at ASL	
Satellite-free powders	Technology for the production of satellite-free powders in gas atomisation
Ultrasonic vibratory atomisation	Used for very narrow size range, perfectly spherical solder powder production for electronics.
Centrifugal (spinning cup) atomisation	Also used in the latest electronic solder powder plants, using rotation speeds of over 50,000 rpm
Use of hot gas for atomisation	This has been installed on the 200 kg production unit at ASL and has allowed both reductions in gas consumption and a simultaneous reduction in median particle sizes, raising yields of fine powders <50 or <25 microns.
Development of ultra-pure (5N) silicon alloy for cancer brachytherapy	This is now accommodated in its dedicated small building and is progressing through clinical trials towards approval

Table 1 Significant technology developments at Atomising Systems

the last decade on the back of much interest in advanced PM in aerospace and oilfield technology, not to mention the very recent surge of interest in Additive Manufacturing.

Past significant technology developments at ASL are identified in Table 1. Of these achievements, most recently the scaling-up of the centrifugal (spinning cup) atomisation technology in both output capacity (t/hour) and melting temperatures (to 1500°C is targeted) is the subject of the company's latest SMART project. This technology is capable of achieving very narrow particle size distributions at good yield levels with standard deviations which can dip below 1.4 compared with a typical 2.0 for gas and water atomised powders.

Current shop floor equipment

The visit was concluded with a tour of the facilities conducted by Rose. Contrary to Dunkley's original feeling, the floor space is now amply utilised. An impressive range of equipment includes a very flexible 15-30 kg melter which can service a range of atomisers including both water and gas, which the company uses for R&D purposes and small lot production of typical batches from 15-500 kgs. There is a large-scale hot gas atomiser with 200 kg, 200 kW melter allowing production of some 600 t/yr of powder with median sizes from 20 to 100 µm with low satelliting and thus excellent flow properties.

Two water atomisers with 750 kg, 500 kW melters have production capacity of over 2000 t/yr of powders ranging from 10 to 500 µm. There are de-watering centrifuges and powder vacuum drying equipment to service the water atomisers.

A total of eight sieves from 500 mm to 1200 mm diameter are on site, with two powder classifiers, both capable of inerted operation. There are two 20 t blenders/homogenisation vessels. A centrifugal (spinning cup) atomiser is currently under development, and this is equipped with a 400 kg, 300 kW furnace.

A laboratory was also viewed which contained hand-held XRF equipment for preliminary chemical analysis (full chemical analysis is sub-contracted out to a local, fully certified analysis laboratory). A laser diffraction particle size analyser by Horiba and a range of laboratory sieving equipment are also in this area.

A Hall and Carney Flowmeter, a tap density machine and weighing equipment for assessment of apparent density and tap density of powders, are complemented by an optical microscope for powder shape evaluation. Filtration equipment for assessment of suspended solids in process water and a loss-in-weight moisture content machine are also available.

The team

A critical question with a company like ASL is succession planning; too often such companies can be too reliant on one man. Dunkley has had a long and distinguished career in the area, publishing numerous papers on atomisation and co-authoring, with Prof A J Yule "Atomisation of Melts" for Oxford University Press. He has also been awarded a PhD by Cambridge University for his published work in the field and was honoured with a Distinguished Service Award by the European Powder Metallurgy Association (EPMA).

Rose, with a PhD in physics joined ASL in 1998 and has been involved in costing and specifying atomisers ever since, travelling extensively to negotiate contracts all over the world as well as gaining hands-on experience of commissioning equipment himself.

After some years of careful searching, Dipl. Ing. Dirk Aderhold, an experienced process development engineer, was recruited in 2004 to train to succeed John Dunkley in charge of atomising technology and development. He has now been involved in several major new developments and has commissioned plants world-wide. He was appointed Technical Director in 2012. After working in the business for nearly twenty years Simon Dunkley took over as Managing Director in 2011, thus John Dunkley is now happy to progressively reduce his workload. The firm remains family-owned and independent and looks forward to the future with confidence.

Outlook

After a very busy two to three years of headlong expansion, followed by a period of consolidation, ASL is now looking out for further premises to accommodate new lines of business to satisfy pressing demands from international clients. With a secure generational transition in place, the team look forward to further advancing the science and practice of metal powder production by atomisation.

Author

Dr David Whittaker is a consultant to the Powder Metallurgy and associated industries.
Contact +44 1902 338498
email: whittakerd4@gmail.com

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Kanthal APMT™: Making high temperature sintering more cost competitive

Kanthal APMT, an advanced Powder Metallurgical FeCrAlMo alloy, has been introduced to the market in a wide range of product forms including hot rolled wide plate, bar, rod, wire and extruded tubes and is therefore a facilitator for furnace components for use at a temperature range where ceramics or cold wall chambers have previously been the only viable options. Bo Jönsson and Roger Berglund, of Sandvik Heating Technology, present the alloy's basic properties and describe several applications within Powder Metallurgy where the alloy provides the potential to increase sintering temperatures and extend lifetime of critical components.

Kanthal® materials and systems from Sandvik have for decades been a standard solution when it comes to electrical resistance heating. The product range has expanded over the years from FeCrAl and NiCr metallic alloys to include a range of ceramic materials and heating elements, such as Kanthal Super ($MoSi_2$) and Kanthal Globar® (SiC) heating elements, as well as seamless extruded tubes and also into components and systems for thermal processing and process heating.

This evolution is now taking another step through the introduction of a range of Kanthal high temperature materials and components. These are intended to withstand and protect from heat in components such as retorts and muffles for PM sintering, furnace rollers and furnace furniture and not to primarily generate heat in heating elements.

Traditionally, wrought or cast Ni-base alloys have been used for this type of high temperature construction, but their limited oxidation resistance above 1100°C and sensitivity to carburising may be a problem in many industrial high temperature processes. As an example, PM steel sintering is largely carried out at the (in this context)

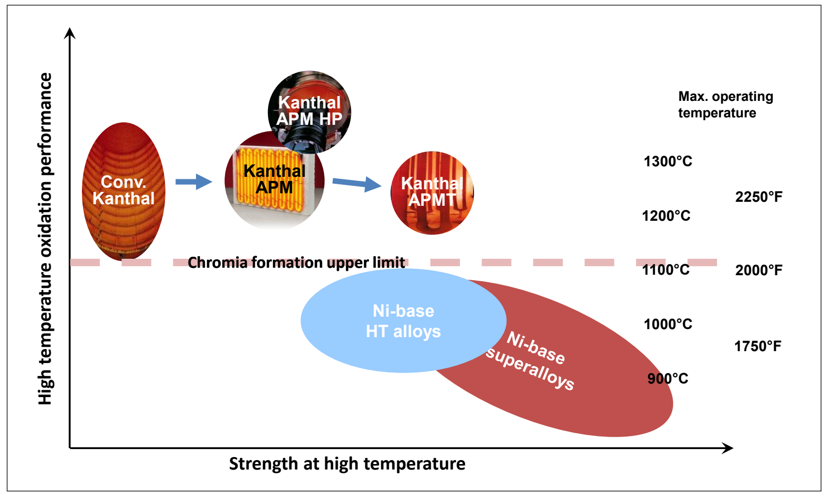

Fig. 1 Schematic relation between main groups of high temperature alloys in terms of creep strength and typical maximum application temperature in oxidising environments. Ceramics may have very good oxidation and corrosion resistance, but are typically relatively brittle and are therefore not included in this schematic view

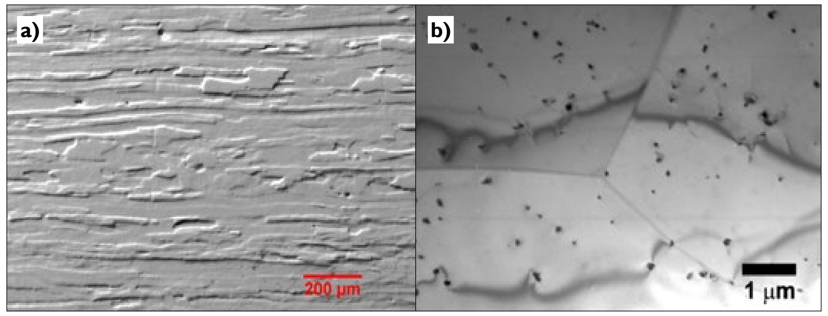

Fig. 2 Polished and etched micrograph from 8 mm hot rolled plate, b) TEM section showing the ferritic base metal, grain boundaries and refractory strengthening particle dispersion

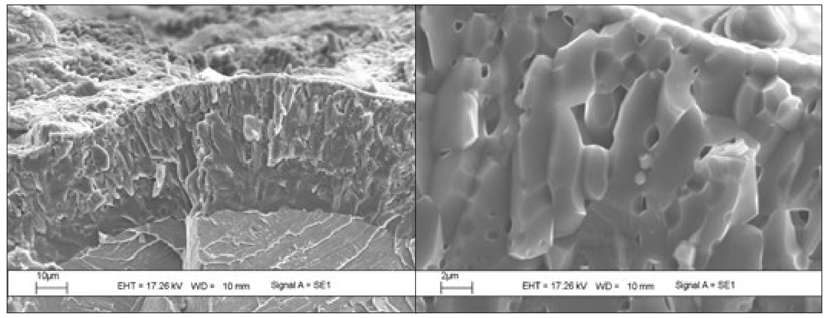

Fig. 3 SEM example from fractured section of APMT exposed to 1300°C for 1100 hours using 20 hour thermal cycles. The dense and coherent oxide is evident even after this fairly tough exposure

	Cr	Al	Mo	RE	Fe
Kanthal APMT (RSPM)	22	5	3	Added	Balance
Kanthal APM (RSPM)	22	5.8	-	Added	Balance
Kanthal AF (Conventional)	22	5.3	-	Added	Balance

Table 1 Nominal compositions of Kanthal APMT and previously developed materials

relatively low temperature of around 1130°C, primarily due to the limits set by available construction materials and the high costs involved in going to conventional vacuum sintering.

Conventional wrought FeCrAl high temperature alloys are, on the other hand, well known for their superior oxidation resistance up to 1300°C or even 1400°C. This outstanding resistance to oxidation is based on their ability to form a very slow growing and protective alumina scale during service. However, the main drawback of these alloys is their relatively low mechanical strength at high temperatures that severely limits their application in mechanically stressed components, such as hot retorts containing a vacuum.

Sandvik researchers were able to enhance the relatively poor high temperature creep properties of FeCrAl alloys as long ago as the late 1980s by inventing a unique rapid solidification powder metallurgical process route (RSP). As a result, Kanthal APM was presented to the wider market in 1989. Since then Kanthal APM has become the preferred choice for high performance and mechanically stable electrical heating elements and radiant tubes within semiconductor and industrial high temperature processing.

This article will give a brief introduction to the further enhanced strength alloy Kanthal APMT, a FeCrAlMo alloy that bridges the gap between high temperature metallic alloys and ceramics, in terms of application temperature (see Fig. 1), and will also look at some application examples within Powder Metallurgy.

Kanthal APMT properties

Kanthal APMT is optimised for hot strength and retained alumina scale protection up to 1250°C or, for shorter periods, even up to 1300°C. This is a temperature range where Ni-base alloys degrade rapidly due to accelerated oxidation and grain boundary softening. Table 1 compares the chemical composition of Kanthal APMT with those of previously developed Kanthal alloys. Fig. 2 illustrates the prevailing strengthening mechanisms in Kanthal APMT.

By combining additions of Mo and trace elements with the unique process route, the result is an alloy that exhibits a unique combination of resistance to oxidation and corrosion and excellent form stability that exceeds that of Ni-base alloys at higher temperatures. An adherent alumina layer on the alloy surface forms spontaneously during service (Fig. 3). This thin oxide scale provides resistance to corrosion attack in most industrial atmospheres and gives great advantages compared with chromia-forming high temperature Ni-base alloys in terms of maximum operating temperature and life.

Oxidation resistance

Alumina formation provides several major advantages; slow oxidation rate, very high scale adherence, chemical stability towards water, carbon and sulphur. These advantages transform into a number of practical benefits in oxidising and carburising environments compared with the best Ni-based chromia-formers, in terms of longer lifetime (of the order of 10 times), 100 to 200°C higher possible operating temperature, as well as very minor particle emissions (spallation) and negligible amounts of gas phase emissions. There is no critical temperature range where oxidation rates are accelerated, although isothermal exposures have shown that oxide growth rate, although very slow, is similar at 800 and 900°C due to a gradual change to the stable and more protective α-alumina phase at higher temperatures.

Lifetime assessment

The lifetime of components is typically limited by creep deformation and/or by oxidation and hot corrosion. Design of components for service at very high temperatures also makes it necessary to understand and control the combined action of simultaneous oxidation and mechanical stresses that may occur from external loads or from thermal gradients. At high temperature and below a certain critical load, the failures are typically determined by the oxidation process. On the other hand, at higher loads and lower temperature, failure is often controlled by the mechanical properties.

During exposure, thermal cycling and also mechanical damage often cause micro-cracks or even macro-defects in the oxide scale. Aluminium then diffuses from the interior of the alloy to the scale interface to repair the scale. The oxidation-limited lifetime is reached when the aluminium level beneath the oxide layer is too low to support the continued alumina formation in those defects. It is found that the critical aluminium level is in the range from one to three wt. % Al remaining in the alloy. Comparative life testing may be assessed by using electric current to heat the samples to a specified temperature as shown in Fig. 4. Based on oxidation tests such as these and theoretical models, it is actually possible to estimate oxidation life of real components fairly accurately.

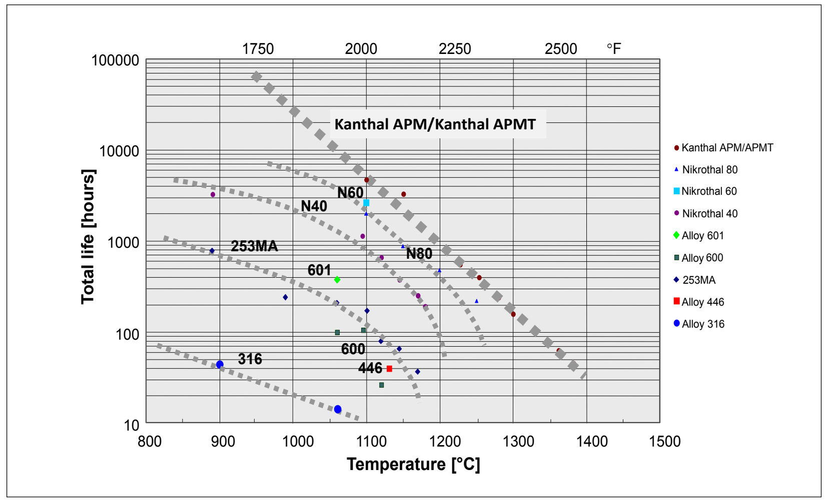

Fig. 4 Oxidation lifetime for various alloys as measured according to ASTM B78 – 81. Thermal cycling 2 minutes on/ 2 minutes off. Samples are Ø 0.7 mm wire

Corrosion resistance in controlled atmospheres

Kanthal APMT forms a protective scale in most commonly-used controlled atmospheres such as endo- , exo-gas and H_2. The only condition in terms of corrosion and where some caution is needed and where Ni-base alloys may be a better choice, due to risk for nitriding, is very dry N_2 or N_2/H_2 mixtures.

Furthermore, due to the extremely dense alumina scale formed and its low permeability of carbon-containing species, the carburisation resistance of Kanthal APMT is excellent in comparison with chromia-forming materials. Kanthal APMT is almost entirely insensitive to metal dusting and its alumina surface is also non-catalysing, which reduces the amount of graphite and coke deposition. Kanthal APMT is also very stable towards sulphur attack compared to Ni-base alloys. As a consequence, Kanthal APMT is highly resistant to combustion atmospheres from fuels such as natural gas and coal up to its maximum recommended temperature.

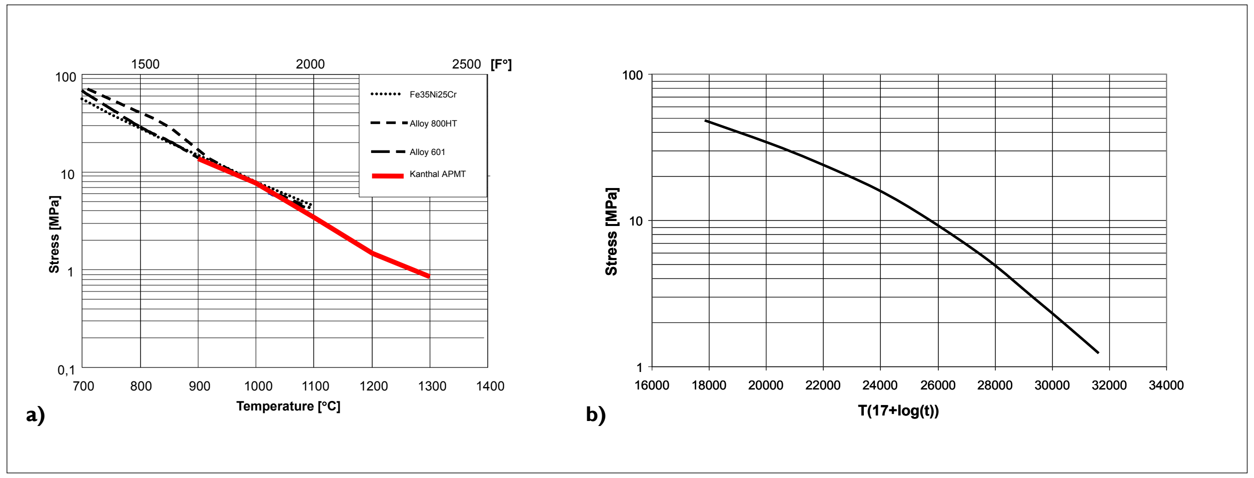

Fig. 5 a) 10,000 hour creep rupture life, b) Larson-Miller plot of creep rupture data. LM constant assumed to 17

Form		Dimensions (mm)	Dimensions (inch)
Plate	Width	≤ 1200	≤ 47,24
	Thickness	3 - 20	0.12 - 0.79
	Length	≤3000	≤ 118,44
Extruded tubes	Outer diameter	26 - 260	1.05 - 10.24
	Wall thick.	2.87 - 11.0	0.11 - 0.43
	Length**	3000 - 13000	118.11 - 511.81
Cold Rolled Strip*	Width	≤ 205	≤ 8.07
	Thickness	0.2 - 3	0.01 - 0.12
Wire	Ø	0.2 - 9.5	0.01 - 0.37
Rod	Ø	5.5 - 12	0.22 - 0.47
Round bar	Ø	≤ 100	≤ 3.94
	Length	≤ 4500	≤ 177.17
Forging blanks	Width	≤ 500	≤ 19.69
	Thickness	35 - 170	1.38 - 6.69
	Length**	≤ 3000	≤ 118.11
Square bar	ϕ	≤ 150	≤ 5.91
	Length	≤ 4500	≤ 177.17

**Cold rolled strip can be delivered as cut to length products*
***Length depending on cross section*

Table 2 Forms of supply. Other sizes and forms can be discussed on request

High temperature creep resistance
In general, the creep rupture strength for APMT alloys is comparable to high temperature Ni-base alloys above 900°C, but the advantage for Kanthal APMT becomes greater, the higher the temperature (Fig. 5). An additional difference in favour of Kanthal APMT is the lower density (7.25 vs. typically 8.1-8.3 g/cm³), which lowers the stresses imposed by gravity. Well defined rupture and creep rate data exist for APMT alloys in the temperature range 1100 to 1300°C, where Ni-base alloys suffer from severe oxidation and, above 1200°C, also from a loss of structural integrity due to initiation of grain boundary sliding.

Welding and joining
The APMT alloy is normally welded using TIG/GTAW. Preheating to approximately 250°C and post-weld annealing at 850°C is however necessary. Some creep strength is lost in the weld and this is normally handled by design so that welds are placed at positions where the stress level is somewhat lower or cross sections are larger. As a rule of thumb, loss of creep strength in an APMT/APMT alloy TIG butt-weld corresponds to a 100°C temperature rise of the base alloy.

Kanthal APMT has slightly lower thermal expansion coefficient compared with common austenitic FeNiCr and NiCr alloys. Although generally an advantage, in dissimilar welds, this will impose stresses during thermal cycling. Welds in general, but dissimilar welds in particular, should therefore preferably be positioned where stress levels or temperatures are below their maximum levels.

In addition to TIG welding there are several other welding techniques that can be used, such as laser or MIG welding. Also brazing has been shown to give good results when it is possible to prepare the gap to adequate tolerance and to perform a heat cycle in vacuum.

Forming and cutting
Kanthal APMT is ductile at room temperature with elongation to rupture between 10 and 25% depending on product form. Since room temperature impact strength is relatively low, it is recommended that plastic deformations are performed using a preheating to ≥250°C when possible, and especially so for heavy dimensions.

Bending over an edge with controlled radius gives less localized stress compared to V bending with a press and is preferred when possible. Using V bending, preheating is generally needed. For preheated plate and strip, the minimum bending radius is $R_{min} = 2t$ using edge and $R_{min} = 3t$ using V bending. Tough forming operations, such as press forming, are readily carried out at red hot temperature where the ductility is extremely high.

Cutting may be performed with different conventional methods, including laser cutting using Ar shield gas, but water-jet cutting has the advantage of not creating mechanical stress and also not affecting the surface conditions negatively.

Application examples

Hot wall vacuum and inert atmosphere bell furnace designs
Most batch processes using vacuum or low pressure at high temperature use cold wall equipment with a graphite or Mo-heated process chamber inside a water-cooled pressure bearing vacuum chamber. The unique combination of properties of Kanthal APMT will provide a number of important opportunities. Due to its combination of adequate oxidation resistance and sufficient mechanical strength, it greatly expands the upper temperature limit for hot wall furnace design, in which the chamber (retort) wall combines the function of keeping a low pressure or vacuum and also constituting the hot wall that transfers heat to the parts inside. This double function obviously puts high demands on the wall material, but, on the other hand, it offers great advantages for batch as well as continuous processes.

Here the alloy is a facilitator, since hot wall retorts using Kanthal APMT can be applied up to at least 1250°C, which is between 100 and 200°C more than is possible with Ni-base alloys. The heat is then provided externally by a conventional electric or gas fired furnace or even induction.

The advantages with hot wall designs are great in terms of increased productivity and lower cost due to:

- Lower investment
- Faster cycle time
- Effective and easy access loading
- Multiple process chambers may be served by one heating system
- Reduced energy consumption due to reduced or eliminated need for water cooling.

The advantages in using a metallic alloy as compared to possible ceramic solutions include weldability, freedom of design, ruggedness and gas tightness. However, depending on temperature and material thickness there is a limitation in size if the wall will have to carry the atmospheric pressure at the full operating temperature. Some application examples are given below.

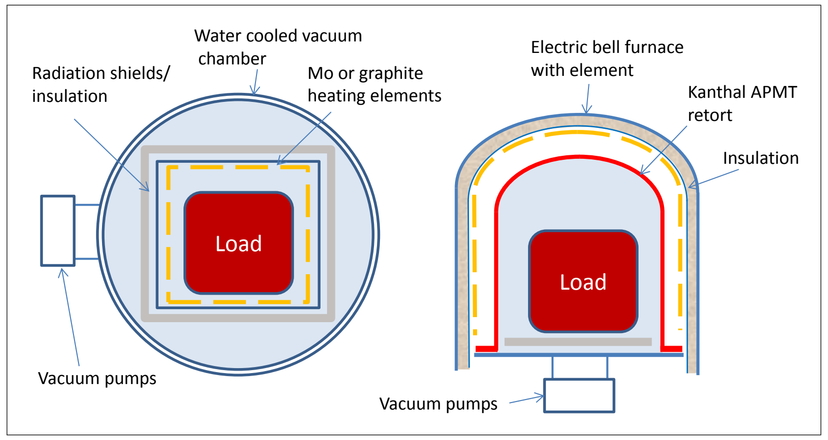

Fig. 6 Principle of cold and hot wall vacuum furnace (blue shaded area indicates vacuum)

Fig. 7 Hot wall vacuum retorts used for high temperature CVD coating. One heating unit serves several process retorts

Retorts
PM vacuum/inert gas sintering of magnetic and medical implant materials such as SmCo and CoCr alloys is carried out at high temperature close to 1250°C in order to achieve almost 100% density and to fulfil high demands on surface quality and mechanical and magnetic properties. To realise the advantages with hot wall technology, the sintering step has been carried out very successfully by using Kanthal APMT retorts made from plate and tube in combination with Kanthal external electric resistance heating systems.

Alumina formation provides additional advantages. This comes from the extremely high affinity to oxygen of the alumina-former Kanthal APMT. While the protective alumina scale forms on its surface, it also acts as a getter for residual oxygen and adsorbed water and thereby further reduces the level of oxygen in the chamber and improves sintering of easily oxidised Cr containing alloys. At very low oxygen levels and dew points in inert or H_2 atmosphere, the thin alumina scale therefore still forms, although very slowly, which prevents metal-metal sticking. In the case of carburising gases, the alumina forms a very protective barrier towards carburisation and also prevents build-up of coke deposits.

Muffles, load support, baskets, trays and belts for continuous processing
Sintering of conventional press and sinter steels is performed using NiCrFe alloy muffle continuous furnaces operating at ~1120°C. This temperature is chosen primarily for reasons of high temperature performance of the available NiCrFe-based furnace construction materials and, therefore, steel powders are mostly optimised to give acceptable properties at this temperature. Increased sintering temperature gives substantial improvement potential for part mechanical properties through a greater freedom of alloy design and there are important threshold levels at which PM parts could qualify into even higher loaded components and gain substantial new market share. Even more interesting is the prospect of increasing the sintering temperature to levels that give closed porosity, thereby making it possible to apply a HIP (Hot Isostatic Press) cycle directly on the sintered parts to reach 100% density. This could potentially be applicable to pressed, MIM (Metal Injection Moulded) or Additively Manufactured parts.

Kanthal APMT therefore offers a possibility for greatly improved properties in PM through the use of relatively conventional all metallic furnace designs, for components such as muffles, load supports, trays

Fig. 8 Vacuum sintering retort made from Kanthal APMT used for five months at approximately 1250°C temperature

Fig. 9 Kanthal APMT tray in steel sintering operation

and baskets. In these components, the alumina formation on Kanthal APMT also provides an inert and non-sticking surface towards the sintered products. The designs will be fairly conventional, although built using a quite unconventional alloy and operating at > 1200°C.

Kanthal APMT trays have also been successfully applied to replace ceramic trays in batch, belt and pusher type furnaces, withstanding thermal shock in mesh belt sinter quench furnaces to enable automated handling.

Other applications

A wide range of tube, bar, wire and plate dimensions is available for high temperature components including radiant tubes. Kanthal APMT can offer great advantages, especially in high temperature operation or in carburising atmospheres due to the clean and inert alumina surface.

Thermocouple protection tubes and thermo-wells see all environments and temperatures possible. At temperatures above 1100°C in oxidising environments, Kanthal APMT replaces brittle ceramic protection tubes and in, for instance, petrochemical processes its outstanding resistance to carburising and sulphidation solve severe corrosion problems experienced with Ni-base alloys.

Furnace rollers made from conventional NiCr(Fe) wrought or cast alloys suffer from severe oxidation and often also carburisation, when applied in oxidising conditions at the high temperature needed for the treatment of certain stainless steel grades, for example. Lifetime in this applicaiton has been improved from six to twelve months to at least four years when changing to Kanthal APMT rollers. By eliminating the need for cooling in many cases, Kanthal APMT also helps reduce energy consumption.

Conclusion

Kanthal APMT is an advanced powder metallurgical dispersion strengthened FeCrAlMo alloy, optimised for continuous service as a construction material in the temperature range up to 1250°C in oxidising and corrosive environments. It has now been introduced in a wide range of product forms, including wide format hot rolled plate, bar, wire and extruded tubes.

With its unique combination of alumina-based oxidation resistance and high form stability, Kanthal APMT opens up new possibilities for design of improved thermal processes within Powder Metallurgy. This will offer the potential for improved product properties, higher productivity, lower costs and large energy savings.

Contact

Bo Jönsson or Roger Berglund
Sandvik Heating Technology AB,
Hallstahammar, Sweden
Tel: +46 220 21752
Email: bosse.jonsson@sandvik.com

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[3] Material datasheet available at www.kanthal.com

AMPM2014 Conference: Controlling properties and performance in Additive Manufacturing

Underlining the growing importance of Additive Manufacturing as a metal-forming technology, the Metal Powder Industries Federation (MPIF) organised and sponsored the Additive Manufacturing with Powder Metallurgy (AMPM) conference in Orlando, Florida, from May 18-20, 2014. The conference ran in parallel with the PM World Congress and proved a popular addition to the event, with many of the sessions running at full capacity. Dr David Whittaker reports on three presentations that discuss issues that are key to the processing of metal AM components.

Additive Manufacturing (AM) has several variants whose abilities to build highly complex shapes, and in some cases shapes that cannot be produced in any other way, are well recognised. These technologies have recently been brought firmly into the general public's awareness through their designation as 3D printing. In order for AM to be considered as a viable technology for serial production, as opposed to its original use as a prototyping method, it is of great importance for it to be able to demonstrate the ability to exercise robust control over the process to ensure reliable and repeatable manufacture of products.

To match the detailed knowledge that has provided the long established foundation for the metal forming technologies with which AM seeks to compete, it is also necessary to gain a detailed understanding of the relationships between processing parameters and product microstructure and, consequently, product properties and performance.

Sessions in the AMPM conference specifically addressed these important issues.

Testing and powder characterisation

A session on the testing and characterisation of powders for AM provided an insight into this aspect of the process.

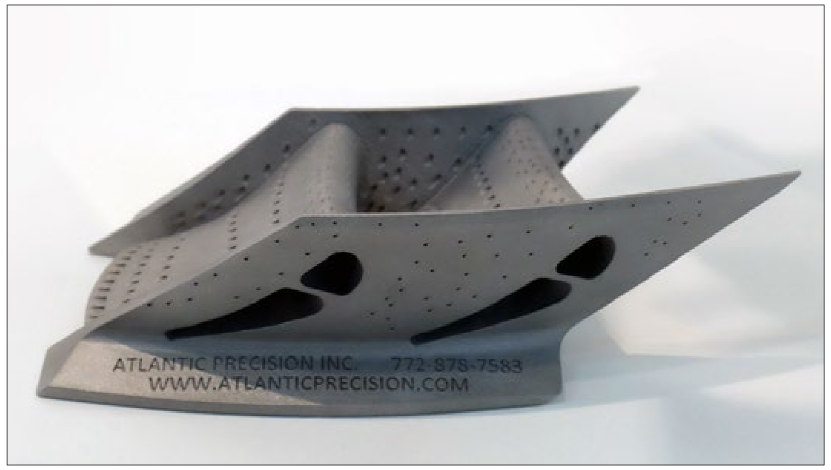

Fig. 1 Accompanying the AMPM conference was an impressive display of AM components, including this Inconel 625 vane manufactured using Direct Metal Laser Sintering (DMLS) by Atlantic Precision Inc.

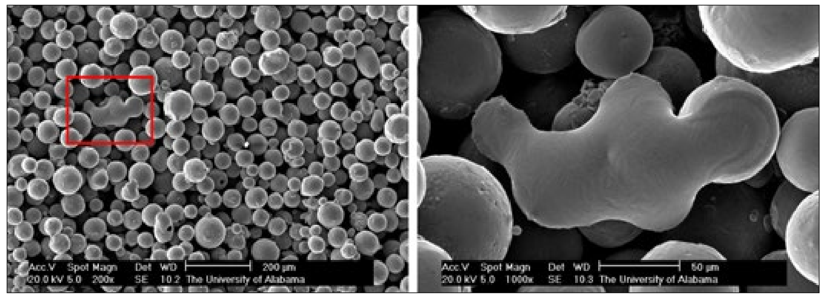

Fig. 2 SEM mages of sintered particles in the Z-plane: (a) low magnification and (b) high magnification [1]

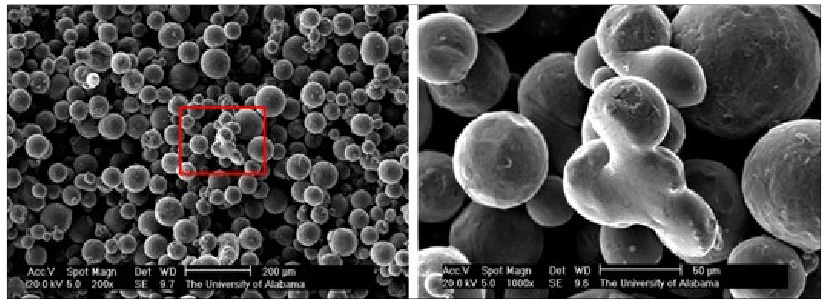

Fig. 3 SEM mages of sintered particles in the X-plane: (a) low magnification and (b) high magnification [1]

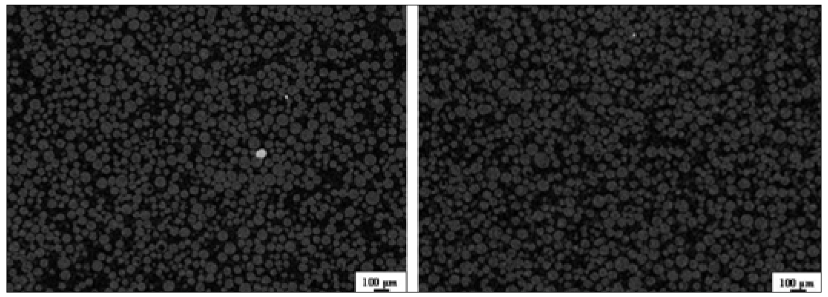

Fig. 4 CT scan images: (a) Z-plane section and (b) X-plane section [1]

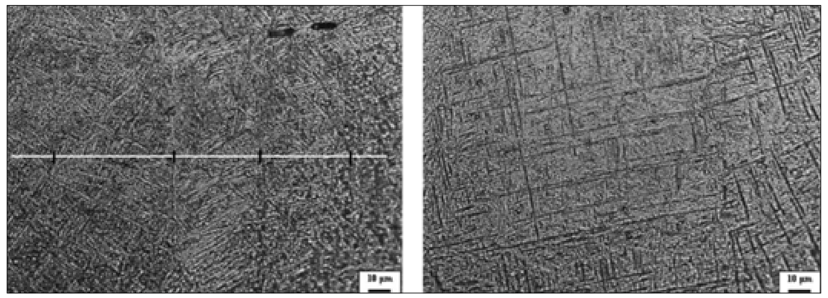

Fig. 5 Optical microscopy images of Ti-6Al-4V microstructures from a sold cylinder sample: (a) top and (b) bottom layers [1]

Characterisation of Ti-6Al-4V powders in Electron Beam Melting

A paper presented by Xibing Gong, University of Alabama, USA, reported on a characterisation study of Ti-6Al-4V powders used in the Electron Beam Melting AM process.

In the EBM AM process, preheating serves to aggregate the precursor powder, lightly sintering it to prevent a spreading effect. Specimens with sintered Ti-6Al-4V powder enclosed were fabricated and prepared for microstructural and morphology examinations by optical microscopy and scanning electron microscopy. In addition, micro-CT scans were conducted on the specimens and were analysed to study the powder porosity and the powder size distributions. The thermal conductivity of preheated powder from EBM AM was also measured and analysed at different temperatures.

The conclusions, drawn from this study, were that:-

1. Preheating results in metallurgical bonds or even partial melting of the powder during the EBM AM process. The phenomenon of neck formation is evident in both the Z-plane and X-plane sections (Figs. 2 and 3). The diameter of the necks is of the order of 1 to 10 μm. In addition, the X-plane seems to have less particles affected by sintering, because of the energy penetration limit.
2. The micro-CT scan images of preheated powder show similar porosity levels in the Z-plane and X-plane surfaces (Fig. 4). The calculated porosity of the preheated powder is about 50%. Moreover, the major diameter range of the powder is from 30 to 50 μm on both planes.
3. The microstructure of a Ti-6Al-4V part from EBM AM exhibits a fine Widmanstätten (α+β) structure and α′ martensite due to rapid cooling during the process (Fig. 5).
4. Ti-6Al-4V powder has significantly lower thermal conductivity than that of a solid counterpart. Also, the thermal conductivity is highly temperature dependent, being about 0.63 W/m•K at room temperature and around 2.44 W/m•K at 750°C for the preheated powder (Fig. 6).

To investigate the preheating effect, further studies will be needed to use different preheating conditions (for example, the beam power and the beam speed) in EBM AM and then to examine the effects on sintering level by metallurgical characterisation. In addition, the influence of preheating conditions on the microstructure of a built part should also be investigated in order to optimise the process parameters in EBM AM.

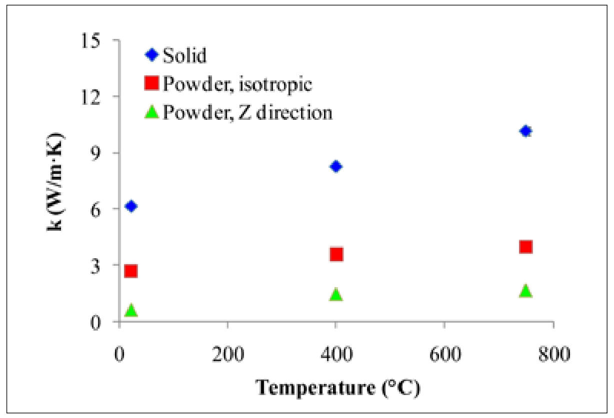

Fig. 6 Measured and analysed thermal conductivity of Ti-6Al-4V specimens (both solid and pre-heated powder samples) [1]

Accurately quantifying process relevant powder properties for AM applications

In a second paper in the same session, Michael Delancy, Freeman Technology, UK and USA, turned attention to the issue of powder flow during the filling of successive layers during powder bed AM technologies.

Any given powder's properties will define its process behaviour and therefore the quality of the final product. Robust methods for characterising powder flow properties are therefore essential. Conventional powder measurements (the Hall Flowmeter for example) rely on basic, insensitive techniques, producing single number parameters that rarely reflect powder behaviour across the range of unit operations used in PM and AM. Modern instrument technology offers a solution; employing multiple precision test methodologies to measure dynamic, bulk and shear characteristics relevant to manufacturing processes.

This paper reviewed the properties of a number of powdered metals used in AM applications and showed that, despite materials being purportedly identical, significant differences in their flow behaviour are observed which have already been shown to translate into process interruptions and/or poor product quality.

Many users rely heavily on a consistent particle size distribution of their powders as a critical quality attribute, but, in many cases, the size distribution alone, or indeed any other single parameter, is not sufficient to enable this to be used to qualify a batch of feedstock fully. The reported study evaluated three batches of stainless steel powder from the same supplier, all having particle size distributions between 15 and 45μm but known to behave differently in AM. These samples were difficult to differentiate using conventional methods.

In order to investigate fully why these materials behave differently in their application, the author stated that it was necessary to study the response of the powders to a range of stimuli that represent their interaction with the unit operations used in their processing.

This investigation was begun by using the company's proprietary characterisation equipment, the FT4 Powder Rheometer, shown in Fig. 7. This equipment was used to evaluate the complete picture of the powders' rheological behaviour by measuring their dynamic flow energy, bulk and shear properties.

The three batches of stainless steel powder studied (A, B and C) were known to show different behaviour when dispensing from a hopper. Batches A and C showed acceptable behaviour across the process, while batch B showed very poor behaviour, routinely blocking the dosing

Powder	BFE, mJ	SI	FRI	SE, mJ/g	CBD, g/ml
Metal Powder A	482 (±1.9%)	0.907 (±5.2%) 1	.05 (±0.5%)	2.23 (±0.1%)	3.98 (±0.4%)
Metal Powder B	591 (±2.67%)	1.04 (±2.5%)	1.18 (±2.0%)	2.94 (±0.6%)	4.16 (±0.1%)
Metal Powder C	529 (±1.3%)	1.06 (±1.0%)	1.1 (±2.1%)	2.32 (±1.34%)	3.95 (±0.1%)

Table 1 Dynamic flow properties of three metal powders [2]

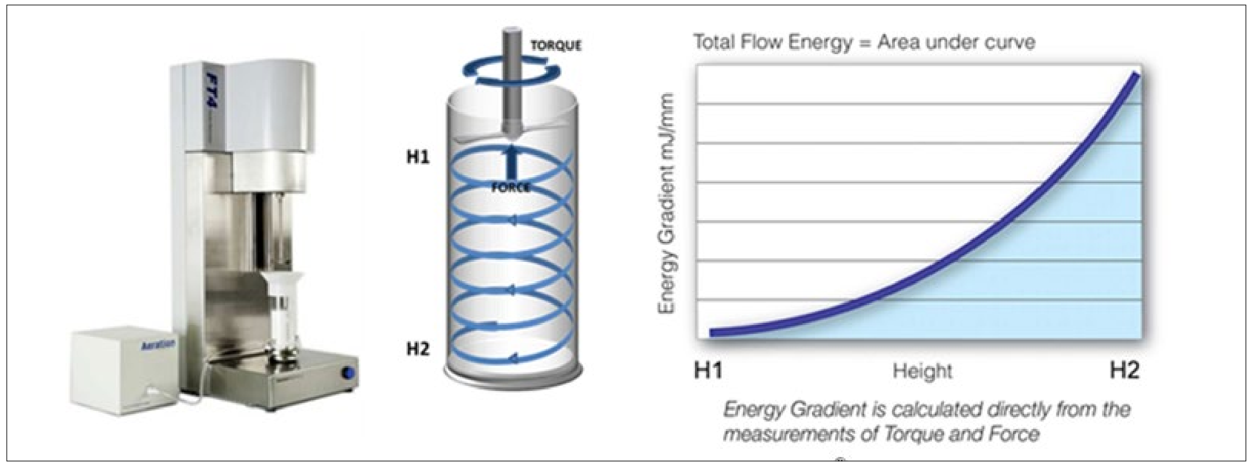

Fig. 7 Measurement of flow energy using the FT4 Powder Rheometer [2]

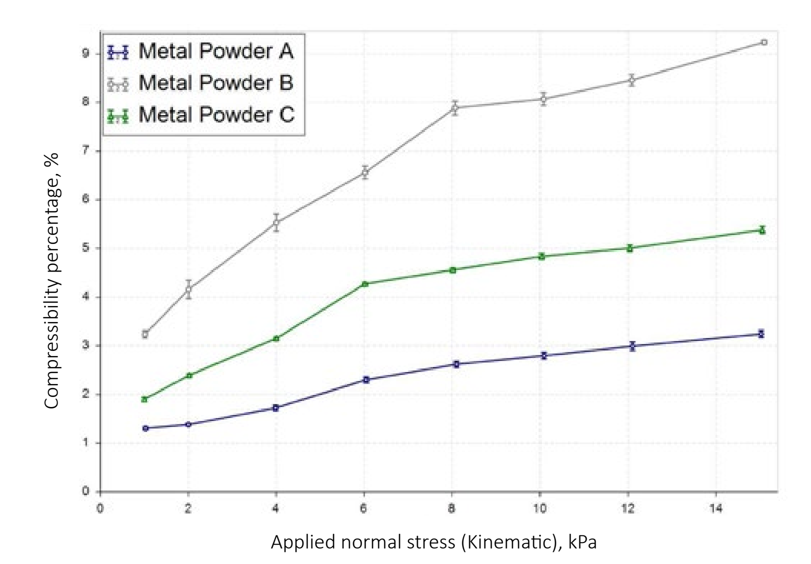

Fig. 8 The compressibilities of the three metal powder samples [2]

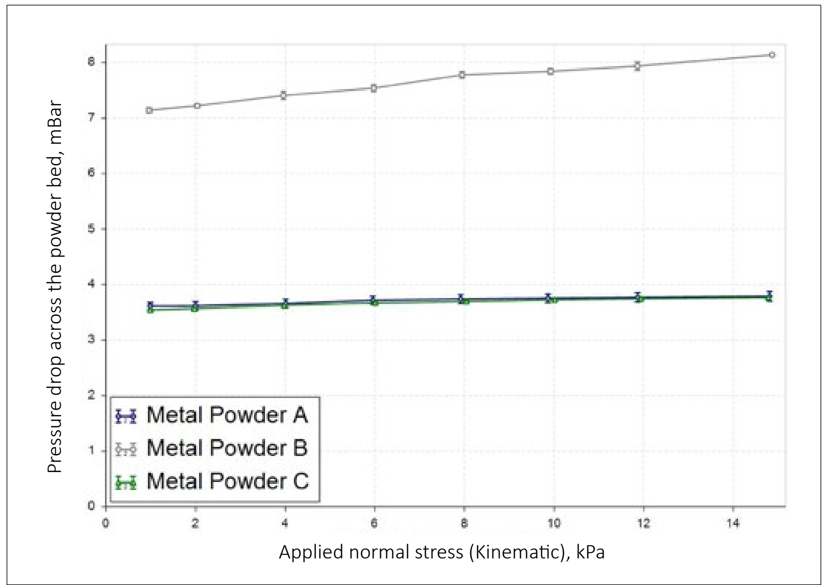

Fig. 9 The permeabilities of the three metal powder samples [2]

mechanism and displaying variable properties in the final product.

The batches were firstly evaluated for their dynamic flow properties, presented as a flow energy in Table 1. The dynamic flow properties are determined by rotating a precision blade down a helical path through the powder. During this downward traverse, the torque and axial force acting on the blade are measured and the resistance to flow is calculated and expressed as the flow energy.

Prior to measuring the flow energy, however, a conditioning process is undertaken (utilising the blade), which moves the powder in a gentle, reproducible way in order to establish a homogenised stress in the powder. This is essential for the subsequent measurement, but also allows the Conditioned Bulk Density to be derived accurately.

Several important measurements and relationships can be derived from this test equipment, which are valuable in understanding how the sample will behave during processing:

- Basic Flowability Energy (BFE) is the energy required to displace a powder during non-gravitational, forced flow, i.e. its resistance to flow in a constrained environment: for example, feeding in a screw conveyer
- The Stability Index (SI) is a measure of the physical stability as function of repeat tests. Particle attrition, segregation, (de)agglomeration and (de)aeration are examples of effects that could result in instability and intermittent flow
- The Flow Rate Index (FRI) measures how sensitive the powder is to being made to flow at different rates through a process by varying the blade tip speed. This measures the response of the powder to changing flow rates, especially relevant for mixing applications and variable feed rate environments
- The Specific Energy (SE) measures the energy required for gravity induced flow, for example, when filling a cavity – the powder sample's resistance to flow in an unconstrained environment
- The Conditioned Bulk Density (CBD).

From the data in Table 1, the high BFE and SE results indicate that Sample B is the least free-flowing and also the measurement repeatability is much lower for this sample. The low Stability Index value for Sample A (SI<1) is also noteworthy, as the presence of agglomerates in this sample resulted in a slightly higher than expected component rejection rate.

Next, the compressibilities of the samples were assessed. The compressibility test measures the ability of the powder to become compacted when subjected to normal stress. It provides an indication of changes in volume and packing during storage and transport, and in processes such as direct compression. Whilst all three powders can be seen to have relatively low compressibility values, they are all very different from each other (Fig. 8). Again Sample B exhibits a notable difference from the other samples.

Permeability measures the ease with which a powder bed will transmit or release air over a range of stress conditions, with higher Pressure Drop values indicating that the material is less permeable. Permeability is relevant for aerosolisation, hopper flow, direct compression, pneumatic transfer and filling applications. Although all three samples can be said to be moderately permeable (Fig. 9), there is a marked difference between Sample B and the other two samples. This significantly poorer permeability undoubtedly contributes to issues with discharge from storage into feeders and inconsistencies in the layering of the powder, resulting in the presence of air voids within layers.

The next type of characterisation test applied was the Shear Cell test, which was intended to determine the stress required to initiate flow in a pre-consolidated powder. This provides an indication of how easily a powder will move from a static condition into dynamic flow; for example, when the outlet on a hopper is opened. The shear data in Table 2 show that Sample B has very slightly higher shear stresses, cohesion and a lower Flow Function compared to the other samples. However, if all the data sets are combined and averaged, as shown in Fig. 10, the resultant variation in the shear point is very small and well within what would be considered experimental variation, making these samples functionally identical in this test regime.

The overall conclusions from the results of the broad suite of characterisation tests applied were that Sample B is distinctly different from the other two samples, having poorer dynamic flow properties; lower permeability (higher pressure drop) and higher compressibility. It was clear that, whilst this batch had the same particle size distribution as the other batches, its noticeably poorer flow behaviour in a range of processing equipment was displayed by a number of different properties.

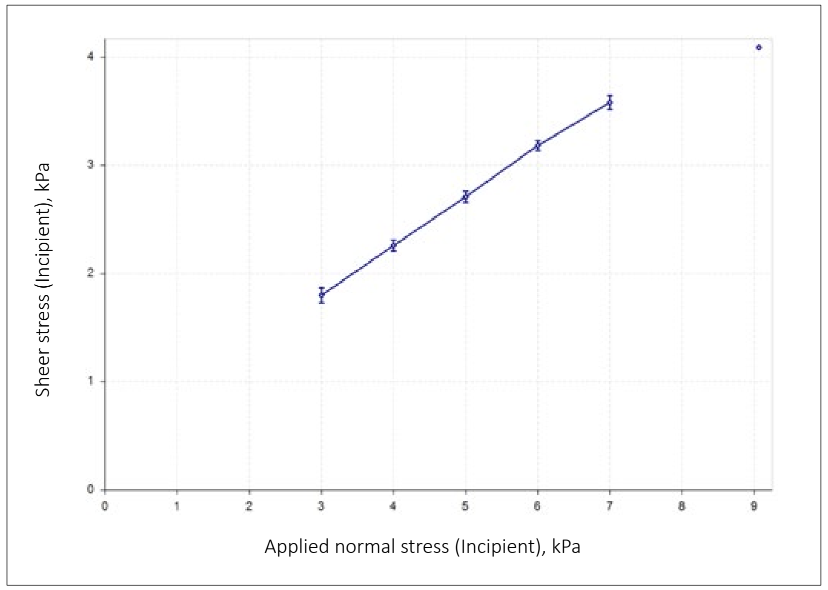

Fig. 10 Combined yield locus derived by averaging the shear cell data from the three metal powder samples [2]

Series Name	Cohesion, kPa	UYS, kPa	MPS, kPa	FF	AIF, °
Metal Powder A	0.34	1.08	12.81	11.83	25.20
Metal Powder B	0.61	1.85	13.59	7.36	23.37
Metal Powder C	0.44	1.37	12.83	9.39	23.87

Table 2 The shear properties of three metal powders [2]

Mechanical properties

Mechanical performance of structures manufactured by Selective Laser Melting: Damage initiation and propagation

In the Mechanical Properties session, a paper, presented by Stefan Leuders, University of Paderborn, Germany, reported on a study of the mechanical performance of structures manufactured by selective laser melting (SLM), with particular reference to damage initiation and propagation.

The report began by identifying the geometrical and economic advantages that SLM is capable of offering, but then went on to outline the drawbacks that can create impediments to the use of SLM for direct manufacturing of highly stressed components. The high cooling rates (>2000°C/s) in the process can introduce residual stresses and generate process-related microstructures. Also, the process can introduce geometrical defects, such as pores (~40 µm), cracks and surface roughness (Ra = 10-15 µm).

The study involved the manufacture of tensile (machined surfaces), fatigue (turned surfaces, tested at R = -1) and fracture mechanical test pieces from Ti-6Al-4V and 316L stainless steel powders. These test pieces were assessed in the as-built condition, after stress relieving and after hot isostatic pressing (HIP). In addition, a heat treatment (2 h at 1050°C) was applied to Ti-6Al-4V test pieces to modify microstructure, as an alternative to HIP.

The tensile test results for Ti-6Al-4V are presented in Fig. 11. The as-built condition offered the highest UTS (~1400 MPa), but low ductility. The stress relieving treatment produced a slight reduction in UTS and a slight improvement in ductility. On the other hand, the microstructure modification heat treatment and the HIP treatment produced more significant reductions

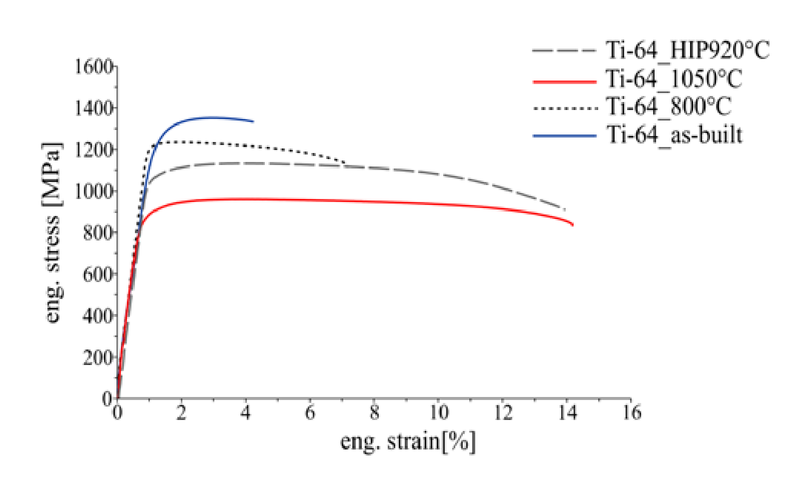

Fig. 11 Tensile (monotonic) performance of Ti-6Al-4V [3]

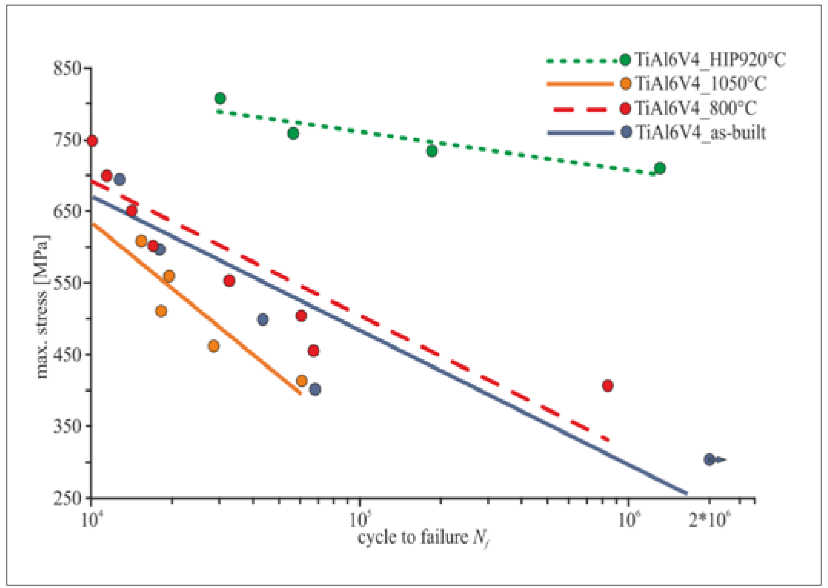

Fig. 12 High cycle fatigue performance of Ti-6Al-4V [3]

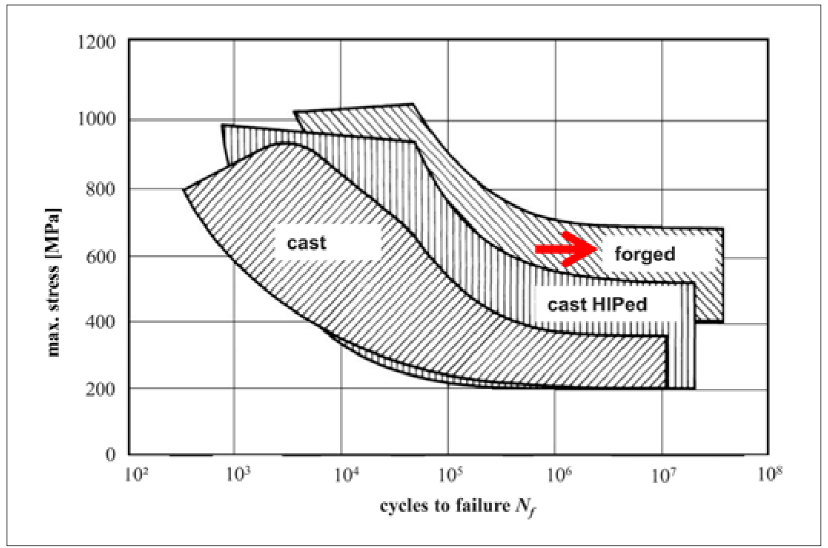

Fig. 13 Fatigue performance of SLM + HIP processed Ti-6Al-4V material, compared with cast and forged materials [3]

in UTS (to ~950 MPa and 1100 MPa respectively), but much improved ductility levels.

The high cycle fatigue test results for Ti-6Al-4V are shown in Fig. 12. The as-built and stress relieved conditions showed similar results – a steep gradient with a knee at around 250 MPa. With the modified microstructure treatment, the fatigue initiation phase did not appear to benefit from the overall ductility. On the other hand, the HIPped samples showed the flattest curve and significantly improved fatigue behaviour. In fact, as demonstrated in Fig. 13, the HIPped SLM materials gave fatigue performance that was superior to cast Ti-6Al-4V materials and comparable with forged material.

Fig. 14 shows the tensile test results for 316L stainless steel. The as-built and stress relieved (650°C) conditions showed similar UTS (~610 MPa) and elongation (~50%) levels, but yield strength was slightly reduced by stress relieving. The HIP treatment generated a small reduction in UTS (to ~550 MPa), a more significant reduction in yield strength, but an improvement in elongation level (~65%).

Fig. 15 presents the high cycle fatigue data for 316L. The as-built and stress relieved conditions showed similar results, with a relatively steep gradient and a knee at around 260 MPa. The HIP treatment, on the other hand, displayed a much flatter curve that, at lower cycles to failure, was below those for the other two conditions. Estimated fatigue endurance limits, however, showed the HIPped condition to be slightly superior at 317 MPa, compared with 267 and 294 MPa for the as-built and stress relieved conditions respectively.

The overall conclusion drawn was that the direct manufacture of highly stressed components by SLM was shown to be feasible, but that the observed benefits of post-treatments were material specific. For Ti-6Al-4V, HIP was shown to improve tensile (monotonic) properties (through generation of a tailored microstructure) and fatigue behaviour (through a reduction of porosity level). Reported fatigue crack growth data also showed a benefit of HIPping through a reduction in residual stress. On the other hand, for 316L, the use of neither of the post-treatments studied was deemed to be mandatory.

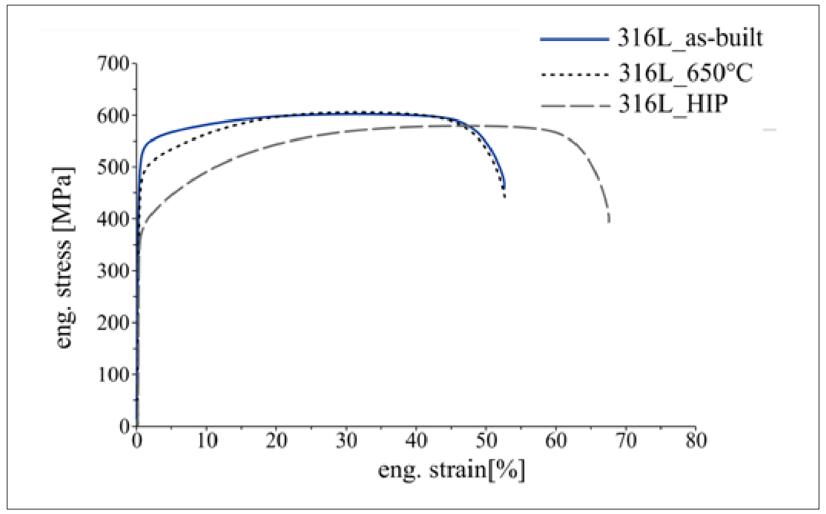

Fig. 14 Tensile (monotonic) performance of 316L stainless steel [3]

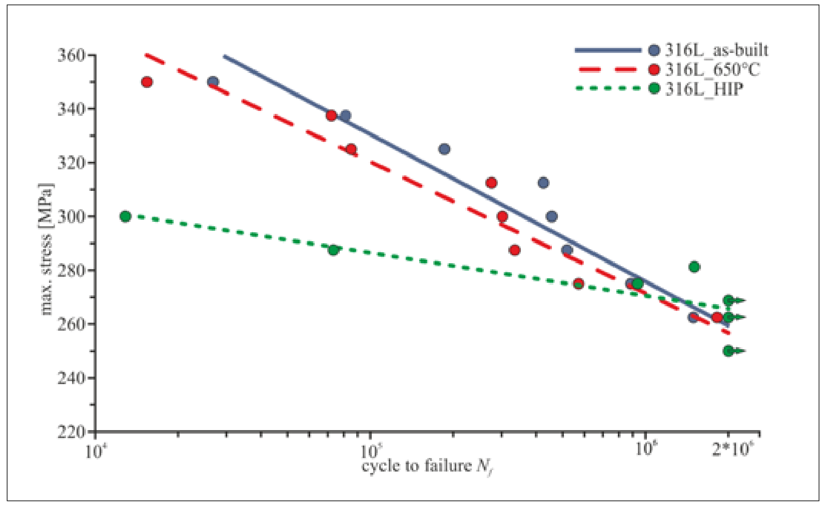

Fig. 15 High cycle fatigue performance of 316L stainless steel [3]

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[2] Accurately quantifying process relevant powder properties for AM applications Michael Delancy, Freeman Technology, UK/USA, as presented at AMPM2014, MPIF, USA

[3] On the mechanical performance of structures manufactured by Selective Laser Melting: Damage initiation and propagation, Stefan Leuders, University of Paderborn, Germany, as presented at AMPM2014, MPIF, USA

Author

Dr David Whittaker is a consultant to the Powder Metallurgy and associated industries.
Contact +44 1902 338498
email: whittakerd4@gmail.com

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Advances in PM materials and process developments presented at the PM2014 World Congress

The PM2014 World Congress on Powder Metallurgy and Particulate Materials, organised and sponsored by the Metal Powder Industries Federation (MPIF), was held in Orlando, Florida, USA, May 18-22. Over five days some 92 technical sessions, posters and special interest programmes highlighted the latest developments in PM technology. Dr David Whittaker reports on a number of key presentations on advances in materials and the processing of PM products.

Soft Magnetic Materials

Emerging markets, including green energy and electric vehicles, are generating new opportunities for soft magnetic components. Soft Magnetic Composite (SMC) materials, processed by Powder Metallurgy (PM) compaction and curing of the inter-particle insulants, have potential advantages in terms of offering isotropic magnetic properties and three-dimensional product design capabilities, but require the enhancement of certain properties (magnetic and mechanical) in order to respond to these new opportunities.

A number of groups have therefore been focussing on material or process developments aimed at generating these property enhancements and these developments were the subject of a series of papers at PM 2014.

Development of new Soft Magnetic Composite material possessing higher levels of magnetic and mechanical performance

In the first of these papers, Francis Hanejko, Hoeganaes Corporation, USA, described developments aimed at increasing the compacted densities of SMC components in order to attain higher magnetic permeability, higher induction and greater mechanical strength.

These developments involved the application of the Ancormax 225 pressing lubricant concepts, previously applied to PM structural parts

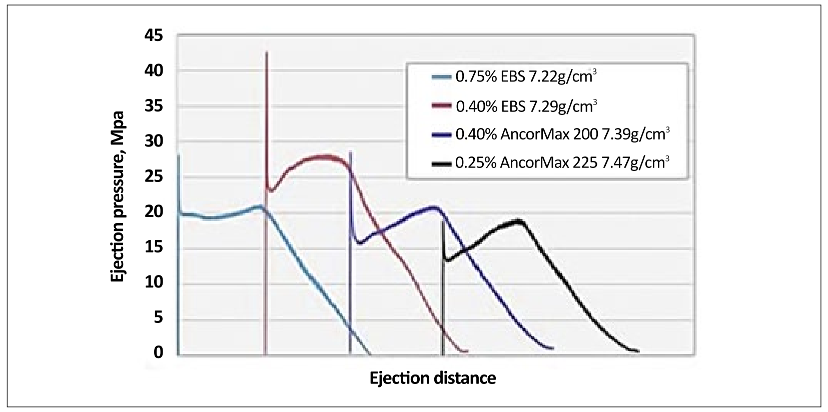

Fig. 1 Strip and slide pressure of various lubricants [1]

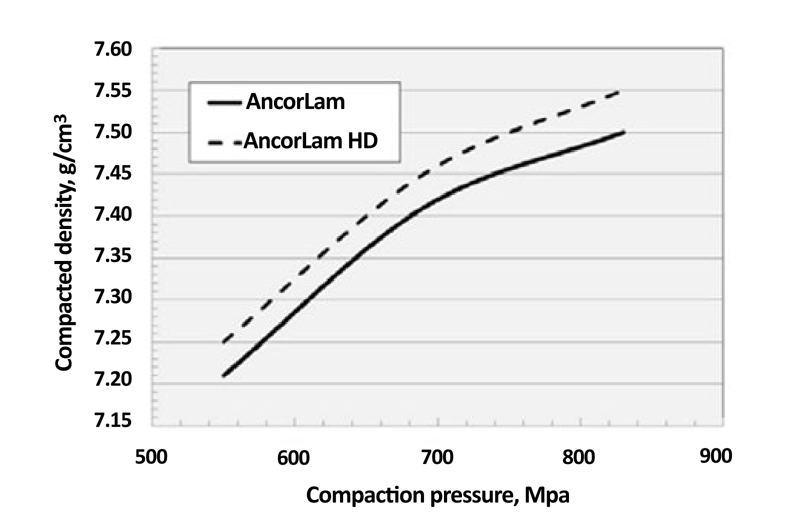

Fig. 2 Compressibility data for AncorLam and Anchorlam HD [1]

Lube System	Compaction Pressure, MPa	Green density, g/cm³	Cured density g/cm³	Cured strength, MPa (psi)	Cured resistivity, uΩcm
Standard AncorLam	550	7.21	7.20	86 (12500)	6,900
	690	7.41	7.39	103 (14950)	6,900
	830	7.51	7.47	107 (15600)	7,000
AncorLam HD	550	7.25	7.24	102 (14850)	8,400
	690	7.43	7.42	119 (17350)	7,400
	830	7.55	7.53	130 (18800)	7,400

Table 1 Compacted and cured densities of AncorLam and AncorLam HD [1]

Material & Density	Frequency, Hz	Max Perm, DC	Residual Magnetism, T	Coercive Field Hc (A/m)	Core loss, w/kg
AncorLam HD at 7.53g/cm³ cured density	DC	515	0.27	264	-
	60	504	0.29	275	7
	400	504	0.32	320	58
	1000	500	0.36	396	178
	5000	450	0.59	870	1955
	10000*	375	0.45	920	2400
AncorLam HR – HD at 7.33 g/cm³ cured density	DC	240	0.14	323	-
	60	235	0.14	330	8.6
	400	230	0.14	350	61
	1000	230	0.15	375	163
	5000	230	0.20	525	1134
	10000*	230	0.17	510	1250

*Tested at induction level of 0.6 Tesla

Table 2 Magnetic properties of AncorLam HD and AncorLam HR – HD tested at 1 Tesla [1]

and described in previous conference reports in *Powder Metallurgy Review.*

This recently developed lubricant can be used at addition levels as low as 0.25 wt%, in combination with warm die compaction with a part ejection temperature of 107°C (225°F), hence the suffix "225". Use of this lubricant concept, together with a compaction pressure of 830 MPa (60 tsi), offers green density levels in excess of 7.5 g/cm^3.

As shown in Fig. 1, this advanced lubricant system offers nearly identical ejection characteristics to the use of a conventional 0.75 wt% EBS lubricant addition, while providing 0.2-0.25 g/cm^3 higher green density.

Two different soft magnetic composite grades were processed using these lubricant/compaction concepts in the reported study:-

- AncorLam HD, a material aimed at achieving high levels of density, permeability and induction.
- AncorLam HR-HD, a material aimed at attaining a moderate density but with high resistivity. High resistivity helps to limit eddy current losses and therefore total core losses in higher frequency operation.

Fig. 2 compares the compressibility of AncorLam HD with the existing AncorLam lubricant system. Reducing the lubricant level from 0.4 wt% to 0.25 wt% results in higher green density over the range of compaction pressures evaluated.

Table 1 shows the green and cured densities attained with these two materials. Higher cured densities are achieved with the lower lubricant addition AncorLam HD. This table also shows that AncorLam HD has a higher resistivity despite having higher cured density and that Ancorlam HD offers an increase of cured TRS strength to 130 MPa, around 20% higher than AncorLam.

The attained magnetic data for AncorLam HD and AncorLam HR-HD are shown in Table 2. AncorLam HD maintains constant permeability up to approximately 1000 Hz in AC operation. Above that frequency, permeability decreases as eddy current losses increase. Despite the higher cured density achieved with AncorLam HD, resistivity and core losses are equivalent to existing materials. AncorLam HR-HD has lower core losses than AncorLam HD at frequencies above 1 kHz and this enables constant permeability up to 10 KHz.

Influence of apparent density on magnetic properties in dust cores, for high-frequency applications

Yuji Taniguchi, Hirofumi Hojo, Hironori Suzuki and Hiroyuki Mitani, Kobe Steel Ltd., Japan, reported on a study of the influence of apparent density on magnetic properties in dust cores for high frequency application.

This study was aimed at reducing hysteresis losses and was based on the concept that low hysteresis loss is associated with the reduction of obstacles to magnetic domain wall movement. The obstacles to domain wall movement are related to disorder of atom arrangement, specifically factors such as purity, crystal grain boundaries, particle grain boundaries and distortion.

In this study, particle shape was modified to influence particle grain boundaries; particle shape is related to apparent density. The apparent density of a high purity iron powder was altered by milling followed by annealing (to eliminate milling distortion). The apparent densities and mean particle sizes of four different powder samples are shown in Table 3. The mean particle size of each powder sample was around 50 µm. These powders were insulated by double layer coating and were then compacted into rings with 7.45 g/cm^3 density by a die wall lubrication method at 130°C. The ring specimens were cured at 500°C, 550°C and 600°C.

As shown in Fig. 3, the hysteresis loss is almost independent of apparent density for the lower curing temperatures of 500°C and 550°C. At these cure temperatures, the main obstacle to magnetic domain wall movement is the residual distortion, with particle surface having hardly

	Apparent Density (g/cm³)	Mean Particle Size (µm)
Powder A	2.76	48.6
Powder B	2.93	52.1
Powder C	3.04	50.7
Powder D	3.18	45.7

Table 3 Apparent densities and mean particle sizes of the powder sampled [2]

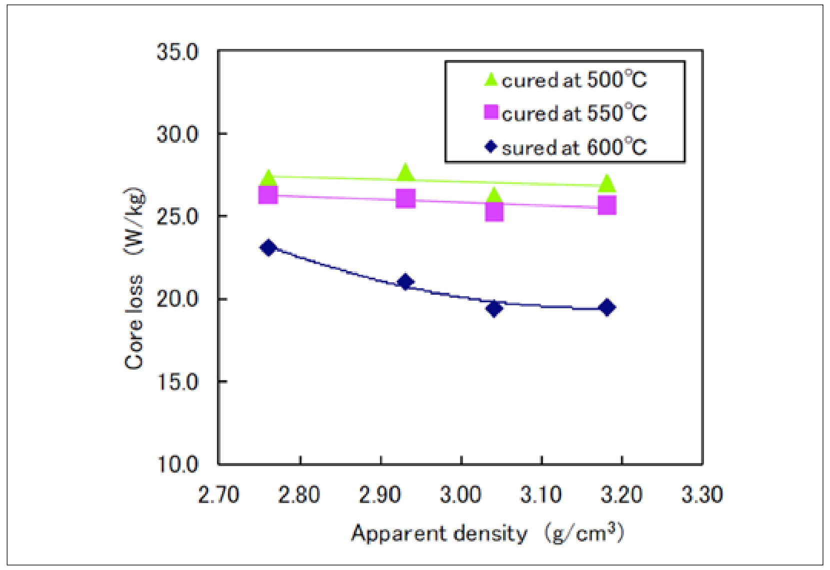

Fig. 3 Relationship between apparent density and hysteresis loss [2]

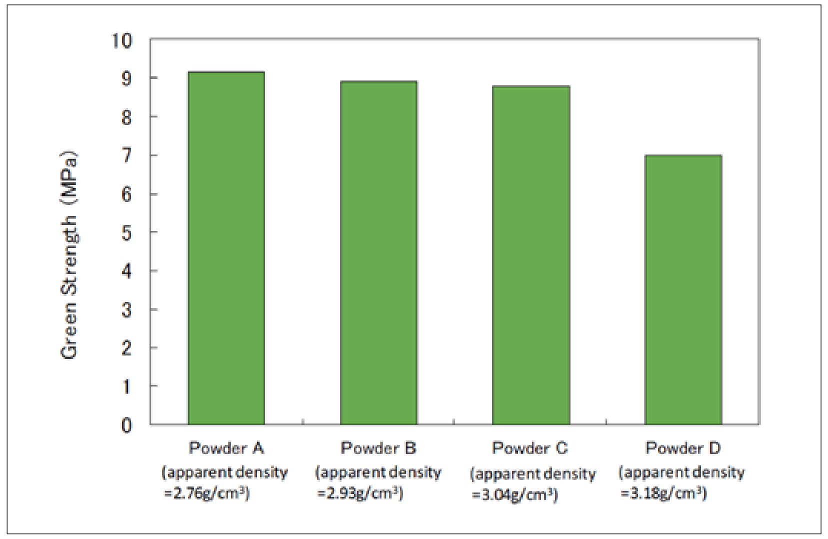

Fig. 4 Green strengths of compacted specimens from the various powder samples [1]

any influence. However, at a curing temperature of 600°C, the residual distortion is sufficiently eliminated that a reduction in hysteresis loss with increasing apparent density is revealed.

However, minimum hysteresis loss is not the sole criterion for deciding optimum apparent density. Through the effects of the milling treatments, smoother powder surfaces are associated with higher apparent densities and this factor leads to a reduction of linkages between compacted particles and, hence, a reduction of green strength. Fig. 4 compares the green

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Voltage	V_{in} : 600VDC	
	V_{out} : 300VDC	
Outpur Current: I_{dc}	20 A (rated) 25A (max)	
Frequency: f	300 kHz	
Inductance: Ls	170 µH min @ 20A 150µH min @25A	
Coil windings	Round copper wire Φ2.2mm	

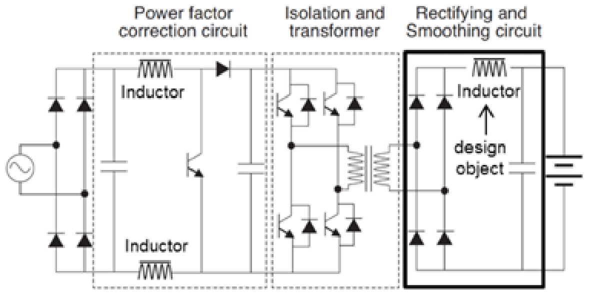


Table 4 Specifications for the rectifying and smooth circuit and inductors [3]

strengths of the compacts from the various powder samples.

It was concluded that powder D may have insufficient green strength to avoid cracking or chipping after compaction and, therefore, powder C, with an apparent density around 3.05 g/cm³, was deemed to offer the optimum combination of low core loss and high strength.

Development of FeSiAl-based low-iron-loss soft magnetic powder cores

A presentation by Tomoyuki Ishimine, Asako Watanabe, Tomoyuki Ueno and Terukazu Tokuoka, Sumitomo Electric Industries, Japan, described the development of FeSiAl-based low iron loss soft magnetic powder cores.

This study was aimed at developing high performance inductors, required for small and powerful power-supply devices with better conversion efficiency for applications in clean power and electric vehicles. The possibility of developing inductors of smaller size and with lower iron losses, using PM SMC cores, was examined.

Especially for automotive applications, inductors are required that operate at temperatures above 100°C. Previous work by this group had identified the benefits of adding Si and Al to the base iron powder composition in reducing iron loss in this temperature range. Based on this prior work, the alloy compositions in this study were selected to minimise iron loss at 100°C.

Two different FeSiAl materials were included in the study:-

- A "conventional" material, a commercially available Fe-9.5Si-5.5Al powder, prepared by a milling method and sieved to -106µm.
- The "developed" material, an Fe-8.5Si-6.8Al powder, prepared by inert gas atomisation and, again, sieved to -106 µm.

Each of these powders was coated with an insulation film and compacted into rings at 980 MPa, with 1 wt% PVA resin added as a compaction binder. The compacts were then heat-treated at 700°C.

In order to investigate the performance of the FeSiAl materials, inductors were designed for an in-vehicle battery charger using the conventional material, the developed material and ferrite. The specifications for designing the inductors are shown in Table 4. The results of designing the inductors in the developed material, the conventional material and a ferrite material are shown in Table 5

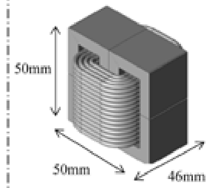
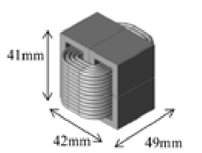
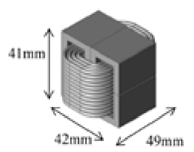
Core material	Ferrite TDK PC90	FeSiAl based PM Soft-magnetic material Conventional material	 Developed material
Overview			
Volume (calculated as cube)	115cm³	84cm³	84cm³
Weight (Core + Windings)	397g (162g + 235g)	325g (126g + 199g)	325g (126g + 199g)
Turns	56	48	48
DC Resistance	34mΩ	27mΩ	27mΩ
Air gap	12mm	1mm	1mm

Table 5 Results of designing inductors [3]

Fig. 5 shows the frequency dependency of iron loss, measured at 25°C, for the two FeSiAl SMC materials. The developed material shows less than half the iron loss of the conventional material at 100 kHz. Fig. 6 compared the eddy current loss and hysteresis loss of the two materials. Eddy current loss of the developed material and developed insulation layer decreased to around a quarter of that of the conventional material.

Fig. 7 compares the temperature dependency of iron loss for the materials in the study. For temperatures of 100°C and above, the developed FeSiAl material shows only around one fifth of the iron loss of the conventional material and this level is low enough to compare with ferrite. Both of the FeSiAl materials delivered higher flux density than ferrite and this attribute can be used to downsize inductor design.

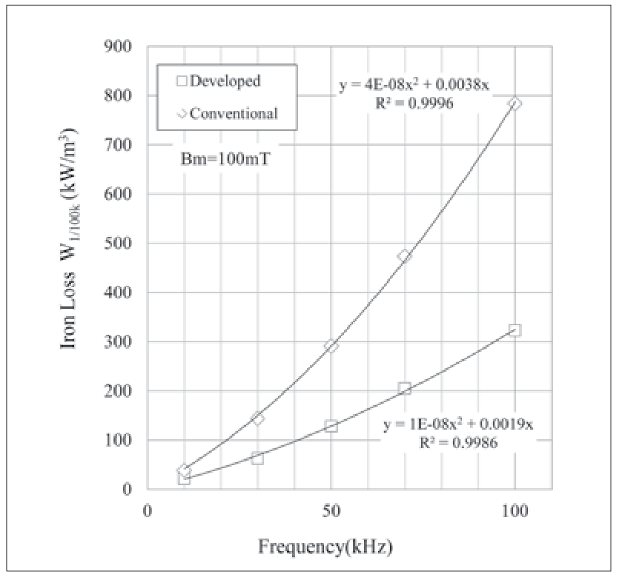


Fig. 5 Frequency dependency of iron loss at 25°C [3]

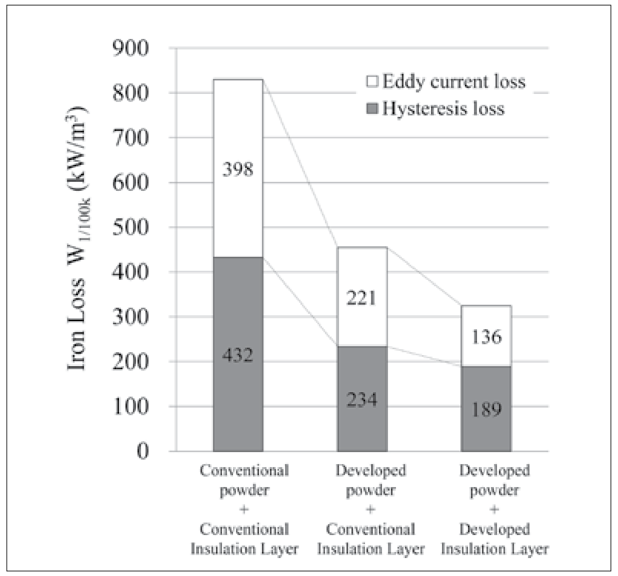


Fig. 6 Eddy current loss and hysteresis loss at 25°C [3]

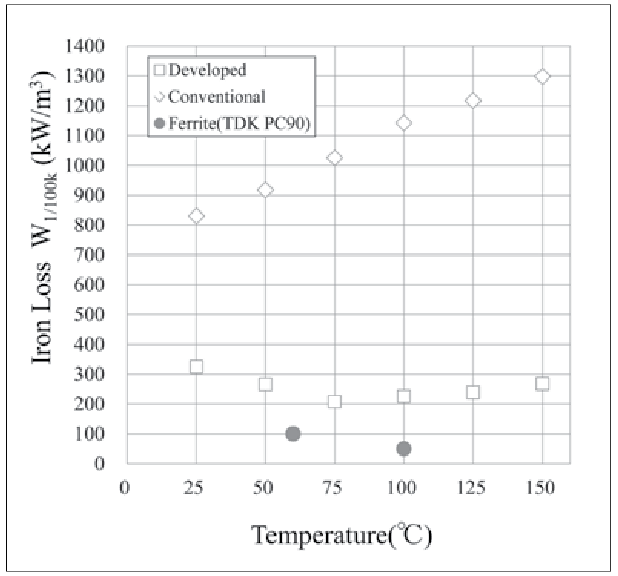


Fig. 7 Temperature dependency of iron loss for the three studied materials [2]

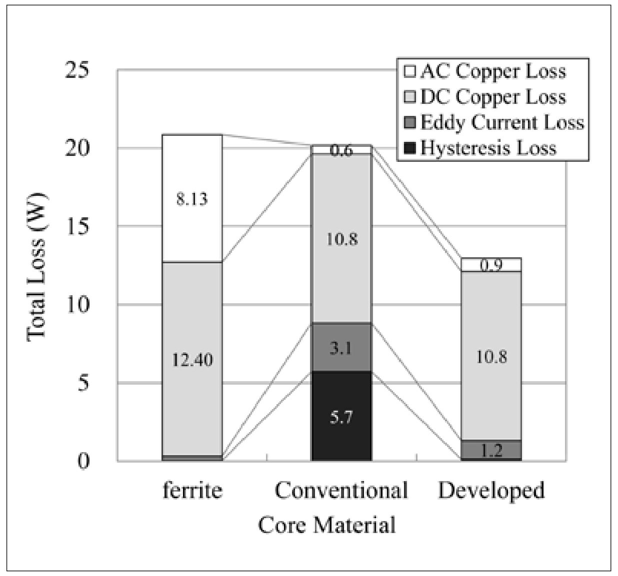


Fig. 8 Loss generation of each of the designed inductors estimated using FEM [3]

Fig. 8 shows a comparison of loss generation during inductor operation for the three materials in the study. Loss is composed of iron loss (the sum of hysteresis loss and eddy current loss) and copper loss (the sum of DC copper loss and AC copper loss). DC copper loss is heat generated by DC current and AC copper loss is an eddy current loss in windings that is generated by leakage magnetic flux passing through the windings. The iron loss property of ferrite material is so superior that the loss generated in the ferrite inductor is largely composed of copper loss and AC copper loss is generated as much as DC copper loss.

For ferrite cores, a magnetic gap is necessary for the modification of DC bias characteristics and this gap generates leakage magnetic flux and increases AC copper loss. On the other hand, for inductors with PM soft magnetic cores, the magnetic gap is smaller than that of ferrite and AC copper loss is barely generated. However, the conventional material shows much larger iron loss than that of ferrite and, therefore, the total loss generated in the conventional material inductor is similar to that of the ferrite inductor. Furthermore, the iron loss generated in the developed material inductors decreases to only one-sixth of the conventional material inductors and, therefore, the resulting total loss is smaller than the conventional material and the ferrite.

Inductors with ferrite cores have a

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Lubricant	Apparent Density g/cm³	Flow Rate s/50g	Green Strength MPa	Spring back %
No lubricant	2.77	29.2	-	-
X-SS Lube	2.84	31.9	13.8	0.16
EBS	2.66	37.8	8.2	0.16
Lithium Stearate	2.90	35.5	4.8	0.20

Table 4 Powder properties of 316L based mixes. Springback was at 6.5 g/cm³ [4]

Lubricant	Apparent Density g/cm³	Flow Rate s/50g	Green Strength MPa	Spring back %
No lubricant	2.85	28.7	-	-
X-SS Lube	2.86	29.9	17.3	0.16
EBS	2.69	31.6	10.3	0.16
Lithium Stearate	2.92	38.2	10.3	0.21

Table 5 Powder properties of 409L based mixes. Springback was at 6.5 g/cm³ [4]

Mix ID	Sintering Designation	Carbon %C	Oxygen ppm O	Nitrogen ppm N
316L-EBS	H	0.040	1990	211
316L-LS	H	0.079	2900	130
316L-X	H	0.040	1940	183
316L-EBS	L	0.023	1230	42
316L-LS	L	0.027	2300	60
316L-X	L	0.023	1230	45
409L-EBS	L	0.006	2730	30
409L-LS	L	0.011	2170	90
409L-X	L	0.009	2310	20

Table 6 Interstitial levels after sintering [4]

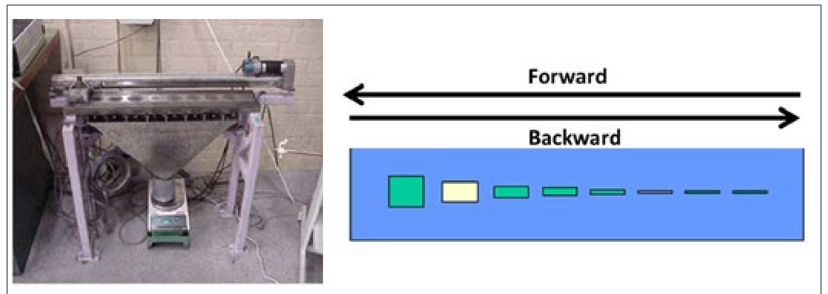

Fig. 9 Die filling simulator and die cavity layout [4]

problem in downsizing and suppression of AC copper loss. The trend for in-vehicle power units is to operate at larger currents and higher frequency conditions. On the basis of this trend, PM soft magnetic material with low iron loss (such as the "developed" material in this study) will play an important role in developing these next generation power control units.

New lubricant system enhances stainless steel powder properties

Compared to low alloy ferrous PM powders, stainless steel powders often have higher flow rate, lower green strength and lower apparent density. These poor powder properties can make the manufacturing of stainless steel PM components more challenging, often leading to lower productivity to meet quality goals. For the stainless steel PM market to expand, improvements in these powder properties are required.

Stainless steel mixes with improved performance

In his paper at PM 2014, Roland T Warzel III, North American Höganäs Inc., USA, reported on the development of a new lubricant system, aimed at improving the critical powder properties compared with industry standard lubricants.

The two most popular lubricants used with stainless steel PM powders are ethylene bisstearamide (EBS) and lithium stearate. EBS provides high green strength in PM stainless steel while lithium stearate is beneficial for improving green density. Lithium stearate is known for providing a high apparent density, however the flow rate is typically poor.

Another important difference between the two lubricants is the difference in their delubrication characteristics. Delubrication is the first step in the sintering process and is of vital importance if PM stainless steels are to achieve acceptable corrosion resistance. EBS is easily removed during the delubrication step and by 425°C is completely removed from the compact. The metal stearates are not completely removed during delubrication and their resultant residue can remain even after the temperature reaches 540°C. Any residual carbon left behind can lead to the formation of chromium carbides during sintering, which will negatively impact on the corrosion resistance of the stainless steel. It is recommended that after the delubrication step, the carbon content should be less than 0.05%.

A 300 series powder (316L) and a 400 series powder (409L) were used in the reported study. From each of these base powder lots, three mixes were manufactured. These various mixes incorporated a different lubricant; EBS, lithium stearate or the experimental lubricant system designated X-SS. In all cases, the lubricant addition was at the 1 wt.% level. Each powder mix was assessed, in terms of:-

- Apparent density
- Flow rate
- Compressibility
- Ejection characteristics
- Fillability index
- Green strength
- Interstitial levels after sintering
- Tensile, impact and transverse rupture properties in the as-sintered condition.

The fillability of each mix was measured using a die fill simulator (Fig. 9), which simulates the filling of die cavities of different sizes. The length and depth of the cavities are kept constant while the widths range from 1 mm to 20 mm. The fill shoe travels from left to right across the die cavities at controlled velocities and then returns back, simulating the fill shoe movement of a production press. The weight of the powder in the cavities is measured and a filling density (FD) is calculated based on the volume of the cavity in question. A filling index is calculated according to the equation:-

$$\text{Filling index (\%)} = (FD_{13\,mm} - FD_{2\,mm}) / FD_{13\,mm} \times 100$$

With a perfect filling material, the filling index would be equal to zero. However with the irregular powders used in PM, this condition is not attainable. A good filling material should have an index as close to zero as possible and be stable over a range of fill shoe velocities.

The major conclusions drawn from the reported comparisons were that the powder properties of stainless steel powders were positively impacted using the experimental lubricant (Tables 4 and 5). An increase in apparent density and faster flow rates were found compared to the EBS mixes. The apparent density was slightly lower than the lithium stearate mixes; however, the flow rate was significantly faster.

Due to the improved powder properties, the experimental lubricant provided improved fillability compared to EBS and lithium stearate (Figs. 10 and 11). In the parallel orientation (Fig. 10), acceptable filling was observed up to a fill

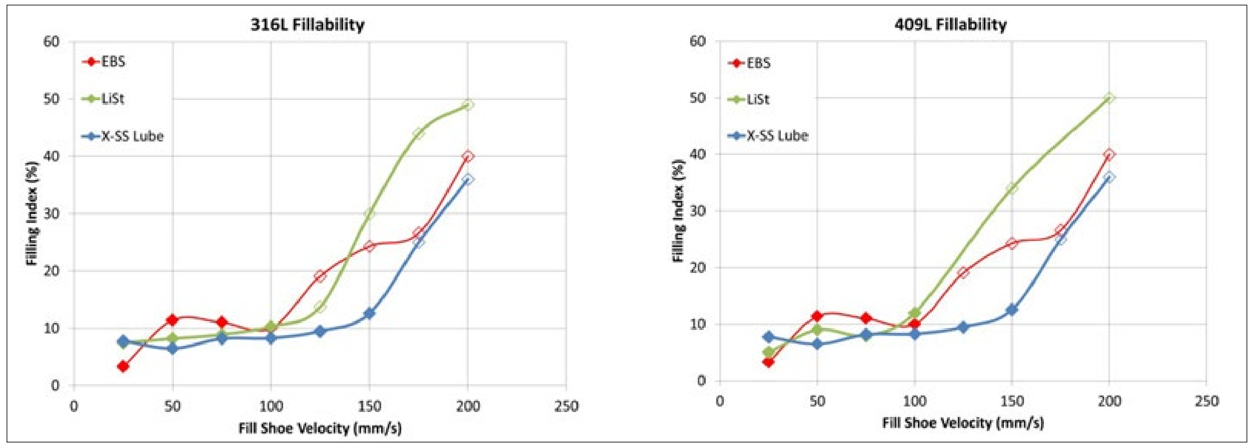

Fig. 10 Fillability of mixes with the cavities parallel to the fill shoe motion (solid markers indicate complete fill, empty markers indicate incomplete fill of the die cavity) [4]

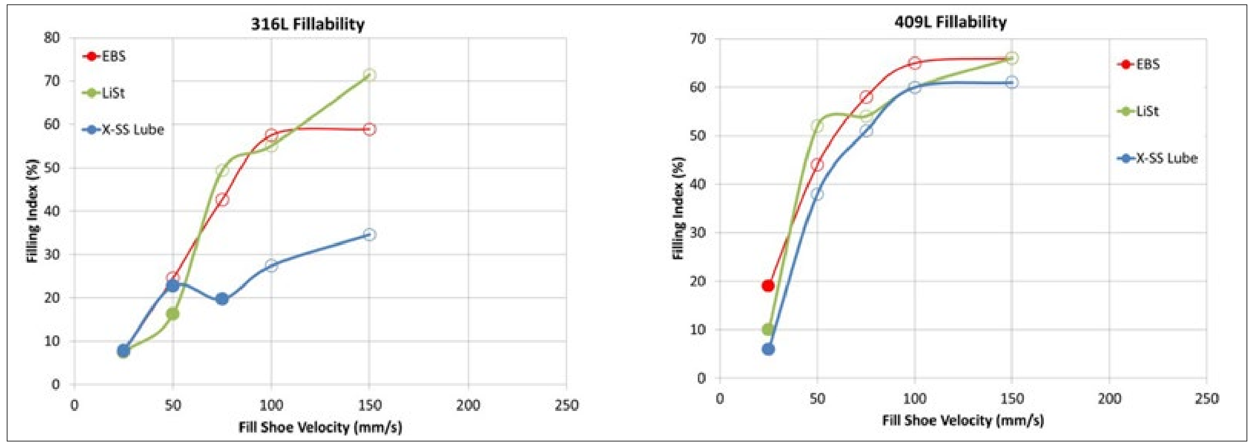

Fig. 11 Fillability of mixes with the cavities perpendicular to the fill shoe motion (solid markers indicate complete fill, empty markers indicate incomplete fill of the die cavity) [4]

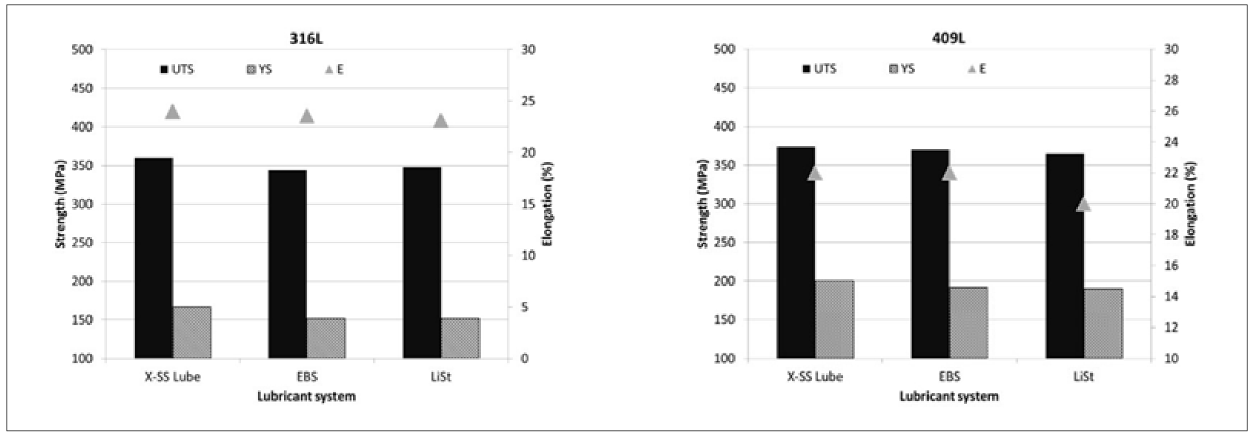

Fig. 12 Tensile properties of 316L and 409L based mixes [4]

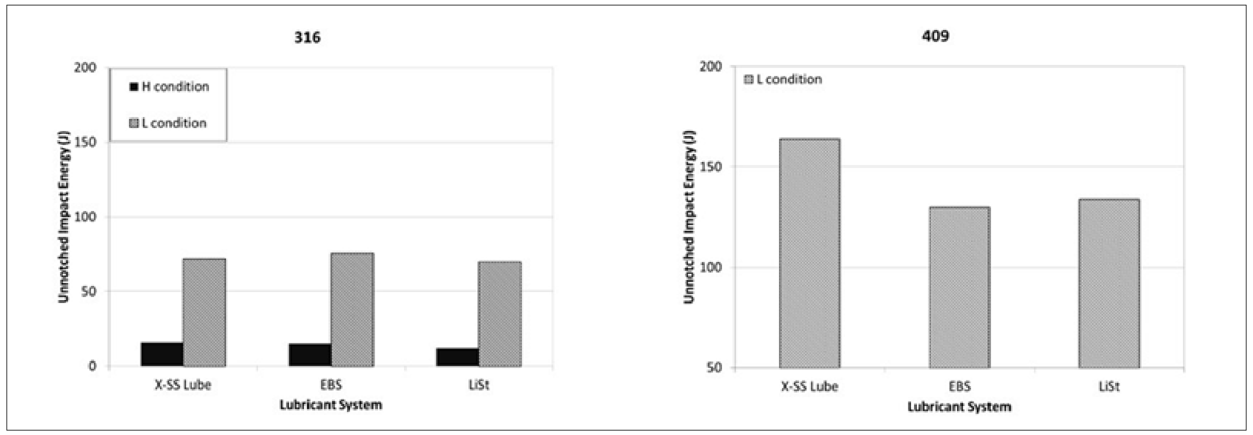

Fig. 13 Impact properties for 316L and 409L based mixes [4]

shoe velocity of 150 mm/s for the experimental lubricant. At the higher fill shoe velocities, the EBS and lithium stearate mixes had incomplete filling. In the perpendicular orientation (Fig. 11), the experimental lubricant provided improved performance compared to EBS and lithium stearate.

A 70% improvement in green strength was observed for the experimental lubricant compared to EBS and lithium stearate (Tables 4 and 5). As stainless steel powders have poor green strength, the experimental lubricant provides the opportunity to reduce failures, which are the result of green compact handling and allows for the compaction of more complex components.

No deleterious effects on mechanical performance were observed for the experimental lubricant (Fig. 12 to 13). It was also reported that interstitial values for carbon indicate a clean delubrication for the experimental lubricant in line with EBS (Table 6).

While corrosion resistance was not evaluated in this study, the low levels of carbon found indicate the corrosion resistance should be as good as the EBS material.

In addition to conducting corrosion testing on the materials investigated in the study, the development is to continue with production trials aimed at determining the benefits of the experimental lubricant with regard to weight scatter, dimensional stability, improved productivity and scrap rate.

Leaner replacements for Mo-Ni-Cu diffusion alloyed PM powder grades

The significant increase in the prices of alloying elements traditionally used as additions to PM structural part materials, such as Mo and Ni, which reached its peak in 2008, has provoked much development work by the major iron powder suppliers, aimed at developing leaner-alloyed alternative materials. Much of this work, reported in previous international PM conferences, has focussed on using alternative alloying elements, such as Cr, Mn or Si.

Development of high compressible leaner alloyed steel powders equivalent to Mo-Ni-Cu diffusion-bonded powders

A more incremental approach to the problem, however, was revealed in a paper by Sylvain St-Laurent and Julie Campbell-Tremblay of Rio Tinto Metal Powders, Canada.

Two different studies were reported that were aimed at developing leaner Mo-Ni-Cu powders as cost-effective replacements for the commonly used diffusion-alloyed grades FD-0405 (0.5Mo-4Ni-1.5Cu) and FD-0205 (0.5Mo-1.75Ni-1.5Cu). The basis for the reported case study on the replacement of FD-0405 was the use of "organic bonding" to bind Ni and Cu additions to pre-alloyed Mo-steel grades. The reported replacement material for FD-0205 used an experimental master diffusion-alloyed powder diluted with Mo-steel powders to achieve the desired chemical composition.

Both studies adopted a similar methodology that included:-

- DOE Taguchi experiments to assess the relative influences on properties of the independent compositional factors (Ni, Mo, Cu and graphite).
- The development of an expert program to aid optimisation of mix formulation to meet a desired set of sintered properties at the lowest possible cost.
- A validation experiment that prepared the predicted optimum material mix and assessed its measured properties against both the properties of the reference diffusion-alloyed grade and the predictions of the model in the expert program.

As the two studies reached quite similar conclusions, this article will report only of the first of the studies.

"Organic bonding" was the term, adopted by the authors, to describe the use of binder treatment of Ni and Cu only, with the graphite and lubricant additions being conventionally admixed, as opposed to a "conventional binder treatment", where all four additions are binder-treated. The choice of the adopted approach was based on data, presented in Fig. 14, on relative dust resistance of a number of alloying approaches. These data indicated that the Ni and Cu bonding offered by the organic bonding approach was significantly superior to conventional binder treatment and was close to that in a diffusion-alloyed material.

Design of experiments (DOE) Taguchi 1.9 was used in this study to firstly evaluate the effects of the four compositional factors: Ni addition (2.5, 3 and 3.5%), graphite addition (0.5, 0.6 and 0.7%), level of pre-alloyed Mo (0, 0.5 and 0.85%) and Cu addition (1, 1.25 and 1.5%). The exact formulations of the prepared mixes are shown in Table 7. A reference mix of FD-0405 was also prepared. The levels of Ni and Cu

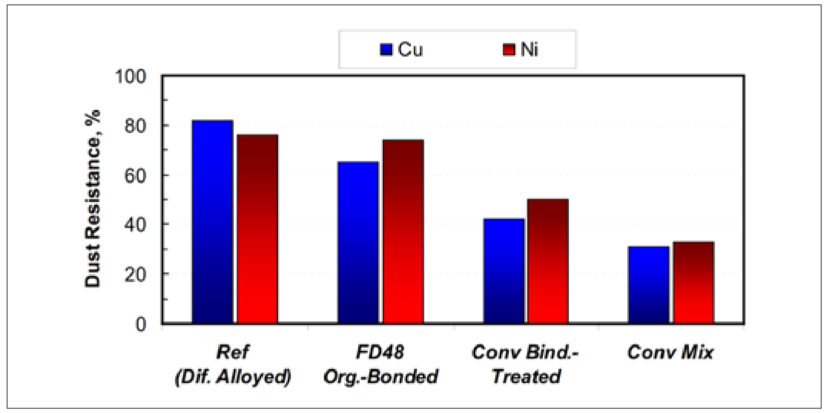

Fig. 14 Ni and Cu dust resistance of various type of 4Ni-1.5Cu-0.5Mo steel powders [5]

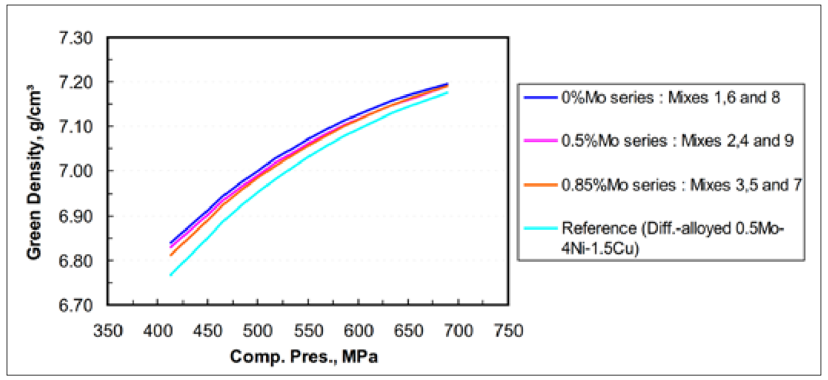

Fig. 15 Compressibility curves for mixes regrouped by level of pre-alloyed Mo [5]

Mix #	Pre-alloyed Mo, %	Admixed Ni, %	Admixed Cu, %	Graphite, %	Cost index
1	0.0 (1001)	2.5	1.0	0.5	0.67
2	0.5 (4001)	2.5	1.25	0.6	0.77
3	0.85 (4401)	2.5	1.5	0.7	0.84
4	0.5 (4001)	3.0	1.5	0.5	0.85
5	0.85 (4401)	3.0	1.0	0.6	0.88
6	0.0 (1001)	3.0	1.25	0.7	0.76
7	0.85 (4401)	3.5	1.25	0.5	0.96
8	0.0 (1001)	3.5	1.5	0.6	0.84
9	0.5 (4001)	3.5	1.0	0.7	0.89
AVG	0.5	3.0	1.25	0.6	0.83
REF	1.25	4.0	1.5	0.6	1.00

Table 7 Description of mixes prepared (Taguchi 1.9). All mixes contained 0.6% EBS wax as lubricant [5]

additions in the formulated mixes were intentionally kept below those of the reference material in order to ensure a cost advantage.

Relative cost indices were calculated for each mix, based on the most recently available alloy prices (Q4 2013) and information on the relative processing costs of diffusion-alloying and organic bonding. These indices are also included in Table 7.

One clear benefit of organic bonding (based on admixing) over diffusion-alloying was an improve-

Mix ID	Process	Chemistry	Cost Index	AD, g/cm³	Flow, s/50g		S. Dens., g/cm³	TRS, MPa	Hard., HRA (HRC)	DC vs green, %	UTS, MPa	YS, MPa	Elong., %
Ref	Diff-Alloyed	0.5Mo-4Ni-1.5Cu-0.6C	1.0	3.10	37	M *	6.99	1570	59.0 (18.2)	-0.22	725	517	1.1
						E *	6.98	1626	58.4 (17.1)	-0.21	788	520	2.2
F48 Lean	Org.-bonded	0.85Mo-2.3Ni-1.15Cu-0.6C	0.80	3.04	32	M *	7.05	1600	59.0 (18.2)	-0.20	757	659	0.5
						E *	7.03	1622	59.6 (19.3)	-0.22	906	636	1.3

* M stands for measured properties and E for properties estimated with the model

Table 8 Estimated and measured properties for a lean organic-bonded powder and an FD-0405 diffusion-alloyed powder. Conditions: compaction at 540 MPa, standard sintering and tempering [5]

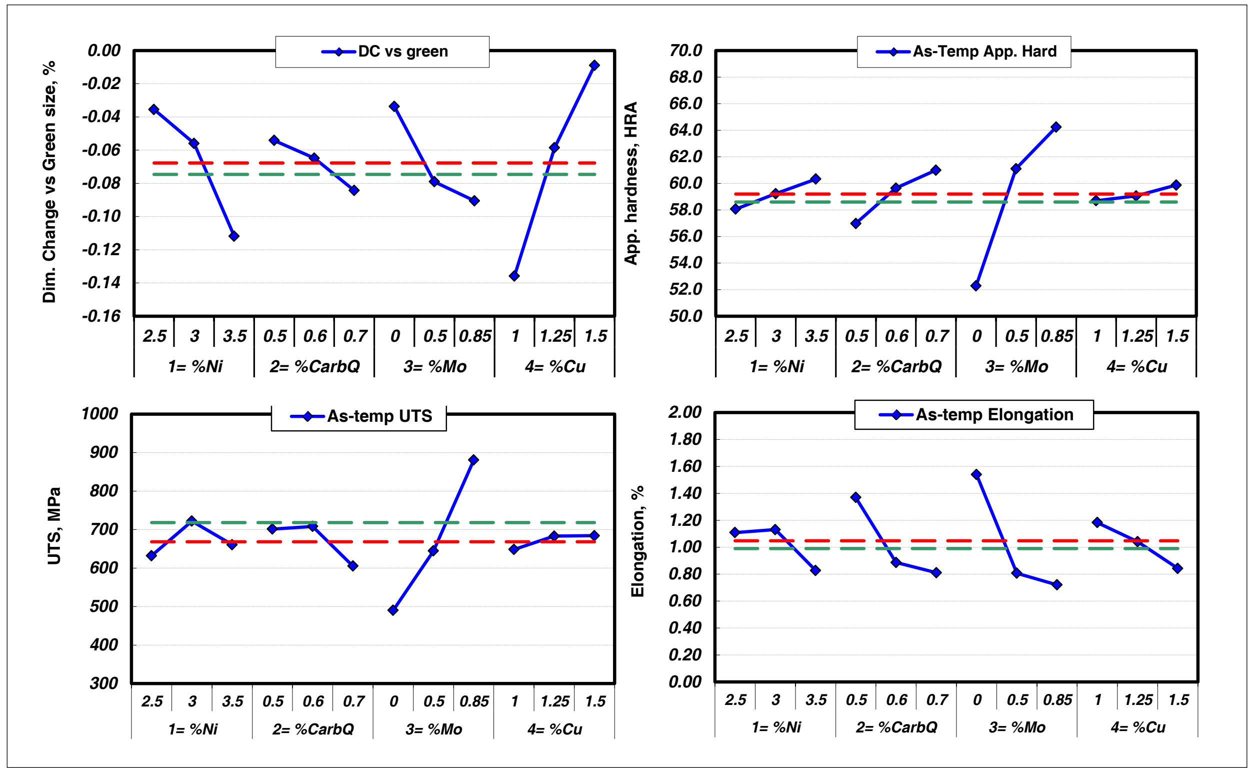

Fig 16 Effect of Ni, graphite, pre-alloyed Mo and Cu content on as-tempered properties at 7.0 g/cm³. Specimens were sintered at 1120°C for 20 min and fast-cooled (1.3°C/sec) [5]

ment in compressibility. Fig. 15 shows the compressibility curves for the mixes grouped by level of Mo. All mixes showed better compressibility than the FD-0405 reference. The typical difference in pressure at 7.0 g/cm³ was around 40 MPa.

The effects of each of the compositional factors on apparent hardness, dimensional change, UTS and elongation are shown in Fig. 16. Mo content was observed to have a stronger positive effect on apparent hardness and UTS than either Ni or Cu content. It was also observed that a graphite content above 0.6% and a Ni content above 3% both led to a drop in UTS. Cu content had a very limited effect on both UTS and apparent hardness.

All of the factors contributed to a reduction in elongation. In the case of Mo, the drop in elongation was mainly between 0 and 0.5%, with further increase in Mo having a much smaller effect.

The effects of each compositional factor were further analysed on the basis of a property-to-cost index, compared to the reference, using the equation:

P/C Index = (PropMix/CostMix)/(PropRef/CostRef)

Fig. 17 illustrates the effect of each factor on the P/C index for hardness, UTS and YS for specimens cooled slowly and rapidly after sintering. The trends in this figure were summarised as:-

- Increasing elemental Ni content lowers the P/C index for all properties, indicating that adding Ni is not a cost effective way to increase sintered properties
- Increasing Mo level is highly beneficial for the static strength (TRS, UTS and YS). On the other hand, it has almost no effect on apparent hardness. Overall, even if costly, Mo allows increasing sintered properties in a cost effective way
- The beneficial effect of Mo on static strength cost ratio is higher under fast cooling conditions
- Increasing graphite level is beneficial for all properties when slow cooled conditions are used. However, 0.6% is the optimum level under fast cooling conditions for the UTS and YS
- Elemental Cu is basically neutral, P/C index remaining quite constant when Cu content is increased
- Except for graphite, none of the factors allows increasing hardness in a cost effective way, Mo and Cu being neutral.

From this analysis it appeared that maximising the Mo level in the base powder and maintaining Ni level low is recommended to maximise sintered strength in a cost effective way. Graphite content of 0.6% is also preferable to maximise strength under fast cooling conditions. Cu does not appear as a key element except for dimensional change; Cu level will be mainly driven by the final part dimensions targeted.

An expert program was next developed, which allowed the determination by iteration of an optimised mix formulation meeting a desired set of sintered properties at the lowest possible cost. An example of the use of this program to develop a lean formulation equivalent to the reference diffusion-alloyed material (FD-0405 with 0.6% graphite) was then provided. The formulation proposed by the model comprised a steel prealloyed with 0.85% Mo as the base material with additions of 2.3% Ni, 1.15% Cu and 0.6% graphite.

A mix was prepared to this proposed formulation and its properties were compared with those of the reference material and with the predictions of the model (Table 8). The proposed formulation gave equivalent or slightly higher sintered properties compared to the reference diffusion-alloyed powder. In general, a very good match was obtained between the measured and estimated properties, except perhaps for UTS and elongation, which were over-estimated by the model for both the binder-treated lean powder and the diffusion-alloyed reference powder.

Differences in the result obtained with the optimised material can be explained by the fact that the DOE used did not allow for the assessment of the effects of interactions. Interactions between factors can be important. Despite these differences, the model proved to be useful in the design of a leaner powder with similar or even slightly better properties than the reference at a much lower cost.

The proposed formulation represents a total cost saving of ~20% compared to the diffusion-alloyed reference powder.

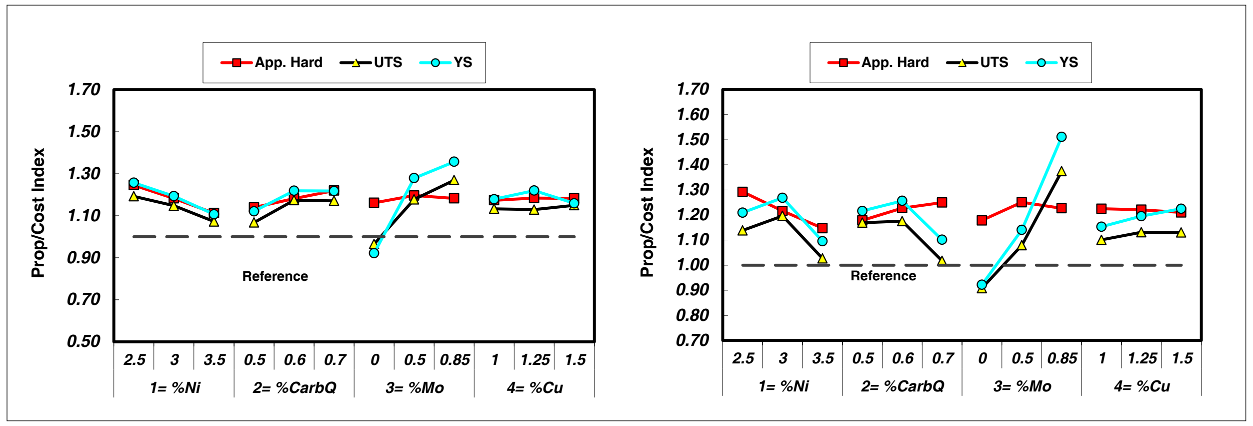

Fig. 17 Effect of Ni, graphite, Mo and Cu content on the P/C Index (relative property to cost ratio) after tempering for (left) standard cooling conditions, and (right) fast cooling conditions [5]

Industrial scale verification of warm die compaction combined with the Hoeganaes AncorMax 225 lubricant system

As previously mentioned, the development and laboratory-scale proving of the proprietary lubricant system, AncorMax 225, has been the subject of past articles in *Powder Metallurgy Review*. Use of this lubricant concept, together with a compaction pressure of 830 MPa (60 tsi), can offer green density levels in excess of 7.5 g/cm³. Fig. 18 illustrates the benefit of reducing lubricant from the traditional 0.75 wt. % to 0.25 wt%; in this idealised data, the increase in pore free density is around 0.25 g/cm³. The combination of the use of the lower lubricant level with the exposure of the powder mass to increased temperatures up to ~150°C, in warm die compaction, delivers the required increase in green density. Fig. 19 shows the effects of raising the die temperature during the compaction of FLN2-4400 and illustrates that increasing the die temperature increases part density by softening the lubricant and consequently lowering interparticle and die wall friction.

The AncorMax 225 lubricant sytem has also been engineered to deliver, at the 0.25wt% addition level, equivalent ejection characteristics and superior green strength to those achieved at the 0.75 wt% addition level with commonly used PM lubricants.

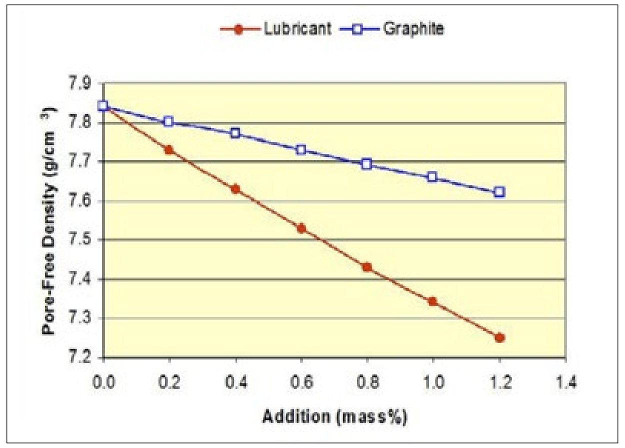

Fig. 18 Influence of lubricant and graphite additions on pore-free density [6]

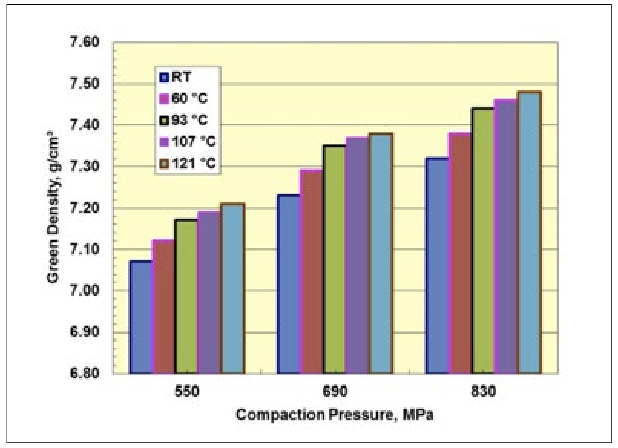

Fig. 19 Effect of die temperature and compaction pressure on green density of an FLN2-4400 [6]

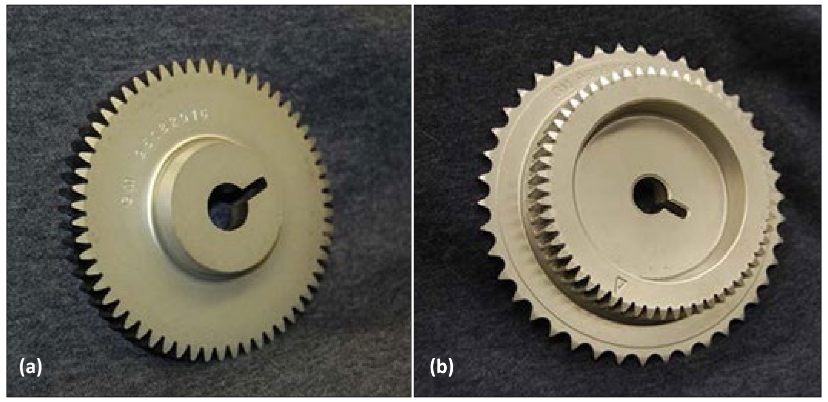

Fig. 20 (a) Main running gear, weight ~450 g; (b) "Spro" gear, weight ~1100 g [6]

Production of high density PM automotive components utilising advanced warm die compaction technology

Industrial experience with this lubricant system and forming approach was the subject of a paper from Gregory Falleur and Suresh Shah (Cloyes Gear, USA) and Francis Hanejko and Sunil Patel (Hoeganaes Corporation, USA).

Cloyes Gear has utilised both warm compaction and warm die compaction to achieve higher part densities through single press/sinter techniques since the early 1990s and has now evaluated and initiated production utilising an AncorMax 225 premix for automotive valve train components. This paper described the manufacturing details and subsequent key production results.

Fig. 20 shows photographs of the components chosen for this study. Both parts were previously manufactured by PM using the warm compaction method (heated powder and heated die) with an ANCORDENSE 450 premix. Both parts were produced, in these trials, from an FLN2-4400 with 0.35 wt% graphite and a total lubricant addition of 0.25 wt%.

During production at Cloyes, the gears were evaluated for the key characteristics of weight variability and press tonnage. Both parts were compacted on standard mechanical PM compaction equipment. The tools were modified to incorporate cartridge heating elements into the stress ring of the die. To measure the repeatability of the bonded premix, multiple set-ups and production runs were completed to ensure product and press performance consistency.

The main gear (Fig. 20a) is compacted to ~7.2 g/cm³ density. In previous experience with the warm compaction route, nominal pressing force was 318 US tons (2830 kN).

Switching to the AncorMax 225 material resulted in an increase in total compaction force of ~8 to 14 US tons (2.5% to 4.4%). This increase in total compaction force was expected because of the reduced powder temperature of the AncorMax 225 relative to the ANCORDENSE 450 process. The consistency of the pressing tonnage part to part (Fig. 21) showed a maximum deviation of less than 1% over two distinct production trials, approximately four weeks apart using two distinct lots of premixed powder.

The part weight consistencies from the two production runs are shown in Fig. 22. The total weight variation of both production runs was less than two grams or approximately 0.5% total weight variation of the part. This translates to a maximum Cpk of ~1.9. Some improvement from the first run to the second run was observed. This can be attributed to the learning curve during production of both the raw material and the part.

Another key aspect of using warm die compaction was the ability to stop the compacting press during the production run without the need to re-establish steady state conditions. This characteristic promotes operational efficiency at the part production level. Further improvements in part-to-part weight consistency are anticipated with additional operational experience and minor modifications to the filling cycle.

Fig. 23 shows the relationship between total compaction force and part weight for the main gear. As noted, this part has approximately 7.2 g/cm³ density and the relationship between pressing tonnage and part weight is nearly linear. This near linear relationship is a direct result of the reduced lubricant in the part giving higher pore free density. Consequently, minor variations in fill will not result in exponential increases in compaction loads and will reduce the potential for die damage and microlaminations within the part from overcompaction.

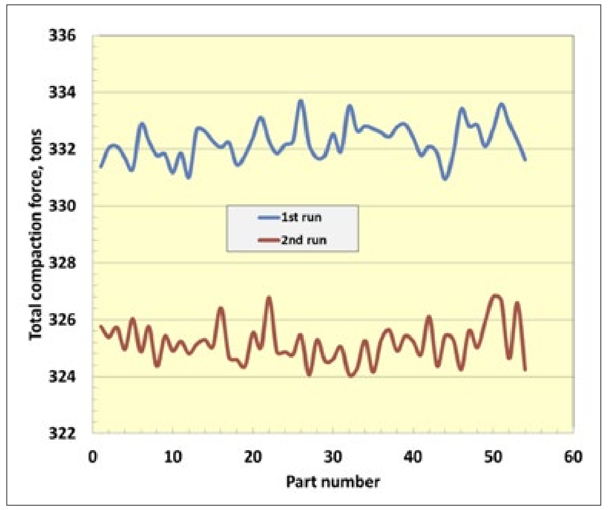

Fig. 21 Main running gear (Fig. 20a), total compaction tonnage from two distinct set-ups approximately one month apart [6]

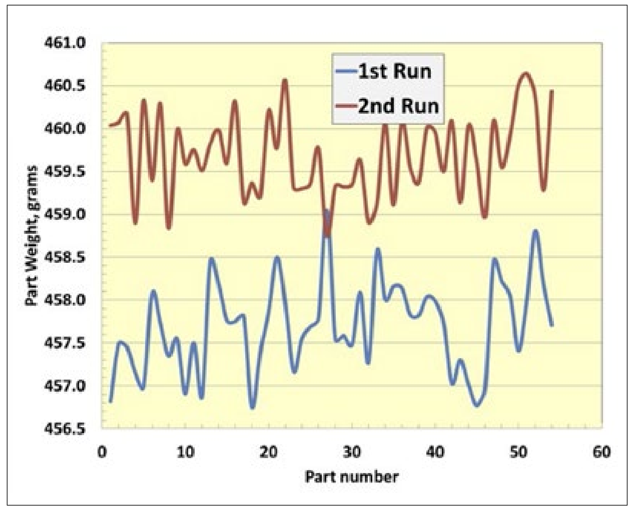

Fig. 22 Part to part weight variation of the main gear shown in Fig 20a [6]

The improvements in part weight and press tonnage control can be correlated with the more consistent powder properties of apparent density and flow. Roughly similar conclusions were drawn from the production trials on the large "Spro" gear.

Another key characteristic necessary for the conversion from warm compaction to warm die compaction was the need to maintain good surface finish. In the trials at Hoeganaes the ejection characteristics of the AncorMax 225 were equivalent or superior to the ANCORDENSE 450. This laboratory experience was replicated in the production trials at Cloyes.

As a final note, a modification to the premix composition was necessary to utilise the existing dies (the modifications still met the MPIF standard for FLN2-4400). The AncorMax 225 material exhibited higher growth than the replaced ANCORDENSE 450 premix. Exact reasons for this difference were not determined and are under investigation.

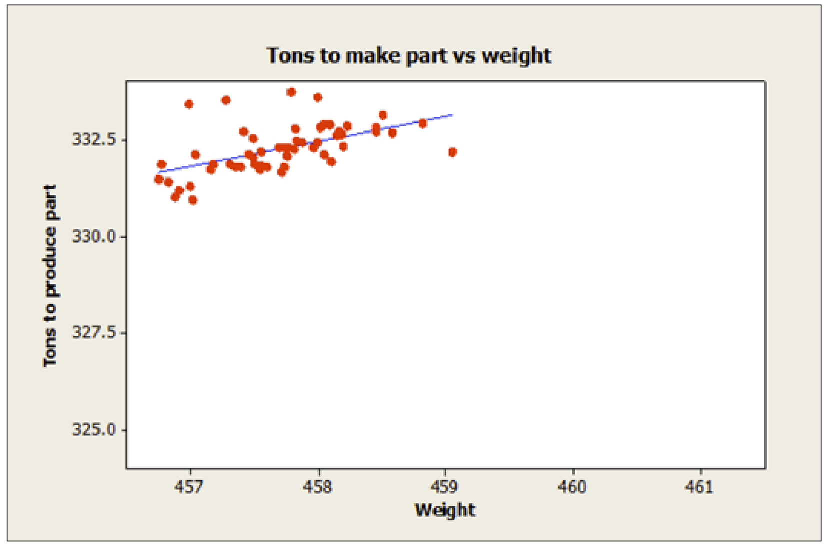

Fig. 23 Part weight vs pressing tonnage for the main gear [6]

PM gear tooth bending fatigue strength

Tooth bend fatigue is a significant potential failure mechanism in gears and needs to be assessed in gear design. Ideally, this assessment ought to be based on fatigue data generated directly on gears. However, such data is often not available, particularly for PM gears. There is, therefore, a strong need to able to transfer fatigue data, derived on test bars, to the design analysis of PM gears.

Predicting PM gear tooth root bending strength

In his paper at PM 2014 Michael Andersson, Höganäs AB, Sweden, reported on a study which examined the comparative merits of four different model types aimed at achieving this transfer and predicting tooth bend fatigue strength in gears.

The four models used to investigate the notch effect in PM materials were:-

- The critical distance method. The criterion in this method is that

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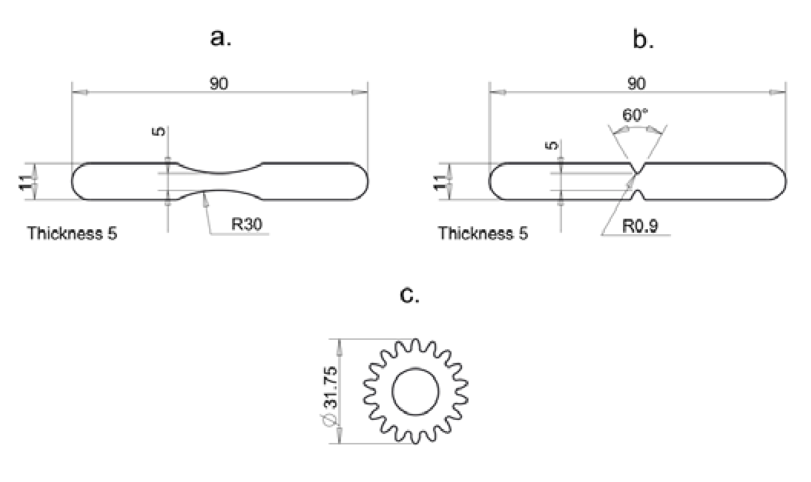


Fig. 24 Test samples: (a) un-notched test bar (ISO 3928), (b) notched test bar and (c) gear [7]

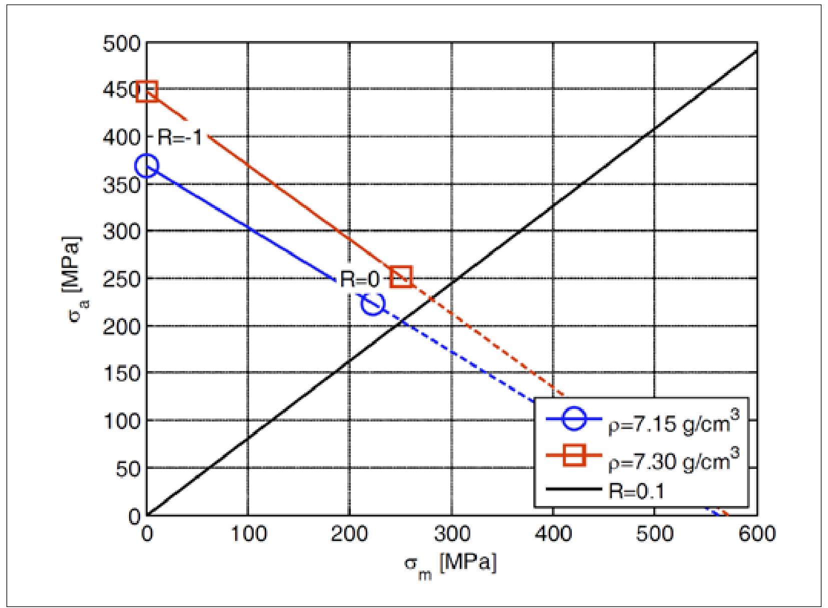


Fig. 25 Haigh diagram, showing mean stress compensation, stress amplitude vs. mean stress and data for un-notched samples [7]

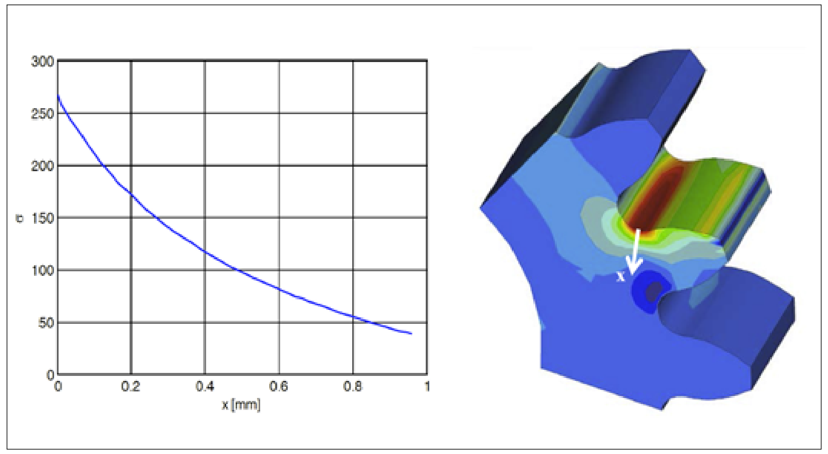


Fig. 26 Stress as a function of distance from the surface at the tooth root and the stress distribution, calculated for a unit load at the tooth tip [7]

the effective stress at a distance dp from the peak stress in a notch should be smaller than the critical stress.

- The gradient method. A similar approach to the critical distance method that introduces a support factor based on the relative stress gradient.
- The highly stressed volume method. This uses a reference volume around the location of peak stress, within which the applied stress falls to no more than a reference level. The reference volume commonly adopted is V90, where the stresses in the volume are 90% or more of the peak stress.
- A fracture mechanics method. This method is based on the observation that, for PM steels, fatigue cracks tend to initiate at the largest pore in the highly stressed volume. By treating crack growth initiation using a fracture mechanics approach, the size of the largest pore can be linked to the fatigue strength of the material. To obtain the size of the largest pore, an approach using extreme value statistics is adopted and the largest pore in a given volume is given by a Gumbel distribution. The parameters that define this distribution can be determined by metallographic investigation combined with image analysis.

Further information on these methods can be found by reference to the full paper.

The experimental procedure, adopted in this study, comprised the production of un-notched and notched fatigue test bars and gear samples (Fig. 24), from Hoganas's Astaloy 85Mo (Fe + 0.85% Mo) + 0.65%C. All samples were through hardened and tempered after sintering.

Fatigue testing in plane bending of the un-notched and notched test bars at load ratios of R = -1 and R = 0 was conducted. To be relevant to the load ratio used in the pulsator testing of gear teeth, the fatigue endurance limits generated in these test bars needed to be recalculated to R = 0.1. This was done through extrapolation in a Haigh diagram (Fig. 25).

The parameters for the fatigue models from the extrapolated results for R = 0.1 were determined. For critical distance, gradient and volume methods, there are two parameters and two data points per density level, allowing the parameters to be calculated. The fracture mechanics model only requires one parameter, which depends only on the structure and thus is density independent. This parameter is determined by a least squares fit of the four available data points. All the resulting parameters are presented in Table 9.

There was finite element analysis of the gear to determine the stress distribution around a gear tooth root arising from point loading on the tooth tip (Fig. 26). By inserting the parameters for the fatigue models, given in Table 9, into the different fatigue models and combining them with the stress analysis for the gears, the strength of the gears was estimated with the different models. Fatigue testing of gears using a pulsator type tooth root bend test was also competed.

The capabilities of the different models were assessed by comparing the predicted strength levels with the experimentally determined values (Table 10). From this table it can be seen that the different models yield results with varying accuracy. Generally, the estimated strengths are reasonably close to the measured values, with a maximum deviation of 13%. However, some models yield significantly better predictions than others.

The volume and fracture mechanics models showed the best accuracy in this study. Both of these models are based on the notion that the notch effect is related to the stressed volume of the component, i.e. a larger stressed volume samples more defects and thus has a lower strength.

Finally, models with a large number of parameters that need to be estimated will also require a large number of experiments. Thus, having a model with only a few parameters,

Model	ρ=7.15 g/cm³	ρ=7.30 g/cm³
Critical distance	d=0.0599 mm $\sigma_{w,cd}$=459.6 MPa	d=0.0981 mm $\sigma_{w,cd}$=459.1 MPa
Gradient	α=0.362 $\sigma_{w,gr}$=364.1 MPa	α=0.843 $\sigma_{w,gr}$=283.7 MPa
Volume	n=43.0 σ_{ref}=495.8 MPa	n=24.9 σ_{ref}=522.0 MPa
Fracture mechanics	ΔK_{th}=4.42 MPa√m	

Table 9 Parameters for the fatigue models [7]

Model	F_w [kN]	
	ρ=7.15 g/cm³	ρ=7.30 g/cm³
Critical distance	2.04 (9.7%)	2.31 (13%)
Gradient	1.99 (6.9%)	2.20 (7.8%)
Volume	1.92 (3.2%)	2.07 (1.5%)
Fracture mechanics	1.96 (5.4%)	2.01 (-1.5%)
Experimental	1.86	2.04

Table 10 Predicted gear strengths using the different models. Differences between predictions and the experimentally determined values are given in parentheses, in percentage terms [7]

but that is able to capture the relevant physical phenomena is a better choice and the fracture mechanics approach has particular attractions in this context. The fracture mechanics model also requires an investigation of the pore structure, but such an investigation is quicker and easier than a full fatigue test. Also, the fracture mechanics approach includes the effect of density in the pore size investigation. For the other models, the effect of density needs to be included separately in the model, introducing further parameters.

Powder forged connecting rods display a further competitive advantage over drop forgings

The operating environment in an engine can have negative effects on the performance of connecting rods, as it is well-known that the strength of most steels quickly deteriorates at higher temperatures. Although the mechanical properties of the materials used to manufacture connecting rods have been widely characterised at room temperature, their strength has not been investigated at higher temperatures

The effect of copper precipitation on mechanical properties at operating temperature of the materials used to manufacture powder forged connecting rods

A paper delivered by Edmond Ilia of Metaldyne LLC, USA, revealed a further and hitherto unrecognised competitive advantage for Powder Forged (PF) connecting rod materials in the contest for market share with Drop Forged (DF) rod materials.

A number of higher strength materials for PF rods, such as the HS150, HS160 and HS170M grades, have been introduced in recent years to match or even exceed the room temperature strength levels (static and dynamic) offered by the higher strength micro-alloyed steel DF rods now being used, 36MnVS4 and 70MnVS4. A comparison of room temperature mechanical properties of HS170M, 36MnVS4 and 70MnVS4 is shown in Table 11.

	Cu	C	MnS	Fe
HS150	3.00	0.48	0.32	Bal.
HS160	3.00	0.58	0.34	Bal.
HS170M	3.25	0.64	0.32	Bal.

Table 11 Mechanical properties at room temperature [8]

	Rm (UTS) (MPa/ksi)	Re (YS) (MPa/ksi)	El (%)	AR (%)
HS170M	1202/174.3	854/123.9	10.0	19.0
36MnVS4[3]	1077/156.1	851/123.4	10.3	42.1
70MnVS4[3]	1077/156.1	701/101.7	9.1	33.7

Table 12 Chemical compositions of HS materials [8]

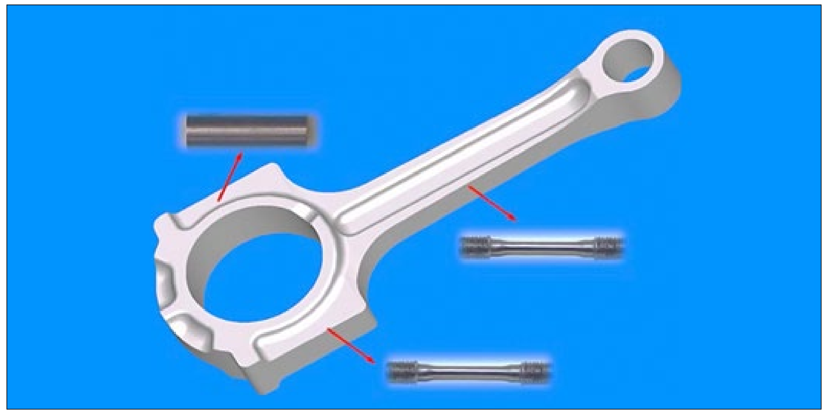


Fig. 27 Tensile and compressive yield strength specimens machined from connecting rods [8]

The development of these new higher strength PF grades has been based largely on the increase of admixed copper content in the powder from the original standard of 2 wt% to levels up to 3.25 wt% in the case of HS170M (Table 12).

The reported study began from the recognition that engine operating temperatures for connecting rods can be as high as 150°C and that, as an aid to connecting rod designers, property data measured at this temperature rather than merely at room temperature should be provided.

The study, therefore, took tensile and compressive yield strength samples from connecting rods (Fig. 27), made from HS150, HS160, HS170M, 36MnVS4 and 70MnVS4, and these were tested at room temperature, 120°C and 150°C.

The results of the tensile tests are overlapped in Fig. 28 and show that:

- The strength levels of the micro-alloyed DF steels reduce as temperature increases
- On the other hand, the strength levels of the high strength PF materials are all higher at 120°C and 150°C than at room temperature
- In terms of YS, all of the HS materials perform better than the micro-alloyed steels at 120°C and 150°C, even though the YS at room temperature for HS150 and HS160 is lower than the micro-alloyed steels.

Similar results were obtained from the compressive yield strength testing (Fig. 29). The author concluded that "when and where it counts, in operation, the strength of the HS materials is significantly higher than that of micro-alloyed steels."

The next stage of the study investigated the microstructural reasons for the difference in reaction of the PF and DF materials to increased temperature. The reduction in strength of the micro-alloyed steels with increasing temperature was assigned to the coarsening (and therefore reduced strengthening effect) of the precipitation hardening vanadium carbonitride precipitates in the as-forged microstructure.

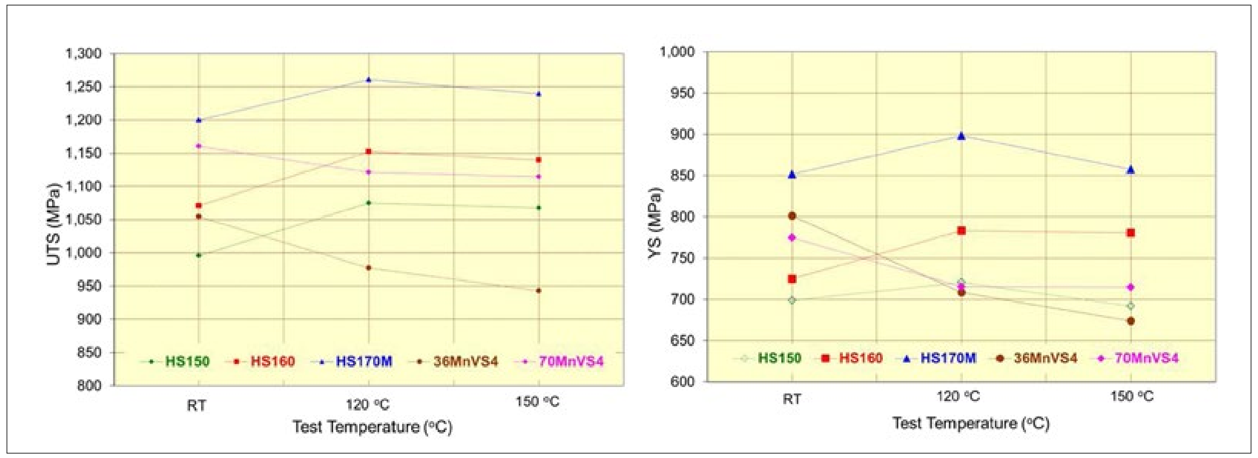


Fig. 28 Strength of both groups of materials as a function of temperature [8]

In contrast, in the case of the HS PF materials, scanning electron microscopy (SEM) studies identified the formation of nano-scale second phase precipitates at temperature. As shown in Fig. 30, precipitates were detected inside the coarse cementite lamellae and, mostly, at the cementite/ferrite interface. In addition, similar precipitates, but finer, heterogeneous, and less defined, were observed in ferrite at higher magnifications. Typically, in all specimens, more precipitates appear to be located in the coarse cementite lamellae than in ferrite. Furthermore, the precipitates are more numerous and finer in the specimen tested at 120°C than in the specimens tested at room temperature or at 150°C. From these first observations, the higher performance of HS materials at 120°C can be attributed to the presence of more numerous and finer precipitates than at room temperature. For the same reason the slight drop in performance at 150°C can be attributed to coarsening of the precipitates.

The nature of these precipitates was then studied using transmission electron microscopy (TEM) combined with X-ray energy dispersive spectrometry (EDS). As shown in Fig. 31, this analysis showed the second phase particles to be rich in copper. This copper precipitation was related to the lower solubility of copper in ferrite at lower temperatures than that in austenite at sintering temperature.

In discussing the design considerations of the results of this study, the author then commented that Powder Forged connecting rods manufactured with HS materials become even more attractive to the automotive industry. In the case of connecting rods manufactured with HS170M, due to the improvement in CYS at 150°C, the safety factor calculated using the CYS at normal operating temperatures would actually be 7.74% higher than the standard safety factor, calculated based on room temperature properties. On the other hand, in the case of steel forged connecting rods manufactured with 36MnVS4, the safety factor calculated using

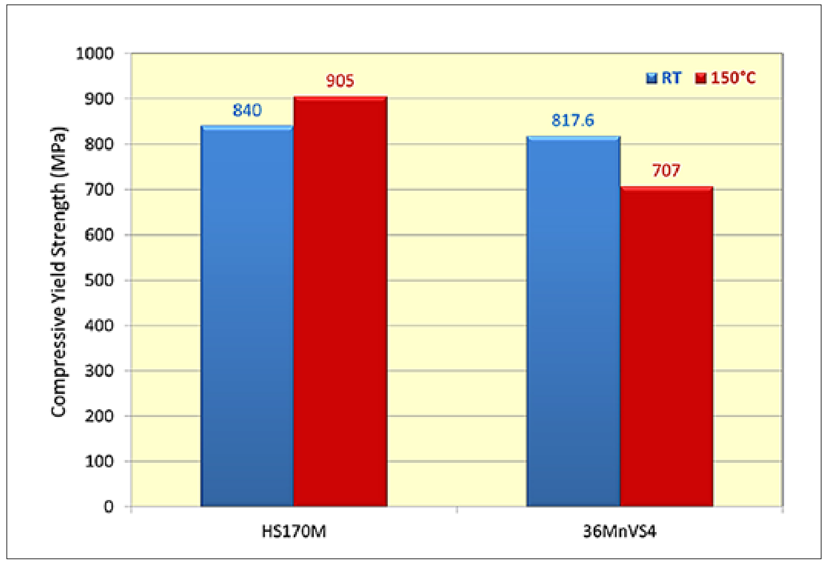


Fig. 29 Compressive yield strength of materials as a function of temperature [8]

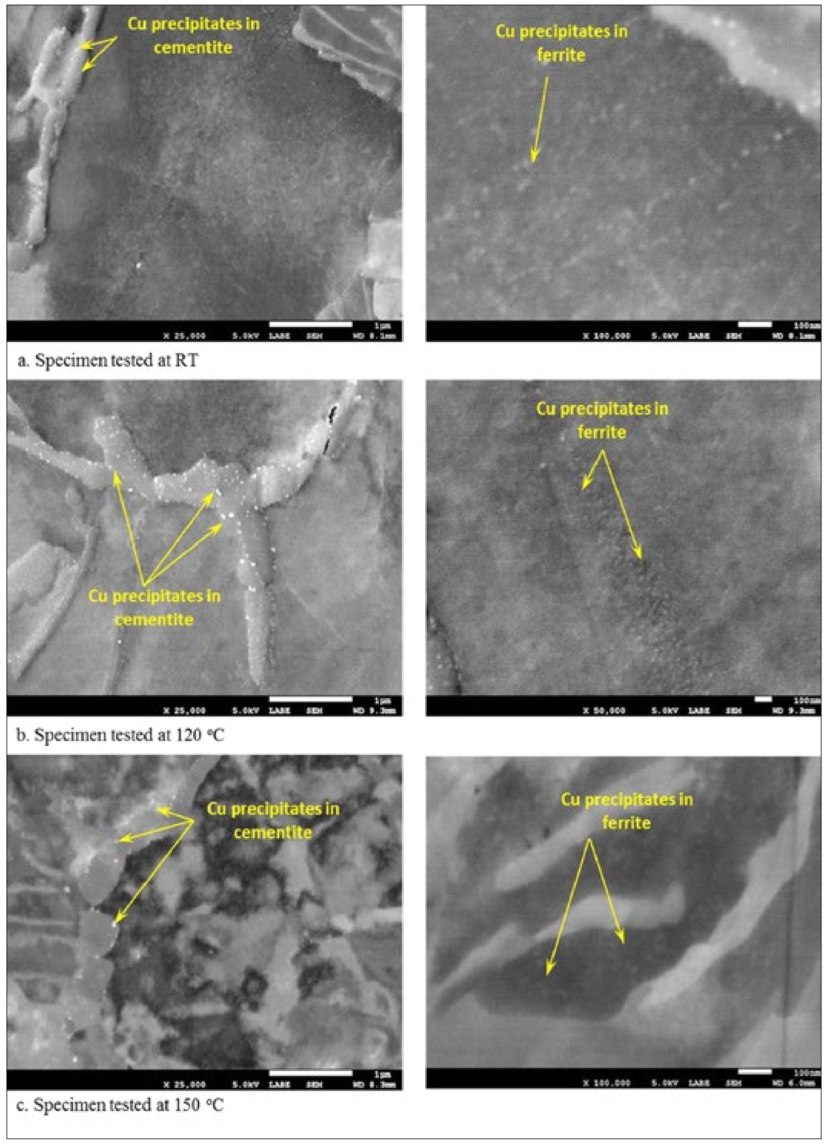
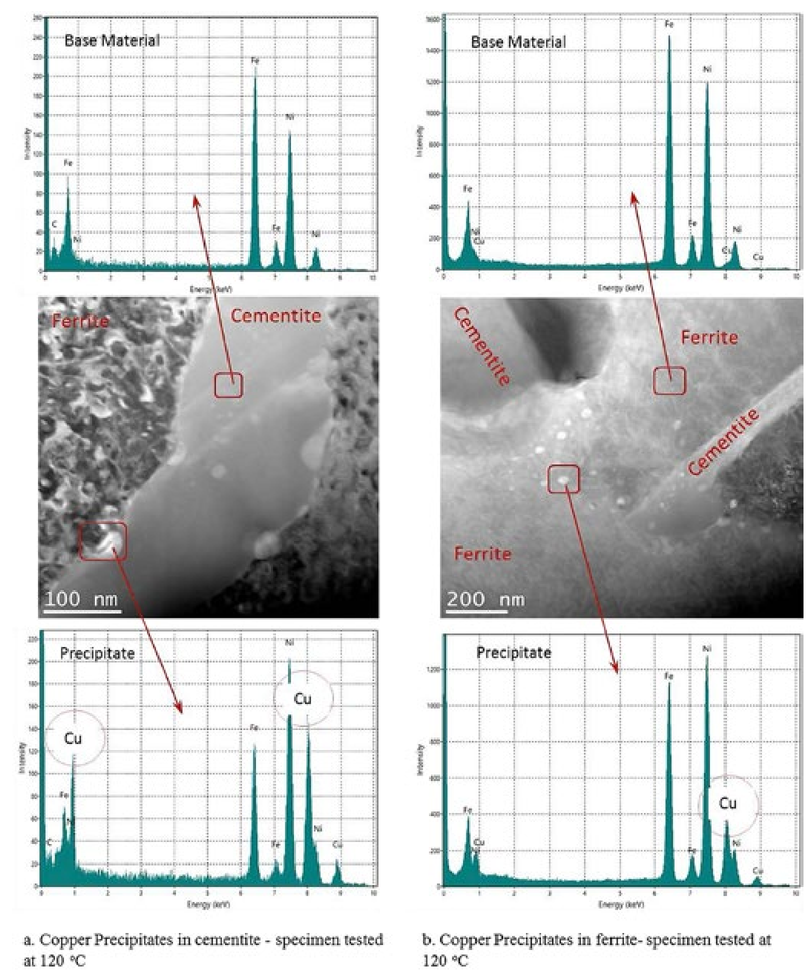


Fig. 30 SEM photos of Cu-rich precipitates observed in tensile specimens in coarse cementite lamellae (left) and ferrite (right) [8]



a. Copper Precipitates in cementite - specimen tested at 120 °C

b. Copper Precipitates in ferrite- specimen tested at 120 °C

Fig. 31 TEM photos and EDS x-ray microanalysis of nano-precipitates in coarse cementite lamellae and in ferrite (tensile specimen tested at 120°C) [8]

CYS at normal operating temperature would be reduced by 13.57%.

As a result, if a typical minimum safety factor for a direct-injection turbo-charged gasoline engine calculated using CYS data obtained at room temperature is 1.2, then the operating safety factor for a powder forged connecting rod manufactured with HS170M is approximately 1.3 (7.74% higher), while the real safety factor in operation in the case of a steel forged connecting rod manufactured with 36MnVS4 is approximately 1.04 (13.57% lower).

Even with this lower safety factor, the design of a steel forged connecting rod is deemed acceptable, based upon historical design and operational data. For this reason, a safety factor of 1.3 in the case of powder forged connecting rods can be considered as unnecessarily high by almost 25%, thus creating an opportunity to reduce the cross section in the I-beam, resulting in significant mass savings. In this way, a leaner powder forged connecting rod with a smaller cross sectional area can be designed, resulting in mass reduction dictated by the improvement in strength due to copper precipitation.

Finally, it was concluded that this hitherto hidden benefit of the copper additions to the PF powder mix "coupled with many others, such as superior raw material utilisation, less mass variation, better machinability and less machining stock, less bore distortion during the fracture splitting operation, better surface condition, less variation in fatigue performance, etc. make powder forging very attractive to the automotive industry, and is able to offer a reliable high performance in operation and additional mass reduction opportunities."

Author

Dr David Whittaker is a consultant to the Powder Metallurgy and associated industries.
Contact +44 1902 338498
email: whittakerd4@gmail.com

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