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CERAMICS / INDUSTRIAL GASES

**SOARING DEMAND FOR
PURIFIED GRAPHITE
SPURS NEED FOR
HIGH-VOLUME FURNACES**

COMPANY PROFILE ///

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SOARING DEMAND FOR PURIFIED GRAPHITE SPURS NEED FOR HIGH-VOLUME FURNACES

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Greetings from Ceramics Expo

I am never not blown away by how much of our daily lives are touched by products that are heat treated. From the time you get up in the morning until the time you hit the sack at night, you are constantly interacting with the heat-treated world.

That being said, thanks for checking out the current issue of *Thermal Processing*, where we're always keeping that window open on the world of heat treating.

And before I dive into what you can find in this issue, let me add that we are very excited to have this issue, as well as part of our *Thermal Processing* team, at this year's Ceramics Expo. Ceramics continues to be a growing part of the heat-treating industry, and being at Ceramics Expo is our way of identifying this crucial niche of the market.

I have been working hard to grow *Thermal Processing's* ceramics coverage to reflect the industry as well. I hope to one day have ceramics information be a major part of the magazine's content, but to do that, I need your help.

So, while we are at Ceramics Expo, drop by our booth (#1713), and say hey. If you have ceramics expertise to share, I would be very excited to discuss publishing your work. I have no doubt our readers will appreciate your contribution.

With this issue focusing on ceramics, as well as industrial gases, our feature articles shine a spotlight on these topics.

Our cover story takes a look at how the soaring demand for purified graphite has spurred the need for high-volume furnaces.

On the subject of industrial gases, our second article take a deep dive into the potential of nitrogen-atomized alloy 625 in the powder bed fusion laser beam process.

In addition to those two articles, our third story continues Gregory Fett's series on carburized steel mechanical properties.

And be sure you check out the latest from our expert columnists.

You'll find all that and more in this month's issue.

Keep in mind that *Thermal Processing* is here to get your message out to your customers, whether that be with news releases that we happily share with our readers or advertising that can drive home what your company can offer. There are options available, and *Thermal Processing's* primary goal is to help you with your company's mission in any way we can.

As always, thanks for reading!

KENNETH CARTER, EDITOR

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The Nutec Bickley contract involves five new lift-up furnaces – two for tempering and three for austenitizing. (Courtesy: Nutec Bickley)

Nutec Bickley gets multi-furnace heat-treat project

Between May and December 2023, Nutec Bickley will manufacture and install five completely new furnaces, along with a fully modernized combustion system, for a leading U.S. manufacturer of high-quality alloy steel and carbon steel closed-die forgings. Onsite work will be completed by Nutec Bickley's expert team one furnace at a time, so that in any given month no more than one furnace will be out of operation, in order to help the customer keep its production schedules fully on track.

This contract involves five new lift-up furnaces — two for tempering and three for austenitizing. Each furnace will be fitted with

a comprehensively modernized combustion system (including replacement fans), latest control systems, complete fiber flues, and new exhaust and pressure control system, plus freshly insulated casings.

Each of these state-of-the-art combustion packages will be fully compliant with NFPA 86 standards, and the installations will comprise the complete supply of materials and instrumentation required to operate the furnace combustion systems, plus new sets of air and gas piping.

Operation will be based on a fuel-only control system (fixed air modulating gas) to allow maximum temperature uniformity potential for all cycles. The retrofitted furnaces will benefit from the incorporation of high-velocity nozzle mixing burners fitted with high-temperature burner blocks. These burners fire with a constant air volume while the control system regulates the gas input by

modulating an impulse-bleed valve.

Meanwhile, the combustion systems will be equipped with an automatic air control valve on the main air manifold that will provide the means to adjust the maximum air volume the system can use, and to lower the air supply to the burners to ensure proper burner ignition conditions.

All controls and instrumentation are mounted in a NEMA-12-rated console, ergonomically arranged for simple and logical operation. Appropriate alarms will be supplied for burner flame failure, loss of air, loss of fuel, and over temperature. The control panel will be installed next to the existing furnace panels and will be prewired and positioned before the furnace replacements begin. They will be wired across the quench pit to the local furnace areas prior to the first furnace being converted. This system will be designed in accordance with the NFPA 70 standard.



SEND US YOUR NEWS Companies wishing to submit materials for inclusion in Thermal Processing's Update section should contact the editor, Kenneth Carter, at editor@thermalprocessing.com. Releases accompanied by color images will be given first consideration.

In order to achieve a completely coordinated command structure, a master PLC will be supplied to integrate the five furnaces and communication with the two existing quench tanks, manipulator/charging machine, the two panel views, the SCADA system, two recording units, and the central hydraulic system. The master PLC will be mounted in a separate NEMA 12 rated cabinet.

When complete, the newly lined units (9in/23cm thick ceramic fiber modules) will work to operational temperature ranges of 900°F–1,950°F (480°C–1,065°C) for the austenitizing furnaces, and 840°F–1,600°F (450°C–1,065°C) for the tempering furnaces. Installed thermal capacities will be 8 million Btu/h (2345kW) for the austenitizing furnaces, and 5.5 million Btu/h (1612kW) for the tempering furnaces.

As with all projects of this type, especially given the exacting timescales involved, carefully planned teamwork between customer and supplier will be a key factor in successful delivery and highest quality outcomes.

“We have long recognized the value of this approach, and we are committed to constant communication and status updates with our customers,” said Rodrigo Gonz lez, VP Metals at Nutec Bickley. “In turn, they reciprocate with the same level of engagement in the

process, resulting in a dynamic and mutually beneficial project result. We are always determined to make this work, and our observation over many years in this sort of undertaking is that the closer the cooperation and the better the flow of information, then the nearer one can get to the optimum progress levels.”

MORE INFO www.nutec.com

Solar Atmospheres of California installs car bottom air furnace

Solar Atmospheres of California (SCA) successfully installed a brand new 14-foot-long car bottom air furnace with a total load capacity of up to 30,000 pounds. The furnace was surveyed in accordance with AMS2750 and is uniform within ±10°F (Class 2). The furnace has a working zone that is 60-inches square by 168 inches long and handles a workload up to 30,000 pounds. With a maximum operating temperature of 1,450°F, this furnace accommodates not only the tempering of large tool steel components but also age hardening of 15-5 PH, 17-4 PH, 13-8PH and nickel-based alloys, and



The new car bottom air furnace has a working zone that is 60-inches square by 168 inches long and handles a workload up to 30,000 pounds. (Courtesy: Solar Atmospheres of California)

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Solar is typically known around the world as a “vacuum-only” heat treater. However, there is a great need for heat treating non-finished parts and materials in accordance with the same specifications (AMS, MIL, Boeing, and Airbus) within different atmospheres where surface oxidation is permissible. This new 14-foot air furnace allows the raw material customer an option, while being more price competitive than with a vacuum environment. This new investment will complement the vast array of vacuum furnaces that Solar operates every day.

“Solar Atmospheres of California is excited to be adding this new furnace and the added capability/capacity,” said Derek Dennis, president of Solar Atmospheres of California. “SCA’s customers have requested this additional capability and it’s our responsibility to meet their needs in supporting the valuable partnerships that we share.”

MORE INFO www.solaratm.com

Bodycote receives science-based target initiative approval

Bodycote, the world’s largest provider of heat treatment and specialist thermal processing services, announced its near-term science-based emissions target has been approved by the Science Based Targets initiative (SBTi).

SBTi is an independent global body enabling businesses to set and validate emissions reduction targets in line with the latest climate science and strict criteria. The initiative is a collaboration between CDP, the United Nations Global Compact, World Resources Institute (WRI), and the World Wide Fund for Nature (WWF) and one of the We Mean Business Coalition commitments.

Science-based targets provide a clearly-defined pathway for companies with ambitious climate goals to reduce greenhouse gas emissions, helping prevent the worst impacts

of climate change and future-proofing business growth. Targets are considered ‘science-based’ if they are in line with what the latest climate science deems necessary to meet the goals of the Paris Agreement — limiting global warming to well-below 2°C above pre-industrial levels and pursuing efforts to limit warming to 1.5°C.

Bodycote, with more than 165 facilities in 22 countries, commits to reduce its absolute scopes 1 and 2 greenhouse gas emissions by 28 percent by 2030 from a 2019 base. Scope 1 includes all emissions directly linked and emitted by Bodycote facilities, and Scope 2 includes all emissions linked to the Group’s purchased inputs, those associated with the purchase of electricity, steam, or cooling. Bodycote measures Scope 3 emissions, in line with the SBTi guidelines, but does not report them as SBTi deems the quantum to be immaterial.

Bodycote’s services are vital to ensuring the performance and longevity of crucial components in almost every part of the mod-

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Ipsen's Pecatonica, Illinois, is being repurposed for vacuum furnace hot zone assembly. (Courtesy: Ipsen)

ern world, enabling — among many other advantages — longer lifetimes, less machining, less waste, and greater fuel efficiency. Both in the products processed and the way they are processed, Bodycote's services support industry to avoid emissions, commonly referred to as Scope 4. By avoiding emissions from the outset, Bodycote is a major contributor to helping industries to reduce their carbon footprint and help to minimize the adverse impact on the climate.

"We are very pleased to achieve approval of our near-term science-based emissions targets," said Stephen Harris, Bodycote Group chief executive. "Managing energy and reducing our environmental impact has long been part of our corporate culture. As a company, Bodycote is focused on ethical and sustainable growth, and proud of our commitment to setting an ambitious target. Leading by example, Bodycote demonstrates the positive impacts of carbon reduction for its stakeholders and encourages other businesses to commit to science-based targets."

MORE INFO www.bodycote.com

Ipsen repurposes ceramics facility for expanded hot zone

Ipsen in Pecatonica, Illinois, (formerly Ipsen Ceramics) is being repurposed for vacuum furnace hot zone assembly. The plant is cur-

rently undergoing major refurbishments, including new lighting, HVAC, roofing, and other interior and exterior upgrades.

The factory, located at 325 John Street, is less than 30 miles from Ipsen's Vacuum Technology Excellence Center in Cherry Valley and will initially employ up to eight material assemblers. Incorporating the Pecatonica location into Ipsen's vacuum furnace production and aftermarket process will provide added benefits to customers.

"Our goal is to reduce delivery times and better control the critical phase of assembly," said Jake Hamid, Ipsen's director and chief operating officer.

In the future, Ipsen is considering other manufacturing activities in Pecatonica to supplement the needs of the Vacuum Technology Excellence Center.

Ipsen is now hiring material assemblers.

MORE INFO www.ipsenusa.com
www.ipsenglobal.com/careers

Plibrico team brings smiles with cards for hospitalized kids

Hospitalized children can go through many different treatments, surgeries, and therapies that can take up long stretches of time. During those very difficult moments, children often feel alone, isolated, and afraid. They miss their friends and life outside of the hospital. For

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A simple act of kindness lets hospitalized children know the Plibrico team cares. (Courtesy: Plibrico Company)

these children, it is a simple act of kindness, a handmade card, that can brighten their day and put a smile on their faces.

Recently, the Plibrico Company's employees came together from Ohio to Washington, including New York, Indiana, and Florida, to volunteer their time and creativity to make handmade cards for Cards for Hospitalized Kids. The charitable organization will distribute the cards to children battling health issues across the country, brightening their day and demonstrating that someone is thinking of them.

Located in Chicago, Cards for Hospitalized Kids (CFHK) spreads hope, joy, and magic to hospitalized kids through uplifting, handmade cards distributed to Children's Hospitals and Ronald McDonald Houses (RMH). More than 500,000 children in hospitals in all 50 states have received cards from CFHK since its inception. Each card is unique, just like the child who receives it.

"I hope that our inspirational cards can give a sick and hospitalized child an added bit of strength, encouragement, and comfort to

get them through a tough time and let them know there are people who truly care about them," said Pamela Gaul, director of marketing at the Plibrico Company. "Creating the inspirational cards was easy to do and fun to make. Everyone that helped build the cards had a great time coming together as a team for a great purpose. I know the cards will help the kids that receive them from CFHK, but this project has really helped the team too."

Handcrafted cards have been proven to improve a patient's mood, emotions, and sense of self-worth. The cards help to help retain the human connection between patients and the outside community and advance feelings of gratefulness. Many hospitals frequently request inspirational cards for their potential to reduce stress and depression, as well as to lighten the weight of serious, lingering illnesses.

Across the United States, the Plibrico Company and its employees are taking an active interest and giving back to the communities they call home. The company has actively participated in or raised funds for

charities that include Shriners Hospitals for Children, St. Jude Children's Research Hospital, Wounded Warrior Project, and Feeding America, among others.

MORE INFO www.plibrico.com
www.cardsforhospitalizedkids.com

L&L ships furnace to titanium steel manufacturer

L&L Special Furnace has delivered a highly uniform, front-loading box furnace to a north-eastern U.S. supplier of titanium castings for the aerospace and power generation fields.

The company deals with exotic metals such as nickel and cobalt-based alloys that are ideal for superior products using the lost wax process for castings.

The L&L model FB435 has an effective work area of 48" wide by 32" tall by 60" deep and has certifiable temperature uniformity of $\pm 10^{\circ}\text{F}$ from 500 to 1,850 $^{\circ}\text{F}$.

The furnace has elements that are very evenly spaced around the chamber. It is lined with ceramic fiber on the sides and top. The door is a pneumatically operated vertical door with a counterbalanced lift system. Included is a NEMA 12 control cabinet with fused disconnect switch, digital program controller, high-limit backup system, thermocouples, fusing, and all interconnection wiring.

The control is a Honeywell DC2500 control with Ethernet capabilities with separate overtemperature protection. The power control is accomplished with six-zone SCRs with digital biasing and one control loop. The floor is composed of castable sections with insulating firebrick backup insulation. There is an 18-inch diameter, air-cooled fan to promote uniformity at lower temperatures. A 6-inch 600 CFM venturi cooling system is included with an outlet port along with a variable speed drive for controlled cooldown rates.

The furnace case is sealed internally for atmosphere control. An inert blanketing gas such as nitrogen is used to displace oxygen within the work chamber. This provides a better surface finish as oxidation is less likely to form on the part. The atmosphere is delivered automatically through a flow panel that is automated by the furnace control.

All L&L furnaces can be configured with



L&L Special Furnace's FB Series electric box furnace. (Courtesy: L&L Special Furnace)

various options and be specifically tailored to meet thermal needs. The company also offers furnaces equipped with pyrometry packages to meet the latest revision of ASM2750.

Options include a variety of control and recorder configurations. A three-day, all-inclusive startup service is included with each system within the continental United States and Canada. International startup and training service is available by factory quote.

MORE INFO www.lfurnace.com

AFC-Holcroft's Disler steps down; new management named

William Disler, president and CEO at AFC-Holcroft, has stepped down from the company, effective March 30, 2023.

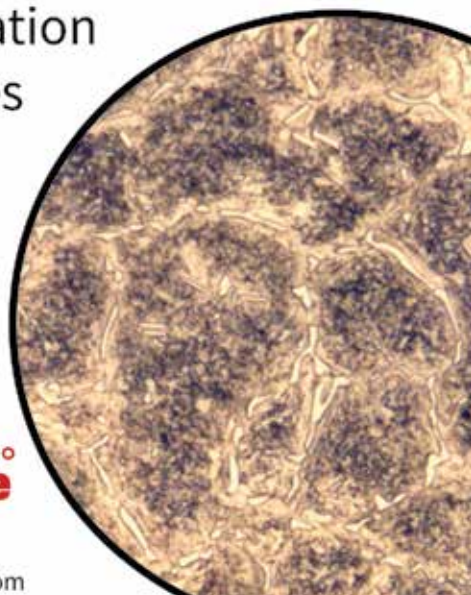
As part of the departure news, the company announced Tracy Dougherty and Ronald

Waligora will share senior leadership responsibility for leading the company. Dougherty, formerly vice president of sales, has been named chief operating officer for sales, applications, marketing, and aftermarket sales while Waligora, formerly senior engineering manager, has been named chief operating officer for project management, engineering, manufacturing, and field services.

"It has been an exciting and rewarding journey with AFC-Holcroft," said Disler. "It is hard to believe it started more than 35 years ago. Over the years I have had a chance to work with many incredible people and have made many friends. I have every confidence that the new leadership will continue to guide the company in a good direction. We have brought together a stellar team, and I feel good that I am leaving the company in such capable hands, and that this company will continue its long history of excellence into the future. I am very proud of AFC-Holcroft and the people that have always worked so hard to make it such a great company."

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“We are proud to once again provide our customers in the Southeastern U.S. with another regional option for aerospace and defense vacuum thermal processes,” said Steve Prout, president of Solar Atmospheres’ Greenville facility. (Courtesy: Solar Atmospheres)

Disler started his career at the Holcroft Company in 1987 as an electrical engineer newly graduated from Lawrence Technical University, where he currently sits on the Advisory Board of the College of Engineering. His career included extensive international involvement, including living in Asia for more than two years coordinating furnace co-builds with multiple customers. He began to transition into engineering and sales roles, such as manager of advanced controls and Far East operations manager, with a growing emphasis on international business, which eventually led to his senior positions at AFC-Holcroft. He traveled throughout more than 25 countries while supporting sales, engineering, and manufacturing activities. After serving as the company’s executive vice president starting in 2005, he was named president and CEO of AFC-Holcroft in 2012.

“I would like to offer my appreciation to Bill for his dedication to AFC-Holcroft over so many years,” said Christian Grosspointner, CEO of Aichelin Group. “And to the new management team, I wish you great success as you lead the company forward.”

“With our new management team in place, I can now take the time to enjoy other private and professional pursuits that I was unable to find time for,” Disler said. “I will always support the AFC-Holcroft team and plan to keep in touch as much as possible with my friends and colleagues. Thanks to everyone—employees, customers, partners, and friends

who helped make my time at AFC-Holcroft so gratifying.”

AFC-Holcroft, founded in 1916, is one of the leading manufacturers of industrial furnace systems used in the heat treatment of ferrous and non-ferrous metals. The company has approximately 120 employees globally and has been part of the Aichelin Group since 2016.

As part of Berndorf AG, Aichelin Group is a leading provider of heat-treatment solutions, such as industrial furnaces, induction hardening plants, industrial gas burners systems, control and automation systems, and industry-4.0 solutions as well as services.

MORE INFO www.afc-holcroft.com

Solar Atmospheres SC awarded Northrop Grumman approval

Solar Atmospheres Greenville, South Carolina, facility announced it has been awarded Northrop Grumman approval. With this approval, all five Solar Atmospheres facilities are now an option for customers with Northrop Grumman requirements for vacuum heat-treating services.

“We are proud to once again provide our customers in the Southeastern U.S. with another regional option for aerospace and

defense vacuum thermal processes, saving them time and money while continuing to deliver the high level of quality required,” said Steve Prout, president of Solar Atmospheres’ Greenville facility.

With the ability to support vacuum thermal processing needs ranging from development cycles to 50,000-pound loads at temperatures of up to 2,400°F, Solar Atmospheres provides AS9100 and Nadcap quality accredited heat treatments, providing customers with the confidence their product is being processed as specified.

MORE INFO www.solaratm.com

Aalberts surface technologies expands austempering

Aalberts surface technologies - HIP | braze | heat treatment announces an expansion of its austempering capabilities and capacity in Canton, Ohio and Ft. Smith, Arkansas.

Three atmosphere-to-salt furnaces will be added at the existing facility in Canton. This expands the number of austempering locations in the Aalberts surface technologies U.S. footprint to four. Infrastructure will be engineered in Canton to allow for up to six austempering furnaces in the future. The expansion in Canton will allow Aalberts

to better serve customers in the eastern United States and those with product flowing through the area. The austempering and marquenching equipment is expected to be online in Q4 2023.

The Aalberts surface technologies austempering facility in Ft. Smith was started in 2018 to bring its capabilities to an underserved market. In response to growing demand in the region, additional capacity will be added at the Ft. Smith plant, with one furnace to be installed in Q3 2023 and another planned for Q4 2024.

The added capacity will allow the company to support its current customers' growth plans as well as demand from new entrants into the fast-growing austempered ductile iron market. Additionally, the expanded U.S. footprint creates redundancy that provides customers greater flexibility, risk mitigation, and cost reduction opportunities.

"We are pleased to announce our latest investment in technology to add capacity that meets our customers' growing demand

in key markets," said Steve Wyatt, president of Aalberts surface technologies – HIP | braze | heat treatment. "The added capacity allows us to maintain our high level of customer service and further strengthens our position as the market leader in austempering and marquenching."

Austempering and marquenching are technical heat treatments that quench iron and steel parts in molten nitrate-nitrite salt instead of more traditional quench media, creating a preferred crystal structure.

Austempered iron and steel have improved mechanical properties (tensile, yield, and elongation) and higher toughness in comparison to traditional quench and temper processes. In addition to improved mechanical performance, austempering and marquenching of components results in substantially reduced distortion, often allowing for finish machining before heat treatment and a reduction in total cost.

MORE INFO www.aalberts-st.com

Busch Vacuum Solutions acquires VESCO-McLaughlin

Busch Vacuum Solutions U.S. announced the acquisition of the business of VESCO-McLaughlin, Inc. (VESCO) in East Windsor, Connecticut.

VESCO is a leading industrial service company specializing in heat treating and metallurgy industries.

"With this acquisition, we are excited to be able to offer our customers an even more comprehensive range of vacuum services in the heat treat and metallurgy industries," said Turgay Ozan, president of Busch LLC. "We are committed to providing our customers with the best possible products and services, and this move allows us to do just that."

VESCO was formerly part of the McLaughlin Furnace Group (MFG), a U.S.



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UPDATE /// HEAT TREATING INDUSTRY NEWS

market leader in engineered vacuum solutions for thermal processing and surface preparation applications. McLaughlin Furnace Group's major customers operate in the aerospace and defense; automotive; energy and environment; metalworking and fabrication; and semiconductor fabrication industries.

With the addition of VESCO, Busch Vacuum Solutions expands its business by adding complementary services to its already broad offering of global field service, overhaul, repair, and installation.

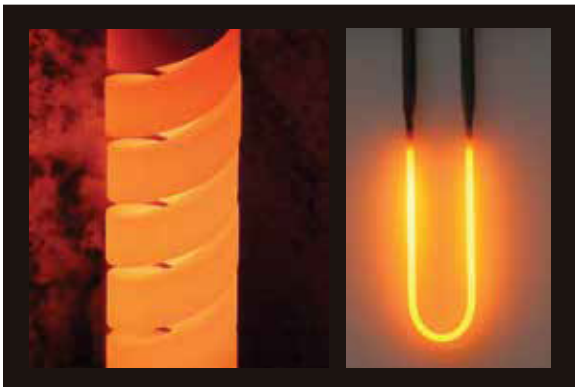
In a strategic move, VESCO will be rebranded and will expand its current business operations under the brand name of VESCO — A Company of the Busch Group, while continuing uninterrupted service for existing customers by maintaining relationships with existing suppliers and transitioning existing VESCO employees to Busch Vacuum Solutions.



VESCO employees with Turgay Ozan, fourth from left, general manager of Busch LLC. (Courtesy: Busch Vacuum Solutions)

MORE INFO www.buschusa.com

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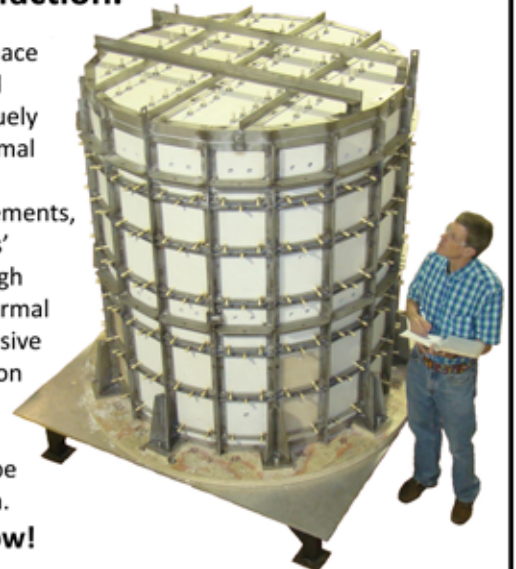
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Optris has CSvision ratio pyrometers for high temperatures

The new CSvision series of ratio pyrometers from Berlin-based infrared specialist Optris can measure the temperature of metals, melts, or ceramics non-contact, safely and reliably from different distances.

Infrared pyrometers must meet high demands. Especially in metallurgy, they are often used under harsh conditions in which they must deliver reliable results at any time. Smoke, steam, or dust often impede a clear view of the measured object and affect the measurement signal. In these conditions, ratio pyrometers nevertheless provide stable measured values compared to single-channel pyrometers, even with dirty optics or with objects that move within the measuring field (e.g. metal rods or wires).

The new CSvision is equipped with the



The CSvision is also usable with the Optris IRmobile app. (Courtesy: Optris)

innovative Smart Ratio Mode (SRM) and can thus master even challenging applications with variable emissivity ratios. The built-in video sight and the motorized focus, which can be operated via software or app, allow the CSvision novelties to be focused very conveniently on the respective object. The switchable two-stage brightness reduction filter ensures optimum viewing conditions even with very hot and therefore bright objects. Together with the crosshair laser, which is also standard, this ensures simple sensor alignment under all conditions.

The CSvision R1M offers an optical resolution of up to 150:1 and a measuring range of 600 to 3,000°C with a spectral range of 0.8 to 1.1 μm in harsh industrial environments up to 65°C without cooling. The R2M has an optical resolution of 75:1 and a spectral range of 1.45 to 1.75 μm . This allows temperatures to be measured from as low as 300 °C to 1,400°C (up to 60°C without cooling).

Optris creates an easy-to-use solution with the CSvision series that can be set up quickly



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and easily. The infrared thermometers have an interface to the IRmobile Android app and CompactPlus Connect software. This allows easy video alignment and real-time process monitoring. Two analog outputs are available for process integration, as well as digital interfaces such as RS485 or Modbus RTU. A variably programmable I/O pin can optionally be used as alarm output, for signal triggering or e.g. for external emissivity/slope settings.

MORE INFO www.optris.global.com

Chinese company buys three Seco/Warwick furnaces

The Chinese manufacturer of a wide variety of vacuum circuit breakers chose Seco/Warwick for the second time by ordering vacuum furnaces for metal heat treatment.

The order includes three Vector® vacuum

furnaces that ensure a very high vacuum level and temperature uniformity within the entire load. Seco/Warwick is one of the few companies in the world providing this type of equipment for electrical equipment manufacturers.

In 2017, this partner purchased two Seco/Warwick furnaces for the first time. Their reliability and high quality of finished production made them decide to develop further production protocols, choosing the Seco/Warwick Group again as their industrial furnace supplier.

“The high-vacuum brazing system consisting of three Vector furnaces will be delivered to the Chinese manufacturer of electrical relays and switches,” said Maciej Korecki, vice president of the Vacuum Furnace Segment in the Seco/Warwick Group. “We are glad that our partner decided to use our solutions again, because it means that the previous systems work flawlessly. It must also be admitted that the specificity of this electric power control production requires incredible precision.

Circuit breakers need a high level of vacuum and temperature uniformity. They are brazed in vacuum furnaces in a very high vacuum; therefore, it was necessary to use an efficient pumping system consisting of a turbomolecular pump and a dry pump. I can proudly say that such a specific solution can be produced by only a handful of companies in the world, and we are one of them. However, our solutions have the best parameters, and this was the decisive factor in choosing Seco/Warwick as the technology supplier.”

Vector is a versatile single-chamber vacuum furnace, operating in more than 70 countries. Every day, Vector vacuum furnace systems thermally treat millions of components that meet the highest standards. Vector vacuum furnaces provide an extensive range of processes, and the compact, modular design allows users to adapt them to specific needs.

The Vector furnace family guarantees high quality and surface protection of machined parts. Thanks to consistently achieving tight temperature uniformity, this technology

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quent Asian companies equip their machine parks with Seco/Warwick furnaces. It is also important that existing customers return after a few years and place new orders. On the Chinese market, long-term relationships with customers are an important value. We want to provide our partners with solutions that will allow them to expand their business and achieve their goals related to production, quality, and profitability.”

The vacuum level in the furnace is maintained by vacuum pumps. Depending on the application — a single pump or a combination of mechanical, diffusion, and/or turbomolecular pumps are used. The turbomolecular pump used in this application is a sophisticated type of mechanical vacuum pump. The turbomolecular pump features high degassing velocity in the particle flow area and high compression ratio, using less energy than diffusion pumps and without oil vapor pollution. ♪

MORE INFO www.secowarwick.com

A new Seco/Warwick order in China includes three Vector® vacuum furnaces that ensure a very high vacuum level and temperature uniformity within the entire load. (Courtesy: Seco/Warwick)

ensures repeatable operational results. It is a perfect furnace to use in production requiring precision. At the same time, it is a furnace characterized by low energy and process gas consumption. The furnace’s low emissivity translates into environmental friendliness,

which is in line with the company’s philosophy — SECO/ECO.

“We are highly active on the Chinese market,” said Liu Yedong, managing director of Seco/Warwick China. “Our reputation is growing; hence, it is unsurprising that subse-

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Flow Meters	Nitrogen, endo, natural gas, air
Controls	Allen Bradley PLC
General	System 1 rear handler



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Max Temp	1450°F
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Max Fuel Demand	1000 CFH, 800,000 BTU
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INTERNATIONAL FEDERATION OF HEAT TREATMENT AND SURFACE ENGINEERING

Industry conference news for 2023, beyond



The 28th IFHTSE Congress, sponsored by the Japanese Society for Heat Treatment, will be November 13-16, in Yokohama, Japan. (Courtesy: Shutterstock)

The 5th International Conference on Heat Treatment and Surface Engineering of Tools and Dies (HTSE-TD), sponsored by the Chinese Heat Treating Association, will be April 24-27, 2023, in Hangzhou, China. This conference will finally resume the HTSE-TD series. At the present time, it is being planned as a face-to-face conference. Please remember that a visa for entry to China is required. Correspondence should be sent to Lihui Liu, chta@chta.org.cn.

» **More info:** htse-td.allconfs.org/meeting/index_en.asp?id=6861

AIM is proud to announce the ECHT 2023 Conference in Genova, Italy, at Magazzini del Cotone. The conference will meet May 29-31, 2023. ECHT 2023 will cover all relevant topics for the heat-treatment and surface-engineering community. The conference will include a special focus on sustainability.

» **More info:** www.aimnet.it/echt2023.htm

Heat Treat 2023 will be co-located with IMAT 2023 in Detroit, Michigan, October 17-19, 2023. It will cover many topics of interest. If an abstract was accepted into the Heat Treat 2023 technical program, the author should submit a full manuscript (6-8 pages) by the deadline date. Complimentary full conference registration will be offered to authors (presenting author only) who submit a full manuscript for the proceedings.

IMPORTANT DATES

» Accept/Reject Notification: April 17, 2023.

» First Draft Manuscript Due: May 31, 2023.

» Final PDF Manuscript Due: July 17, 2023.

» **More info:** www.asminternational.org/web/heat-treat/event-info

28TH IFHTSE CONGRESS

The 28th IFHTSE Congress, sponsored by the Japanese Society for Heat Treatment, will be November 13-16, 2023, in Yokohama, Japan. This wide-ranging conference offers participants the opportunity to network and hear papers on a wide-ranging series of topics, including the thermal processing of steel, surface hardening, additive manufacturing, and modeling and simulation of industrial processes.

IMPORTANT DATES

» Deadline of abstract submission: April 28, 2023.

» Notification of acceptance: May 31, 2023.

» Preliminary program release: June 30, 2023.

» Deadline of extended abstract: July 14, 2023.

» Deadline of early registration: July 31, 2023.



The ECHT 2023 Conference will be in Genova, Italy, May 29-31.

» Deadline of full paper submission: September 22, 2023.

A special issue of JSHT is scheduled to be published in March 2024. Applicants can submit a full paper to the special issue. Only the presenters of the 28th IFHTSE Congress can submit full papers for this special issue.

The 29th IFHTSE Congress, held jointly with the ASM IMAT annual meeting September 30-October 3, 2024, is planned for Cleveland, Ohio. Details will be announced soon.

SPOTLIGHT ON MEMBERS

Quaker Houghton Inc.

Quaker Houghton Inc. in Conshohocken, Pennsylvania, is a global supplier of industrial process fluids. It is the largest supplier of quenchant, both oil and polymer, to world-wide customers. In addition to heat-treating product technology, it can provide process modeling, filtration, metering and monitoring devices, quenchant selection, customized proactive maintenance programs, and complete technical service and training.

The company operates in 25 countries and has more than 4,700 employees, including chemists, metallurgists, and engineers to partner with customers to improve its operations in a more efficient and sustainable manner.

UPCOMING IFHTSE EVENTS

APRIL 24-27, 2023

5th International Conference on Heat Treatment and Surface Engineering of Tools and Dies

Liangzhu Dream Town, Hangzhou, China

MAY 29-31, 2023

ECHT23

Genova, Italy | www.aimnet.it/echt2023.htm

OCTOBER 17-19, 2023

Heat Treat 2023

Detroit, Michigan | www.asminternational.org/web/heat-treat

NOVEMBER 13-16, 2023

28th IFHTSE Congress

Yokohama, Japan

For details on IFHTSE events, go to www.ifhtse.org/events



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INDUSTRIAL HEATING EQUIPMENT ASSOCIATION

IHEA Combustion Safety Training: One-Day Safety Standards and Codes Seminar



IHEA's Safety Standards & Codes seminar is designed for individuals involved in the design, manufacture, service, or operation of ovens, furnaces, kilns, dryers, thermal oxidizers, and a wide range of industrial applications.

Explosions and fires in industrial heating systems can result in injury or loss of life, as well as loss of property and production. Understanding the proper use of National Standards governing the compliant design and operation of ovens and furnaces is essential for everyone involved with this type of equipment.

IHEA's Safety Standards & Codes seminar is designed for individuals involved in the design, manufacture, service, or operation of ovens, furnaces, kilns, dryers, thermal oxidizers, and a wide range of industrial applications.

Don't miss this important training event as IHEA presents a one-day Safety Standards and Codes seminar in conjunction with the Process Heating and Cooling Show Tuesday, May 23, in Rosemont, Illinois. The seminar covers critical safety information for those

involved with a wide range of industrial thermprocess applications. This abridged, one-day course will focus on recent updates in the newly released *NFPA 86 Standards for Ovens & Furnaces, 2023 Edition*. Attendees will also learn identification of principles and concepts for several critical safety topics.

One-Day Program agenda includes:

- » Overview of NFPA 86 standards for ovens and furnaces including administration, references, and definitions.
- » General requirements, location, and construction.
- » Classification of furnaces: A, B, C, and D.
- » Gas line evacuation (purging) and charging.
- » Commissioning, operations, maintenance, inspection, and testing.



Jeff Rafter



Jason Safarz



Bob Sanderson

»Hydrogen introduction.

»Burner management systems and PLC applications.

IHEA speakers are directly involved with the NFPA committees and will discuss the changes in the newly released edition of NFPA 86.

Speakers include:

»Jeff Rafter, Selas Heat Technology Co. LLC.

»Jason Safarz, Karl Dungs, Inc.

»Bob Sanderson, Rockford Combustion.

The seminar registration fee includes classroom instruction, seminar materials, and handouts; lunch; and full conference registration to the Process Heating and Cooling Show. Attendees will also receive a printed copy of the 2023 edition of *NFPA 86 Standard for Ovens and Furnaces*. Upon completion of the course, seminar attendees will be issued a certificate documenting six Professional Development Hours (PDHs).

» **More info:** www.ihea.org/event/SSMay23



Safety Standards and Codes seminar is being presented in conjunction with the Process Heating and Cooling Show. Seminar registration includes full conference registration for the show.

IHEA CALENDAR OF EVENTS

MAY 16

IHEA Induction Seminar

Alabama Power Technology Applications Center | Calera, Alabama

Classroom instruction and hands-on demonstrations in the lab on the basics of heat induction, including induction theory, heat sources and simulation problems. Includes copy of IHEA's *Induction Process Heating Handbook for Industrial Applications*. Registration fee is \$125.



MAY 23

Safety Standards & Codes Seminar

Donald E. Stephens Convention Center | Rosemont, Illinois

MAY 24

Process Heating & Cooling Show

Donald E. Stephens Convention Center | Rosemont, Illinois

The 2023 Process Heating & Cooling Show is a place for anyone involved with industrial heating and cooling processes. The effective application of heat, or the removal of it via cooling, involves critical, transformative steps during the manufacturing processes of many products. This event will bring together industrial cooling and heating equipment makers with plant and process personnel from numerous companies in the process industries, including those working in oil and gas, electronics, pharmaceuticals, food, beverages, packaging and plastics.

» Register at www.process-heating.com/heat-cool-show

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For details on IHEA events, go to www.ihea.org/events

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The importance of carbide formation and reheating steps during simulation.

Low pressure carburization simulation of steel alloys with strong carbide forming elements

Low pressure carburizing (LPC) is quickly gaining popularity across many industries due to LPC's reduced cycle time, lack of oxidation/decarburization at and near surface, better efficiency and repeatability, and power savings when compared to conventional gas carburization. [1,2] However, designing the boost-diffuse schedule is anything but straightforward, especially for steel alloys containing strong carbide-forming elements (Cr, Mo, V, and W). [3] In light of this difficulty, many furnace manufacturers now include software with their LPC furnaces which are capable of designing the LPC schedule based on a few simple inputs. This works great for certain alloys, but once strong carbide-forming elements are introduced to the alloy system, a simple diffusion model, based on Fick's Second Law, will no longer suffice.

During a boost step, carbon quickly reaches the saturation limit in austenite on the surface of the part. If alloy elements with an affinity for carbon are present, carbides will form and grow. Once the diffuse step begins and the carbon-carrying gas is evacuated from the furnace, the carbides are able to dissolve, providing additional nascent carbon for diffusion. If the carbides are not allowed to dissolve significantly during the diffuse step, they can block further carbon diffusion and result in unacceptable microstructural features in the final part. Therefore, the modeling of carbide formation and dissociation must be included for accurate LPC simulation predictions.

In addition to including a carbide model, the LPC process simulation should also include any relevant steps where carbide dissociation and/or carbon diffusion can occur. For any process specification requiring a slow cool and reheating step after carburizing, as opposed to quenching directly from the carburizing temperature, the reheating step should be considered for an accurate carbon profile prediction. Including any additional process heating steps in the simulation will help ensure the predicted carbon profile matches the carbon profile witnessed in production.

The importance of including carbides and a reheat step for AISI 9310 undergoing an LPC process, with a subsequent reheat, will be demonstrated in this article. By controlling the carbon potential in the furnace, atmospheric carburizing of 9310 results in few carbide issues. However, the formation of detrimental carbides when using LPC to carburize 9310 is a real possibility. Figure 1 shows unacceptable carbides at the near surface during LPC trials for AISI 9310. The process described in "The Effect of the Quenching Method on the Deformations Size of Gear Wheels after Vacuum Carburizing" is simulated using DANTE's VCarb software tool and the results compared

to the measurements reported in the publication. The reported process uses three boost – diffuse pairs (6 – 13, 4 – 34.5, 3.5 – 16 minutes) and the part is cooled to room temperature after carburizing. The part is then subjected to a hardening cycle, which includes austenitization, soaking, and quenching in oil.

The first two simulations executed exclude carbide formation. This is a common assumption made to simplify the model and reduce the

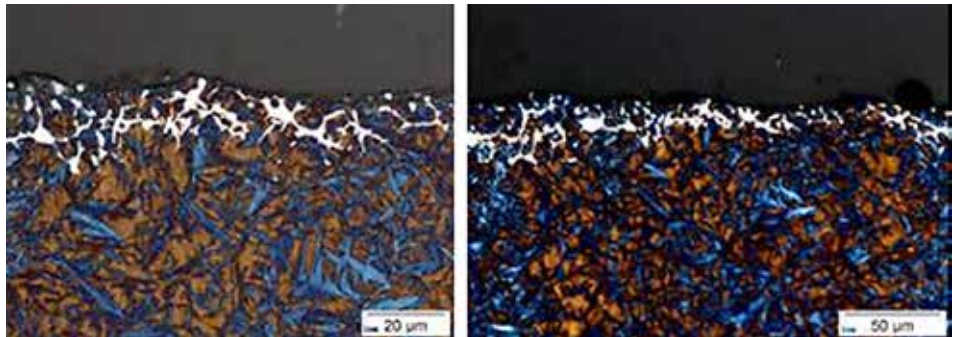


Figure 1: Carbides shown near surface for AISI 9310 after LPC processing.

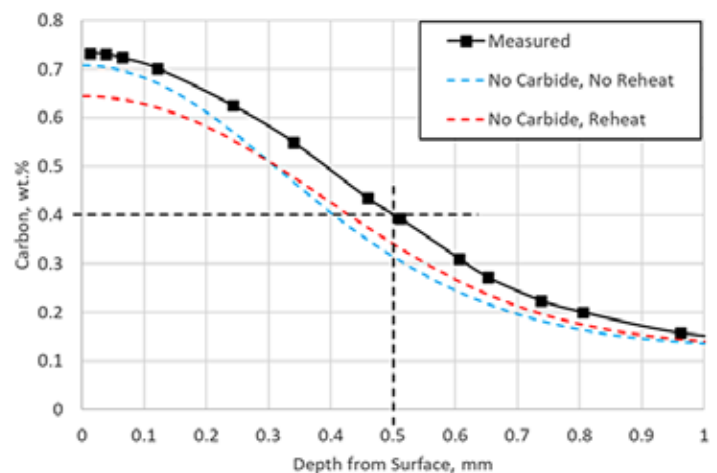


Figure 2: Comparison of measured data with simulations with and without a reheat step and with no carbide predictions.

amount of material data needed. Models describing carbide kinetics can be challenging and time-consuming to develop and validate. Once developed, each alloy to be simulated would need to be characterized during LPC processing. Figure 2 shows the carbon profile prediction when carbides are not considered, with and without a reheating step. Without including the reheat, the surface carbon concentration is predicted to nearly match the measurement, but the effective case

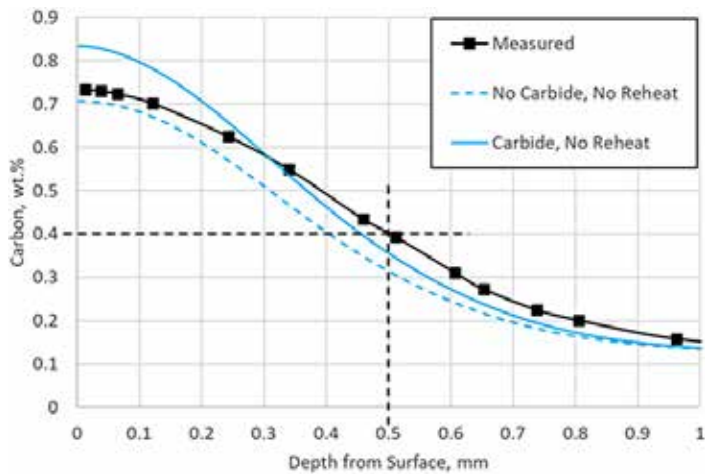


Figure 3: Comparison of measured data with simulations with and without carbide predictions and with no reheat step.

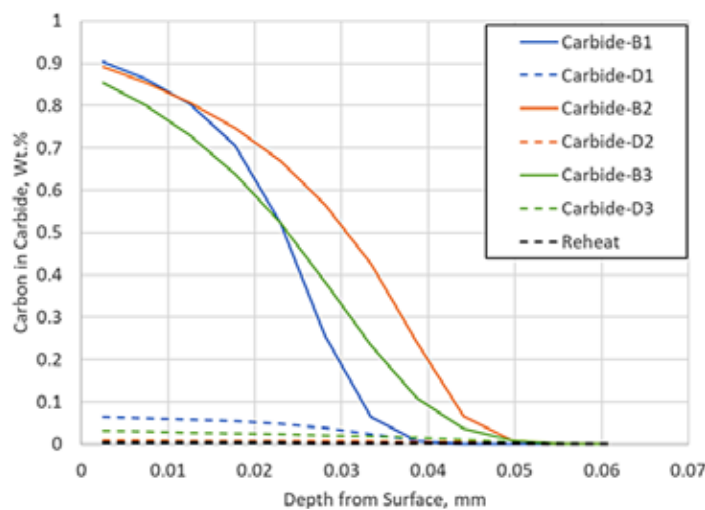


Figure 4: Carbon in carbide form at the end of each boost and diffuse step.

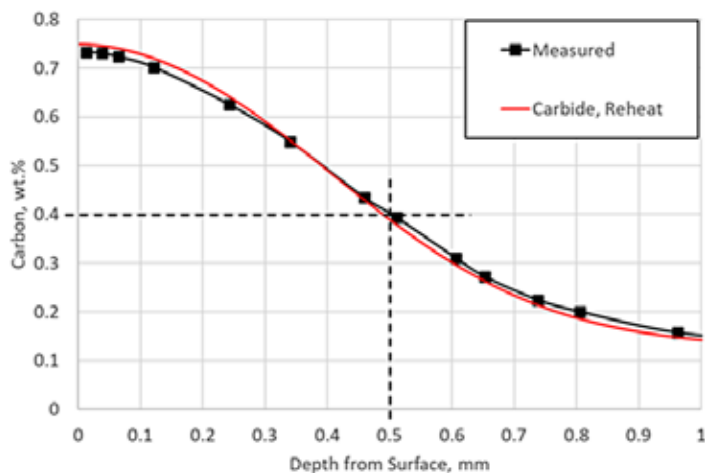


Figure 5: Comparison of measured data with simulation that includes carbide predictions and a reheat step.

depth (ECD) is underpredicted by 0.1 mm. Once the reheat is included, the ECD prediction is closer to the measurement, but the surface carbon is underpredicted by 0.1 percent.

DANTE, including VCarb, includes a standard carbide model which includes model parameters for AISI 9310 carbide kinetics during LPC processing. Figure 3 compares the predicted profile with and without carbide formation, neglecting the reheat step, to the measured data.

Including carbide formation overpredicts the surface carbon by 0.1 percent, but the ECD prediction is nearing the measured data. The addition of the carbide prediction serves to provide more than additional carbon to the surface; it also provides an additional carbon source for diffusion. Figure 4 shows the carbon in carbide form for each of the three boost – diffuse pairs. It is clear that a substantial amount of carbon is taken up into carbides during the boost step, 0.9 percent at the near surface, and then dissociates into nascent carbon and diffuses into the part during the diffuse step.

There is very little carbon in carbide form at the end of this particular LPC schedule, but the reheat step ensures there are no carbides present as the part enters quench; in practice, a long final diffuse is generally used to ensure no carbides persist after carburizing. Including the reheat step with the carbide prediction in the simulation yields an identical match to the experimental data, as shown in Figure 5.

CONCLUSION

The importance of including carbide formation and dissociation when simulating an LPC process for steel alloys containing strong carbide-forming elements was demonstrated and it was shown that carbide models are critical to an accurate carbon profile prediction. It was also shown that it is possible to form carbides during the LPC process, but not have any after the carburizing process. It is therefore imperative to characterize medium- and high-alloy steels for carbide formation if undergoing an LPC process.

While LPC recipe development can be accomplished through trial and error, it has been reported that some high-alloy steels used in the aerospace industry can take years to develop a single LPC recipe yielding acceptable results. This painstaking process does not even consider any effect of geometry on diffusion and carbide kinetics, which can be substantially more than atmospheric carburizing if the LPC process is not well controlled and/or the recipe is not well designed. Therefore, simulation is a more efficient method than trial and error for LPC recipe design. While it is true that characterizing the carbide kinetics for a steel alloy is time-consuming, though once complete, the model can be used to optimize a unique LPC recipe for each new geometry using that steel alloy. Simulation can also be used to optimize any surface carbon or case depth discrepancies between convex (higher surface carbon and shallower case depth) and concave (lower surface carbon and deeper case depth) geometric features. ☞

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////////// ABOUT THE AUTHOR

Justin Sims is a mechanical engineer with Dante Solutions, where he is an analyst of steel heat-treat processes and an expert modeler of quench hardening processes using Dante software. Project work includes development and execution of carburization and quench hardening simulations of steel components and analysis of heat-treat racks and fixtures. He has a mechanical engineering degree from Cleveland State University.



A discussion on the methods used to measure salt concentration and procedures to recover polymer from a high salt-containing solution.

Quenching from molten salt into polymer quenchants

In this column, I will discuss quenching aluminum from molten salt into a polymer quenchant.

A molten salt bath is a common method of heating aluminum components to the solution heat-treating temperature. The salt is heated to the solution heat-treating temperature, and parts are immersed in the bath. The salt is usually a mixture of sodium and potassium nitrate, and usually complies with MIL-S-10699 Type I or II compositions (Table 1).

Parts are solution heat treated in salt for many different reasons—because the part is big, because of rapid heating, or cost. After solution heat treatment, the part is removed from the molten salt using some sort of crane, and the part is lowered into the quench tank containing the polyalkylene glycol (PAG) solution. This typically must be done in under ten seconds or less to minimize part cooling. As described in an earlier column, polyalkylene glycol solutions are used effectively to control distortion and residual stresses in many different aluminum product forms (extrusions, castings, forgings, cast, sheet metal, etc.). The molten salt is very liquid at the solution heat-treatment temperature of aluminum, and depending on the geometry of the part, significant drag-out of the molten salt can occur.

If the solution was strictly water, the specific gravity of the water and molten salt (50% NaNO₃ and 50% KNO₃) would increase. This increase is linear with the percentage of sodium nitrate and potassium nitrate present in the water (Figure 1). The solubility of sodium nitrate is 848 g/L at 25°C, and the solubility of potassium nitrate is 3830 g/L at 25°C. These salts are both very soluble in water.

Typical polymer quenchants have a specific gravity of approximately 1.09. However, the diluted solutions have a much lower specific gravity of approximately 1.015 at a concentration of 20 percent.

Polyalkylene glycol quenchants exhibit inverse solubility in water. PAG quenchants are completely soluble at room temperature but become insoluble at elevated temperatures. This phenomenon provides the unique mechanism for quenching hot metal by surrounding the metal piece with a polymer-rich coating that serves to govern the rate of heat extraction into the surrounding aqueous solution. The inverse solubility of PAG quenchants occurs at approximately 165°F, known as the cloud point.

If the water and PAG quenchant are heated to above the cloud point, the polymer and water separate, with the polymer sinking to

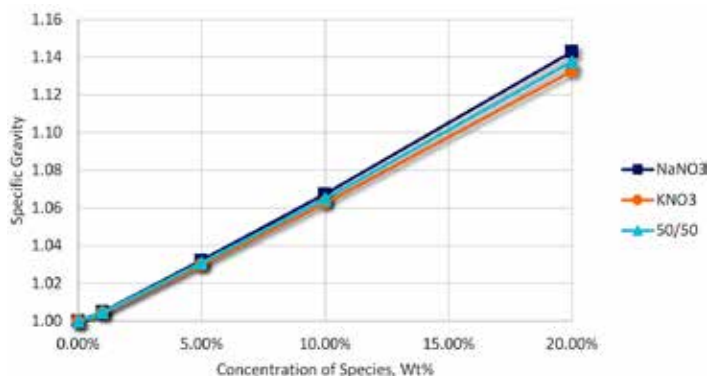


Figure 1: Specific gravity of sodium and potassium nitrate as a function of concentration (Wt%) in water.

the bottom of the beaker (or quench tank). This is due to the density differences between water with a specific gravity of 1.0, and the polymer quenchant with a specific gravity of approximately 1.09.

However, as the salt content of the water and PAG solution increases, the cloud point of the polymer quenchant decreases. This is mainly a function of inorganic content of the water/polymer solution and specific gravity. If the cloud point decreases to approximately room

Class	Working Range (°F)		Working Range (°C)		Composition (%)								
	Min	Max	Min	Max	NaNO ₂	NaNO ₃	KNO ₃	Na ₂ CO ₃	NaCl	KCl	BaCl ₂	BaCl ₂ plus SiO ₂	CaCl ₂
1	325	1099	163	593	37-50	0-10	50-60	-	-	-	-	-	-
2	550	1099	288	593		45-57	45-57	-	-	-	-	-	-

Table 1: Typical compositions for molten salts used for the solution heat treatment of aluminum per MIL-S-10699 [1].

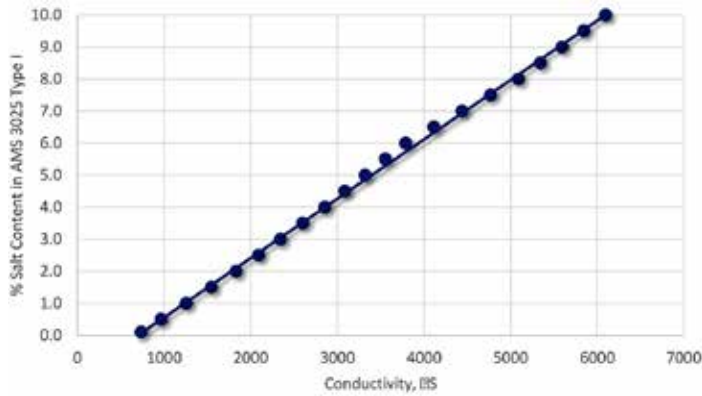


Figure 2: Conductivity of a 23 percent AMS 3025 Type I PAG quenchant, mixed with varying concentrations of a MIL-S-10699 Class I salt.

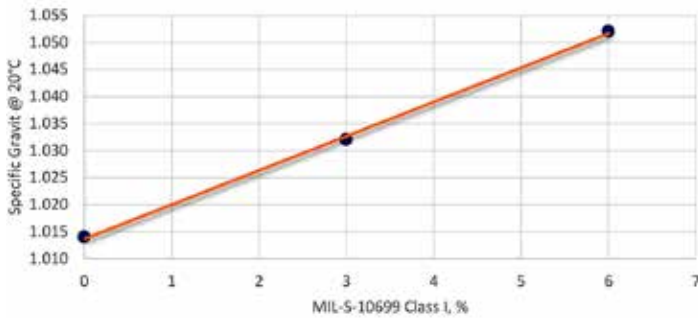


Figure 3: The variation of specific gravity of a water and 23 percent AMS 3025 Type I polymer quenchant, and varying concentrations of MIL-S-10699 Class I salt.

temperature, the polymer and water solution will separate, with the polymer now on the top of the bath, and water on the bottom. This reversal is due to the specific gravity differences between pure water and water containing the molten salt. This can result in an aluminum part being quenched through a layer of 100 percent polymer, and into the lower layer of water. This lower layer of water has a much faster quench rate due to the presence of salt. This will occur at approximately 8-12 percent salt content.

CORRECTIVE ACTION

From AMS 2770N [2], paragraph 3.4.9.4 Salt Contamination:

“Salt content in polymer/water quenchants shall not exceed 6.0 percent by weight. Water/polymer quenchants used with salt bath furnaces shall be tested for salt content weekly. The method used shall be calibrated against solutions containing known amounts of both polymer and salt, and the procedure documented. Meters used to determine the salt concentration shall be calibrated every 90 days. Quench tanks which exceed 6.0 percent salt content shall not be used until the salt content has been reduced below 6 percent or the quenchant has been replaced.”

There are two methods to determine the amount of salt present in a polymer quenchant bath. The first method is conductivity of the solution, and the other method is using specific gravity.

A conductivity meter is used, and consists of a probe and, usually, a handheld display. The probe is placed in the polymer solution, and the meter applies voltages between two electrodes inside the probe. The conductivity of a solution is measured in micro-Siemens or mS. The conductivity of the solution depends on the concentration of the ions present. Usually, some sort of temperature compensation is needed. Measuring at a constant temperature of 25°C provides consistent reading. Meters are usually calibrated using standards

available from the conductivity meter supplier.

As the salt content increases, the conductivity of the solution also increases. This is generally linear for salts associated with molten salt (KNO_3 and $NaNO_3$). This is shown in Figure 2.

At a conductivity of approximately 4000 mS, the salt content is approximately 6 percent, and time to perform corrective action.

Specific gravity can also be used to determine salt content of solutions. Specific gravity of a liquid is measured using a hydrometer. These simple devices, based on Archimedes’ principle, consist of a hollow tube that is weighted at the bottom, and a long stem that is graduated in specific gravity. To conduct the test, a graduated cylinder is filled with approximately 100 ml of polymer/water solution, and the hydrometer placed inside the graduated cylinder. The lower the specific gravity of the fluid, the deeper the hydrometer sinks. The specific gravity of the solution is read from the stem of the hydrometer. Hydrometers are available inexpensively from chemical supply houses.

The specific gravity of the polymer/water will increase linearly as the content of salt increases. This is shown in Figure 3.

Should the specific gravity of the solution exceed approximately 1.05, then corrective action is needed. This specific gravity value can change depending on the concentration of polymer present. However, for most installations, this value represents a typical number.

To eliminate the salt content, there are two things that can be done. The simplest is to dump and recharge the quench tank. However, this is not usually cost-effective. The second alternative is to thermally separate the water and polymer.

Performing a thermal separation of the water polymer solution requires heating the solution to above the cloud point, turning off the agitators, and allowing the polymer to separate from the water. Depending on the relative specific gravity (and salt content), the polymer may be on the top or bottom. The inorganic salts report to the water phase. The polymer can then be recovered by pumping it to totes or drums. The water containing the salt is then disposed. The polymer is reused, and new polymer added to the tank to meet the desired concentration. New water is then added. In very sophisticated operations, the water containing the salt is heated to boiling, and the water recovered using a retort. The salt deposits on the retort, and can then be completely reused.

CONCLUSION

In this article, the problems of excessive salt in a water/polymer solution were explained. Methods to measure salt concentration were detailed, and methods to recover the polymer from a high salt-containing solution were described.

Should there be any questions regarding this article, or suggestions for additional articles, please contact the author or the editor. ☞

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In a profession marked by techniques, technicalities – maybe even a little technobabble? – there is a room for creativity as well.

Developing a ‘C’reative culture in heat treat process

Hitchiner Manufacturing’s motto is, “imagination in metallurgy.” As a heat treater, there are certainly opportunities to use a bit of creativity in creating a process that yields a successful outcome in the customers’ requirements. In his book “Creativity: The Psychology of Discovery and Invention”, psychologist Mihaly Csikszentmihalyi defines creativity as “any act, idea or product that changes an existing domain or that transforms an existing domain into a new one.”

The heat-treat domain is the black and white customer specification — the pyrometry requirements of AMS2750, and metallurgical understanding of thermodynamics and kinetic principles, along with the people involved with all of the creation, maintenance, and implementation of these items.

And it can be seen from the customer’s basic drawing requirement of only mechanical properties that the heat treater must be creative in determining the methodology for heating up the furnace and fixturing the parts to minimize distortion in meeting such requirements.

But Csikszentmihalyi defines this sort of creativity with a lower case “c”. Not that this type of creativity isn’t valued. Instead, creativity with a capital “C” represents the impact of such novel ideas on the overall culture.

So how can we do this for the employees? How can a culture come alive with such a strong desire to see a process grow and find everyone engaged toward a successful heat treat of parts meeting customer demand? Where furnaces are almost singing when parts go in and out with effortlessness and ease? Where the timing of pyrometry testing such as SATs (system accuracy test) or even TUSs (temperature uniformity survey) don’t miss the Takt time of production, the steady heartbeat for moving parts through a given process? Where the environment does truly feel like imagination in metallurgy?

Csikszentmihalyi shows us a way to create such an environment through four components to be practiced every day, at every level of the organization.

I. PROMOTE CURIOSITY AND INTEREST

“Try to be surprised by something every day.”

For example, when we have an issue with the furnace — say, the ohm reading on the rear element is not reading right — our first reaction isn’t to get all frustrated and bent out of shape knowing we

will get scolded for missing production that day. Instead, allow this to challenge our thinking. Be surprised in the sense that something of opportunity can be taken from this. What part of the maintenance program can we improve upon?

“Try to surprise at least one person every day.”

This one can be tricky. Most people think the only thing employees look forward to most are their weekends, vacations, and their paychecks. Surprise them by remembering something they did last weekend as you work together on a particular heat-treat setup that takes a long time to build. Surprise them by showing up and being present, ready to go, and take on the day’s challenges. It doesn’t always have to be a gift card around the holidays or bonus after each year.



II. CREATION OF FLOW

“Wake up in the morning with a specific goal to look forward to.”

Work provides clear goals. With clear goals, the expectations are straightforward. However, goal setting can be challenging sometimes as the black and white numbers of heats to produce each day become monotonous. Shake it up sometimes by making small games throughout the day, by spending time building a cycle with the operators to liven up the energy. Shake it up by giving a special assignment during a heat-treat cycle to be on the watch for thermocouple behavior and atmosphere irregularities on special heat-treat cycles. Everyone wants to continually learn on some level.

“If you do anything well, it becomes enjoyable.”

These little goals start to add up. I have seen operators go from being students in the classroom of my training on heat-treat to being engineers or technicians by simple application of continuing to build on what they enjoy doing. Even operators who don't view their careers as technicians or engineers still thrive in knowing that what they do they can still be engaging every day. This mindset can be taught.

“To keep enjoying something, you need to increase its complexity.”

This engagement is achieved through adding a little complexity each time to a given task. For the engineer, give them a sense of design in the experiments. Build upon layers of more integration of awareness of customer specifications and metallurgical phenomena.

III. HABITS OF STRENGTH

“Take charge of your schedule.”

Scheduling is probably the most challenging thing I see at work with employees, even with well laid out goals. With expectations of production numbers, boss' expectations, and where a person is that day, empowering the employee to own what they are going to do comes with encouragement and building a desire to want to do it. Make it bite-size chunks toward achieving the main goal.

“Make time for reflection and relaxation.”

This is often best expressed when allowing time for proper reflection. We recently had a thermocouple failure and, before panicking when Operations was going to arrive screaming, we took a little bit of time

to relax and think about the situation as it was new and different. Coming from this place, we were then able to make the best decision that has now been integrated into the practice.

“Find out what you like and what you hate.”

This time of reflection is critical for the employee in that we begin to see what he or she does like or maybe even hates.

“Start doing more of what you love, less of what you hate.”

Recognizing these patterns, employees can now begin to embark down a path of growth.

IV. PROBLEM SOLVERS

“Constantly surprised.”

Problems arise on this path all the time. Often, we are surprised by them. But imagine seeing instead an opportunity for a new solution.

“Implement the solution.”

To be Creative — with a capital “C” — in the world of heat treat, we must continue to encourage the imagination of the employees with proper understanding of pyrometry, metallurgical, and customer-related requirements as they continue to solve the everyday challenges of heat treat. 🔥



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Tony Tenaglier is the heat treat process engineer at Hitchiner Manufacturing. He earned both a B.S. in material science engineering and an M.A. in psychology. You can contact Tenaglier at tony_tenaglier@hitchiner.com.

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*SOARING DEMAND FOR
PURIFIED
GRAPHITE
SPURS NEED FOR
HIGH-VOLUME
FURNACES*



Growth industries such as electric vehicles, battery energy storage systems, and next-generation semiconductors require higher-purity graphite.

By DEL WILLIAMS

The global demand for graphite is surging and expected to continue for decades, driven by the broad use of graphite for a range of products such as batteries for EV cars and energy storage systems, LEDs, solar equipment, high-performance semiconductors, and critical components in high-temperature furnaces.

In an ironic twist, the furnaces that produce high-purity graphite also require components manufactured from graphite and related materials such as fiber-reinforced carbon. Graphite's unique atomic structure gives it the ability to withstand extreme furnace temperatures in a corrosive environment, making it an ideal choice as a critical material in hot zones used in industrial furnaces.

"Graphite found in nature is crystalline in form; when extracted from a mine, it usually has about 90 percent carbon content," said Thomas Palamides, senior product and sales manager — Industrial Furnaces at PVA TePla America, a global supplier of custom industrial furnace equipment to the graphite industry. "Specialized high-temperature furnaces create synthetic graphite with approximately 99.5 percent carbon content. If the application requires higher purity, specialized equipment can reduce the impurities to the parts per million range."

For graphite suppliers, the growing demand for synthetic graphite with high-carbon content and federal government initiatives to restore U.S. domestic semiconductor production is driving a need for increased capacity electrical furnace systems that produce a greater payload in less time. Because the material purification process involves extreme temperatures and noxious gases, these industrial tools require highly specialized process controls and safety features.

RISING SILICON CARBIDE DEMAND

In various industries, there are many uses for silicon carbide, in the production of which graphite is an essential component.

The superior surface hardness of silicon carbide facilitates its use in engineering applications where a high degree of sliding, erosive and corrosive wear resistance is necessary for components. The most straightforward process to manufacture silicon carbide is to combine silica sand and carbon in a graphite electric resistance furnace at a temperature between 1,600°C and 2,500°C.

However, silicon carbide's use as a semiconductor material is one of the areas for the highest growth potential. The demand for graphite is growing substantially as silicon carbide replaces silicon as the semiconductor material of choice in many future-generation electronic products. Compared to traditional silicon wafers, silicon carbide is superior for higher voltage operation and provides significantly broader temperature ranges and increased switching frequencies.

Federal legislation to spur domestic semiconductor manufacturing and strengthen the supply chain will also increase demand for silicon carbide and graphite. The CHIPS and Science Act appropriated \$52.7 billion to fund semiconductor incentive programs authorized by the CHIPS for America Act of 2021.

In the semiconductor industry, one of the main drivers of graphite use is growing silicon carbide mono-crystals refined in various downstream processes. The crystal growth starts with a consumable silicon carbide powder as the source material. The powder evaporates when exposed to temperatures more than 2,000°C inside a crystal growth machine reactor. In the process, silicon and carbon molecules formed in the gas phase slowly crystallize on a very high-quality disc composed of silicon carbide.

Graphite is used in many other forms to allow equipment to withstand high temperatures, such as furnace linings, heat exchangers, foundry accessories, and electrodes. As such, the process occurs in a graphite crucible surrounded by graphite thermal insulation.

FURNACES CUSTOMIZED TO THE APPLICATION

In the industry, graphite suppliers often operate furnaces that are decades old and may be ready to extend capacity by replacing equipment or building new installations. Even among next-generation furnace options, there can be substantial differences in how the manufacturer addresses the issues of safety, reliability, configuration, and control in equipment design.

Of course, ensuring the safe, secure operation of industrial furnaces is a priority. Any fire risk resulting from the very high operating temperatures and the large masses or charge inside the furnace must be avoided by intelligent design solutions. Therefore, both hardware and software components of the system are properly engineered and equipped with redundant safety features, among other factors.

"Generally, about four metric tons of graphite material are loaded into a furnace reactor heated to 2,400°C, which is a huge amount of energy, so safety is essential," said Dr. Thomas Metzger, a PVA TePla senior product manager in Germany. "Abnormal conditions in the process or media must be considered in the system design to ensure the safety of the equipment operators. Whether there's a failure of cooling water, processed gas, compressed air, or electrical power, the system must be capable of returning to a safe state to protect the operators, equipment, payload, and environment."

Additionally, most furnaces are custom designed around the graphite suppliers' specific requirements, such as available space, loading method, and payload.

"When one customer created a new (furnace) workshop for a facility, he designed it with a pit for a bottom loading system, which facilitates loading the charge without lifting from a trolley on the ground floor," Metzger said. "With bottom load furnaces, the whole bottom of the vacuum chamber drops down to place the charge in, and then it is raised on an electrically powered system into the furnace."

Metzger noted other customers prefer top-loading systems, which can be loaded using an overhead crane.

"The top would slide to the side 180 degrees to open," he said. "A boom crane or overhead crane would be used to load the charge into the furnace from the top."

Selecting the process gases used is an important consideration,



The furnaces that produce high-purity graphite also require components manufactured from graphite and related materials such as fiber-reinforced carbon. (Courtesy: PVA TePla America)

and each manufacturer uses slightly different chemistry, or time, for their process.

“We typically utilize process gases like fluorine or chlorine,” Metzger said. “Although these are highly corrosive and dangerous at high concentrations, we have to be flexible to the specific requirements of the process.”

HIGH-TEMPERATURE PURIFICATION

The inner configuration of the industrial furnace is unique to high-temperature purification.

“We keep all the process gas inside the containment reactor, and outside we have an inert gas like argon or nitrogen,” Metzger said. “This configuration ensures high product purity and avoids contamination of the heating elements, insulation, and other vessel components. The reactive volume is kept completely separated from the heating elements.”

Generally, furnaces with diameters up to two meters can accommodate graphite-machined parts of different geometries, producing payloads of four metric tons or more.

“Typically, machined parts are placed in the furnaces for purification and then packed, sealed, and shipped after the items are processed,” Metzger said.

Customizing the system allows the accommodation of certain process conditions and installation requirements.

“Some customers like to have an additional dedicated pump for

handling corrosive process gases,” Metzger said. “Others prefer to use the same pump first to evacuate the system, before heating, and later for process pressure regulation.”

He added that design flexibility can extend to auxiliary equipment and the control system to best address environmental concerns.

“Graphite suppliers generally choose to include a scrubber system to clean exhaust gases,” Metzger said. “Corrosive gases used during processing are, for the most part, consumed in the process, but the process exhaust gases must meet the legal requirements for emission protection.”

He added that a technician can operate the furnace with a PC or use remote connections for monitoring. An overview screen displays the real-time status of all the pumps, valves, and critical components with alert indicators for problems and required maintenance.

“Today’s furnace systems are essentially turnkey with sufficient integration,” Metzger said.

To meet the rising global demand for refined high-purified graphite safely and reliably, proactive graphite suppliers that consult with experienced furnace suppliers to customize their systems to current and future market demand will have a lasting edge over the competition. ♣



ABOUT THE AUTHOR

Del Williams is a technical writer based in Torrance, California. For more information, go to www.pvateplaamerica.com.

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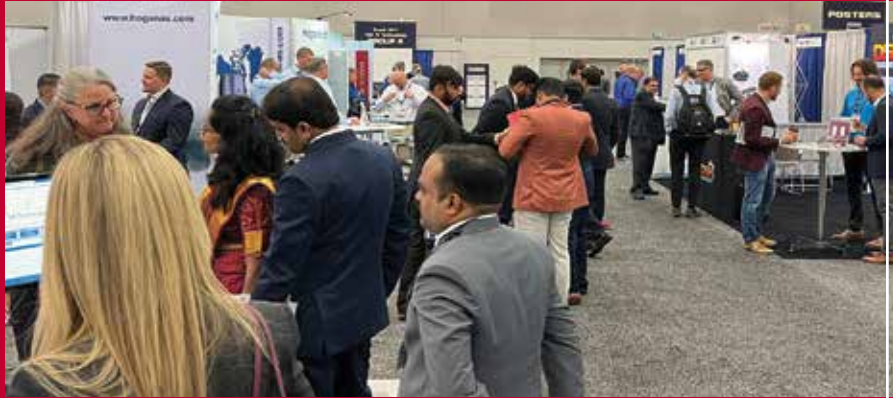
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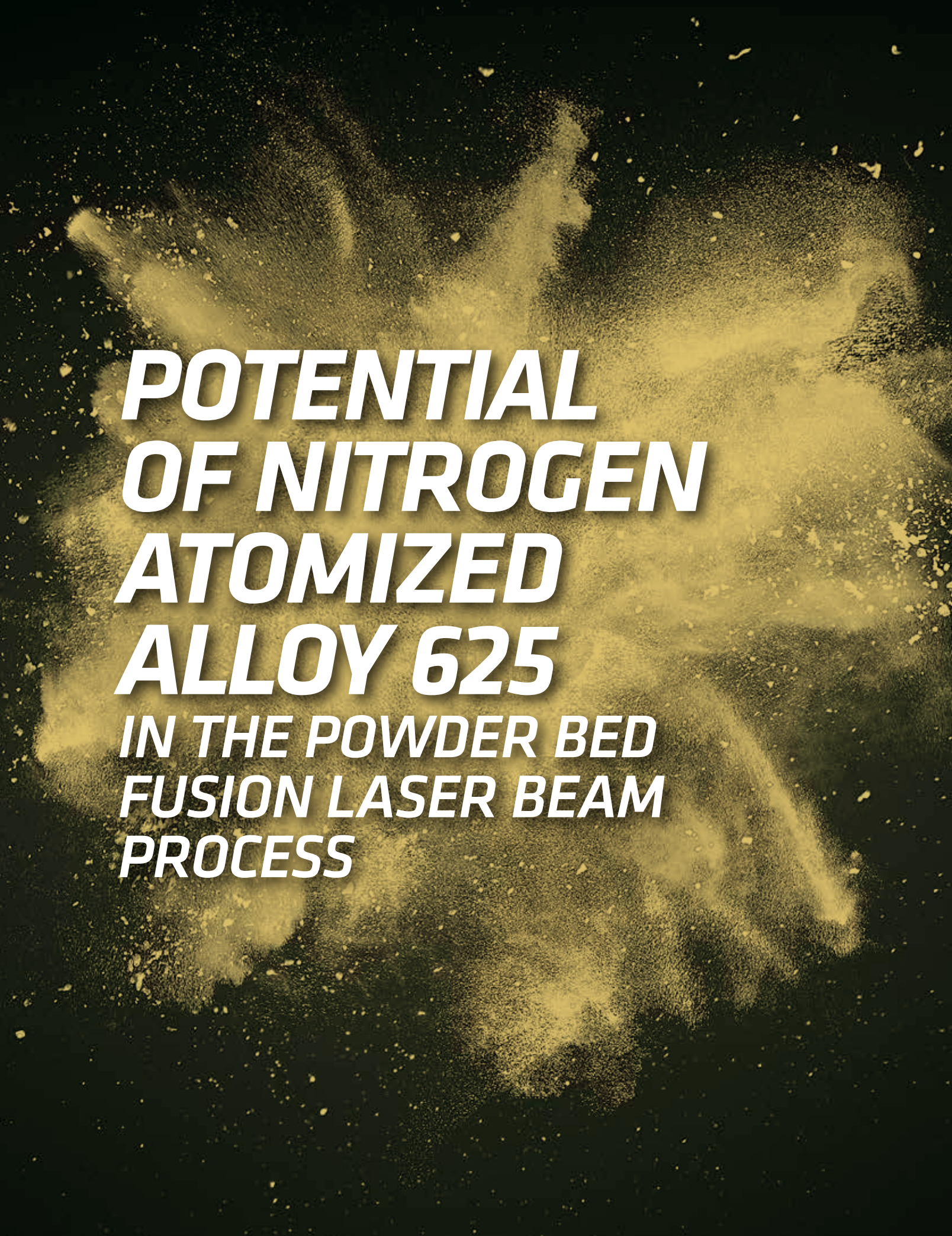
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The background of the image is a dark, almost black, space filled with a dense spray of fine yellow particles. The spray originates from the top and bottom edges, creating a central area of higher concentration. The particles vary in size and are scattered in all directions, giving the impression of a laser cutting through a powder bed or a high-speed spray. The overall effect is dynamic and industrial.

***POTENTIAL
OF NITROGEN
ATOMIZED
ALLOY 625***

***IN THE POWDER BED
FUSION LASER BEAM
PROCESS***

This study investigates the possibility of using nitrogen instead of argon during atomization of Alloy 625 powders intended for the PBF-LB process, which would be environmentally beneficial, but to do this, the 625 alloy must be tailored to limit deleterious effects stemming from the use of nitrogen gas.

By C.J. HASSILA, M. PASCHALIDOU, P. HARLIN, and U. WIKLUND

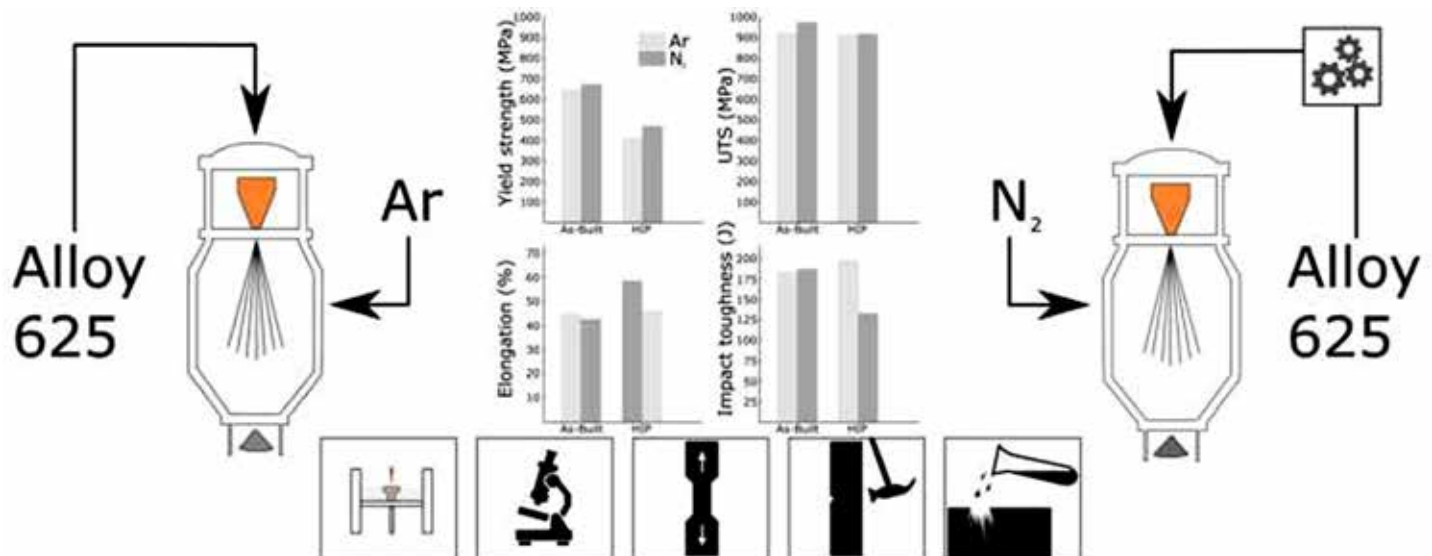
Powder-based metal additive manufacturing processes like powder bed fusion — laser beam use gas-atomized metal powders as feedstock material. Typically, for nickel-based alloys such as Alloy 625, argon gas is used during the atomization process. Considering the larger environmental impact of argon gas compared to nitrogen gas, and the increasing use of gas-atomized metal powders, the environmental impact of powder-based additive manufacturing techniques could be mitigated if gas atomization of alloys such as Alloy 625 using nitrogen was possible. This work investigates the feasibility of tailoring an alloy to allow atomization using nitrogen gas while remaining within the Alloy 625 specification. This is achieved by limiting the nitrogen pick-up during the atomization process, primarily by reducing the titanium content. The metallurgical implications of this tailored alloy and the subsequent atomization using nitrogen, as well as the attained microstructure from the powder bed fusion — laser beam process is investigated and compared to a more common 625 alloy composition, which was atomized using argon. Furthermore, the microstructural development of the alloys after heat treatments is evaluated. Lastly, corrosive properties, as well as tensile and impact properties, are evaluated both in the as-built and hot isostatic pressed condition.

1 INTRODUCTION

Alloy 625 is a Ni-based alloy, which, since its introduction in the 1960s,

has found widespread application in marine, nuclear, and chemical processing industries [1]. This is because the alloy possesses high strength and excellent corrosion resistance in a variety of environments, also in cryogenic and high-temperature applications. The resistance to corrosion is derived from the formation of a passivating Cr-oxide layer, similar to that of other alloys such as stainless steels, for example 316L. The maintained strength at extreme temperatures is mainly derived from the solution strengthening of the γ -Ni phase by additions of Mo and Nb.

Traditionally, Alloy 625 components have been cast or wrought, but with the increased capabilities of metal powder production, near net shape hot isostatic pressing (NNS HIP) has become a common manufacturing route [2]. Lately, additive manufacturing (AM) has become an additional manufacturing alternative thanks to the alloy's excellent weldability. Depending on the manufacturing technique, different microstructures will be achieved. Typically, casting produces coarser grains with larger grains located toward the core of the casting, while the NNS HIP process result in a much more isotropic material with small equiaxed grains throughout the component. Additive manufacturing, on the other hand, often results in a highly textured material with elongated grains oriented parallel to the build direction[3]. This texture is a consequence of the large thermal gradients present during the AM processes, resulting in an epitaxial growth. A consequence of this texture is anisotropic mechanical properties



Argon gas compared to nitrogen gas in the use of gas-atomized metal powders.

	Ni	Cr	Mo	Nb	Fe	Ti	Al	Si	C	Mn	N	NO
Alloy A	Bal.	21.10	8.80	3.80	1.10	0.18	0.14	0.07	0.01	0.01	0.003	0.004
Alloy N	Bal.	21.5	9.20	3.72	2.4	0.01	0.05	0.04	0.02	0.01	0.118	0.096

Table 1: The composition (wt%) of the alloys A and N after atomization, NO is the nitrogen content in the as-built material, evaluated using melt extraction.

which are commonly evaluated in tensile testing [4], [5], [6].

In the AM process known as powder bed fusion — laser beam (PBF-LB), a component is produced in a layer-by-layer process. At first, a thin layer of pre-alloyed metal powder is spread on top of a build plate. This layer is then selectively molten by the means of a focused laser beam, which is scanned over the areas that correspond to a cross section of the intended component. The process continues iteratively with spreading and melting of additional layers until a complete component has been produced. The relatively small volume of molten material at any given moment results in large thermal gradients, which promote rapid cooling and hence the pronounced texture previously mentioned. Furthermore, when the focused laser beam is scanned over the region that is to be molten, not only is the powder of that specific layer molten, but also the material directly beneath it. This is known as back-melt. Further below the scanned area, the material will not be remolten, instead it will be affected by a temporary temperature increase, forming what is known as a heat-affected zone (HAZ)[7]. Consequently, materials produced via PBF-LB have been subjected to both cyclic melting and cyclic heating, which has a direct influence on the resulting material texture, precipitation, and the segregation of elements. For some alloys, this cyclic melting and heating result in severe segregation and the precipitation of large amounts of brittle and incoherent phases[8]. These attributes are common for alloys less suitable for welding and might be challenging for additive manufacturing using the PBF-LB process.

The material feedstock for the PBF-LB process is gas atomized powder. The atomization process of Alloy 625 is conventionally performed using argon gas. The production of argon gas by fractional cryogenic distillation of air results in several byproducts, one of these being nitrogen gas[9]. Since air typically consists of 0.9% argon and almost 80% nitrogen, it is considerably more challenging to extract argon gas than nitrogen gas. Hence, production of nitrogen gas has a smaller environmental footprint compared to that of argon gas[10]. Consequently, replacing argon gas with nitrogen gas would reduce the environmental footprint of the atomizing process.

However, one element included in Alloy 625, titanium, has a high affinity for nitrogen and forms primary nitrides such as TiN. Such precipitates may affect the mechanical properties and render the material brittle and less resistant to fatigue[11]. Fortunately, the alloying specification of Alloy 625 with UNS designation N00625 is quite wide, especially regarding the amounts of Mo, Nb and Ti[12]. This makes it possible to tune the alloy to allow for higher nitrogen content by reducing the amount of Ti, possibly avoiding precipitation of primary nitrides.

An earlier study on nitrogen gas atomized Alloy 625 has shown that nitrogen gas atomized powders can result in a larger amount of prior particle boundaries in NNS HIPed components[13]. Also, attempts on tailoring the 625-alloy composition to better suit atomization using nitrogen gas has been undertaken by Rizzo[14], where the titanium content was reduced to limit TiN precipitation during atomization and NNS HIP processes. In that study, it was found that the higher nitrogen content had an insignificant impact on thermal stability, while the ductility in the resulting material was impaired. There are few examples of use of nitrogen gas atomized 625 powders in the PBF-LB process[15], and to the authors' knowledge, no comparison between argon and nitrogen gas atomized powers in said process.

This work investigates and compares two variants of pre-alloyed Alloy 625 powders atomized using argon and nitrogen, respectively, to determine if acceptable material performance can be attained while reducing the environmental footprint. The microstructure is characterized using SEM, and the mechanical properties are evaluated by tensile and impact testing as well as with indentation techniques. Lastly, the corrosive properties of the alloys are evaluated and compared. The alloys are investigated both in the as-built condition and after hot isostatic pressing (HIP). HIP was used both as a solution heat treatment and to reduce any porosity which might impact the mechanical properties.

2 EXPERIMENTAL

2.1 Alloying and atomization

All samples in this study were produced using Sandvik's Osprey® metal powders, which had been produced using vacuum induction melting with subsequent gas atomization with either argon or nitrogen gas. Impact, tensile, and corrosion test samples were manufactured using powders atomized from two different compositions of Alloy 625, henceforth denoted Alloy A (argon) and Alloy N (nitrogen). The alloying composition after gas atomization of alloy A and N is shown in Table 1. Also shown in Table 1 is the nitrogen content in the as-built material. Alloy A was atomized using argon gas and has a "typical" alloying composition with regards to titanium. Alloy N on the other hand was atomized using nitrogen gas and, therefore, has lower titanium content in order to avoid excessive precipitation of TiN during both the atomization and the PBF-LB processes. The produced powders A and N had the same particle size distribution (+15/-45 µm) and equally good processability, meaning that qualitatively, the powders behaved equally good when spreading the power layers. Consequently, the same process parameters for the PBF-LB process could be used for both alloys. The nitrogen content in the two alloys in the as-built material conditions was evaluated using melt extraction according to standard ASTM E1019.

2.2 PBF-LB

The investigated samples were produced in and EOS M100 PBF-LB system [16] equipped with a 200W Yb-fiber laser having a focus spot 45 µm in diameter. The build chamber was purged using high purity argon. The resulting protective atmosphere of argon was used throughout the entire PBF-LB process. All samples were produced using a layer thickness (d) of 20 µm and a 67-degree rotating scan strategy. This means that the scanning direction of each subsequent layer was rotated 67 degrees relative to the previous layer. A laser power (P) of 160 W and a scan speed (S) of 1,400 mm/s was used. In combination with a hatch distance (h) of 60 µm a resulting input energy density of 95 J/mm³ was calculated using Equation 1.

By the means of optical microscopy and image analysis on polished cross-sections of the produced material a density above 99.9% could be determined for both alloys. Sample dimensions for tensile and impact samples were Ø10×60mm and 10×10×60mm, respectively. All samples were built with the longest axis of symmetry parallel to the build direction.

$$E = \frac{P}{d * h * S} \quad \text{Equation 1}$$

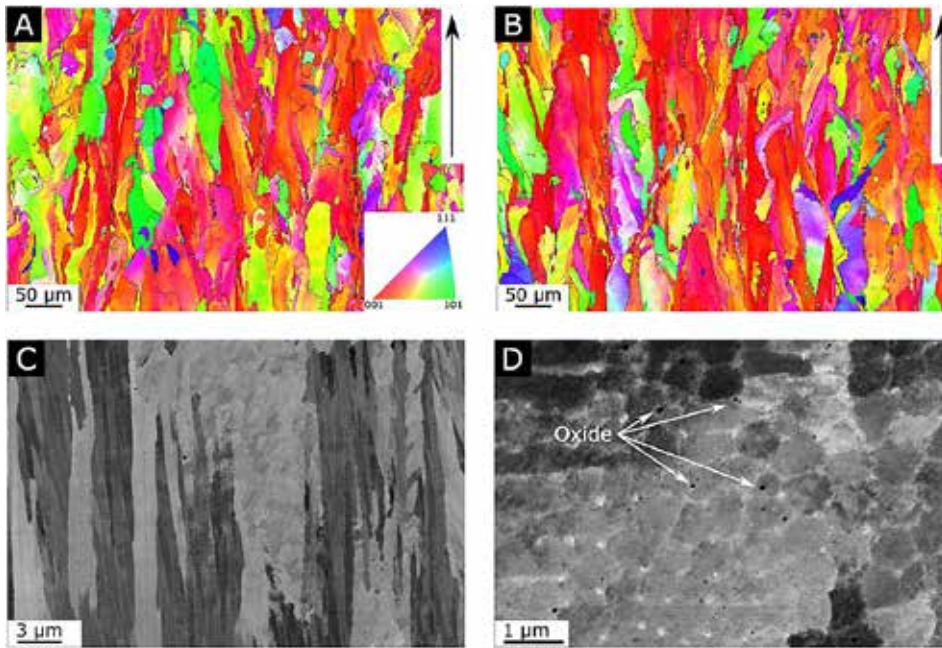


Figure 1: EBSD-IPF images showing the general grain structure and texture for alloy A (A) and alloy N (B), the build direction is indicated by the black arrow. SEM-BSD images at higher magnifications display a distinct columnar sub grain structure (C) as well as interdendritic regions (D).

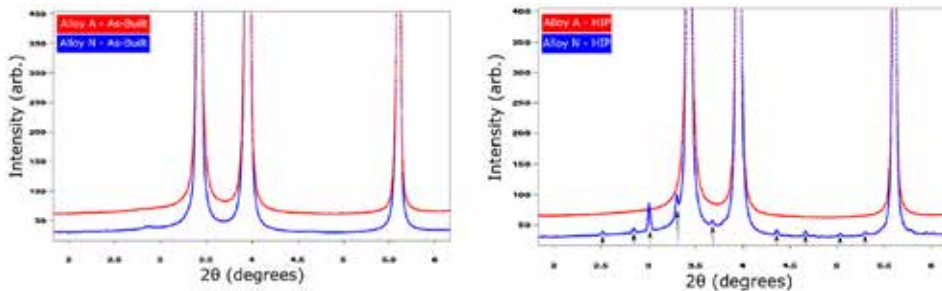


Figure 2: WAXS diffraction patterns for alloy A and N in the as-built condition (left) and the HIP condition (right). Arrows indicate reflections from NbCrN in alloy N in the HIP condition.

2.3 Sample preparation & mechanical testing

After the L-PBF process, half of the produced samples were heat treated using HIP where the samples were heated to 1,120°C at a pressure of 100 MPa for 2 hours. All tensile samples were then machined to ASTM E8M standard 4C20 rods and tested in an INSTRON 4505 using an initial strain rate of 2.5·10⁻⁴/s. A total of three samples per material condition was tested. To reduce the total test time for tensile testing, the strain rate was increased after material yield (at 1.7% elongation) to 5.0 × 10⁻³/s until fracture occurred. Impact samples were machined to full sized 10 × 10 mm Charpy V-notch specification and tested in a Zwick PSW 750 equipment in accordance with ISO-14556. The impact samples, built vertically, attained an impact plane with its normal parallel to the build direction. The mechanical properties of Alloy A and N were further investigated using 300 gf micro-Vickers indentation both in the as-built and HIP condition in order to evaluate hardness in the macro scale. In order to evaluate differences in hardness coupled to variations in microstructure, nanoindentations in a 10 × 10 matrix were performed using a Berkovich tip and 100 mN loads.

2.4 Corrosion tests

Prior to the electrochemical experiments, samples produced using alloy A in the as-built and HIP condition, as well as samples produced using alloy N in the as-built and HIP condition, were polished to a 1 μm diamond suspension finish and subsequently ultrasoni-

cally cleaned in acetone, ethanol, and, lastly, deionized water. An in-house three electrode cell was used, using each sample as a working electrode, an Ag/AgCl (3 M NaCl) (0.205 V vs. SHE) as reference electrode and a Pt wire as counter electrode. The cell was connected to a Metrohm Instruments Autolab PGSTAT302N potentiostat/galvanostat. A circular surface area of 0.2 cm² of the samples were exposed to the 0.5 M H₂SO₄ electrolyte, which had been degassed in N₂ for 60 minutes to remove any dissolved oxygen. All samples were left under open circuit potential (OCP) for 60 minutes, after which electrochemical impedance spectroscopy (EIS) measurements were performed under OCP. The EIS plots were recorded using a sinusoidal AC perturbation of 10 mV and a frequency range from 100 kHz to 100 mHz. Following that, polarization curves were recorded to display the corrosion resistance. To evaluate the significance of native oxides on the corrosion resistance, two different experimental approaches were selected when performing the polarization measurements, both using a scan rate of 1 mV/s. In the first approach, the starting potential was -0.2 V vs. OCP i.e., about -0.2 V vs Ag/AgCl, and the polarization curve was terminated at 1.1 V. For the second approach, the starting potential was -0.6 V vs. Ag/AgCl and scanned to 1.1 V. Also, for the second approach, the native oxide was first reduced by applying a stable potential of -0.9 V vs Ag/AgCl prior to the recording of the polarization curve. Corrosion potentials and corrosion current densities were determined by using graphical extrapolation involving anodic and cathodic currents close to the corrosion potential i.e., Tafel plots.

2.5 Material characterization

Microstructural and fractographic studies were conducted in a Zeiss Merlin Field Emission SEM equipped with EDS. WAXS diffraction using synchrotron light was performed at DESY P.21 using a wavelength of 0.1239 Å while the beam had a 0.6 × 0.6 mm² cross sectional area. A VAREX XRD4343CT detector was used, placed 1.75 meters behind the sample. The attained data was analyzed using the FullProf Suite.

3 RESULTS

3.1 Microstructural characteristics

3.1.1 As-built condition

The general microstructures in the as-built condition, shown in Figure 1a and 1b, exhibit elongated grains oriented parallel to the build direction, having an overall (1 0 0) texture-oriented parallel to the build direction. At the same time, grains with an apparent random orientation could be seen. Grains analysis, see Figure S1 in the supplementary material, shows the grains in the two alloys had a similar size distribution and were limited in length to a maximum of a few 100 μm. For both alloys, the grains were comprised of a sub-

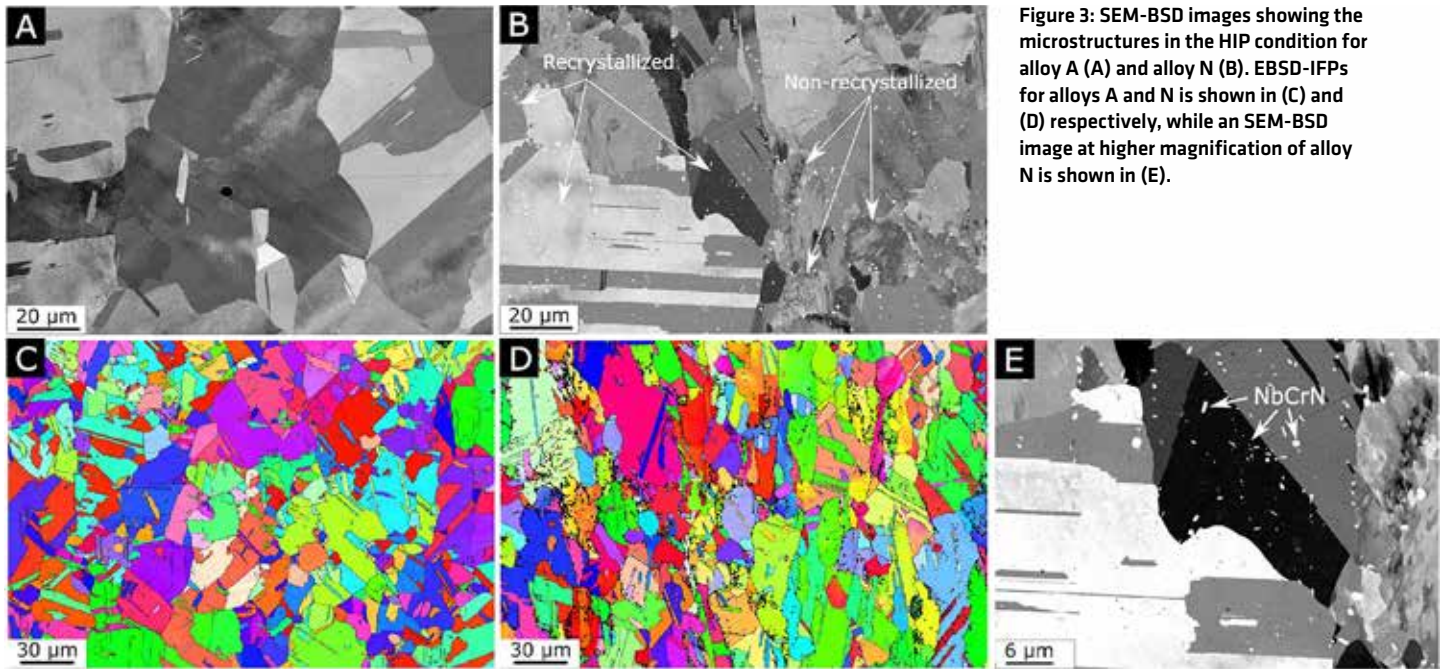


Figure 3: SEM-BSD images showing the microstructures in the HIP condition for alloy A (A) and alloy N (B). EBSD-IFPs for alloys A and N is shown in (C) and (D) respectively, while an SEM-BSD image at higher magnification of alloy N is shown in (E).

structure revealing the dendritic growth of the γ -Ni phase, see Figure 1c. Depending on the orientation of the grains, this substructure can appear either cellular or columnar for grains oriented either perpendicular to or parallel to the prepared sample surface. Segregation of larger elements towards the interdendritic regions, as well as evenly distributed oxide inclusions can clearly be seen in high resolution SEM-BSD image shown in Figure 1d. WAXS measurements on materials in the as-built condition showed no evidence for Laves phase for either of the two alloys, only peaks corresponding to the γ phase could be found, see Figure 2. In summary, no distinguishable differences could be found when comparing alloy A and N with regards to texture, segregation, or precipitates in the as-built condition. This is quite remarkable as chemical analysis using melt extraction reveal, as expected, that there was a considerably larger amount of nitrogen in alloy N compared with alloy A.

3.1.2 HIP condition

As can be seen in Figure 3, after the HIP heat treatment, a dramatic change in the microstructure can be seen. For alloy A shown in Figure 3a and c, the columnar grains seen in the as-built condition have undergone recrystallization, resulting in a microstructure with larger and more equiaxed grains and a substantial amount of twinning, and there are no precipitates present apart from the oxide inclusions previously observed in the as-built condition which are still in the sub-micron range and uniformly distributed within the grains. Furthermore, the recrystallized grains no longer exhibit the same cellular substructure as seen in grains in the as-built condition. When comparing alloy A with N in the HIP condition, again, a similar grain size distribution can be seen, see Figure S2 in the supplementary material. However, while all grains in alloy A have undergone complete recrystallization, some of the grain in alloy N appears to not have undergone such recrystallization process. As can be seen in Figure 3b and 3d, there are grains that appear to still display an elongated appearance with clear signs of a cellular substructure. Although, the morphology of these non-recrystallized grains is similar to grains seen in the as-built condition, they too appear to have been affected by the heat treatment to some extent. Also present in alloy N are unevenly distributed precipitates, clearly seen in Figure 3e. These precipitates are found in the

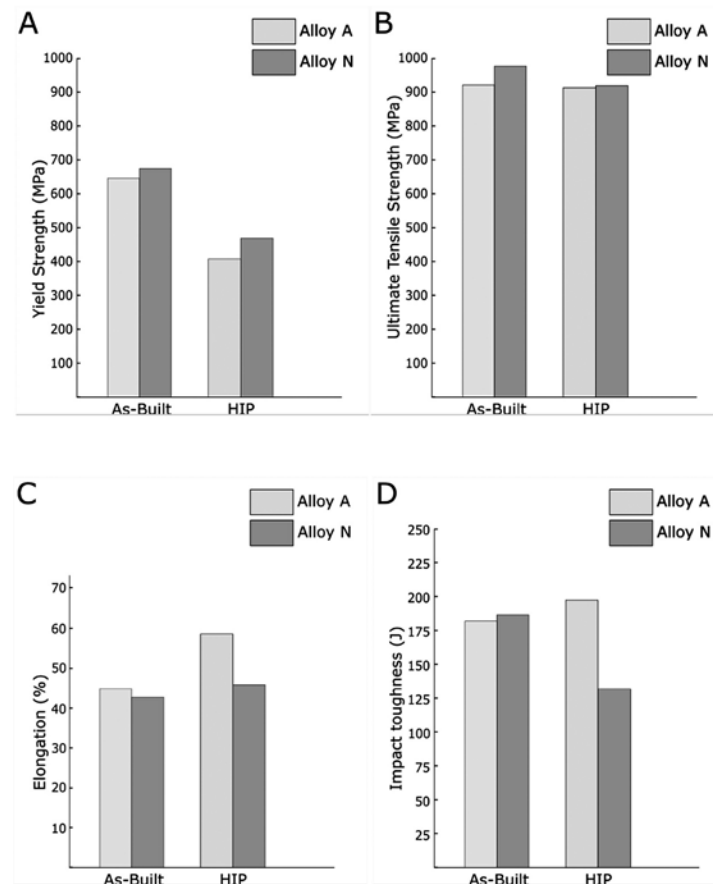


Figure 4: Tensile test and impact test results for the two alloys A and N in their respective material condition.

grain boundaries of grains that have not undergone recrystallization or within grains that have undergone recrystallization. The distribution of precipitates within the recrystallized grains seems to indicate previous grain boundaries, i.e. grain boundaries that were present in the as-built condition prior to recrystallization. The precipitates were identified as NbCrN using WAXS and EDS data, see Figure 2, Figure 3.

		As-Built		HIP	
		Alloy A	Alloy N	Alloy A	Alloy N
Yield Strength	MPa	664 (3)	694 (3)	421 (3)	483 (6)
Ultimate Tensile Strength	MPa	915 (3)	978 (1)	891 (1)	895 (4)
Elongation	%	45.8 (1)	43.6 (1)	59.8 (1)	46.8 (1)
Impact toughness	J	181 (7)	186 (7)	197 (2)	126 (3)

Table 2: Tensile and impact test results for the two alloys A and N in their respective material condition. Standard deviation is given in the brackets.

	Alloy A		Alloy N	
	As-Built	HIP	As-Built	HIP
Micro-Vickers (HV)	336 (10)	227 (8)	352 (12)	269 (15)
Nanoindentation (HV)	489 (20)	408 (16)	480 (11)	399 (38)*

*The values for alloy N are further displayed in Figure 6. Standard deviation is given in the brackets.

Table 3: Micro-Vickers and nanoindentation hardness (recalculated to Vickers) of the four specimens using either 300 g load or 100 nm indentation depth.

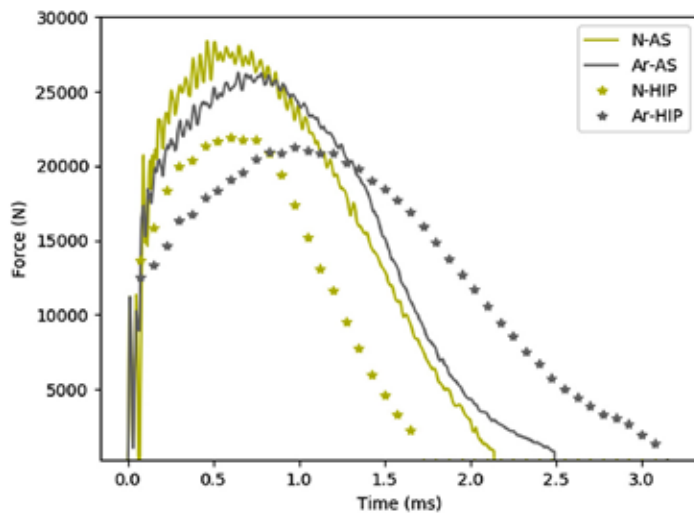


Figure 5: Force-time plot during impact for the two alloys in their respective material condition.

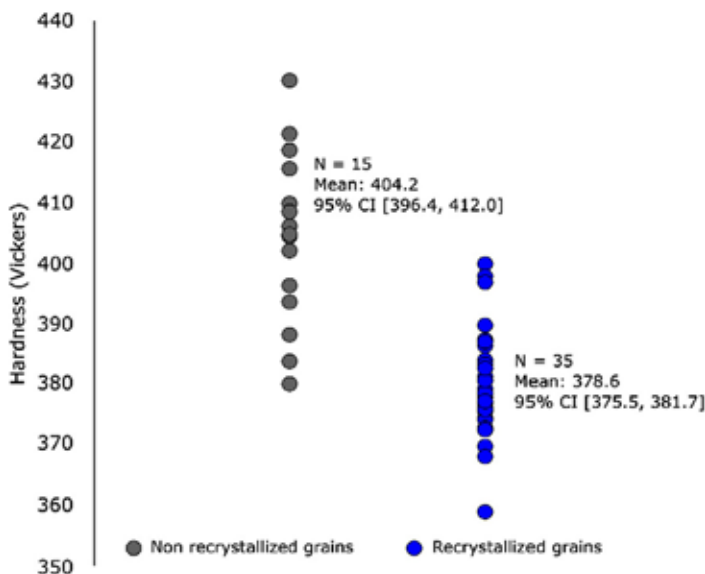


Figure 6: Hardness from individual indents (excluding 50 indents on or in the direct vicinity of precipitates) for alloy N, as separated using SEM into two groups; recrystallized and non-recrystallized grains.

3.2 Mechanical properties

3.2.1 Tensile and impact testing

Results from tensile tests shown in Figure 4 and Table 2 reveal there is a noticeable reduction of the yield stress after HIP heat treatment for both alloys. When comparing samples produced using alloy A and N with respect to material condition, a significant difference in yield stress can be seen. Both in the as-built and in the HIP condition, a higher yield stress was noted for alloy N samples. For both alloys, the HIP heat treatment had limited effect on the ultimate tensile strength. While the ultimate tensile strength was higher for alloy N in the as-built condition compared to that of alloy A, no difference in ultimate tensile strength could be noted between A and N after the HIP heat treatment. The elongation to failure was almost

indistinguishable between alloy A and N in the as-built condition. However, after HIP, the elongation to failure increased considerably for alloy A while alloy N remained unaffected.

When analyzing the impact results, no significant differences between the alloys in the as-built condition could be observed, see Figure 4d. To evaluate impact results more extensively, the force-time curves for the respective samples were compared with one another, see Figure 5. When analyzing the impact-toughness results in this way, the similarities between alloy A and N in the as-built condition were evident. However, after HIP heat treatment, a significantly larger amount of absorbed energy could be noted for alloy A when compared with alloy N.

3.2.2 Micro-Vickers and nanoindentation

The micro-Vickers hardness values were very similar for Alloy A and N in the as-built condition, and both alloys exhibited a reduced hardness after HIP heat treatment. However, in the HIP condition, a higher hardness could be observed for alloy N than for alloy A. When performing nanoindentation on the samples, see Table 3, the reduction in hardness post HIP was evident for both alloys, while a larger standard deviation was noted for alloy N. When differentiating between indents located within recrystallized and non-recrystallized grains for alloy N, and excluding indents located on, or in the direct vicinity of, precipitates or grain boundaries, a higher hardness was measured for non-recrystallized grains in alloy N, see Figure 6.

3.2.3 Fractographic study

SEM micrographs on fractured surfaces from impact tests display transgranular ductile fractures both in the as-built and HIP condition for both alloys A and N. The appearances of the fractured surfaces are overall similar when comparing alloy A with alloy N in the as-built condition. In the HIP condition, there is no significant difference in appearance of the fractured surface of alloy A. For alloy N, however, there are a significant number of unevenly distributed craters, at the bottom of which precipitates with high Nb content could be found, see Figure 7 and Figure S3 in the supplementary material for EDS map. These precipitates are likely the previously identified NbCrN, as this must be the most frequent precipitate found in alloy N in the HIP condition given the diffraction results shown in Figure 2. Several of the precipitates found in these craters are fractured, and the crater walls are typically covered with dislocation slip lines, indicating a high ductility of the surrounding matrix.

3.3 Corrosion

After immersion of the samples in the 0.5 M H_2SO_4 electrolyte for 60 minutes, the OCP values for all samples showed similar trends. At first, the OCP dropped after about 100 seconds, after which they tended to remain relatively stable without significant fluctuations. Only the sample produced using Alloy N in the as-built condition showed some fluctuations during the OCP measurements. During the last 1,000 seconds of the process, however, the OCP value became stable also for the Alloy N samples in the as-built condition, indicating a re-passivation of the surface. The OCP plots are included in the supplementary material, see Figure S4, while the attained OCP values after 60 minutes are presented in Table 4. The EIS data, measured under OCP, including Nyquist plots, see Figure S5a, feature straight lines close to the imaginary axis, while in the Bode phase plot, see Figure S5b, the phase angle was between 79.6 degrees to 80.5 degrees at the lowest frequency value (100 mHz).

The polarization curves, see Figure 8, differed between the two experimental approaches but showed no significant difference between samples tested using the same experimental conditions. For the first approach shown in Figure 8a, where any native oxides were still present, all samples displayed a corrosion current of about $4.5 \cdot 10^{-8} A/cm^2$ at a corrosion potential of $-0.03 V$ vs. $Ag/AgCl$, after which a current increase was observed when the potential became more positive than that. For the second approach, where native oxides were first reduced, the current value was about three orders of magnitude higher for all samples while the corrosion potential was about $-0.2 V$ vs. $Ag/AgCl$, see Figure 8b.

For the first approach, a passive region with a stable plateau ranging from 0.55 V to 0.9 V vs. $Ag/AgCl$ was observed for all samples, having a current of $1 \cdot 10^{-5} A/cm^2$, see Figure 8a. For the second approach, a similar plateau with currents at about $1 \cdot 10^{-5} A/cm^2$ was observed for all samples, ranging from $-0.16 V$ to 0.9 V vs. $Ag/AgCl$, see in Figure 8b. Both experimental conditions resulted in a shift from the passive to the transpassive region at about 0.9 V vs. $Ag/AgCl$.

SEM investigations of the corroded surfaces, representative images are shown in Figure 9, revealed for both alloy A and N, the typical cellular substructure of PBF-LB produced material in the as-built condition. The interdendritic regions, which are rich in Nb, see EDS mapping in Figure S6, appear bright and are indicated by an arrow in Figure 9a. For alloy N in the HIP condition, Nb-rich precipitates are visible on the corroded surface, very likely in the same areas were the NbCrN precipitates were seen. These too appear bright in contrast as indicated by arrows. Both Nb-rich precipitates and interdendritic regions in samples in the as-built condition protrude above other areas of the surface where corrosion had been more pronounced.

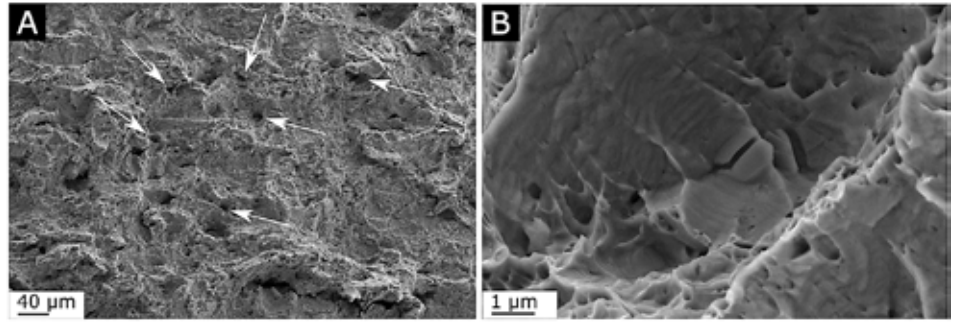


Figure 7: SEM micrographs on a fracture surface of alloy N, showing presence of craters (indicated by white arrows in A) and a NbCrN precipitate located in one of these craters (B).

Sample	Alloy A (As Built)	Alloy N (As Built)	Alloy A (HIP)	Alloy N (HIP)
OCP	0.1 V	0.08	0.08 V	0.07 V

Table 4: Stable OCP after 60 minutes in the electrolyte.

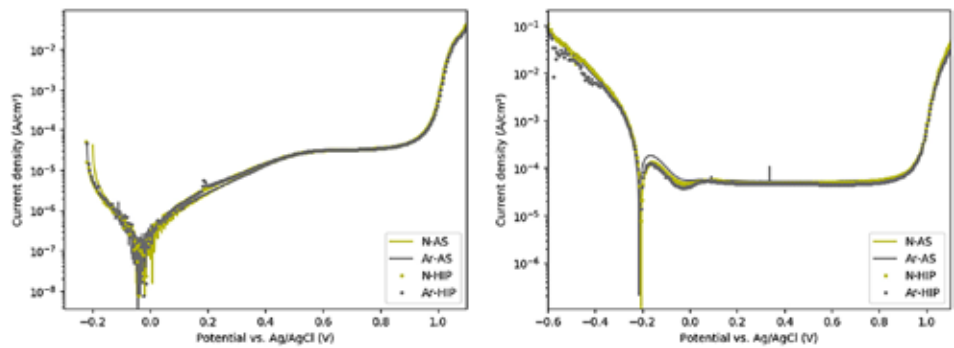


Figure 8: Potentiodynamic polarization curves for alloy A and N in both the as-built and HIP condition and for both experimental conditions. Starting potential $-0.2 V$ vs. OCP (left). Starting potential $-0.6 V$ vs. $Ag/AgCl$ (right).

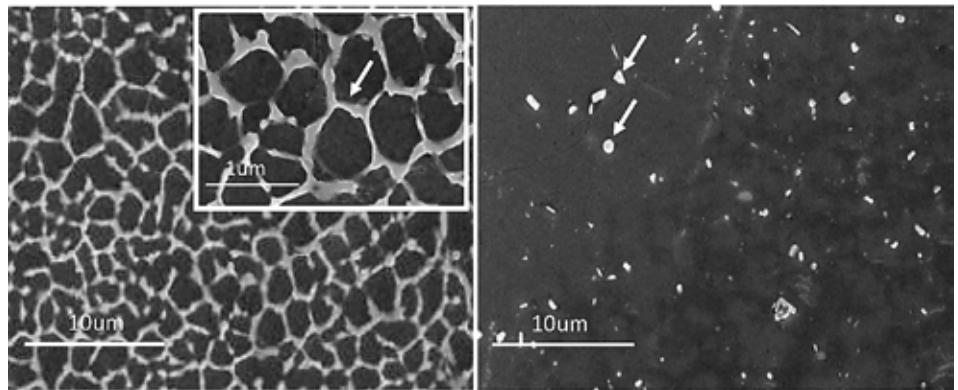


Figure 9: SEM of sample surfaces after polarization tests, with build direction parallel to image normal. (a) Alloy A in the as-built condition (but representative for both samples in the as-built condition). (b) Alloy N in the HIP condition.

4 DISCUSSION

4.1 Microstructure

4.1.1 Nitrogen

Even though the PBF-LB process is conducted in a protective argon atmosphere, there are still some presence of atmospheric gases i.e., oxygen and nitrogen, during the process. In the EOS M100 system, the process starts once the oxygen level is below 0.12%, and the value then quickly falls below 0.05% once the process starts. Although there is no nitrogen sensor in the system, given the fact that there is roughly 3.7 times more nitrogen than oxygen in air, one can predict the level of

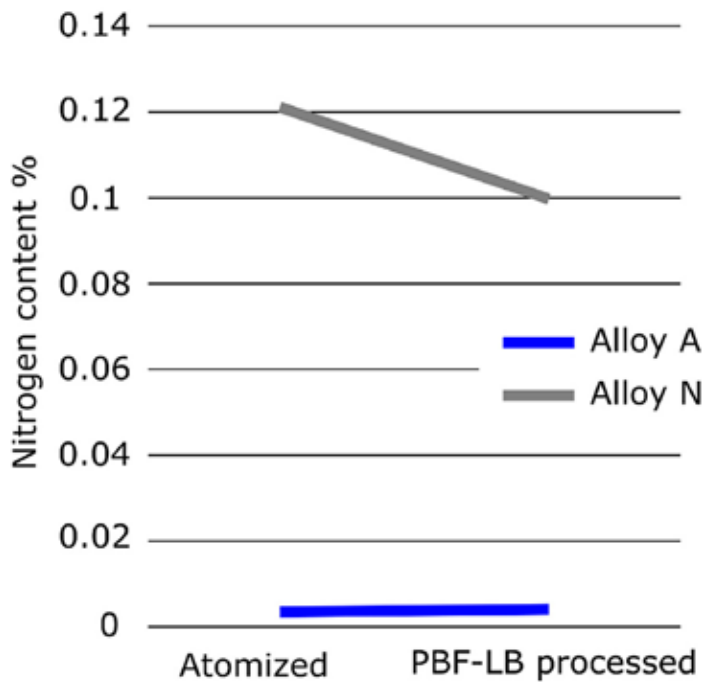


Figure 10: Nitrogen content before and after the PBF-LB process for the two alloys.

In the as-built condition, the microstructures of the two alloys are virtually impossible to distinguish from one another. Consequently, no significant differences were found in tensile, impact, hardness, or corrosion measurements.

nitrogen in the build chamber is close to 0.5% at the beginning of the process. In other words, nitrogen pick up is plausible during the printing process. When comparing the nitrogen content of the powders after atomization with the nitrogen content after printing in the as-built condition, see Figure 10 and Table 1, an increase of nitrogen can indeed be seen for alloy A. For alloy N however, a reduction of nitrogen was noted. This is most likely the result of nitrogen release during the melting of the powder. This would imply alloy N was supersaturated in nitrogen after the atomization process. Nevertheless, the reduction of nitrogen in alloy N is small compared to the overall difference in nitrogen content when comparing alloy A with N.

4.1.2 As-built microstructure

Both in the as-built and in the HIP conditions, oxide inclusions can clearly be seen uniformly distributed throughout the material for both alloys, see Figure 1d. The size and distribution of these inclusions are seemingly unaffected by the heat treatment. Furthermore, as these precipitates are in the same size range as those known to be present in the pre-alloyed powders [17], [18], [19], it is concluded that the PBF-LB process had no direct effect on these oxide inclusion. Given this, no drastic effect on other types of primary precipitates,

having a similarly high melting point is expected. One such type of primary precipitates is nitrides. In Alloy 625, titanium nitride (TiN) is of particular concern. Precipitation of TiN most likely occurs in both alloys in this study, but it is limited by the amount of titanium and nitrogen available in the two alloys. For alloy A, precipitation of TiN is limited by the low availability of nitrogen, while in alloy N, it is limited by the low availability of titanium. In this work, no presence of TiN could be confirmed when using SEM and WAXS for any material condition. This implies the amount of TiN present in either alloy is well below the detection limit for the utilized techniques, hence the tailoring of alloy N, as described earlier, was successful in limiting precipitation of primary nitrides.

Given the discussion above, only alloy A is expected to have free titanium present during the final stages of solidification in the PBF-LB process. Generally, for Alloy 625, titanium is added for its ability to participate in carbide formation and hence limit the amount of free carbon. If instead the titanium is allowed to form TiN precipitates, early onset of grain boundary precipitation of secondary M₂₃C₆ carbides during operating temperatures has been reported [20]. This is because as TiN precipitates, the available titanium in the alloy is reduced and limits the formation of titanium carbide. From this follows that more carbon will be left free in the material that may react with other elements such as Nb to form less stable carbides. In alloy N, the lack of free titanium could result in an excessive amount of secondary carbide formation, possibly also during use at high operational temperatures. Although not within the scope of the current work, the possible effect of the latter is addressed in the last section of this work.

Another important result is the excess of nitrogen that must be present in alloy N in the as-built condition i.e., nitrogen, which has not participated in the formation of primary nitrides. As there are no obvious primary nitride formers apart from titanium in the alloy, much of the remaining nitrogen must be dissolved into the solidified material. This excess nitrogen, given the solubility of nitrogen in Ni-based alloys, must be concentrated to grain boundaries [21].

4.1.3 Effects of the HIP heat treatment

After the HIP cycle used in this work, the microstructure changed dramatically compared to the as-built condition. For alloy A, complete recrystallization can be seen with a total absence of the cellular substructure seen in the as-built condition. For alloy N however, recrystallization has occurred, but there are still many grains present that exhibit the cellular substructure. Such grains are hence denoted as non-recrystallized grains, although they, too, have been affected by the heat treatment. This is evident since the interdendritic regions are much less defined than in the as-built condition as can be seen when comparing Figure 1c with Figure 3e. Also, a significantly lower hardness was measured for these grains compared to their as-built equivalents, see Figure 6 and Table 3.

Another major differentiating feature is that only alloy N in the HIP condition has the presence of 0.5–1.0 μm NbCrN precipitates; these are known as z-phase. Presence of z-phase is commonly observed in high Cr martensitic steels but scarcely reported for Alloy 625. As these z-phase precipitates are observed in either grain boundaries or within recrystallized grains, they must have been formed in grain boundaries already present in the as-built condition before any substantial recrystallization had taken place. This corresponds well with the increased amount of nitrogen in the grain boundaries described in the previous section. Most likely, the presence of these precipitates obstructed the recrystallization process, hindering a complete recrystallization of alloy N.

The purpose of the HIP cycle used in this work was to close any

remaining porosity in the PBF-LB produced samples while at the same time homogenizing the material. However, it should be noted other heat treatments, with or without pressure assistance, could be used to affect the microstructure in different ways, but this is left open for future studies.

4.2 Influence of microstructure on mechanical properties

The microstructural similarities and differences reported above are reflected in the mechanical properties of the alloys. In the as-built condition, the microstructures of the two alloys are virtually impossible to distinguish from one another. Consequently, no significant differences were found in tensile, impact, hardness, or corrosion measurements.

The HIP heat treatment strongly affects the microstructures of both alloys, and differences in mechanical properties emerged. More specifically, a reduction in yield stress and hardness was found for both alloys after HIP heat treatment, shown in Figure 4 and Table 3 respectively. These results, combined with the microstructural investigation, make it possible to conclude the decrease in hardness and yield stress is attributed to the recrystallization and grain growth in alloy A and a combination of recrystallization, grain growth, and recovery via dislocation annihilation processes in alloy N.

The grains in alloy N in the HIP condition, which have only undergone recovery, display a higher hardness compared to fully recrystallized grains, see Figure 6. The maintained hardness of these non-recrystallized grains likely contributes to the larger yield stresses measured for alloy N in the HIP condition while also the presence of z-phase precipitates contributed.

The difference in the amount of recrystallization in alloy A and N, and the presence of z-phase precipitates in alloy N is also the reason for the much lower increase in elongation to failure observed for alloy N post HIP compared to alloy A. A similar increase in elongation to failure is not observed for alloy N for two reasons. Firstly, unlike alloy A, alloy N has not achieved complete recrystallization, which results in a less ductile material. Secondly, the presence of z-phase precipitates inhibits plastic deformation, i.e. limits dislocation motion, resulting in an elongation to failure like that seen in the as-built condition. Surprisingly, despite the significant differences in microstructure before and after HIP heat treatment, no significant impact on the ultimate tensile strength was noted for the alloys. Hence the precipitation of z-phase in alloy N could not be correlated to any major change in the overall strength of the material.

However, results indicate that the z-phase precipitates significantly affected impact-toughness test results. While both alloys displayed an overall ductile fracture in both material conditions, the presence of z-phase precipitates in the HIP condition for alloy N allowed the formation of voids in the material. The formation of these voids reduced the ductility of the material, caused a decreased impact toughness and distinct craters on the fractured surfaces, see Figure 7b.

4.3 Corrosion

The results from the OCP measurements showed both alloys A and N in as-built and HIP conditions were able to reach similar OCP values after 60 minutes of immersion in the electrolyte. In general, the OCP is considered as a mixed potential, resulting from anodic and cathodic reactions, which are affected by any passive layer present on a sample surface. Hence, the stable OCP values measured indicate that surface passivation was possible for all samples. Also, the EIS results, see Figure S5a and S5b, indicate the corrosion resistances were very similar for all samples under OCP conditions. The passive layers formed on the sample surfaces resulted in an almost straight line in the Nyquist plots, indicating a “capacitance”-like behavior,

which is typically seen for corrosion-resistant materials. Moreover, the phase angle values in the Bode phase diagram range between 79.6 degrees to 80.5 degrees for the tested samples. These values are close to the 90 degrees seen for an ideal capacitive behavior, which confirms that the occurrence of corrosion reactions in the studied corrosive environment are limited.

The results from the polarization curves clearly show the used experimental approaches, i.e. different starting potentials, affect the shape of the polarization curves. However, no significant difference was seen among samples tested using the same starting potential. The distinctly different polarization curves attained for the two experimental approaches can be explained by the fact the native passive layer was not completely reduced in the first approach, as the starting potential was not sufficiently negative. According to thermodynamic data presented in the supplementary information, starting a polarization curve from 0.2 V vs. OCP, i.e. about 0.2 V vs Ag/AgCl, means the native oxide layer should not have been fully reduced prior to recording of the polarization curve. However, when applying a sufficiently negative potential on the samples, as in the second experimental approach, a large portion of the native oxides should have been removed. As the corrosion potential represents the potential where the rate of the anodic and cathodic reactions become equal, its value depends on how fast a passive layer can be reformed during a potential scan.

By employing the two different experimental approaches and the fact the corrosion current density was very similar for all samples tested with the same experimental conditions, it has been shown, using the first approach, that the native oxide layers on the sample's surfaces perform equally well, indicating corrosion resistant materials. Regarding the second approach, the similar corrosion behavior seen for all samples testify for a similar ability for all samples to reform the passivating layers on their surfaces.

5 CONCLUSIONS AND FUTURE OUTLOOKS

This study investigates the possibility of using nitrogen instead of argon during atomization of Alloy 625 powders intended for the PBF-LB process. This would be environmentally beneficial. But for this, the 625 alloy must be tailored (alloy N) to limit deleterious effects stemming from the use of nitrogen gas. The resulting material properties attained after the PBF-LB when using nitrogen gas atomized powders were compared to those of a more common alloy composition atomized using argon gas (alloy A).

» The two alloys Alloy A and N were virtually indistinguishable from one another both in terms of microstructure and corrosive properties when evaluated in the as-built condition. Also, their mechanical properties were similar, with Alloy A having only marginally higher elongation to failure than Alloy N. This makes alloy N a promising candidate for manufacturing using PBF-LB.

» After HIP heat treatment, complete recrystallization was seen for alloy A. Alloy N showed only partial recrystallization and presence of z-phase precipitates. As z-phase precipitates were found in grain boundaries of non-recrystallized grains, and within fully recrystallized grains, it is concluded they precipitate early during the HIP cycle in the grain boundaries of the as-built material where nitrogen content is high. Their presence then hinders the complete recrystallization of the Alloy N.

» Both alloys exhibited a decrease in hardness and yield stress after the HIP cycle. This is attributed to the recrystallization and grain growth in alloy A and a combination of recrystallization, grain growth and recovery in alloy N.

» After HIP, the elongation to failure was increased for alloy A thanks to complete recrystallization of the material. For Alloy N,

where recrystallization was limited due to z-phase precipitates, no change was seen.

» In impact testing, z-phase precipitates in Alloy N contributed to a lower impact toughness.

» Irrespectively of material condition, no difference between the two alloys could be noted in their corrosion resistance in 0.5 M H₂SO₄. Hence, the passivating performance of the native oxides is equally good for alloy both A and N and there is no difference in their ability to passivate their surfaces.

6 FINAL REMARKS

Although Alloy A outperformed Alloy N with regards to ductility after HIP, it should be noted the performance of Alloy N is still within specification. Furthermore, by adjusting the nitrogen content and by optimizing the heat treatment after the PBF-LB process, the presence and amount of z-phase should be possible to control. Such further development is motivated by environmental benefits that can be achieved.

It should also be noted the presence of z-phase precipitates also can affect other material properties. Although outside the scope of the current work, it is plausible the presence of z-phase in the grain boundaries of alloy N could limit grain-boundary sliding at elevated temperatures, which would imply an increased creep resistance. Yet another plausible effect from the presence of z-phase precipitates is reduced resistance to fatigue, due to their incoherency to the surrounding matrix and its brittle nature. Lastly, the impact of free carbon in Alloy N and the possibility of secondary carbide formation during use at high operating temperature should be addressed.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

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DATA AVAILABILITY

The raw data required to reproduce these findings cannot be shared at this time due to technical or time limitations. The processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

APPENDIX A. SUPPLEMENTARY DATA

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.matdes.2022.110928>. 📄

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*CARBURIZED STEEL
MECHANICAL PROPERTIES
**BENDING
ULTIMATE
AND IMPACT
STRENGTH,
TEST BARS VS.
ACTUAL PARTS***

In bending, the stress should be maximum at the surface and zero at the center of the cross section; based on hardness, carburizing is ideal because it places the high hardness and strength at the surface where it is needed most.

Editor's note » This is part two of a three-part series on carburized steel mechanical properties.

By **GREGORY FETT**

In part one, it was determined the carburized case was not as strong as the hardness would predict. How are carburized gears able to function as well as they do since in bending, the applied stress is greatest at the surface? Most of the information in this part and part three has been in the public domain since 1988, but it is timeless and will help to understand this question.

In the mid-1980s, there was an issue with low-impact life on a light truck rear axle during vehicle durability testing. The differential gears were failing after 3-5 cycles of a vehicle clutch dump test (rev engine up and slide foot off clutch). This was back at a time when the teeth on differential gears were still machined rather than net formed. The Revacycle machined gears were made from 8615 carburized steel with a case depth of about 0.89 mm and a core hardness of about 30-35 HRC. The failure mode was bending overload at the tooth root radius of the differential pinion.

STUDYING GRADES OF CARBURIZING STEEL

A study was initiated to look at different grades of carburizing steel and the effects of tempering temperature [1]. Unnotched Charpy bars were used because of a concern the properties of notched bars might be too low to show any meaningful difference. The data for this study is shown in Table 1. Tempering at elevated temperatures was evalu-

ated because it was being used to increase impact strength in some drag racing applications where gears were changed out every weekend and longevity was not an issue.

In bending, the stress should be maximum at the surface and zero at the center of the cross section. Based on hardness, carburizing is ideal because it places the high hardness and strength at the surface where it is needed most. As the tempering temperature increases, the surface is softened, which should decrease the strength. However, if we look at Figure 1, we can see the ultimate strength actually increases with increasing tempering temperature and reaches a maximum at about 316°C where the surface hardness has decreased from 66 HRC to about 52 HRC.

Likewise, the yield strength as determined by the Johnson Elastic Limit or JEL (50% change in slope) reaches a maximum at a tempering temperature of 260°C where the surface hardness has decreased to 59 HRC. This indicates the carburized case is not as strong as the hardness indicates. As we saw in part 1, it is suffering from a form of embrittlement called quench embrittlement [2]. The carburized case is not able to reach its full potential strength based on hardness because it fails in the elastic region before any significant plasticity occurs. This is not an anomaly, but rather it is normal for carburized components. It is a prime example of a brittle material that has been around for a long time and still functions very well in demanding applications.

Tempering Temperature versus Bending Properties of Carburized Unnotched Charpy Bars

Sample Number	Steel Grade	Tempering Temperature Degrees C	Hardness HRC Surface	Hardness HRC Core	Case Depth Visual mm	Case Depth Effective mm	Charpy Impact Energy Joules	Yield kN	Ultimate kN	Deflection mm
1	8615	As Quenched	66	36	0.89	1.02	16-20	19.6	30.2	0.86
2	8615	150	63-64	37	0.97	1.02	24-26	27.6	33.2	1.02
3	8615	205	59-61	35-36	0.91	1.02	26-30	27.6	35.1	1.07
4	8615	260	58-59	35-36	0.91	1.02	19-31	34.3	39.2	1.42
5	8615	315	55-56	36	0.84	1.02	43-56	32.0	42.9	1.45
6	8615	370	51-53	34	0.58	1.02	53-144	28.0	42.2	2.39
7	8615	425	48-49	32	0.36	1.02	175-231			
8	8615	480	45-46	29-30	1.02		264-302	23.6	35.1	5.08
9	8620	As Quenched	64-66	45	1.17	1.14	24-30	22.2	34.6	1.09
10	8620	150	62-65	45-46	0.91	1.14	34-39	32.9	37.4	1.09
11	8620	205	59-60	45-46	1.09	1.14	33-60	29.8	38.7	1.12
12	4320	As Quenched	64	46	1.40	1.52	26-28	26.7	34.3	1.17
13	4320	150	61-63	46	1.65	1.52	38-41	27.1	36.9	1.14
14	4320	205	58-59	46-47	1.40	1.52	43-47	30.2	38.4	1.17
15	8617	150	60-61	38	0.99	0.91	22-45	28.9	36.1	1.12
16	4815	150	58	42-43	1.22	0.91	53-79			
17	4820	150	58	40-41	0.89	0.86	58-68	28.0	37.0	1.40

Bars 1-14 Carburized in One Batch, Bars 15-17 Carburized in Second Batch, All Bars Direct Quenched in Cold Oil

Table 1

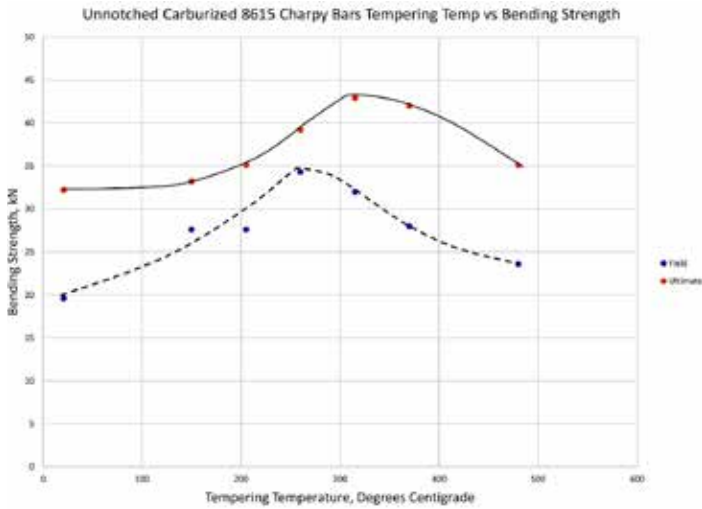


Figure 1

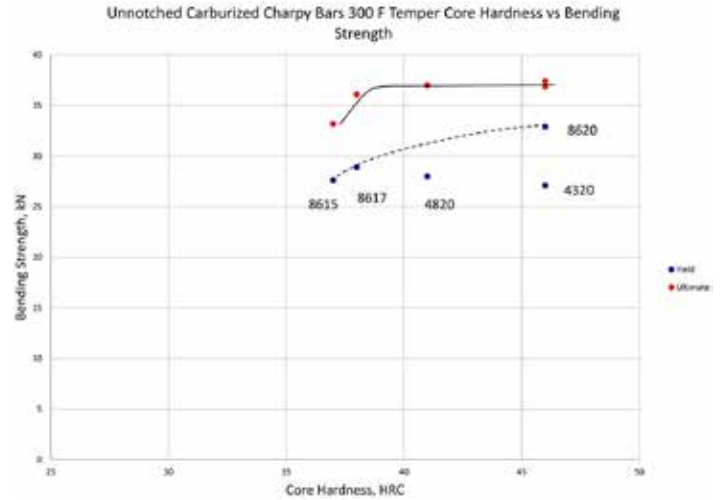


Figure 2

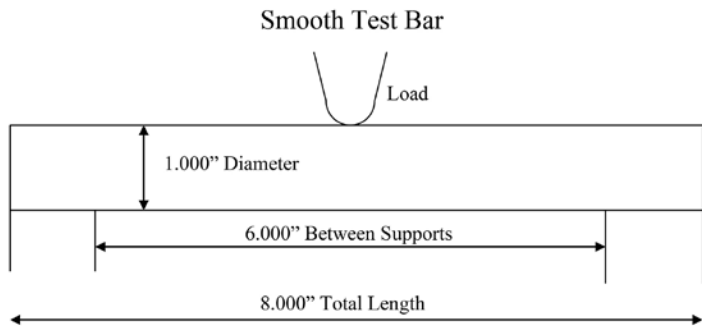


Figure 3

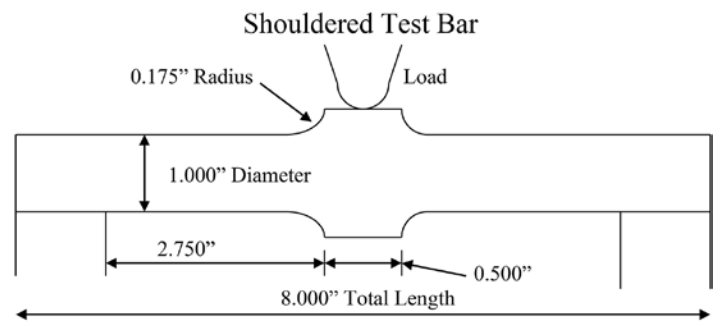


Figure 4

EMBRITTLMENT ISSUE

Tempering at an elevated temperature will certainly address the embrittlement issue to a degree; however, wear and fatigue life will then become major concerns.

Quench embrittlement does not normally occur with hardened lower carbon steels. However, a similar issue due to temper embrittlement was documented on a medium carbon 1038 induction hardened shaft tempered on the same scanning equipment used for hardening [3]. Elevated temperature tempering after the fact had the same effect of increasing the bending strength with decreasing hardness. With furnace tempered non-embrittled shafts, the bending strength and ductility remained constant up through 300°C. The embrittled shafts exhibited an intergranular fracture at the origin, while the normal furnace tempered shafts also exhibited intergranular fracture but with a small amount of dimple rupture fracture.

INCREASING IMPACT STRENGTH

The Unnotched Charpy bar data in Table 1 indicated 4320 steel should be a better choice for increasing the impact strength of the differential gear compared to 8615. The 4320 tempered at 150°C showed an increase in ultimate strength from 33.2 kN to 36.9 kN, and the impact energy increased from 24-26 J to 38-41 J. The test bar data also showed the increase in core hardness with 4320 should not be an issue. Figure 2 shows increased core should increase bending strength. Gears were made with 4320 steel and the truck impact test was rerun, and the life decreased to only 1-2 cycles. The test bar data did not appear to agree with the actual parts.

The main difference between the test bars and actual parts was the parts had a root radius to increase stress while the test bars did not. As a result, a new series of one-inch-round test bars with six

inches between supports was designed. There was a smooth test bar without a radius, which is shown in Figure 3 and shouldered bar with a radius shown in Figure 4.

EVALUATING CORE HARDNESS

To evaluate the effect of core hardness, the carbon content and resulting hardenability was changed within the 8600 series low nickel family (8615, 8620, 8625, 8630, 8640), and the hardenability was changed using alloy content within the medium nickel family (4620 and 4320). These bars were tested under slow bend, drop tower impact, and fatigue conditions. The slow bend results for the smooth bar are shown in Figure 5 [4].

The smooth bar results were similar to the unnotched Charpy data in that higher core hardness appears to provide higher strength. The spread between bending yield, as determined by the JEL, and bending ultimate decreases as the core hardness increases. The medium nickel steels 4320 and 4620 and the high nickel steel 9310 appear to provide a significant improvement over 8600 series low nickel steels. Once again, this is not in agreement with the actual differential gears.

The shouldered test bar results are shown in Figure 6 [5]. With the shouldered bar, there is an increase in bending yield and ultimate strength with increasing core hardness but only to about 30 HRC where the yield remains constant and the ultimate strength decreases to meet the yield strength at about 40 HRC. This potentially explains why the 4320 differential gears with a core hardness of 42-45 HRC performed worse than the 8615 gears with a core hardness of 30-35 HRC. The data does indicate the rules that govern carburized parts in bending are significantly different depending on whether a radius or stress concentration is present or not. A smooth bar without a stress

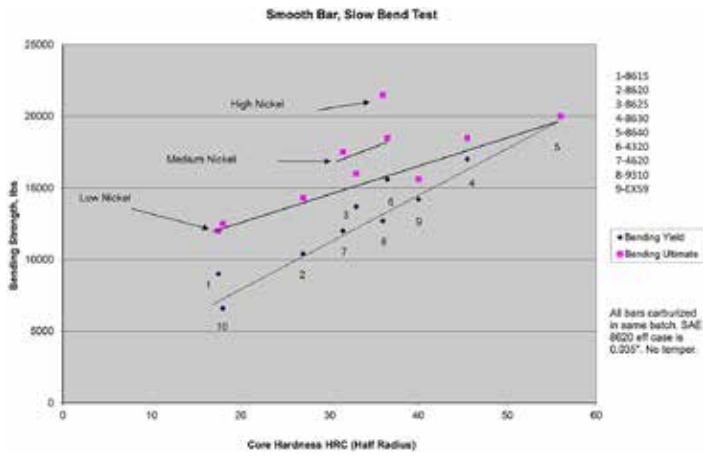


Figure 5

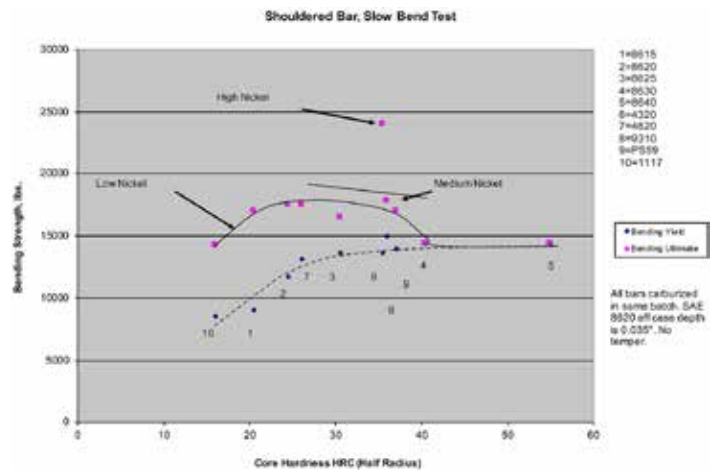


Figure 6

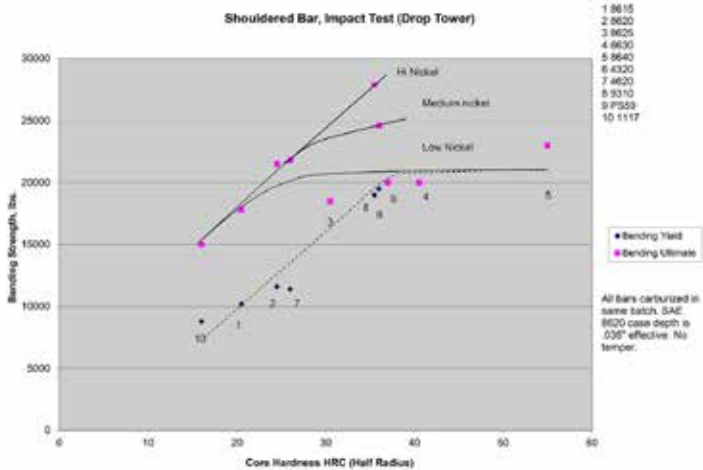


Figure 7

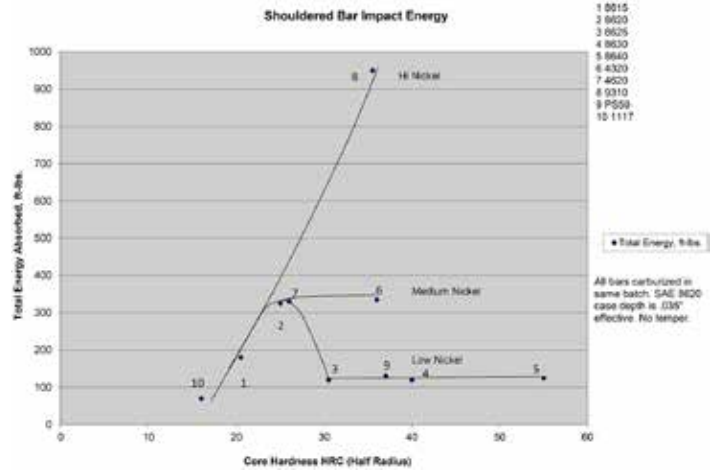


Figure 8

concentration will not provide the correct information for a gear tooth. Higher core hardness is beneficial for the smooth bar, and it is detrimental for the shouldered bar. With the shouldered test bar, the optimum core hardness for low nickel steels and possibly medium nickel steels is about 30-35 HRC. It provides the highest yield and ultimate strength. Below this hardness, the yield and ultimate strength will decrease, and above it, the ultimate strength will decrease to meet the yield strength.

IMPACT STRENGTH DATA

Figure 7 shows the shouldered bar impact strength data from the instrumented drop tower test [5]. Like the slow bend test, the strength increases with increasing core hardness but only to a point. The yield and ultimate strength also become the same at about 40 HRC. Unlike the slow bend data, 4320 does show an improvement over the 8615 steel. However, the 4320 core hardness is considerably lower than the actual gear due to the one-inch cross section of the test bar. Another notable difference from the slow bend test is the bending strength is higher under impact conditions.

Figure 8 shows the shouldered bar absorbed impact energy from the drop tower test [5]. The energy initially increases with increasing core hardness to about 350 ft-lbs at 25 HRC. The energy then decreases for the low nickel steels to 120 ft-lbs at 30 HRC remains level above that. The energy for the medium nickel 4620 and 4320 steels remains level at 350 ft-lbs at 25-35 HRC. The high nickel 9310 steel has the highest energy at about 950 ft-lbs at 35 HRC. This data also indicates there should be an improvement in impact life for the 4320 steel over

the 8615 steel. However, due to the test bars cross section, the core hardness values are lower than the actual gears.

In part three, a smaller test bar that duplicates the core hardness of the actual gear will be discussed, as well as the effect of case depth. ☞

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Gregory Fett retired from Dana Corporation in 2016 where he was chief materials engineer for nearly 35 years. He has done considerable research and authored numerous publications in the areas of carburized steels and induction hardened steels. He currently is a materials engineering consultant at Fett Engineering LLC. For more information, contact him at fetteng@gmail.com.

COMPANY PROFILE ///

BUSCH VACUUM SOLUTIONS



LEADING VACUUM TECHNOLOGY

Busch Vacuum Solutions has a team of applications engineers and sales staff who are experts in vacuum technology.
(Courtesy: Busch Vacuum Solutions)

Busch Vacuum Solutions offers leading edge vacuum and overpressure technology to a variety of industries around the globe.

By **KENNETH CARTER**, Thermal Processing editor

Not to be content with being a global leader for industrial vacuum technology, Busch Vacuum Solutions has constantly branched out in the industrial sector since its founding in 1963.

“Busch has begun to offer a more comprehensive range of vacuum services, such as leak detection, remote condition monitoring of pumps, and full vacuum furnace maintenance and reconditioning,” said Tom Burke, vice president of business development at Busch. “With our recent acquisition of VESCO in East Windsor, Connecticut, we can now offer full vacuum furnace service, hot zone repair and replacement, controls upgrades, leak detection service, and of course vacuum pump service and upgrades.”

ADVANCED SYSTEM BUILDING

With its state-of-the-art facility in Virginia Beach, Virginia, Busch Vacuum Solutions boasts advanced system-building capabilities and cutting-edge vacuum pump manufacturing.

“For the heat-treating industry, we supply a wide range of vacuum pumps, gauges, hardware, engineered systems, and leak detector solutions to OEMs and end users,” Burke said.

But the company really excels in custom solutions, according to Burke.

“We specialize in engineered solutions,” he said. “I would say that is where Busch really stands out. We have experienced substantial growth with our customized engineered solutions. These systems can range from small laboratory pumping systems with filtration to massive systems up to several hundred thousand cubic feet per minute used for steel degassing.”

DRY VACUUM PUMPING TECH

“Dry vacuum pumping technology is becoming increasingly accepted in the heat-treat industry,” Burke said. “In my over 43 years of experience in the vacuum industry, I’ve seen all of the different types of dry pumping technology and configurations, and I feel that, by far, the COBRA is the best product on the market.”

COBRA dry screw vacuum pumps can be used in industrial applications that require reliable and contaminant-free extraction of gases and vapors. The operating principle is based on screw technology, which involves two screw rotors that rotate in opposite directions. The pumped medium is trapped between the cylinder and the screw chambers where it is compressed and transported to the outlet. During this process, the screw rotors do not come in contact with each other or the cylinder.

By offering products that are designed to be more environmentally friendly, Burke said Busch is always looking for ways to give

its customers what they need and expect.

“The Busch philosophy is that our customers have always been the focus of the business, but we also focus on our employees, the environment, and the community,” he said. “And within the heat-treat industry, we’re passionate about our solutions for even the toughest applications. We’ll take on any, and all.”



Busch offers a wide range of vacuum pumps, gauges, hardware, engineered systems, and leak detector solutions to OEMs and end users. (Courtesy: Busch Vacuum Solutions)

EXPERT TEAM

Accomplishing this takes a dedicated staff, according to Burke.

“We have a team of applications engineers and sales staff who are experts in vacuum technology,” he said. “Our experienced industry-leading staff utilizes the most advanced simulation tools and then produces a customized system that ensures maximum performance.”

Their dedication and knowledge serves as a testament to the



“We have a team of applications engineers and sales staff who are experts in vacuum technology. Our experienced industry-leading staff utilizes the most advanced simulation tools and then produces a customized system that ensures maximum performance.”



COBRA's operating principle is based on screw technology, which involves two screw rotors that rotate in opposite directions. (Courtesy: Busch Vacuum Solutions)

Busch team's ability to step up and identify the correct solution, according to Burke.

"It's pretty much just tackling any problem, whether it's a tough application or expanding our reach of service by adding service centers," he said. "For our customers, downtime is typically not acceptable, so we are prepared with many service options to keep them up and running."

EXPANSION

And with its recent acquisition of VESCO, Burke said it only serves to expand the expert services Busch has to offer in the heat-treating industry.

"We wanted to look at this industry holistically, and this seemed like a great way to offer more than just a vacuum pumping solution, but a solution for the entire system," he said.

VESCO was formerly part of the McLaughlin Furnace Group, a U.S. market leader in engineered vacuum solutions for thermal processing and surface preparation applications. MFG's major customers operate in the aerospace and defense, automotive, energy and environment, metalworking and fabrication, and semiconductor fabrication industries.

With the addition of VESCO, Busch Vacuum Solutions was able to expand its business by adding complementary services to its already broad offering of global field service, overhaul, repair, and installation.

The company was founded in 1963 by Dr. Carl Busch and his

wife, Ayhan, according to Burke, and has seen a lot of expansion over the years.

"Busch remains a family-run business at the forefront of industrial vacuum technology serving customers in industries such as chemical, plastics, food, steel, and semiconductor production with almost 70 companies serving customers in over 40 countries," he said. "We have established ourselves as an international leader. As far as our U.S. presence, our headquarters is in Virginia Beach and has a capacity of over 200,000 square feet with close to 500 employees."

MARKET LEADER

A 60-year-old base of expertise and a strong commitment to its customers has kept Busch going strong and will only help to serve the company as it continues offering the best expertise to the heat-treat industry and beyond.

"We've had great success recently in the steel degassing industry by offering some of the largest customized vacuum systems solutions in the world," Burke said. "The way I see the industry progressing is that customers will be looking for who's the most responsive supplier and who offers the widest range of solutions. We want to be that leader in the metallurgical market, and we will continue to look for creative ways to achieve that goal." ♪



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COBRA dry screw vacuum pumps can be used in industrial applications that require reliable and contaminant-free extraction of gases and vapors. (Courtesy: Busch Vacuum Solutions)

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TOM HART /// PRODUCT MANAGER – VACUUM FURNACES /// SECO/VACUUM

“Pit LPC can be used with many applications, particularly with very large, bulky parts or with many small parts.”

What is Pit LPC, and what went into its development?

Pit LPC, or low-pressure carburizing, is an alternate method of low-pressure carburizing, and it's conducted in a vacuum furnace. The desire to go to the pit-style construction is that, in a typical horizontal configuration, it's hard to load a horizontal furnace with heavy loading. Subsequently, a pit-furnace design vertically loaded is much easier to produce, and material handling is easier.

With typical pit-style components, they're large, and large components oftentimes require deep-case steps. To do a deep-case step takes numerous hours. If you're, for example, trying to do a case step of a 1/8" or 3 mm, you could be in a typical atmosphere furnace for 24 hours. That's the time it takes the endothermic atmosphere, which is the carbon gas, to diffuse into the surface. With low-pressure carburizing, the diffusion can happen much faster, because in a vacuum-style furnace, you can run that furnace at much higher temperatures. The materials within the furnace can withstand those environments.

The atmosphere-design furnaces are limited in their temperature ratings, and if you run them at higher temperatures, they're going to fail on you sooner. Whereas a Pit low pressure carburizing furnace utilizes materials that will respond well when open to the air environment when you're transferring a load from the furnace to the oil quench tank, for example. The real benefits are that you can run that Pit low-pressure carburizing furnace at much higher temperatures, which now drastically reduces your carburizing cycle time because of the kinetics and the rate at which the carbon diffusion into the steel.

What are some of the other benefits of Pit LPC over traditional atmosphere carburizing?

It's much safer and environmentally friendly. Being able to reduce those cycle times and getting higher throughput are major advantages. With sustainability being predominant in today's industry, people are paying a lot more attention to safer, environmentally friendly pieces of equipment. You can walk up to a vacuum furnace and put your hand right on it. That's how they're constructed. You couldn't dream of doing that with an atmosphere's tight design. Vacuum furnaces provide a better environment for your employees and operators and, therefore, become a lot more desirable to work around, in addition to the other benefits associated with the process.

Could you go into more detail about the sustainability advantages for the Pit LPC?

You're going to utilize much less process consumables, i.e. your carbon gas. You're not required to use an endothermic generator that uses a methanol or acetone-type gas carrier. You're not emitting carbon dioxide and carbon monoxide, so you're getting that benefit as well. We don't emit those carbon gases to the environment.

What are some of the potential applications for Pit LPC?

Pit LPC can be used with many applications, particularly with very large, bulky parts or with many small parts. It will lend itself to wind turbines, power generation, heavy mining, heavy machines, railway, mining, oil and gas markets — basically anything that's going to have a large gearing component or a large bearing component. Because the Pit LPC furnace houses a large working volume, it is optimized for large components and heavy loading capabilities. Those types of markets that use those larger gearing components will benefit greatly from this type of technology.

In what ways is Pit LPC more economical than the traditional method?

As I mentioned before, the process time can be drastically reduced due to the elevated processing temperature, so you're not heating the parts as long, so you're able to get that faster throughput. You have the benefits of utilizing just the amount of carbon gas that you need. We put in just what you need to cover all the surface areas that you would like to have carburized. We're putting in low-pressure, very small amounts of carbon (measured in lpm), rather than filling up the furnace with the carbon potential of a traditional atmosphere furnace (measured in CFH). You're not doing that with low-pressure carburizing. We put in just enough, then we stop, let it diffuse, then we'll insert some more, let it diffuse in, and as that diffusion happens, you're getting your case depth.

What's been the industry response so far?

So far, it's good. It's newer to the industry, so it's off and running now. We've got a number of customers that are engaged in it. It's going to really help users get better returns on their investment by being able to heat-treat faster. With low-pressure carburizing, the case uniformity is much better than an atmospheric type. It's because the carbon goes right to the surface, and it diffuses uniformly throughout the entire geometry of the part, so you don't get these instances where you can have a light case and a deep case in various areas of your component.

Anything else you'd like to mention?

Our company, SECO/VACUUM, which is a SECO/WARWICK Group company, not only manufactures this type of equipment, but we manufacture various other vacuum and atmospheric equipment, so if anybody needs solutions to their heat-treatment issues or troubles, they can contact us. We can lend our expertise and help them through their needs and solutions. 📞

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