

Technologies and Processes for the Advancement of Materials

Thermal processing

ISSUE FOCUS ///

CERAMICS / INDUSTRIAL GASES

HEAT WORK TO THE FORE

COMPANY PROFILE ///

Metal Treating Institute



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HEAT WORK TO THE FORE

The sixth annual Ceramics Expo 2020 tackles the burning issues.

SPECTROSCOPIC STUDY OF PLASMA NITROCARBURIZING PROCESSES WITH AN INDUSTRIAL-SCALE CARBON ACTIVE SCREEN

In this study, the absolute concentrations of CH_4 , C_2H_2 , HCN , CO , and NH_3 were monitored in dependence on the total gas flow of the feed gas and the gas pressure in the reactor.



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COMPANY PROFILE ///

ENHANCING THE IMAGE OF HEAT TREATERS

MTI strives to fulfill its mission of enhancing the image and value of the heat-treating industry through advocacy, training, and business intelligence.

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New Products, Trends, Services & Developments



- Gasbarre has orders for three batch steam treaters.
- AHT now Nadcap® accredited for gas and ion nitriding.
- AMPM2020 technical program now available.

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MICHAEL HAGER
BUSINESS UNIT MANAGER ///
VERDER SCIENTIFIC/CARBOLITE GERO



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Industrial Heating Equipment Association (IHEA)



In this section, the national trade association representing the major segments of the industrial heat processing equipment industry shares news of the organization's activities, upcoming educational events, and key developments in the industry. **14**

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The effects of partial decarburization on the residual stress profile of a through hardened M50 and a case carburized M50NiL bearing ring. **16**

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Many of the issues in induction hardening can be directly related to the amount of contamination in the quench system, and improper or inadequate cleaning. **18**

QUALITY COUNTS ///

Once having no intention to work in quality, spending time cross-training in other departments quickly ignited a desire to help improve that part of the business, and work to find solutions to complex problems. **20**

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FROM THE EDITOR ///



Keep alert and stay healthy!

That being said, the April issue of *Thermal Processing* is still here to keep the window open on the world of heat treating.

Our main focus article concerns Ceramics Expo. Even though the show has been postponed four months, it will still boast the same schedule it had planned for May. In our cover article, an official with the show details what will be on tap come September. They've got an incredible show planned with some amazing programs running throughout the event. If you couldn't go in May, then definitely shoot for a trip to Cleveland in September.

Industrial gases are also a part of our April issue, and *Thermal Processing* is excited to present an article that looks at how different gases such as CH₄, C₂H₂, HCN, CO, and NH₃ were monitored during a spectroscopic study of plasma nitrocarburizing processes.

In addition to those articles, I had the pleasure of speaking with Tom Morrison, CEO of MTI Management, for our company profile. The Metal Treating Institute has been around since 1933, and for years, the organization has been responsible for the Furnace North America trade show. That show, still scheduled for the end of September, serves as the perfect opportunity to discover a massive amount of information about heat treating. Check out the article for what attendees can expect, as well as a look at the history of MTI. Morrison also takes a look in his crystal ball and predicts what could be around the corner for the industry in the decade to come.

And as always, this month's issue has a wealth of information from our regular columnists, so make sure you check them out as well. I am always learning something new from them.

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.

In the meantime, practice social distancing, and, as always, thanks for reading!

KENNETH CARTER, EDITOR

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Gasbarre's equipment is designed by experienced engineers and metallurgists who understand customer needs.

Gasbarre has orders for three batch steam treaters

Gasbarre Thermal Processing Systems recently received three separate orders for batch steam treating equipment. The batch steam treaters produce an oxide layer that promotes corrosion and wear resistance properties and provides an attractive surface finish. The three unique orders range in size from 18" to 30" in diameter and 12" to 48" deep. The gross load weight capacity ranges from 300 pounds to 1,800 pounds with Gasbarre supplying the production tooling. The equipment is electrically heated and has a maximum operating temperature rating of 1,400°F. Steam-treating processes are used in many different industries. As such, these

orders will be shipped to companies that provide products to the medical, additive manufacturing, automotive and consumer products industries.

With locations in Plymouth, Michigan; Cranston, Rhode Island; and St. Marys, Pennsylvania; Gasbarre Thermal Processing Systems has been designing, manufacturing, and servicing a full line of industrial thermal processing equipment for nearly 50 years. Gasbarre's product offering includes both batch and continuous heat-processing equipment and specializes in temper, tip up, mesh belt, box, car bottom, pit, and vacuum furnaces as well as a full line of replacement parts and auxiliary equipment which consists of atmosphere generators, quench tanks, and charge cars.

MORE INFO www.gasbarre.com

AHT now Nadcap® accredited for gas and ion nitriding

Advanced Heat Treat Corp., a recognized leader in heat-treat services and metallurgical solutions, announced that it has added gas nitriding to its Nadcap® accreditation. Advanced Heat Treat Corp. has held Nadcap® accreditation for ion nitriding since 2013.

President Mikel Woods said, "We are very excited to add gas nitriding to our growing list of Nadcap® accreditations. Our Nadcap Merit Status in both ion and now gas nitriding displays our ongoing dedication to quality and growth within the aerospace market."

Having demonstrated its ongoing commitment to quality by satisfying customer requirements and industry specifications, the Nadcap Task Group determined that Advanced Heat Treat Corp. has earned special recognition. This means that, instead of having its next Nadcap audit in 12 months, Advanced Heat Treat Corp. has been granted an accreditation that lasts until April 30, 2022.

"Nadcap accreditation is universally acknowledged as a significant undertaking. Validating compliance to industry standards, best practices and customer requirements, Nadcap has long been incorporated by the aerospace industry into their risk mitigation activity. Some companies, such as Advanced Heat Treat Corp., go even further achieving Nadcap accreditation to obtain Merit status and they should be proud of it," said Michael J. Hayward, executive vice president and chief operating officer at the Performance Review Institute®.

The Nadcap Heat Treating accreditation was achieved at its MidPort Boulevard location in Waterloo, Iowa. The company also has locations in Cullman, Alabama; Monroe, Michigan; and another on Burton Avenue in Waterloo, Iowa.

MORE INFO www.ahtcorp.com



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.

AMPM2020 technical program available ahead of conference

The program for the Additive Manufacturing with Powder Metallurgy Conference (AMPM2020) is now available. Metal additive manufacturing (AM) is one of the powder metallurgy (PM) industry's fastest growing fields. Join the metal AM industry in Montréal, Canada, June 27-July 1, 2020 at the Montréal Convention Center for the leading North American metal AM conference. Explore the latest technological developments, meet with industry experts, and discover the inherent capabilities of metal AM.

AMPM2020 attendees will also have access to two co-located conferences: The World Congress on Powder Metallurgy & Particulate Materials (WorldPM2020) and the International Conference on Tungsten, Refractory & Hardmaterials (Tungsten2020).

Conference attendees can expect:

› More than 140 sessions and nearly 500 technical presentations between the three co-located conferences.

› The world's largest trade exhibit featuring

more than 150 exhibitors of PM; metal AM; and tungsten, refractory, and hardmaterials equipment, powder suppliers, and component manufacturers.

› Networking with more than 1,200 attendees from around the world.

› Even more time in the trade exhibit – this year's conference features two networking luncheons adjacent to the trade exhibit.

› An optional metal AM tutorial focusing on basic information and guidance on the use of metal powders, including safety, handling, and storage to maximize the quality of the powders and subsequent performance of the consolidated parts.

Montréal is North America's No. 1 host city for international events," said James P. Adams, executive director/CEO, Metal Powder Industries Federation. "Combined with the region's strong technology center, delegates should leave Montréal with new ideas, industry contacts, and lasting memories."

There are two co-located conferences:

› Held once every six years in North America, World Congress on Powder Metallurgy & Particulate Materials (WorldPM2020) is the largest global hub of PM. Delve into materials, material characteristics, powder production, and more.

› The International Conference on Tungsten, Refractory & Hardmaterials (Tungsten2020) was last held in 2014. The conference addresses recent developments in the tungsten, refractory, and hardmaterials field encompassing processing, microstructure, properties, and applications. Multiple sessions will be held on 3D-printing specific topics.

At press time, this show's date had not changed.

MORE INFO AMPM2020.org

Larson Forgings celebrates 125th anniversary

Larson Forgings, the leading manufacturer of open die and rolled ring forgings, is celebrating its 125th anniversary of providing quality forged products.

The company was founded in 1895 by Charles E. Larson, who began hammering out horseshoes and small tools from his blacksmith shop near the Chicago River. Today, Larson Forgings is a global industry resource that has helped build aerospace,



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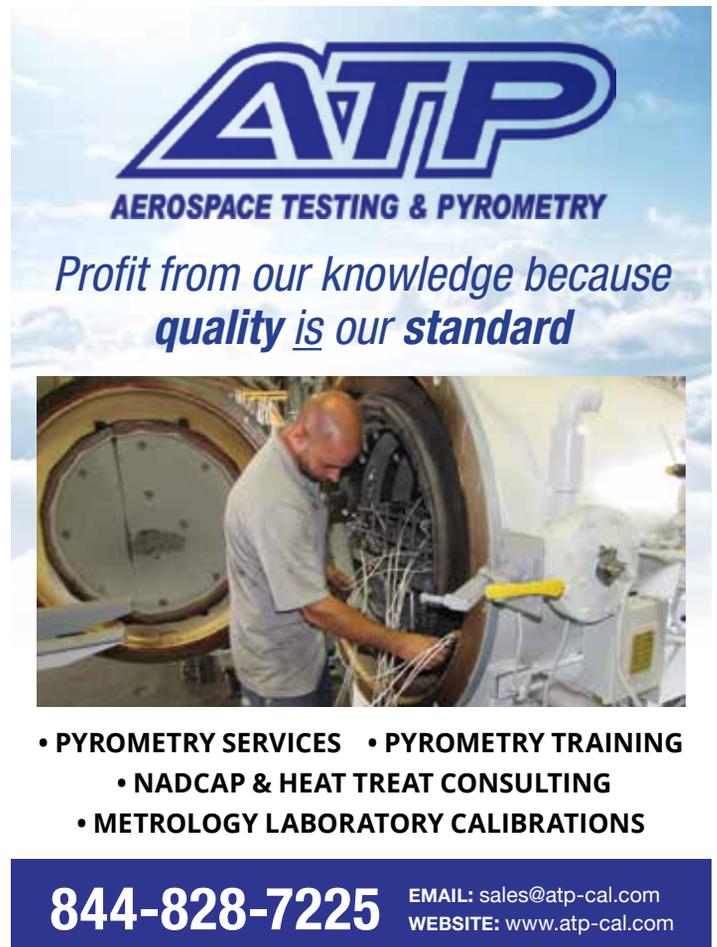
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Above: Larson Forgings' workers pose near a power hammer in the early 1900s; founder Charles E. Larson is the man holding a paper in his hand. (Courtesy: Larson Forgings)

Right: Larson Forgings' employees in 2019. (Courtesy: Larson Forgings)



construction, mining, nuclear, oil and gas, pharmaceutical, and many other industries.

"Larson Forgings' dedication to quality, continuous improvement and high levels of customer service has defined our company throughout its history," said Keith Zawrazky, president of Larson Forgings. "No other company has such a committed and highly skilled team. With our strong team and dedication, we are poised to continue to shape the needs of industry worldwide for another century."

"Flowserve has relied on Larson Forgings expertise since 1909," said Lee Holtz, procurement specialist, Flowserve. "Larson's unmatched quality and delivery are critical to our success."

Providing specialized technical expertise and problem solving along with customized sizes, shapes, and quantities of open die and rolled ring forgings, Larson Forgings

has a rich history of setting quality benchmarks and exceeding customer expectations. The company is AS9100 certified, National Aerospace and Defense Contractors Accreditation Program (NADCAP) accredited, and America Society of Mechanical Engineers NCA-3800 Quality System certified (nuclear material).

MORE INFO www.larsonforge.com

ASME offers reduced partial-year rate for new members

Join ASME by May 30 and enjoy access to all of its valuable member benefits at a reduced partial-year rate, plus receive a free gift.

ASME's membership year runs from October to September. This reduced partial year rate offer is for membership through September 30, 2020.

ASME offers the right information and resources to succeed, as well as delivering high-quality technical and career benefits including:

- › Free subscription to *Mechanical Engineering* magazine.

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MORE INFO go.asme.org/MembershipNow

Abbott gets order for stainless steel brazing furnace

Abbott Furnace Company reports that a diversified global automotive supplier, focused on metal forming, aluminum casting, fluid systems, and flexible assemblies to help automakers meet their lightweight requirements, has placed an order with the company for a continuous belt stainless steel brazing furnace to be installed in Mexico in the second quarter of 2020. Abbott Furnace will design, manufacture and install the five-zone electrically heated industrial furnace that is rated for 2,150°F and includes a 30-inch wide belt, silicon carbide muffle, and will feature Abbott Furnace's Varicool convective cooling system.

Abbott Furnace is an industrial furnace manufacturer with 35 years of experience designing and producing some of the industry's most reliable and high performing industrial continuous process furnaces. It



Abbott Furnace Company's five-zone electrically heated industrial furnace that is rated for 2,150°F. (Courtesy: Abbott)

is a privately owned company located in St. Marys, Pennsylvania.

MORE INFO www.abbottfurnace.com

QuesTek Innovations announces joint venture

QuesTek Innovations LLC has long been known as a creator of new materials and metal alloys to meet user-defined properties for applications in aerospace, automotive, consumer electronics, and other technology markets. The customer base for these high-performance alloys is international in scope. To better serve customers and prospects in Japan, QuesTek International has announced a joint venture agreement with Tokyo-based Itochu Techno-Solutions (CTC). CTC provides sales and consulting services for a range of materials-related software products, including those for alloy design, materials process design, materials characterization, and related databases, while providing support and consulting services. The new joint venture is called QuesTek Japan K.K.

QuesTek has been a pioneer in Integrated Computational Materials Engineering (ICME) technologies and its Materials by Design® methodology, which combines physics-based models, vetted thermodynamic and kinetic databases of the elements and predictive property models to allow for efficient materials design, process optimization, modeling, and simulation. A recent example of QuesTek's application of ICME was the development of the first high-temperature aluminum alloy for use with additive manufacturing (3D printing).

As a company, CTC is involved in providing application support for software that creates comprehensive simulations to predict the properties of materials. Founded in 1972, CTC reported sales in the last fiscal year of

more than \$4.1 billion and has 8,600 employees worldwide.

QuesTek Chief Science Officer and Massachusetts Institute of Technology Thermo-Calc Professor of the Practice Dr. Greg Olson, said, "With more than 30 years of experience in Japan's technology markets, CTC is well-positioned to be a strong part-

ner for QuesTek. As a lead-up to this joint venture, we've worked closely with CTC and have found them to have the technical skills, the market knowledge, relationships, and track record to be the ideal partner for us in Japan."

MORE INFO www.questek.com

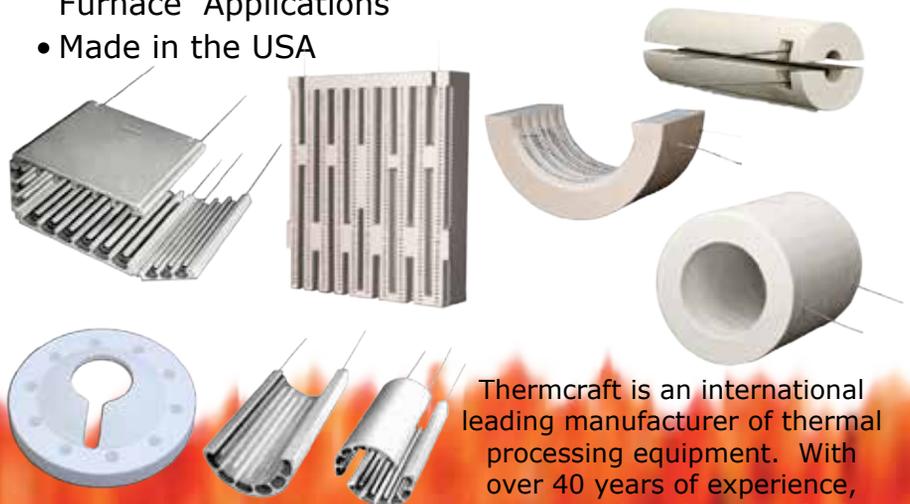


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L&L Special Furnace's XLE3636 tempering furnace is used to temper hot formed parts. (Courtesy: L&L Special Furnace Co)

L&L ships furnaces for hot forming, tempering

L&L Special Furnace Co, Inc. shipped two furnaces to a manufacturer of medical implant components in the northeastern United States.

The furnaces are a floor standing forging furnace for use in hot forming of medical implant parts and a floor standing tempering furnace to a manufacturer of medical implant components.

The forging furnace, an L&L model FWE422 with working dimensions of 48" wide by 24" high by 24" deep, is heated to 1,800°F. The parts that are blanks are heated in the furnace — once at temperature, they are moved to the hot forming press and formed into the required shape. The electrical heating system provides a clean atmosphere with no products of combustion which could affect the work load.

The furnace features a vertical door with adjustable stops. These stops allow the door to be stopped at a predetermined location during the heating process for minimal heat loss.

The tempering furnace is an L&L model XLE3636 with a vertical door and 12" diam-

eter, air-cooled convection fan and roller hearth. It has an effective work zone of 34" wide by 30" high by 32" deep. The furnace is used to temper hot formed parts and other thermal processing duties.

Both furnaces are complete with floor standing NEMA12 control panels with program controls and overtemperature protection systems.

MORE INFO www.llfurnace.com

International Tungsten2020 set for June

The 10th International Conference on Tungsten, Refractory & Hardmaterials (Tungsten2020) program is now available. Since the conference was last held in 2014, explosive developments have been made in numerous fields, notably metal additive manufacturing (AM) using refractory metals and alloys, and their carbides. Explore the latest developments in Montréal, Canada, June 27–July 1, 2020, at the Montréal Convention Center.

Tungsten2020 conference attendees can expect topics including developments in:

- › Metal AM.
- › Cobalt-free carbide and alloy compositions.
- › Field-assisted sintering technology (spark plasma sintering).
- › Cold spray for refractory materials.
- › Nanocrystalline materials.
- › Nano-phase refractory metals and alloys.

Tungsten2020 attendees will also have access to two co-located conferences: Additive Manufacturing with Powder Metallurgy Conference (AMPM2020) and the World Congress on Powder Metallurgy & Particulate Materials (WorldPM2020).

Co-located conference access includes:

- › More than 140 sessions and nearly 500 technical presentations between the three co-located conferences.

- › The world's largest trade exhibit featuring more than 150 exhibitors of PM; metal AM; and tungsten, refractory, and hardmaterials equipment, powder suppliers, and component manufacturers.

- › Networking with more than 1,200

attendees from around the world.

- › Even more time in the trade exhibit — this year's conference features two networking luncheons adjacent to the trade exhibit.

- › An optional Metal AM Tutorial focusing on basic information and guidance on the use of metal powders, including safety, handling, and storage to maximize the quality of the powders and subsequent performance of the consolidated parts.

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- › Held only once every six years in North America, World Congress on Powder Metallurgy & Particulate Materials (WorldPM2020) is the largest global hub of PM. Delve into materials, material characteristics, powder production, and more.

- › Focusing on metal additive manufacturing, the Additive Manufacturing with Powder Metallurgy Conference (AMPM2020) will feature worldwide industry experts presenting the latest technology developments in this fast-growing field.

At press time, this show's date had not changed.

MORE INFO Tungsten2020.org

Abbott Furnace receives order for calcine furnace

A multinational corporation that specializes in producing chemicals and precious metals and operating in more than 30 countries worldwide has placed an order with Abbott Furnace Company for an electrically heated continuous belt calcine furnace to be delivered in the second quarter of 2020. Abbott Furnace will design, manufacture, and install the industrial furnace that is rated at 1,850°F and includes an 18-inch wide inconel belt, silicon carbide muffle, and data acquisition system.

MORE INFO www.abbottfurnace.com

Milacron aids career development at AIM with equipment

Milacron Injection Molding & Extrusion is enabling career development in the plastics industry by delivering a 55-ton Milacron-FANUC Roboshot with iMFLUX technologies to the AIM Institute.

Milacron is a leading industrial technology company serving the plastics processing industry. The Roboshot excels in its resilience to handle many injection molding applications. The industry-standard-setting technology found in the Roboshot all-electric injection molding machine is a direct result of years of partnership and insight between Milacron and FANUC.

Integrating iMFLUX technology into machine controls is a more economical route to faster cycles and advanced part quality. iMFLUX offers quality benefits through improved balancing of high-cavitation molds, such as caps, closures, and consumer product applications. Additional benefits can be attributed to iMFLUX capabilities in recognizing a change in melt viscosity and adjusted filling to maintain consistent low pressure while achieving consistent part weights.

“Milacron was the first to present iMFLUX integrated directly into the machine’s controller, allowing the operator to modify settings through a single screen,” said Andy Stirn, director of new product development for Milacron Injection Molding & Extrusion. “At Milacron, we’re always looking for ways to help our customers succeed. Milacron began offering this capability in 2018 and have set the standard for fully integrated iMFLUX capability inside our control systems.”

The American Injection Molding (AIM) Institute offers a variety of courses for beginner injection molders through high-level experienced processors. AIM teaches that, regardless of job function in the industry, it is critical to understand all four disciplines in the injection molding process: plastic materials, mold design, injection molding processing, and part design; and how each of these components affect the final part. Their Molders’ Series curriculum is designed for processors of all experience levels and has the potential to benefit hundreds of

students each year. Students can enter the series at the level appropriate to their current experience and exit when they have achieved their desired knowledge and skills.

This machine will benefit multiple courses offered through AIM. It will further the AIM Institute partnership with iMFLUX, a wholly owned subsidiary of P&G,

to provide training on their proprietary process control technology. Classes held at AIM’s headquarters in Erie, Pennsylvania, include “Introduction to iMFLUX” and “Applied iMFLUX” processing. Students who complete both courses have the ability and intelligence to set up and adjust an iMFLUX process, as well as the skills to convert a



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conventional molding process to one using IMFLUX.

“The demand for experts in this industry is not slowing. We are working to eliminate knowledge gaps through catered courses that fill the needs of working professionals,” said David Hoffman, director of AIM. “Our students inherit our research-based practices and skills from our specialized educators, geared toward their specific occupation. Students will be capable of adapting the latest techniques to tackle new obstacles in the industry that are relevant now and in the future.”

Milacron shares this commitment to pioneer new developments and solve complex gaps in the plastics industry. The partnership between Milacron and AIM is critical to conduct new research and techniques for methods in parts processing and manufacturing. This analysis will be distributed among AIM graduates, but also to leaders in the industry on how to reform plastics processing.

Milacron Injection Molding & Extrusion is a global leader in the manufacture, distribution, and service of highly engineered and customized systems within the plastic technology and processing industry.

MORE INFO www.milacron.com

Blue M Ships mechanical convection ovens

Thermal Product Solutions, a global manufacturer of thermal-processing equipment, has shipped three Blue M mechanical convection ovens to a company in the semiconductor industry.

These Blue M convection ovens have a designated temperature range of 15°C above ambient to 250°C. The interior of the work chambers has a dimension of 22 W x 50 D x 55 H. The spacious chambers will allow

for epoxy curing. The interior of the ovens is all-welded heavy gauge, reinforced type 304 stainless steel for maximum heat and corrosion resistance. The exterior of the ovens is all-welded heavy gauge reinforced cold-rolled steel. The doors use ball-bearing hinges and industrial type latching to provide positive door seal and minimize fume leakage.

There are three main components to the control system which control the temperature ramp/soak capability, alarms, and events.

“These particular ovens have a direct drive ball bearing motor for heating and air delivery system featuring a balanced stainless steel multi-bladed blower wheel. The heating elements use Nichrome wire strung through high temperature, high dielectric strength refractory disks,” said Jonathan Young, product manager. 📡

MORE INFO www.thermalproduct.com

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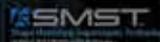
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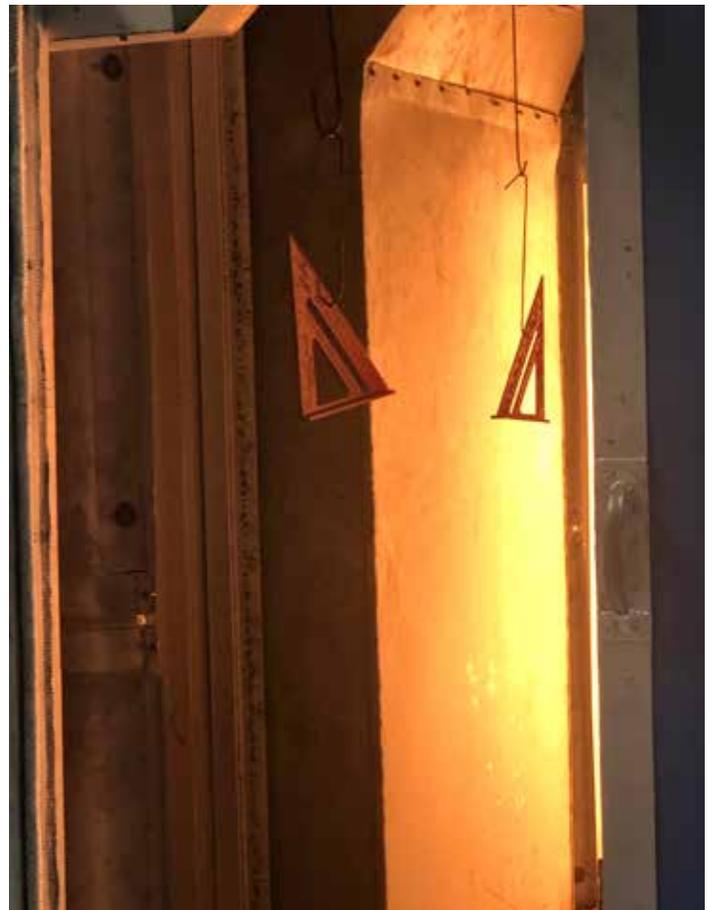
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The one-and-a-half-day agenda will cover a variety of topics presented by members of companies dedicated to the education and growth of the industry. IHEA's IRED members will present a section on infrared curing, which reviews the basics of infrared technology, several useful applications both electric and gas, and case studies to prove the benefits for those who use it. Attendees will also benefit from seeing how to cure parts during the lab demos.

- › Curing: Infrared Basics — Scott Bishop, Alabama Power.
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The effects of partial decarburization on the residual stress profile of a through hardened M50 and a case carburized M50NiL bearing ring.

Surface stress is key in rolling contact fatigue

Most bearing failures can be attributed to some form of rolling contact fatigue. Many factors influence rolling contact fatigue, but two main factors are steel cleanliness and residual stress. [1] Generally, steel cleanliness has improved dramatically over the years and is becoming less of an issue. [2] Residual stress has been shown to have a significant effect on rolling contact fatigue performance and can be affected by many variables. [3-5] A variable often overlooked is partial decarburization, which can have an extremely negative impact on the residual stress profile of through hardened and case carburized components. [6] This article will examine the effects of partial decarburization on the residual stress profile of a through hardened M50 and a case carburized M50NiL bearing ring with the use of modeling.

A bearing ring with a 4-inch outer diameter was modeled using the DANTE heat treatment simulation software to show the effect of partial decarburization on residual stress. Figure 1 shows a 270° CAD section of the bearing ring. An axisymmetric model was used for the study, with the assumption that the cooling conditions act uniformly in the circumferential direction. Figure 2 shows the cross-section of the bearing ring and the line of nodes used for plots of carbon, hardness, and stress as a function of depth from the surface.

Using DANTE, the predicted residual stresses of a through hardened M50 and a case carburized M50NiL bearing were compared. It is well known that the compressive residual stresses from a case carburized bearing can substantially improve rolling contact fatigue resistance for heavily loaded bearings. [1-6] However, decarburization has the potential to alter the residual stress state at the surface of the component and affect rolling contact fatigue performance. A situation was therefore derived by which the amount of decarburization still resulted in a fully martensitic microstructure, with a hardness of approximately 60 HRC. Figure 3 compares the predicted carbon distributions for a through hardened M50 bearing and a case carburized M50NiL bearing, with and without decarburization.

As shown in Figure 3, the amount of decarburization is approximately equal for both materials and can be classified as partial decarburization. The decarburization extends to approximately 0.1 mm, with a surface carbon value of 0.4 percent. With the carbon at 0.4 percent, the surface hardness is approximately 60 HRC for both cases, as shown in Figure 4. This means that both components would most likely be accepted by quality control. While the hardness would lead to the belief that the component would function well during rolling contact, a closer look at the surface stress state tells a different story.



Figure 1: 270° section of the CAD model used for the study.

Most through hardened bearings have a slight tensile stress at the surface of the component. This is due to the phase transformation progressing from the surface to the core. Figure 5 shows the martensite and stress relationship for the M50 bearing with and without decarburization. It should be noted that the core point lies approximately 1 mm below the surface and the maximum principal stress shown corresponds to the hoop stress in the bearing's outer raceway when that stress is tensile. Figure 5 shows that after the initial thermal contraction of the surface, followed by thermal contraction of the core point, the martensite transformation starts on the surface. This transformation causes the surface to go into compression, while the core balances the stress with tension. Approximately 15 seconds after the surface transformation, the core begins to transform to martensite. The core transformation acts to push out on the hardened surface, reducing the compression and eventually driving the surface into tension. Both conditions exhibit this behavior, but the decarburized transformation progresses much faster on the surface, such that there exists a large difference in the amount of martensite formed on the surface relative to the core. This slight difference has a very significant impact on the residual stress state.

Carburized components are just the opposite, with the transformation beginning subsurface in the lower carbon region and progressing out toward the surface. This means the surface layer is the last to transform to martensite, leaving the surface in a state of compression from the volume expansion of the martensite transformation. Decarburization has the same effect with respect to martensite phase transformation timing as the through hardening behavior; the surface transforms before the subsurface material does. This behavior acts to reduce the compression generated from the carburized transformation behavior. For the amount of decarburization in this example, the surface compression of the carburized M50NiL bearing is reduced approximately 100 MPa, as

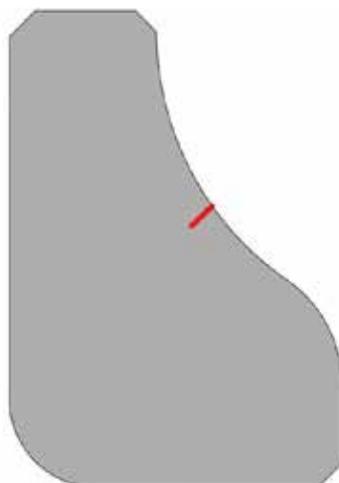


Figure 2: Cross-section of the bearing ring with the line of nodes used for line plots shown in Figures 3-6.

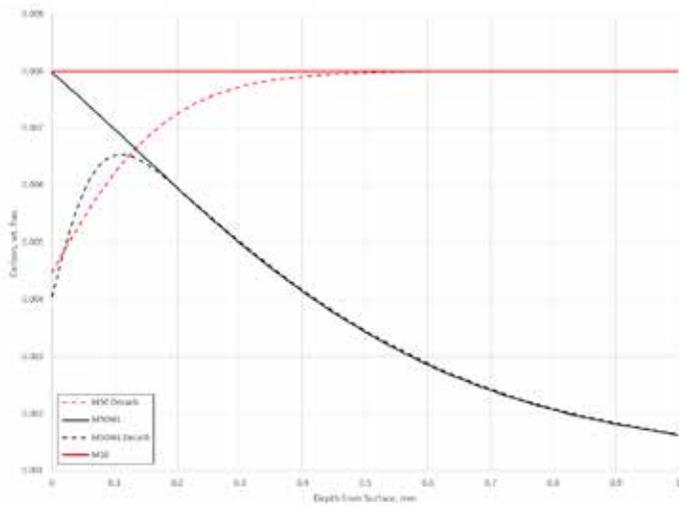


Figure 3: Plot of carbon as a function of depth from the surface for the carburized ring (good carbon) and the decarburized ring (bad carbon).

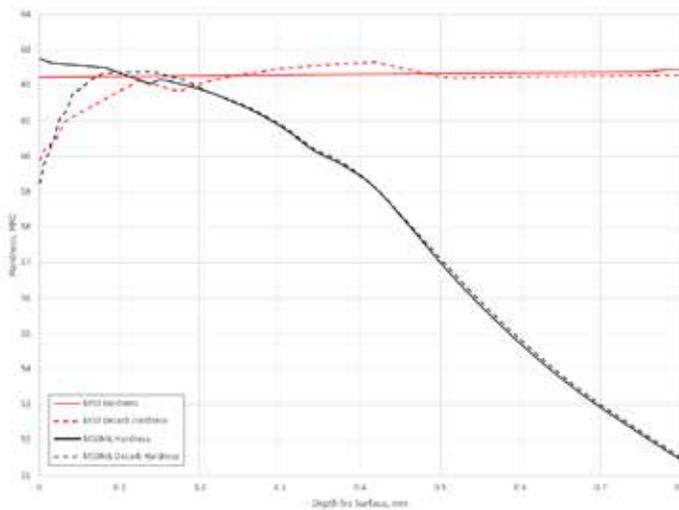


Figure 4: Plot of hardness as a function of depth from the surface for the carburized ring (good carbon) and the decarburized ring (bad carbon).

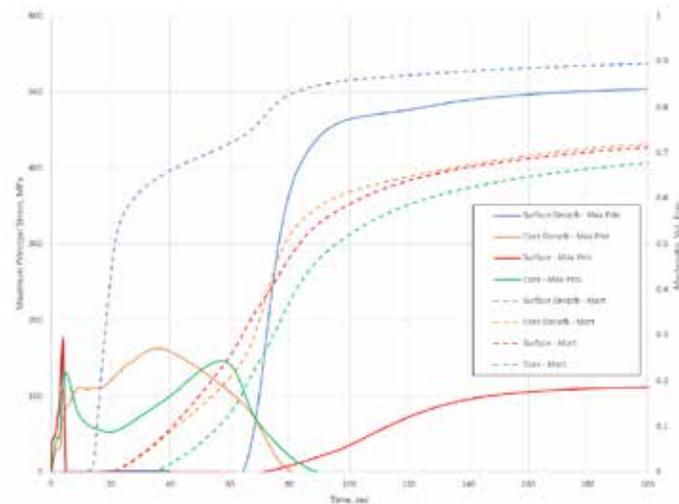


Figure 5: Plot of martensite and maximum principal stress versus time for the M50 bearing ring, with and without decarburization.

shown in Figure 6. This may not be enough to seriously hurt the rolling contact fatigue performance of this bearing. However, the through hardened M50 bearing saw a 500 MPa increase in tensile stress for the hoop and axial directions. Besides being extremely detrimental to rolling contact fatigue, this level of stress may be enough to cause

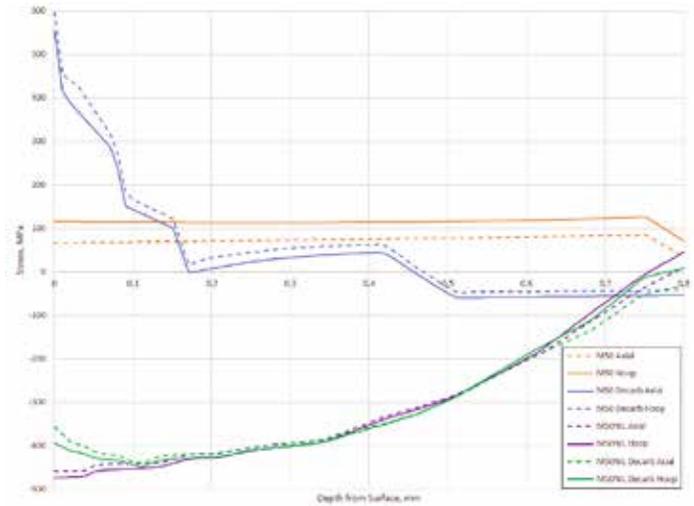


Figure 6: Plot of residual stress in the hoop and axial directions as a function of depth from the surface for the carburized ring (good carbon) and the decarburized ring (bad carbon).

surface cracking if a minor surface defect is present.

CONCLUSION

In summary, surface stress plays a fundamental role in rolling contact fatigue performance of bearing components. While through hardened bearings usually have some residual tensile stresses at the surface, they are generally low in magnitude. While these low magnitude tensile stresses are not helpful to performance in rolling contact, they can be easily converted to compressive by post-hardening finishing operations such as shot peening or burnishing for high-load applications. However, with mild decarburization these operations may not completely remove the tensile stresses, let alone impart compressive residual stresses. Case carburized bearings, on the other hand, use a carbon gradient to induce relatively high magnitudes of residual surface compression. Mild partial decarburization for a case-hardened bearing is far less severe, simply reducing the compressive magnitude slightly. ☞

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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.



Many of the issues in induction hardening can be directly related to the amount of contamination in the quench system, and improper or inadequate cleaning.

Troubleshooting induction hardening problems: Part 1

Induction hardening is a unique method used to harden steels. The process uses a power supply, RF generator, induction coil, and quenching mechanism (spray or immersion) to yield a high surface hardness and advantageous residual surface stresses. The process can be highly automated and results in high production rates with uniform results. Heating is very fast, with selective heating of the desired part. An induction hardening line can be integrated readily into cellular manufacturing.

It is commonly used to heat treat gear teeth, shafting, and other parts that require a high surface hardness for wear or strength. The native hardenability of the steel is used to obtain the surface hardness. For this reason, medium- to high-carbon steels are used. Typically, carbon contents between 0.4 to 0.6 percent carbon are used in induction hardening. Alloy content can vary, depending on the desired depth of hardening and the required core hardness. The resulting hardened part offers excellent control of distortion compared with conventional carburizing, with practically identical mechanical properties [1]. A comparison of an induction-hardened gear tooth and a carburized gear tooth is shown in Figure 1.

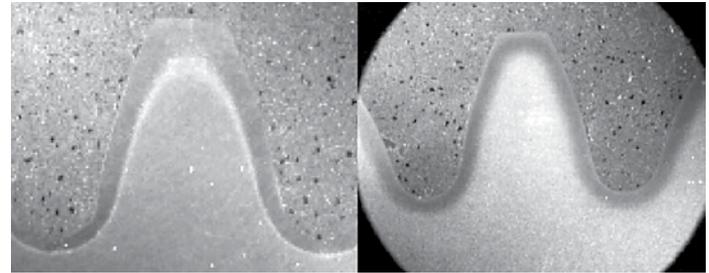


Figure 1: Comparison of an induction hardened gear tooth and a carburized gear tooth: Left is induction-hardened tooth; Right is carburized tooth [1] 5X.



Figure 2: Blocked holes and non-uniform quenching can create soft spots. It can also increase the chance of cracking.

PROBLEMS DURING INDUCTION HARDENING

There are many problems that can occur in induction hardening that can have nothing to do with the power supply, RF generator, or coil. These are process-related issues that can be traced back to improper or inadequate process control. These problems can manifest themselves as improper part hardness or cracking; improper pattern; quenching issues such as foaming or excessive drag-out; corrosion issues; or biological issues such as bacteria and fungus, or odors. In this article and next month's, we will discuss several of these issues and ways to troubleshoot corrective actions. This month, I will cover hardness or cracking, excessive polymer consumption or drag-out, and foaming.

LOW HARDNESS AND MECHANICAL PROPERTIES OR DISTORTION AND CRACKING

Low hardness and mechanical properties are usually associated with poor quenching. Occasionally, it is found that the part has not been properly heated to the austenitizing temperature, or that inadequate dwell time has been used. However, this is not common. Poor properties can often be traced to either improper quenchant concentration, excessive quenchant temperature, or poor or non-uniform agitation. Contamination of the quenchant can also cause problems with achieving proper quenching [2].

The quenchant used (predominantly polymer quenchants) need to be controlled to achieve a stable concentration. This is usually accomplished using a simple optical or digital refractometer. The refractometer should be properly calibrated according to the manufacturing recommendations. Concentration is usually determined

by taking a reading in °Brix on the optical or digital refractometer. This reading is then multiplied by the factor supplied by the manufacturer to determine the concentration of the polymer. This concentration should also be verified by the manufacturer at routine intervals, or in-house using kinematic viscosity. The multiplying factor can change due to differences in water — reverse osmosis vs. hardwater — or due to contamination. Contamination and harder water tend to make the factor drift downward due to the increased solids present.

Concentration should be determined at least once per shift. The actual number depends on the parts processed per hour, and the amount of drag-out experienced. Even if the drag-out is low, the concentration can creep upwards due to the evaporation of water. In most cases, water is added more often to quench tanks than is polymer due to evaporation. For most applications, the concentra-

tion should be controlled within ± 2 percent (or 1°Brix depending on the quenchant multiplying factor). Tighter control is better, but measuring the difference becomes problematic, depending on the refractometer chosen.

The optical refractometer should have a narrow range. This increases the reliability and reproducibility of the measurement. For most optical refractometers, it is difficult to measure tighter than $0.5 - 1.0^\circ\text{Brix}$. For digital refractometers, it is much easier to achieve repeatable measurements within $\pm 0.25^\circ\text{Brix}$.

Polymer quenchants are very sensitive to the bulk temperature. Polymer quenchants tend to slow down the heat extraction rate as the bulk temperature is increased. Only 10°C can have a marked change in the cooling rate. To achieve a consistent process control, the temperature of the quench bath should be controlled within $\pm 3^\circ\text{C}$.

Agitation is very important. The quench ring or spray head should supply a uniform flow of quenchant to the part. Blocked holes or improper pressures can result in soft spots or even cracking (Figure 2). Filtration should be used prior to the quench ring to prevent clogging of the ports on the spray ring.

In summary, low hardness and soft spots can usually be overcome by decreasing concentration; increasing agitation and the uniformity of agitation; and decreasing the bulk quenchant temperature. Preventing contamination is also important.

Distortion and cracking of an induction-hardened part is usually related to non-uniform quenching. It can also be from a low concentration of quenchant. Overheating a part is unfortunately common from misaligned coils. Proper centering of the part within the coil is important for uniform depth of hardening and preventing cracking.

The fixes for excessive distortion or cracking are generally the opposite for low hardness. Increasing concentration or changing to a slower polymer can slow down the quench, reducing the chance for distortion. Increasing the bulk temperature of the quenchant will also slow down the quench. Increasing the dwell time (or distance) between the time the heat is turned "off" and the time the quench is turned "on" will reduce the thermal gradients in the part, reducing the chances of distortion or part cracking. Reducing the flow or pressure of the quenchant will also slow down the quench.

EXCESSIVE POLYMER CONSUMPTION OR DRAG-OUT

Excessive polymer drag-out or consumption can be related either to the quenchant used or the geometry of the part. Polymers work by creating a polymer film on the part instead of a stable vapor phase. During the vapor phase, heat transfer is slow, and usually by radiation. Non-uniform rupture of the vapor phase can result in distortion. A polymer creates a film of polymer around the part, and heat transfer is by conduction. Heat transfer is more uniform and rapid. At a certain point, depending on surface temperature and other factors, the film will rupture uniformly. This creates uniform heat transfer, with resulting uniform residual stresses. Distortion is reduced.

If the part exit temperature from the quenchant is too high, or inadequate time is allowed for the polymer to dissolve back into solution, then excessive drag-out can result. Usually a cure for this is longer dwell times in the quenchant, allowing the part to cool below the cloud point of the quenchant, or allowing the polymer to dissolve back into the water.

Alternatively, different types of polymers can be used to reduce drag-out. Use of lower solids quenchants can reduce residual films on parts and reduce polymer consumption. Excessive polymer drag-out can also be reduced or recovered by recycling or reclaiming polymer from exiting parts. However, care must be taken to prevent removing

additives that may prevent other problems like rusting and biological growth.

FOAMING

Foaming is usually a mechanical issue. Leaky seals on pumps, or excessive pressures can contribute to foaming. Excessive spray pressures can also create excessive foaming. Inadequate fluid levels resulting in a vortex on the suction side can entrain air, which comes out of solution at the pressure drop at the spray, creating foam. Defoamers can be added, but usually it is better to increase fluid levels or repair pump seals so that the problem is fixed, instead of just maintained.

In Part 2 next month, I will discuss biological and odor problems, and corrosion issues.

Should you have any questions regarding this article, or have suggestions for new articles, please contact the editor or the author. ☞

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Once having no intention to work in quality, spending time cross-training in other departments quickly ignited a desire to help improve that part of the business, and work to find solutions to complex problems.

Willingness to change course leads to satisfying career

Editor's note: Beginning with the March issue of *Thermal Processing*, we added Tony Tenaglier, a heat treat process engineer at Hitchiner Manufacturing, as a contributor to the Quality Counts column. Conrad Kacsik's Jason Schulze will continue to write occasional columns. This month, meet Shaun Kim, quality director at Byington Steel Treating.

My name is Shaun Kim, and I'm the quality director at Byington Steel Treating, Inc (BST). As this is my first time writing for *Thermal Processing*, this column will be about the impact quality has on heat treatment. My intent is to offer readers an alternative view into how quality is a key factor in all areas of heat treatment, and its importance to compliance to statutory and regulatory requirements.

First, a little background about my company. BST's facility is in Santa Clara, California. It's a family-owned business that dates to 1952. We are in our third generation of ownership, with Sean Byington leading the company as CEO. Our facility is NADCAP accredited for heat treating, hardness, and conductivity testing. Our location in the Silicon Valley allows us to serve aerospace, semi-conductor, medical, and many other commercial industries.

Now, a little about myself. I was born and raised in the California bay area, and attended San Francisco State University where I focused on communication studies. (In the following months we'll see how much knowledge I retained in creative writing. Fingers crossed.) I've been with BST since 2015, and director of the quality department since 2016. A little irony to this story is that my father was a metallurgical engineering major, but now works in the technology sector. I guess you can say that I followed in his footsteps to a degree. Maybe the apple really doesn't fall far from the tree. When I got into the heat-treating industry, I knew virtually nothing about it. I wasn't content with the path that my career was going, but was still young enough to make a change. I wanted more. At that point, I decided to take a position at BST operating the vacuum furnaces. It quickly changed when I was asked to cross-train in multiple departments, including receiving and quality. I had no intention of going into quality, but I noticed quickly that I was intrigued by the quality side of heat treating and how my own pragmatic nature was helping to improve the quality side of the business. Additionally, I enjoyed the work of finding solutions to complex problems. My CEO recognized that and inserted me into the QA department working closely with the quality director at the time. Long story short, I was promoted to quality director and here I am five years later.

Until now, the biggest accomplishment for myself is leading the

company in obtaining our NADCAP accreditation for heat treating through the Performance Review Institute (PRI). BST was primarily a commercial heat-treating business upon my arrival but had aspirations of elevating our processing capabilities into the NADCAP level with the quality to back up any certification given. This was probably the most difficult task I've been assigned on any professional level. Because of that, I would say it's the one I'm most proud of.

As I think about how this column will progress, I realized that there are many areas where I can begin. First are the continuous changes to AMS and ASTM specifications and how quality determines how those changes are interpreted and flowed down to processing. Second would be training, and its importance to be able to follow your company procedures.

It's extremely important for the quality department to know and understand any changes to standards or process specifications. They can and will affect the way your company operates from a processing perspective. Your company may have been doing things a certain way for several years, but a change in a standard can alter or completely change how you do things after it's released. There are certainly other areas that can be affected besides processing, but I'll limit my discussions to the quality side of things when writing about this topic for the next column.

In my experience, training is the most crucial element in aligning your operators to your heat-treating procedures. All your hard work of interpreting a new standard and updating procedures means nothing if you can't train your team to follow. In a future column, I'll discuss the challenges of training, what I've learned that works great, and things that didn't work out so well. I'm a firm believer that you will learn the most when you make mistakes along the way. Some are big mistakes, and some are small ones. Either way, lessons learned along the way are invaluable to the efficiency of your quality system.

I'd like to share my experience and insight on these topics and more. I look forward to contributing to *Thermal Processing* in the coming months. My goal is to cover topics that are on the minds of the readers, as well as those that may have been overlooked. I always say those of us in quality rotate on a slightly different axis than others. Similar to our modern world, quality is always changing, and I hope that this column will provide a resource to keep up with those changes. 🔥

In my experience, training is the most crucial element in aligning your operators to your heat-treating procedures.

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HEAT TREAT EVENTS 2020



AeroMat 2020 > May 4-6 | Palm Springs, CA

AISTech2020 > May 4-7 | Cleveland, OH

WorldPM2020 > June 27-July 1 | Montreal, Canada

AMPM2020 > June 27-July 1 | Montreal, Canada

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ISSUE FOCUS ///

CERAMICS / INDUSTRIAL GASES

HEAT WORK TO THE FORE



The sixth annual Ceramics Expo 2020 tackles the burning issues.

By CHARLIE WALLIN on behalf of CERAMICS EXPO

Supporting its new tagline — Enabling a Clean, Efficient & Electrified Future — the highly regarded Ceramics Expo will once again head for Cleveland's I-X Center (September 22–23) for its sixth annual technology showcase. The organizer, Smarter Shows (Tarsus) Ltd., has confirmed that this is a virtually sold-out event, and it's now expected that more than 300 manufacturers and system providers from 25 countries across the world will come together to deliver a dynamic technology presentation and establish the ideal platform for a wide-ranging forum.

Fuel-efficient and consistent drying, curing, firing, and sintering are central to smooth operation and enhanced performance in the various parts of the ceramics, glass, and allied-products industry, and therefore it's no surprise to see these elements sitting front-and-center of North America's largest free-to-attend event for this community. Pre-registrations are at an encouraging level, and more than 3,000 visitors are expected to make the trip to the CLE come May.

"This is a high-temperature industry, and inevitably, there is closely focused attention on dependable, efficient, and safe thermal processing, as well as advanced combustion monitoring and control," said Exhibition Director Danny Scott. "We are proud to note that many of the leading international exponents in this critical area of our sector — alongside those organizations responsible for materials innovation — will be represented on the exhibit floor, with the scope and size outstripping anything we've seen previously."

The shorter two-day format with pre-show networking, introduced to overwhelmingly positive reaction last year, is repeated. Also featured once again is the dedicated 1,200-square-foot space within the exhibit area for staging the B2B Meetings initiative, a concept that draws together interested parties for discussions that lead to well defined and mutually beneficial actions. Brand new for 2020 is the Ceramics Expo Start-Up and Academia Pavilion, scheduled to present about 30 exciting organizations, and a feature that Scott describes as "the ideal forum at which to maximize all the exciting opportunities for furthering discussion and cross-fertilization and for widening perspective."

With all the various facets of this event in place — many tried and tested plus those enjoying their first run-out — a bustling atmosphere can be expected to be generated by visitors traveling from about 40 countries. For all those with an interest in, or pushing advancement of, industrial heat work then Ceramics Expo is a show not to be missed. Exhibitor offerings include:

FIRED UP

Innovative, flexible, and volume-capable kilns and furnaces sit at the heart of ceramic manufacturing facilities, and the leading innovators will participate in Ceramics Expo 2020.

Renowned U.S. company Harper International will be in Cleveland. Harper tailors its batch and continuous systems to each application, making the company ideal for processing in controlled and specialty atmospheric environments and at temperatures from 300°C to 3,000°C (570°F to 5,430°F). The company boasts extensive experience in designing for the production of silicon nitride, tungsten carbide,

boron nitride, and aluminas in gas, electric, and microwave heating. Additionally, Harper kilns are widely used to calcine powders and sinter components such as thermistors, varistors, and monolithic and multi-layer capacitors. It also has interesting technologies available for silicon carbide (SiC) fiber processing and carbon fibers.

Also from the U.S., visitors will meet with Swindell Dressler, whose customers include the structural clay, whitewares, technical ceramics, and carbon graphite industries and whose roots go back more than 100 years, as well as the team from Thermaltek, the designer and manufacturer of custom high-temperature kilns and metallic resistance heating elements for a broad spectrum of industrial applications, including technical ceramics, electronics, fuel cells, optical fibers, calcining, glass, crystal growing, and non-ferrous metal melting and holding.

Backed up by more than 80 years' experience, WISTRA (on the Cerinnov Group booth) is another globally recognized name in the business of thermal engineering design for technical and advanced ceramics. It produces everything from tempering units operating at just 350°C (660°F) right up to specialty kilns required to fire to 2,000°C (3,630°F). A whole host of products are manufactured in WISTRA systems, including structural and electronic ceramics, multi-layer ceramics, catalysts, filters, compacts, ceramic-metal composites, bioprotheses, abrasives, beads, and high-purity aluminas and zirconias. Kilns can also be designed for insulator firing.

Returning to Ceramics Expo is the Mexican kiln, furnace and oven constructor Nutec Bickley. Depending on the specific application, it offers shuttle, elevator, Carbell®, or tunnel kilns for firing an array of technical ceramics. A recent success in this area has been the commissioning of a special, high-temperature (1,700°C/3,100°F) tunnel kiln for a producer of technical ceramics in the U.S. Top-level performance is reported.

In the more traditional sector of the ceramic industry, Nutec Bickley has also been putting the finishing touches to its latest shuttle kiln for one of the world's largest sanitaryware manufacturers. With an operational temperature of about 1,205°C (2,200°F), this kiln is a nine-car, three-deck model with a setting height of 2.2 meters (7.2 feet). With this design, the total weight of kiln furniture is reduced by 20 percent as compared with more traditional set-ups, and the fuel usage can be significantly reduced. Further, the kiln is fitted with the company's Jointless® insulation modules, which can withstand temperatures up to 1,350°C (2,460°F), providing for minimal maintenance, improved fuel economy and extended service life. This is a one-piece system that eliminates the joints between modules and also the spaces typically in the exhaust ports. It doesn't need to be covered with cordierite, which reduces fuel consumption.

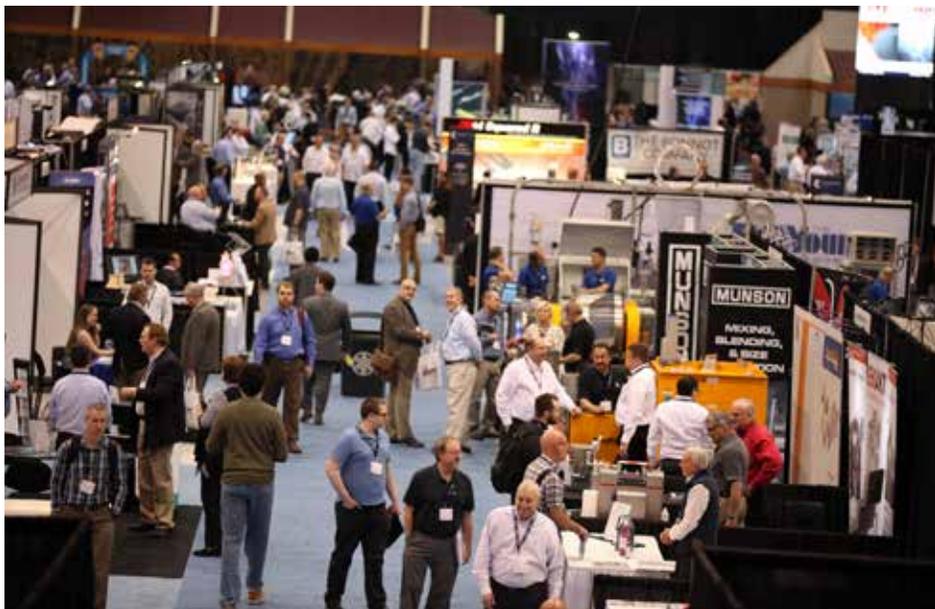
Visitors will also be able to catch up with news from the kiln and furnace subsidiaries of some leading international ceramic equipment suppliers, such as Germany's Riedhammer (on the Laeis booth, part of the \$1.6 billion SACMI Group). One of the global sanitaryware groups recently chose Riedhammer to design and build a new kiln for a Russian facility, investing to double the current shuttle kiln capacity for the fire/re-fire of glazed pieces. The kiln comprises five

cars, has a total effective volume of 50 cubic meters (1,765 cubic feet) and allows the manufacturer to load pieces on two or three levels, depending on the items.

STRONG SUPPORT

Of course, all the above-mentioned systems rely on excellent kiln furniture refractories in order to function properly. IPS Ceramics USA came up with several new approaches recently. In one case, it developed adjustable systems based on recrystallized silicon carbide (ReSiC) toothed support posts and slide-in batts. ReSiC has very low levels of high temperature creep, meaning the posts remain straight after many firings. Already supplied in lengths of nearly two meters, they have seen prolonged service at temperatures of up to 1,600°C (2,910°F) without any distortion or breakage. The post design can be optimized so the spacing of the teeth matches the customer's specific product line.

IPS has also introduced a grade of cordierite suitable for injection



Ceramics Expo's shorter two-day format with pre-show networking, introduced to overwhelmingly positive reaction last year, will be repeated. (Courtesy: Ceramics Expo)

molding. This allows a wide variety of complex shapes to be produced, with features such as through holes, slots, and fine surface detail. A good example is support blocks for plate heaters, which require a complex zig-zag slot arrangement to retain the wire element. Good market penetration and increasing growth saw the company relocate in February from Cornelius to Huntersville, North Carolina, a move designed to provide the facilities required to increase the company's scope of operations and offer a more efficient customer service across North America.

Another exhibitor offering a wide range of primary and secondary kiln furniture is SELEE Advanced Ceramics, incorporating Engineered Ceramics® solutions and custom-designed systems using its Micromass®, Tylar®, and HYcor® product lines to meet virtually every firing need. There are tiles, saggars, setters, hearth plates, burner blocks, and element holders in materials including alumina, clay bonded silicon carbide, fused silica, mullite, and other specialty formulations.

For a number of highly specialized applications, the Korean company YJC has been working on a number of novel solutions. One of these is a multi-hole ceramic setter featuring 0.45 mm diameter perforations and delivering superior debinding and thermal conduction

of the fired object. Another is a YJC box sagger whose main use is heat treatment of ceramic chips and powders such as MLCC. These saggars can be coated with YSZ (yttria-stabilized zirconia) or CSZ (ceria-stabilized zirconia), depending on requirements, and are optimized to perform where thermal shock conditions are found in the firing process. Meeting the needs of new industry demands, YJC has now developed a box sagger used for the high-temperature reaction and synthesis of lithium and metal oxides in the production of positive electrode materials, for instance in lithium ion secondary battery production.

More specialisms to investigate, particularly aimed at the technical ceramics industry, can be found at the Sunrock Ceramics booth. Sunrock's HPA family of high alumina materials ranges from 85-percent alumina to 99.7-percent alumina with porosity of 15 percent to 18 percent to enhance thermal-shock resistance. Additionally, the company produces the HPA-CG material designed specifically for pusher plates used on pusher furnaces. Sunrock also offers an insulating 99.7-percent alumina material called B-99, used primarily in furnace linings for reducing atmosphere applications. Where required, the company has very flexible processes for economically and rapidly making new custom shapes, either from pressing or by thixotropic casting.

WHEN THE PRESSURE'S ON

Advanced thermal processing, particularly in the field of technical ceramics, will often involve the simultaneous application of heat and pressure to achieve densification at elevated temperatures of a ceramic powder compact with the aim of the bonding process being to achieve production of a polycrystalline body. The sintering conditions allow for various mechanisms of atom movement to occur, such as viscous flow, liquid phase solution-precipitation, surface diffusion, bulk diffusion, and evaporation-condensation. This can be applied to bodies formed of oxides, nitrides, borides, and carbides, but also comes into play for some cermets, ferrites, abrasives, ceramic composites, and nanoceramics.

Ceramics Expo exhibitor Thermal Technology has decades of experience in specialized thermal processing and its hot press furnace systems employ state-of-the-art hydraulics for extremely high precision force and/or position control. Automatic operation is conducted by PLC-based control using a touch screen HMI. Automatic hydraulic safety systems limit force application while the furnace chamber is open in order to mitigate die breakage and operator injury. Standard sizes are available up to 2,000kN with a maximum pressing temperature of 2,500°C (4,530°F). Larger, custom-built units can be produced for specific applications. Thermal Technology also manufactures spark plasma sintering systems.

American Isostatic Press (AIP) is also active in the field and offers furnace designs with temperatures ranging from 1,200°C to 2,200°C (2,190°F to 3,990°F), typically made from Kanthal, molybdenum, tungsten, and graphite. Special oxygen-capable furnaces are available in platinum and molybdenum disilicide types. Furnaces for pressure impregnation and carbon-carbon production are offered for reactive gas containment. Both top and bottom loading furnaces are available, as are special automated loading systems.

New for lab conditions is the recently launched lab sized Series 45

top loading furnace for ultra-high temperature research and development work from Centorr Vacuum Industries. Capable of achieving 3,200°C (5,790°F) in an inert gas atmosphere, the hot zone size of 6 inches (150 mm) in diameter by 9 inches (230mm) tall is ideal for heat treatment of ceramics, graphite and carbon composites, process development, and lab studies. The furnace features solid graphite panel elements designed for long service life within a cylindrical hot zone of rigid graphite insulation to provide long-term service even in the presence of process off-gassing.

There's also plenty of pedigree on show by AVS Inc, with more than 1,000 installed systems worldwide for sintering, hot pressing, HIP and CVD/CVI process routes spanning aerospace, defense, and additive manufacturing. With fabrication capabilities ranging in size from 4 inches (100mm) in diameter mini furnaces, to hot zones in excess of 15 feet (4.6 meters), AVS can fabricate graphite, ceramic, or refractory metal hot zones in-house. This can be for applications involving a combination of high temperatures, up to 3,000°C (5,430°F), high vacuum down to 10-6 Torr, gas pressures up to 3,000 psig (200 bar), and even hot pressing that ranges from five tons to more than 2,000 tons of hydraulic force.

The other side of the densification coin encompasses the possibility of reduced temperature input. Anyone interested in this should get to the California Nanotechnologies (Cal Nano) booth. The company has been collaborating with Idaho National Laboratory and talked in detail about a novel sintering process in a recent article (Materials Today Communications, December 2019), co-authored by Cal Nano's CEO, Eric Eyerman. The work concerns the "densification of graphite under high pressure and moderate temperature," and it's been demonstrated that graphite can be sintered at lower temperatures than previously thought possible. The technical and potential economic benefits shown by this effort can lead to lowering the cost of producing graphite components to meet current and future technical challenges.

Another benefit that can be brought into the equation is reduced cycle times, something addressed by German exhibitor Dr Fritsch and its FAST/SPS sinter presses. During FAST/SPS-sintering, current flows directly through the sintering mold and even through the sinter material if it is electrically conductive. The electrical resistance of the mold and the sinter material leads to rapid heating. This is also referred to as Joule's Heating. Temperature is consequently generated only where it is needed. The sinter cycles are very short since the atmosphere in the sinter chamber does not need to be heated up. The short cycles result in reduced grain growth, higher densities, and better microstructure of the materials. Since grain growth is reduced to a minimum, nanomaterials stay nano after sintering.

SUPERMATERIALS AID EXCELLENCE

While all the central equipment needs to be optimized, we can't ignore the contribution of newly formulated, high-performance materials. One such comes from CFOAM LLC. CFOAM® carbon foam is a macroporous carbon with foam-like structure, providing the advantages common to other foam-like insulation materials. However, unlike other insulating foam materials, CFOAM® can be used at very high temperatures, up to at least 3,000°C (5,430°F). It demonstrates high thermal shock resistance and dimensional stability over temperature. CFOAM just announced a distribution partnership with Composites One, a leading North American supplier of composite materials – the latter company will stock CFOAM in its network warehouses in the USA and Canada.

Also new to the scene is Covaron, an early stage, fast-growing, and award-winning advanced materials company. It's an R&D company that specializes in materials technology to control inorganic poly-

mers, forming ceramic-matrix composites, ceramic-organic composites, and ceramic-fiber composites. Covaron has several new ceramic coating products, among them its ceramic-like thermal barrier coatings that combine the formulation and process versatility of polymer coating.

Formulating thermal materials that will serve advanced industries in their future endeavors is crucial, and Ceramics Expo remains a prime platform. The 2020 show will include Naieel Co, for instance, an expert in boron nitride nanotubes (BNNT) and their manufacture and exploitation. BNNT presents similar thermal conducting and mechanical properties to carbon nanotubes, while its thermal/chemical stabilities, biocompatibility, electric insulating, and thermal neutron absorbing properties are much higher. Due to these superior properties, BNNT is under exploration in many areas of IT, space/nuclear, bio-medical, and energy.

COMPELLING CONFERENCE

In addition to the many exhibits in Cleveland, the experience is substantially uplifted by the Ceramics Expo 2020 Conference – all elements of which are free-to-attend, just like the expo. Attending the sessions is easy as the conference forum areas sit alongside the exhibit floor on the same level. These include:

➤ **Solving Thermal Management Challenges for Electronic Packaging and Assembly Applications Using Advanced Ceramic Materials (panel, Day 1, Track 1, 10:45 a.m.).** Many electronics manufacturers are unaware of the benefits that ceramics can bring. This session will explain the advantages of using ceramic materials; end users and manufacturers cannot afford to miss this disruptive growth area. For the supply chain, this session will also explore how to maximize their usage of ceramics in electronic circuitry. It will also allow electronic packaging manufacturers who are already using ceramics in their products to outline the benefits for those who have not yet begun integration.

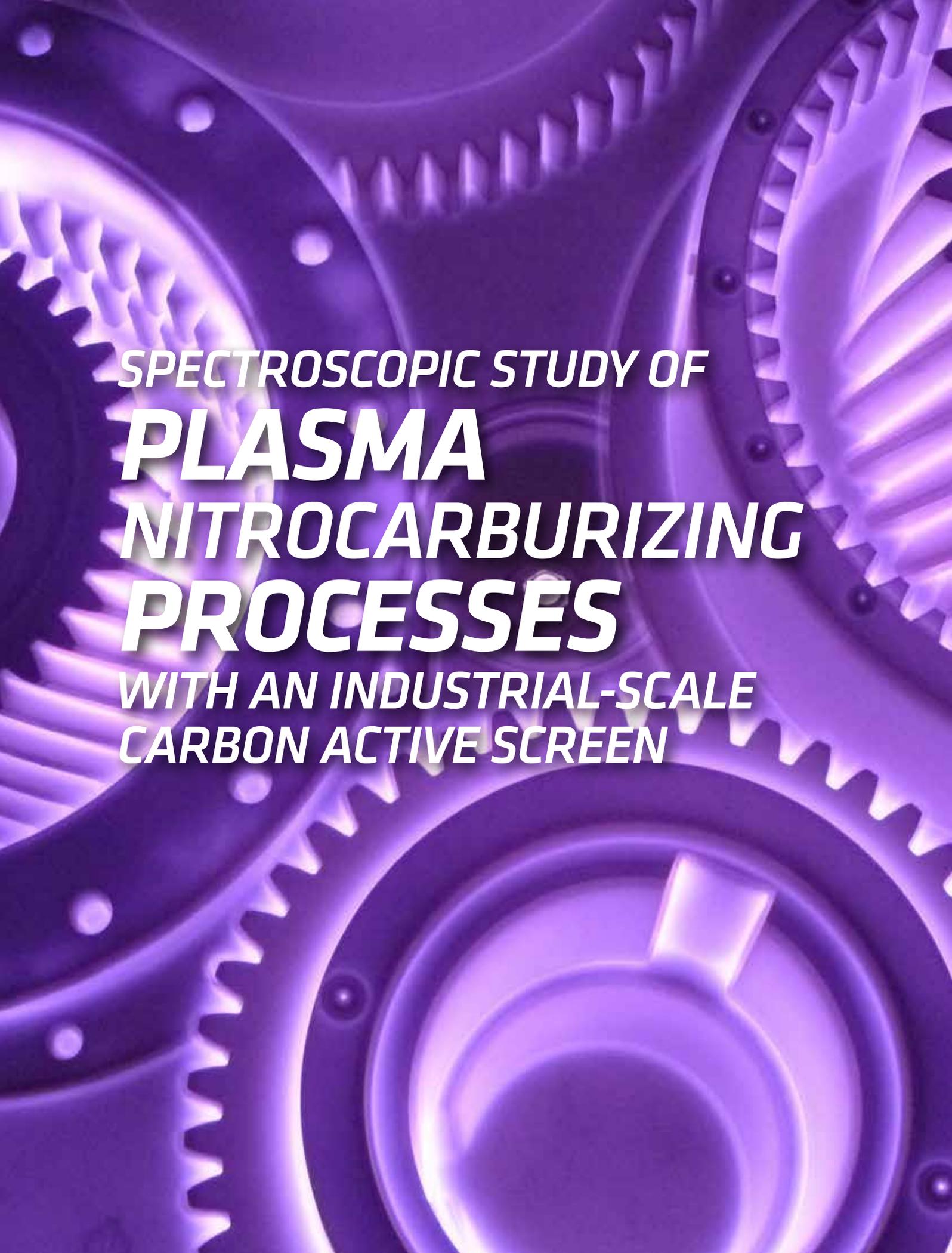
➤ **Strides Forward in Machining and Process Improvement (Day 1, Track 2, 1 p.m.).** This Innovation Spotlight is an opportunity for ceramic manufacturers and raw material suppliers to learn about improvements in the field of machining and processing. There is always room for innovation and for implementing new and exciting manufacturing methods. This can save time, cost, and improve efficiency. Whether it is machining, sintering, debinding, etc., this is the session to find out what the competition is up to. With three expert speakers presenting on their cutting-edge findings, this session will cover improvements to processing and sintering, the cost efficiency of ceramic production methods, and the benefits of these changes to the end product.

➤ **Ironing out the Wrinkles: Addressing the Challenges of Integrating Ceramic Matrix Composites (Day 2, Track 1, 1 p.m.).** The last five years have seen the growing use of ceramic matrix composites (CMCs) in industry. The benefits of these hybrid materials over sometimes inferior metal counterparts have undeniable benefits when it comes to thermal management, strength and efficiency. Yet, there are still many challenges to be resolved before they can be applied in a number of areas. We are already seeing CMCs being used in the aerospace industry, and they are expanding to energy harvesting applications also. This session will explore the challenges of CMC manufacturing and explore some of the latest applications for these unique materials. ☞



DETAILS

Ceramics Expo is scheduled for September 22-23 in Cleveland, Ohio. Register online for free at: ceramicsexpousa.com



SPECTROSCOPIC STUDY OF
PLASMA
NITROCARBURIZING
PROCESSES
WITH AN INDUSTRIAL-SCALE
CARBON ACTIVE SCREEN

In this study, the absolute concentrations of CH₄, C₂H₂, HCN, CO, and NH₃ were monitored in dependence on the total gas flow of the feed gas and the gas pressure in the reactor.

By A. PUTH, L. KUSY'N, A.V. PIPA, I. BURLACOV, A. DALKE, S. HAMANN, J.H. VAN HELDEN, H. BIERMANN, and J. RÖPCKE

The active screen plasma nitrocarburizing technology is an improvement of conventional plasma nitrocarburizing by providing a homogeneous temperature distribution within the workload and reducing soot formation. In this study, an industrial-scale active screen (AS) made of carbon-fiber-reinforced carbon serves as the cathode as well as the carbon source for the plasma-chemical processes taking place. The pulsed dc discharge was maintained at a few mbar of pressure while simultaneously being fed with a mixed gas flow of hydrogen and nitrogen ranging from 10 to 100 slh. Using *in situ* infrared laser absorption spectroscopy with lead-salt-tunable diode lasers and external-cavity quantum cascade lasers, the temperatures and concentrations of HCN, NH₃, CH₄, C₂H₂, and CO have been monitored as a function of pressure and total gas flow. To simulate industrial treatment conditions, the temperature of the sample workload in the center of the reactor volume was kept at 773 K by varying the plasma power at the AS between 6 and 8.5 kW. The resulting spectroscopically measured temperatures in the plasma agreed well with this value. Concentrations of the various species ranged from 6×10¹³ to 1×10¹⁶ cm⁻³ with HCN being the most abundant species.

1 INTRODUCTION

In the field of thermal treatments of materials, nitriding and carburizing and the combination of both, called nitrocarburizing, are standard practices to improve the wear and corrosion resistance of steel components. The basic mechanism for this improvement is the diffusion of nitrogen, carbon, or both, respectively, at temperatures of up to 823 K into the lattice structure of the material, which typically expands and thus forms expanded austenite or the so-called s-phase. Due to the expansion, the material is locally hardened, generating hardness profiles that correspond to the diffusion depth from the interface. The benefit of the combination treatment of nitrocarburizing is a smoother transition of hardness and thus typically a higher load-bearing capacity [1]. While the diffusion mechanism can also be achieved using gas or salt-bed reactors, the use of plasma nitrocarburizing (PNC) has the advantage of a lower environmental impact at a reduced processing time [2]. In part, this is due to an in-process activation of the diffusion interface by removal of the oxide layer. However, current industrial implementations of the PNC technology still are limited in their variation of the carburizing potential as the carbon content within the process is regulated using carbon-containing admixtures to the feed gas. This can lead to oversaturation of the process atmosphere, in turn causing soot production and generation of cementite in the compound layer [3]. So far, process control in industrial applications is based on the operator's empirical experience. Already in 1997, Mittermeijer and Somers published about the prospect of a general nitriding process control with process control for PNC being far out of reach [4].

Current industrial applications of plasma-assisted nitrocarbu-

rizing are commonly conducted by conventional plasma nitrocarburizing. Therein the workload is negatively biased relative to the grounded reactor walls. At a few mbar of pressure, a pulsed dc glow discharge is maintained, producing reactive species from the feed gas of H₂, N₂, and carbon-containing species. The plasma heats the workload to a temperature between 673°K and 823°K, thus enabling diffusion processes to take place. However, depending on the workload geometry, the temperature distribution may not be homogeneous and therefore causes uneven treatment progress, the so-called edge effect [5]. The plasma may as well sputter the workload surface, requiring post-treatment polishing.

These drawbacks have led to the development of the active screen plasma nitrocarburizing (ASPNC) technology. In this approach, the glow discharge is placed at an intermediate steel screen, called active screen (AS), which surrounds the workload and allows a gas flow to pass from the plasma region to the workload providing the reactive species required for the hardening process. Even though, for industrial-scale reactors, a secondary plasma directly at the workload is still required to achieve a reasonable nitrocarburizing response, the secondary plasma is operated at a fraction of the power of the AS plasma. As a consequence, thermal inhomogeneity and sputtering are reduced [6–8].

Several studies and proposals already discussed admixtures to the feed gas to affect the process conditions and treatment results [9–11]. However, with the AS being introduced as a cathode in a reactive plasma, it can be sputtered and thus serves as an additional solid chemical source. To investigate the mechanism of nitriding with an ASPNC reactor, Hubbard *et al* studied the mass transfer from an AS made of steel to the workload and found that sputtered material does not contribute to the nitriding of the substrate [6]. Independent on this study, other groups investigated co-alloys to the AS, such as aluminum and silver, to produce e.g. antimicrobial surfaces, again via the mass transfer from AS to the workload [12, 13]. A different approach is the substitution of steel as screen material with a solid made of carbon, such as graphite or carbon-fiber-reinforced carbon (CFC). The use of a solid carbon source had been first proposed by Lebrun *et al* and realized by Crespi *et al* for CN_x deposition on a polymer substrate [14, 15]. The application for ASPNC had then been reported by Burlacov *et al* and Hamann *et al* in 2017, for industrial- and laboratory-scale reactors respectively [16, 17]. In both works, it has been found that an AS made of CFC affects the process atmosphere most drastically in the concentrations of HCN and C₂H₂, which increased by a factor of 30 and 70 respectively compared with 1% admixture of CH₄ in the H₂-N₂ feed gas typical for ASPNC processes using a steel AS. This is of particular interest since beneficial diffusion conditions created by the highly reactive HCN molecule were first reported in the early 1990s [18, 19]. In a later study, Burlacov *et al* identified the concentration of HCN as a control parameter for the carburizing potential of the nitrocarburizing process [20].

To monitor the concentrations and temperatures of HCN and further species, non-intrusive *in situ* diagnostics are required. In the past, spectroscopic methods such as optical emission spectroscopy (OES), Fourier transform spectroscopy in the infrared, and laser absorption spectroscopy (LAS) have been used for this purpose [16, 17]. Therefore, LAS is exemplary due to the high-quality quantitative information and high sensitivity it offers. Depending on the selected laser source, different species are available for detection. In previous studies, lead-salt diode lasers (TDL) have been used to measure the transient CH_3 radical as well as CO , CO_2 , C_2H_4 , and C_2H_6 [21]. During the early 2000s, external-cavity quantum cascade lasers (EC-QCL) became commercially available and found widespread application. While technologically limited in their achievable spectral range, they tuned over a much broader range than TDLs, allowing a near-simultaneous multi-species detection. In the study of ASPN processes, both types of sources were extensively used in combination with OES and other spectroscopic techniques. This way, 14 species were detected within the plasma process, and, as mentioned before, partially linked with the treatment behavior [17, 20, 22, 23].

Some of these measurements were conducted at the laboratory-scale plasma nitriding monitoring reactor (PLANIMOR) [24]. This reactor was specifically constructed to simulate the plasma-chemical conditions present in an industrial-scale plasma nitriding process, while simultaneously offering improved access for diagnostics, including spatially resolved LAS measurements of the afterglow region. A comparative study between PLANIMOR and an industrial-scale reactor has proven the general similarities in respect to the plasma-chemical behavior of both types [25]. Consequently, the plasma-chemical similarities also are reflected in the nitriding treatment of samples in both systems, resulting in a comparable thickness of the compound layer.

Recently, the results of investigations on plasma processes using an AS made from CFC in PLANIMOR have been published [22]. In this study, the concentrations, temperatures, and conversion efficiencies of nine molecular species were measured as functions of the plasma power at the AS, the pressure, and the nitrogen fraction in the feed gas. Additionally, the carbon mass flow from the AS and the efficiency of the carbon mass flow in relation to the applied power were presented. The present study is a continuation of the former and investigating the plasma processes of an AS in an industrial-scale ASPNC reactor. To find the influence of process pressure and feed gas flow, a constant temperature is maintained

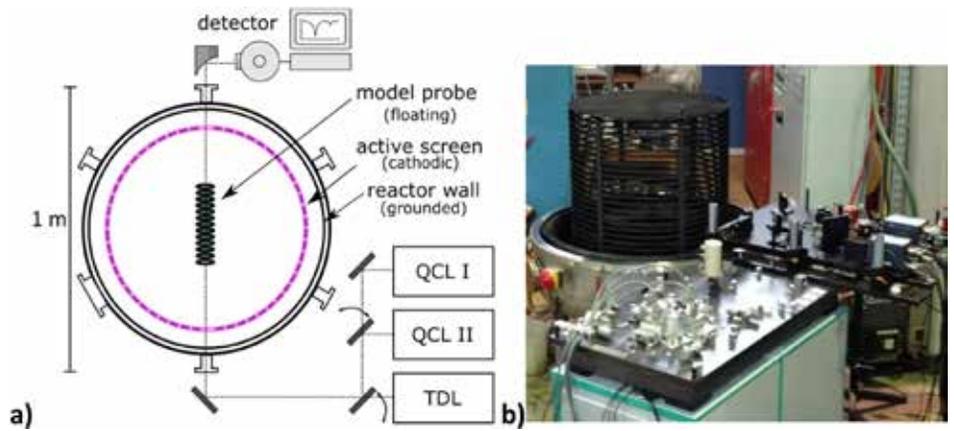


Figure 1: (a) Schematic top view of the experimental setup and (b) photograph of the open reactor (left background), QCLs (right), and IRMA (left foreground). Both QCL and the TDL absorption spectrometer share the same beam path and detector. The beam path passes through the reactor, including the plasma at the AS made of CFC depicted in pink. A scale of the absorption length is given.

Species	Spectral position [cm ⁻¹]	Absorption line strength [cm ⁻¹ /(molecule cm ⁻²)]	Limit of detection [molecules cm ⁻³]	References
$\text{CH}_4^{(Q)}$	1356.4868	1.784×10^{-20}	2×10^{13}	[29]
$\text{CH}_4^{(Q)}$	1356.5974	1.190×10^{-20}	2×10^{13}	[29]
$\text{NH}_3^{(Q)}$	1388.0552	2.726×10^{-22}	2×10^{14}	[30]
$\text{NH}_3^{(Q)}$	1767.5181	6.090×10^{-21}	2×10^{13}	[30]
$\text{C}_2\text{H}_2^{(Q)}$	1356.8305	5.899×10^{-22}	5×10^{14}	[31]
$\text{C}_2\text{H}_2^{(Q)}$	1356.8881	8.920×10^{-21}	2×10^{13}	[31]
$\text{HCN}^{(Q)}$	1356.9389	4.636×10^{-23}	4×10^{14}	[32]
$\text{HCN}^{(Q)}$	1388.3225	3.592×10^{-22}	1×10^{14}	[33]
$\text{CO}^{(T)}$	2150.3409	1.840×10^{-21}	2×10^{11}	[34]
$\text{CO}^{(T)}$	2150.8560	1.826×10^{-19}	2×10^{13}	[34]

Table 1: Species, spectral positions, and line strengths, at room temperature, used for infrared LAS measurements and their estimated limits of detection. The laser source for the given spectral position is denoted with a Q for an EC-QCL and a T for TDL sources. Data taken from the HITRAN database [28].

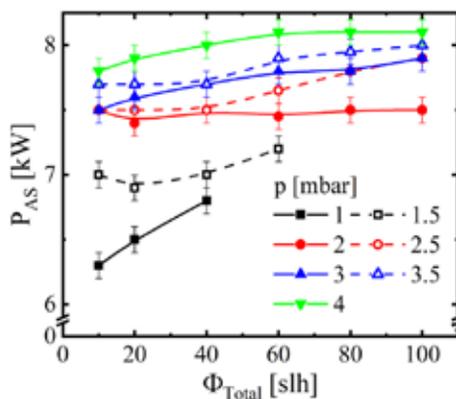


Figure 2: Plasma power at the active screen P_{AS} at different pressures measured as a function of the total gas flow. Conditions: $\text{H}_2:\text{N}_2 = 1:1$, $T_{set} = 773 \text{ K}$. Lines are guides for the eye.

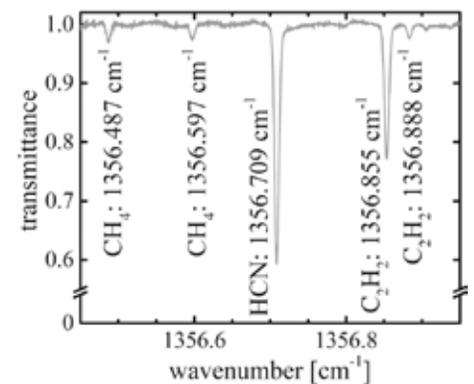


Figure 3: EC-QCL absorption spectrum of CH_4 , HCN and C_2H_2 around 1356.7 cm^{-1} . Conditions: $\text{H}_2:\text{N}_2 = 1:1$, $\Phi_{Total} = 80 \text{ slh}$, $T_{set} = 773 \text{ K}$, $p = 3 \text{ mbar}$.

inside the reactor while the concentrations and temperatures of HCN, NH_3 , CH_4 , C_2H_2 , and CO were measured using EC-QCLAS and TDLAS.

The current study focuses on the production rate of the five molecular species, their dependence on the gas flowrate, and finally, the scalability of the concept for an AS made of carbon materials. Further

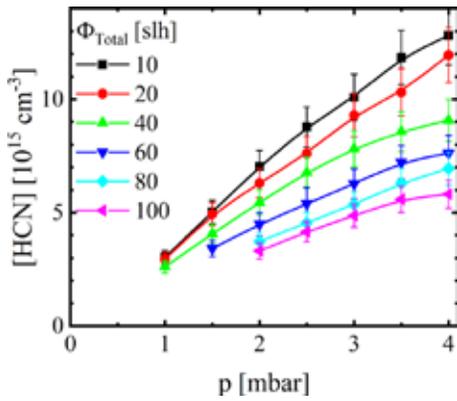


Figure 4. Concentration of HCN at different total gas flows measured as a function of the gas pressure. Conditions: $H_2:N_2 = 1:1$, $T_{set} = 773$ K. Curves are guides for the eye.

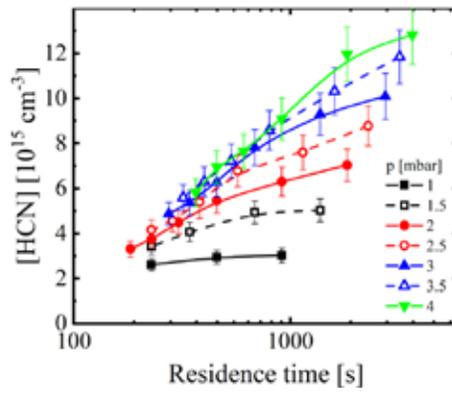


Figure 5. Concentration of HCN at different gas pressures measured as a function of the residence time. Conditions: $H_2:N_2 = 1:1$, $T_{set} = 773$ K. Curves are guides for the eye.

observations concern the sole detected oxygen-containing species CO as a measure of contamination with oxygen, since previous studies by Burlacov *et al* pointed out the importance of contamination in industrial-scale reactors [20].

2 EXPERIMENTAL

A crosscut schematic of the industrial-scale reactor with the LAS setup including three laser sources is shown in Figure 1. As indicated by the presence of two flipping mirrors, the laser sources are co-aligned and cannot be used simultaneously. Furthermore, two Daylight Solutions EC-QCL, labelled as QCL I and QCL II, provide mode-hop free spectral ranges of $\nu_I = 1,345\text{-}1,400$ cm^{-1} and $\nu_{II} = 1,770\text{-}1,850$ cm^{-1} , respectively [26]. In addition, the infrared multi-component acquisition system (IRMA) [27] has been used, which combines three lead-salt tunable diode laser (TDL) sources to a single acquisition system. Table 1 presents a list of detected species and their respective spectral positions, line strengths, and limits of detection, whereby the superscript Q and T designate the measurement with an EC-QCL or a TDL source respectively. The reference and etalon spectra of the EC-QCLs were measured simultaneously using a three-channel setup. For the TDL sources, they were measured separately.

The shared beam path enters and exits the reactor through two KBr windows at a distance of 1 meter, which is assumed identical to the length of absorption. A model sample composed from 26 equidistant punched discs is placed in the center of the reactor, aligned to the beam path. It serves to simulate the influence of a workload for nitrocarburizing and can be biased for this purpose. Finally, a liquid nitrogen cooled HgCdTe detector positioned at the focus of an off-axis parabolic generates an analogue signal for data acquisition.

The reactor itself has a cylindrical volume of approximately 1 m^3 , at a diameter of 1 meter. It contains the AS made of CFC with a diameter of 0.8 meters and a height of 0.75 meters. Besides the top plate, the screen is composed of circular sections made of CFC each covering 60° of the cylinder's perimeter with 20 mm radial depth, distanced 20 mm in height between each section. The thickness of the CFC base material is 5 mm. All components included, the AS weighed approximately 30 kg. For plasma generation, a pulsed DC power supply with a maximum power of $P = 15$ kW at a frequency of $f = 1$ kHz and a duty cycle of 60% is connected with the AS. The steel reactor wall is grounded, and the model probe is on a floating potential. During the measurement, the system regulated the power at the AS between 6.0 and 8.5 kW to maintain a stable temperature of $T_{set} = 773$ K, measured with a thermal probe integrated in the

model probe. The plasma power at the AS as a function of the total gas flow for different pressures is shown in Figure 2. At the lower limit of the pressure range, the discharge was not stable, reflected by a reduced power consumption.

The precursor composition is controlled by mass flow controllers. In turn, the feed-gas is led into the reactor via a showerhead integrated into the reactor top. This way H_2 , N_2 , and Ar were introduced, the latter only during starting process of the reactor to provide a stable plasma until the chosen treatment temperature was reached. The total mass flow varied from $\Phi_{Total} = 10\text{-}100$ slh, at a mixing ratio $H_2:N_2$ of 1:1. The pumping system is connected to the bottom of the reactor, including a butterfly valve allowing

the control of gas pressure in the range of $p = 1\text{-}4$ mbar.

An EC-QCL absorption spectrum in the spectral range near 1356.7 cm^{-1} with specified absorption lines assigned to CH_4 , HCN, and C_2H_2 recorded with EC-QCLAS is shown in Figure 3. The conditions were as follows: $\Phi_{Total} = 40$ slh $H_2 + 40$ slh N_2 , $T_{set} = 773$ K, $P_{Bias} = 0$ W, $p = 3$ mbar. For the determination of the temperature, line profile analysis has been used to obtain the translational temperature, associated with Doppler broadening. The laser linewidth, determined at a room temperature reference, was taken into account. We calculated concentrations with Beer-Lambert law using temperature dependent line information from the HITRAN database [28] and derived mole fractions with the ideal gas law. The production rate $r_{Production,i}$ of a species i is given by

$$r_{Production,i} = n_{mole,i} \cdot \Phi_{Total}, \quad \text{Equation 1}$$

whereas $n_{mole,i}$ is the mole fraction of the species i and Φ_{Total} is the total gas flow through the reactor with the unit slh. We assume that the plasma-chemical reactions are in a steady state of equilibrium. The rate of production has the unit of slh. The carbon mass flow Φ_C is given by

$$\Phi_C = \sum_i r_{Production,i} \cdot N_{C,i}, \quad \text{Equation 2}$$

with $r_{Production,i}$ being the production rate of species i in slh, $N_{C,i}$ being the number of carbon atoms within species i and the sum including all species in the process. The carbon mass flow has natively the unit of slh, which is converted to $mg\ h^{-1}$ using the atomic mass of carbon and the ideal gas law to better relate it with the macroscopic mass of the AS. Finally, the carbon consumption efficiency E_{CC} is given by

$$E_{CC} = \frac{\Phi_C}{P}, \quad \text{Equation 3}$$

whereas Φ_C is the carbon mass flow in $mg\ h^{-1}$ and P is the plasma power at the AS. Accordingly, ECC has the units of $mg\ h^{-1}\ W^{-1}$. Additionally, the overall conversion X_i of a species i is given by

$$X_i = \frac{\sum_j n_j \cdot N_{i,j}}{n_i}, \quad \text{Equation 4}$$

with n_i being the concentration of species i , meanwhile $N_{i,j}$ is a proportionality factor for correlation of the resulting species j to the feed gas species i . The right hand side of the equation chain is equivalent to Equation 2 with the consideration that the feed gas species are diatomic molecules. The concentration has the unit of cm^{-3} .

3 RESULTS AND DISCUSSION

Due to the temperature dependence of the absorption line strength, the temperature has been determined for the available species using line profile analysis. For the transitions of the available species HCN, CH₄, CO, NH₃, and C₂H₂ the line profiles correspond to the set process temperature of $T_{\text{set}} = 773$ K. Apart from that, a thermal equilibrium between rotational and translational temperatures is assumed. Even if the rotational temperatures are not directly available in this study, in [35] comparable values for the process temperature measured at the model probe and for the rotational temperature measured in the plasma at the steel screen have been found. However, it should be noted that LAS is a line-of-sight method allowing only line-averaged results and as such does not allow any spatially resolved information in the used geometry. Thus, the active plasma zone at the carbon screen may have a higher temperature or even no thermal equilibrium at all.

Using the line strengths for 773 K, the absolute concentrations of the detected species have been determined. To demonstrate the chronological order of measurement, the absolute concentration of HCN at different gas flows measured as a function of pressure is presented in Figure 4. The measurements were conducted at a set gas flow, varying the pressure until moving on to a new set gas flow starting with a total flow of 10 slh. As the pressure increases, the concentration of HCN increases linear within the margin of error from 2.5 to $14 \times 10^{16} \text{ cm}^{-3}$ in case of the lowest gas flow. On the other hand, increasing the total flow decreases the concentration from 14 to $6 \times 10^{16} \text{ cm}^{-3}$ at the pressure of 4 mbar. At lower pressures, the decrease is less pronounced, and in some low pressure and high total gas flow settings no stable discharge was achieved.

In Figure 5, the concentration of HCN is shown at different gas pressures as a function of the residence time with a logarithmic x-axis. With increasing residence time, a monotonous increase of the concentration of HCN can be seen at every pressure, typically with a steeper increase at higher pressure. Due to the high reactor volume, the residence time ranges in the minutes. Since the concentration of HCN is dependent on the resident time – even at high values of the residence time, one has to assume that surface reactions are contributing to the production of HCN. Similar increases were observed for the concentrations of C₂H₂, CH₄, and CO, with only the concentration of NH₃ decreasing with residence time. Accordingly, for the generation of all detected species, surface processes are non-negligible.

Subsequent figures present the mole fractions in dependence on the total gas flow. Accordingly, Figures 6a-6e shows mole fractions

The release of adsorbed oxygen-containing species from the large surface area of the carbon cathode during the plasma process is the likely source for oxygen in the formation of the CO molecule.

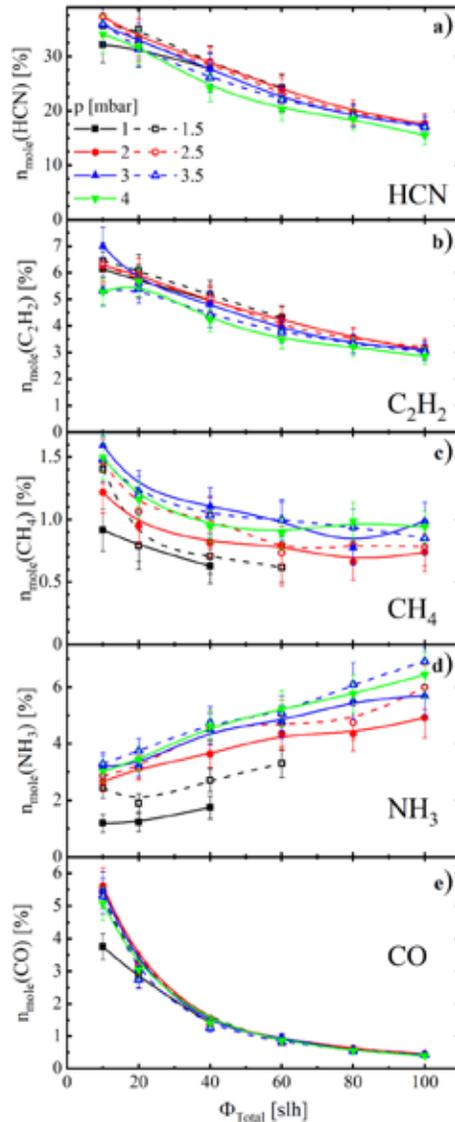


Figure 6: Mole fractions of (a) HCN, (b) C₂H₂, (c) CH₄, (d) NH₃, and (e) CO at different gas pressures measured as a function of the total gas flow. Conditions: H₂:N₂ = 1:1, $T_{\text{set}} = 773$ K. Curves are guides for the eye.

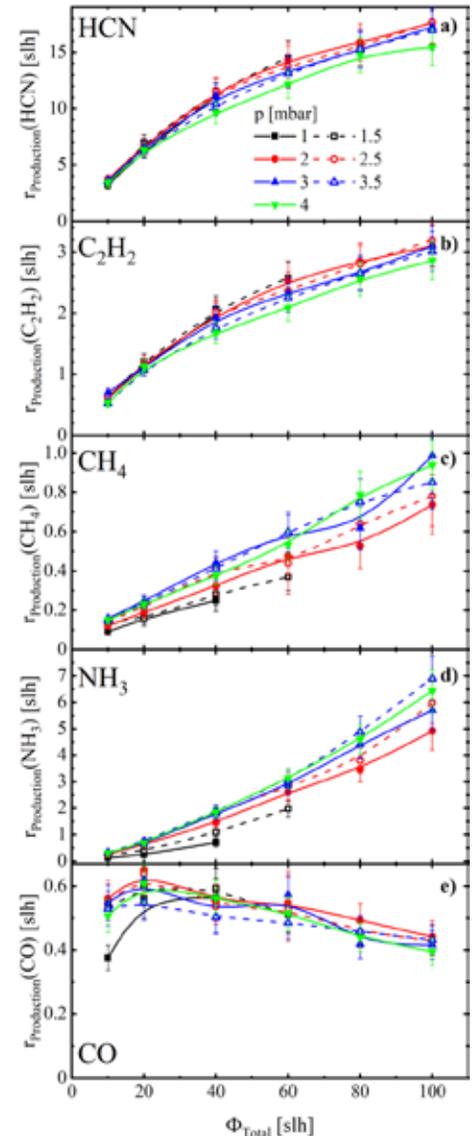


Figure 7: Production rates of (a) HCN, (b) C₂H₂, (c) CH₄, (d) NH₃, and (e) CO at different gas pressures measured as a function of the total gas flow. Conditions: H₂:N₂ = 1:1, $T_{\text{set}} = 773$ K. Curves are guides for the eye.

of HCN, C₂H₂, CH₄, CO, and NH₃ measured as a function of the total gas flow for different pressures. As the total gas flow increases, the mole fractions of HCN, C₂H₂, CH₄, and CO decrease. Concerning HCN and C₂H₂, the respective mole fractions share very similar trends. Accordingly, the ratios of the two mole fractions, which is equal to the ratios of the two concentrations, can be approximately given as $[\text{C}_2\text{H}_2]:[\text{HCN}] \approx 0.17$ at all gas flow settings. Both mole fractions, as well as the mole fraction of CH₄, decrease by a factor of 2 between

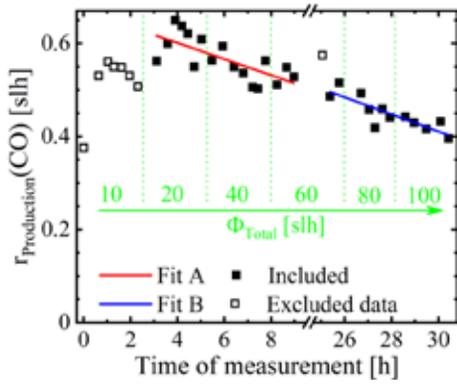


Figure 8: Production rate of CO as a function of the time of measurement. During the indicated break in the x-axis, no plasma was applied, and the reactor was actively evacuated. The stepwise increased gas flowrate is indicated in green, as well as two linear fits before and after the break.

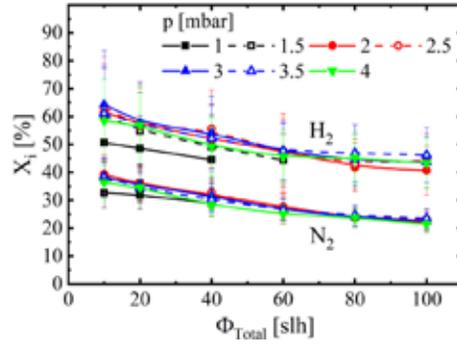


Figure 9: Conversions X_{H_2} and X_{N_2} at different gas pressures as a function of the total gas flow. The higher set of curves is associated with H_2 , the lower for N_2 . Conditions: $H_2:N_2 = 1:1$, $T_{set} = 773$ K, $P_{Bias} = 0$ W.

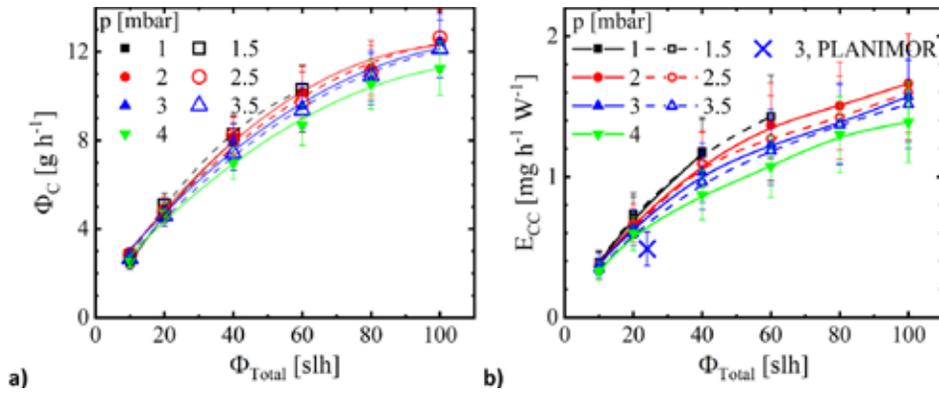


Figure 10: (a) Carbon mass flow Φ_C and (b) carbon consumption efficiency E_{CC} measured at different pressures as a function of the total gas flow. Conditions: $H_2:N_2 = 1:1$, $T_{set} = 773$ K, $P_{Bias} = 0$ W. In (a) second order polynomial fits are used as guides for the eye. In (b) the carbon consumption efficiency at 3 mbar in PLANIMOR [22] is added with a x as comparison. Curves are guides for the eye.

a total flow of 10 and 100 slh. However, the mole fraction of CH_4 reaches a plateau at 60 slh of total gas flow. Furthermore, the mole fraction of CO decreases by approximately a factor of 10 within the same range of the total gas flow, from 5.5% to 0.5%. In that relation, only NH_3 is an outlier of the five species, its mole fraction increasing linearly with the total gas flow by a factor of 2 over the full range of the total gas flow. The mole fractions of HCN, C_2H_2 , and CO indicate only a weak or no dependency on pressure, while those of CH_4 and NH_3 show a clear dependency on pressure. The mole fractions of both species increase with growing pressure.

For further analysis of the data, the production rates at stationary conditions of each molecular species were determined by applying Equation 1. The production rates of HCN and C_2H_2 for different pressures measured as a function of the total gas flow are shown in Figures 7a and 7b respectively. While both are independent on the pressure and increase with the total flow, the slopes decrease with higher total gas flow values. For the production rate of CH_4 in Figure 7c, a nearly linear increase with the gas flowrate can be identified, while the production rate of NH_3 , shown in Figure 7d, increases over proportionally with the gas flow. However, the production rate of CO, as shown in Figure 7e, decreases with the total gas flow with the exception of the lowest gas flow setting. It is independent on pressure. Since we supply no oxygen-containing species, the two available sources for CO formation can be wall attachment and leakage, with the wall attachment contribution typically decreasing with time.

Figure 8 shows the production rate of CO as a function of time of measurement. The measurement was taken in two sessions; the first session concluded after circa 9 hours and the second session began at 25 hours as indicated by the break in the time axis. During the break, no plasma was active; the total gas flow was stopped, and the reactor chamber was actively evacuated. From data point to data point, the pressure was changed; the stepwise increase of the total gas flow is indicated in green. With the exception of the lowest total gas flow setting, the production rate of CO decreases linearly from 0.6 to 0.4 slh during the active time of the plasma. Even the break has little influence on the trend, as indicated by the good continuity of two linear fits, Fit A before and Fit B after the break. Therefore, the most likely oxygen source is the contamination of the reactor wall and the AS with oxygen-containing species. With CFC being a highly porous material, this assumption is well founded. The observed dependency of the production rate of CO on the total gas flow, as shown in Figure 7e, should therefore be interpreted as a dependency on the active time of the plasma. Since the measurement procedure created a direct correlation between the active time of the plasma and the total gas flow, deriving a pure dependency of the production rate of CO on the total gas flow is impossible.

Focusing again on the intentionally admixed feed gas, in Figure 9 the conversions of hydrogen, X_{H_2} , and nitrogen, X_{N_2} , are shown at different pressures measured

as functions of the total gas flow, as determined by Equation 4, assuming that only the detected species and the feed gas species are present in the reactor. While measurements at PLANIMOR have shown that other species are created, such as C_2N_2 or CN, their concentrations are negligible [22]. The higher set of curves corresponds to the conversion of hydrogen and the lower set to the conversion of nitrogen. Both set of curves in Figure 9 decay from 62% to 45% and from 35% to 25% respectively, while at the same time showing no dependency on the pressure. Figure 9 also allows to calculate the mole fractions of hydrogen and nitrogen according to

$$n_{mole,i} = \frac{(1 - X_i)}{2}, \quad \text{Equation 5}$$

with $n_{mole,i}$ being the mole fraction of species i . One can see the decrease of the mole fraction of the most abundant species HCN with increasing total gas flow being reflected in a decreased conversion of both hydrogen and nitrogen. This is only partially compensated by an increased mole fraction of NH_3 , resulting in a larger mole fraction of both hydrogen and nitrogen at high total gas flow compared with a low total gas flow. In order to explain this trend, the third constituent of HCN, i.e. carbon, was investigated.

The detected species can be separated in carbon-containing species such as HCN, C_2H_2 , CO, and CH_4 , and carbon-free NH_3 . Consequently, the carbon mass flow is determined by using Equation 2, as shown in Figure 10a for different pressures measured as a function of total

gas flow. The carbon mass flow reflects the consumption rate of the CFC cathode of finite mass, as previously shown at PLANIMOR [22]. It can be assumed that no carbon-containing species of noteworthy concentrations remained undetected. Since HCN is the most abundant carbon-containing species, the trends of the carbon mass flow are similar to those of the absolute production rate of HCN, increasing with the total gas flow but being independent on the pressure. The slope of the carbon mass flow in dependency on the total gas flow decreases at high values of total gas flow, indicating a limit to the release of carbon.

Such a limit to the release of carbon may explain the trends of the production rates as seen in Figure 7. At low total gas flow settings, the release of carbon is sufficient to create large amounts of carbon-containing species. In turn, these carbon-containing species bind a large fraction of the available hydrogen and nitrogen, leading to lowered concentrations of NH_3 . As the total gas flow increases, the release of carbon can only scale weaker than linear. This limits the production rates of carbon-containing species, thus e.g. the mole fraction of HCN is reduced. Accordingly, the conversions of the feed gas species are lowered, and higher amounts of hydrogen and nitrogen are available for the formation of NH_3 . Finally, the mole fraction of NH_3 increases, as observed in Figure 6d. The production rate of CO is the only outlier of the carbon-containing species, due to its dependence on oxygen concentration, which is not intentionally admixed.

Using Equation 2, the carbon consumption efficiency E_{CC} has been calculated and found to range between 0.13 and 1.9 $\text{mg h}^{-1} \text{W}^{-1}$, as presented in Figure 10b. The trends of ECC strongly correlate with the trends of the total carbon mass flow, as the power only slightly varies for different gas flows and different pressures. For comparison, we can again refer to investigations on the laboratory-scale PLANIMOR [22] where, for a pressure $p = 3$ mbar and a feed gas mixture $\text{H}_2:\text{N}_2 = 1:1$, the carbon consumption efficiency ranged between 0.7 and 0.9 $\text{mg h}^{-1} \text{W}^{-1}$ depending on the power at the AS.

Taking the cross section of both reactor designs into account to compare flow speeds, the mass flow of PLANIMOR of 20 sccm corresponds to a flow of 24 slh in the industrial-scale reactor. The closest measured value of gas flow is 20 slh with a carbon consumption efficiency of $E_{CC} = 0.6 \pm 0.07 \text{ mg h}^{-1} \text{W}^{-1}$, being in quite good accordance. Considering previously established differences in temperature and contamination, a small mismatch can be expected.

4 SUMMARY AND CONCLUSION

In the current *in situ* spectroscopic study, an industrial-scale PNC process with an AS made of carbon-fiber-reinforced carbon was investigated using TDL and EC-QCL sources in the mid-infrared. This way the absolute concentrations of CH_4 , C_2H_2 , HCN, CO, and NH_3 were monitored in dependence on the total gas flow of the feed gas and the gas pressure in the reactor. To approximate common industrial conditions, the plasma power at the AS was regulated to maintain a steady temperature $T_{\text{set}} = 773$ K. The translational temperatures of the five molecular species were found to agree with the set temperature.

The plasma-chemical environment was strongly influenced by reaction products, which accounted for 50%–30% of the mole fraction at low- and high-total gas flow respectively. Furthermore, the mole fraction of C_2H_2 , HCN, and CO were found to be independent on the pressure, while for CH_4 and NH_3 the mole fractions increased with the pressure. Since all the concentrations of all detected species increased monotonously with the residence time, surface reactions should play a role in the production of the five species. HCN was the most abundant measured reaction product, ranging in the 10s of percent in mole fraction and followed by C_2H_2 and NH_3 , both

in the high percent of mole fraction range. Since a stable plasma-chemical equilibrium was assumed, the production rates could be derived from the concentrations of the species. This allowed to distinguish CO from the other species, since its production rate is the only one to drop with the increase of flow. Further investigation then highlighted the linear decay of the CO production rate with the progressing active time of the plasma. The release of adsorbed oxygen-containing species from the large surface area of the carbon cathode during the plasma process is the likely source for oxygen in the formation of the CO molecule. The influence of the oxygen-contamination of the reactor was unique to the CO molecule and made conclusions about the dependency of its production rate on the total gas flow impossible.

However, for the production rates of the remaining four molecular species, dependencies on the total gas flow trends could be established. The dominating production rate is of HCN with up to 18 slh, followed up by the production rate of NH_3 , which increased from 0.25 to 7 slh with increasing total gas flow. While the production rates of all oxygen-free species increase monotonously with the total gas flow, the production rate scales over proportionally only for NH_3 . In addition, it shows the highest relative increase over the given range. For CH_4 , a linear scaling was observed. However, HCN and C_2H_2 production rates flatten with higher total gas flows. This is mirrored in the carbon consumption rate also flattening with higher total gas flows, indicating a limit to the amount of carbon released by the AS. The largest value for the carbon consumption rate in this experiment was approximately $\Phi_C \approx 12 \text{ g h}^{-1}$, which for an AS with a mass of 30 kg allows for 2,500 hours of treatment time, assuming ideal circumstances. However, with the current data, a reduction of screen size is considered for further experiments.

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MTI strives to fulfill its mission of enhancing the image and value of the heat-treating industry through advocacy, training, and business intelligence.

By **KENNETH CARTER**, Thermal Processing Editor

Heat treating is an important and essential part of so many industries, that it only made sense that heat-treaters would see the need to come together to advance their standing and widen their recognition.

A group of seven heat treaters did just that when they met in Detroit, Michigan, in 1933 and formed the Metal Treating Institute, more commonly referred to as MTI.

Since then, the group has been a driving force in the world of heat-treating, helping to enhance the image and the value proposition that heat treaters bring to the marketplace, according to Tom Morrison, CEO of MTI Management.

“The founding group wanted to be able to do the things that we do today, which is, they wanted to be able to coordinate their efforts together, and not have any of those efforts rely on any one person. Then they are able to share those costs, which is what we call our membership dues,” he said. “The key behind all that is, since 1933 we’ve got several strategic plans. When you go back to all of them, you see the same thing. Their key challenges were with government regulation, healthcare costs, costs of doing business, forecasting the future, and things like training, and professional development. MTI was created to be able to do those things effectively and together, for their members. Those are all the marquee things that we still do today.”

ADVOCATING OUTSOURCING

Heat treating is often essential, but it also can be complicated and expensive. That’s why one of MTI’s goals is to advocate outsourcing jobs to commercial heat treaters that are equipped to handle it, instead of attempting to do jobs in-house, according to Morrison.

“When manufacturers have an opportunity to heat treat, the question is: Do you heat treat it in-house, or out of house?” he said. “MTI spends a fair amount of resources in publications and on the web trying to do two things: 1) Educating captives on the benefits of outsourcing heat treating, due to the fact that heat treating is a very capital-intensive environment, with labor, EPA, OSHA, and equipment. So, we spend a lot of time trying to educate captives on the benefits of outsourcing the heat treating to a commercial heat treater. And 2) MTI is heavily involved in performing tasks that are unproductive for members, such as online technical training.”

The recent coronavirus outbreak is a prime example, according to Morrison.

“We’re going out and bringing resources to the table that they don’t have to go out and search for themselves,” he said. “Our focus is safety, quality, training, benchmarking, and getting the most out of our productivity and people.”

FURNACES NORTH AMERICA 2020

One of the more visible functions of MTI is its role in the biannual Furnaces North America tradeshow, and Morrison said this year’s show, scheduled for September 30-October 2 in Louisville, Kentucky, is expected to be packed with a plethora of must-know information in the heat-treat world.



FNA 2020, scheduled for September 30-October 2 in Louisville, Kentucky, will focus heavily on the technical programs and connecting attendees to the latest developments in technology on the show floor. (Courtesy: MTI)

“MTI is the producer and owner of the Furnaces North America show,” he said. “We’ve been doing this so long that it’s really a systematic operation that we have. We have a playbook that we put together, and we utilize our Education Committee, which is generally around 15 commercial heat treaters and suppliers, to develop the technical programming. Then, we have our management team at our office, who coordinates the selling of the booths, and the booths have sold out the last four shows in a row. It’s just a total team effort between our Board, volunteers, and our management team who coordinates all that.”



FNA 2020 will be a good opportunity for the industry to regroup. (Courtesy: MTI)

Morrison stressed that this year's show will focus heavily on the technical programs and connecting attendees to the latest developments in technology on the show floor.

"We just want to bring more people to the show because we have an incredible technical program every single time we do the show," he said. "It's a who's-who list of exhibitors that come to the show. Our focus is on giving people the opportunity to come see the latest in equipment and trends, because disruption is huge in the industry right now. It's moving very fast, but this is one opportunity for you, as a heat treater, whether commercial or captive, to come to one place and see all the latest stuff in equipment, sensors, controls, refractory — you name it, it's sitting on that showroom floor. FNA is very budget friendly because it provides a central place for a heat treater to see the latest and greatest in technology, trends, and all things heat treating. That's what we're really trying to bring to the table — a huge cost and time savings for people looking to upgrade their equipment and see the latest technology."

TECHNICAL SHOWCASES

To get show-goers excited about what's coming to the show, MTI is developing technical showcases over the next six months, according to Morrison.

"They're going to be seven-minute online interviews with some of the top people offering equipment and sensors, controls, and those kinds of things, sharing with potential attendees about what they can expect when they come to the show," he said.

The FNA show will be a good opportunity for the industry to regroup once the coronavirus runs its course, according to Morrison.

"It's obviously a blip on the screen," he said. "It's here; it's real; it's serious. It's going to come, and it's going to go just like 2008 and 2009 came and went. Somewhere soon, we're going to see some kind of shift, and things are going to hopefully get back to normal. We just have to be ready to serve our members with excellence when normal returns."

Morrison said that economists were already predicting an eco-



MTI is the producer and owner of the Furnaces North America show. (Courtesy: MTI)

a healthy marketplace.”

MILLENNIAL INVOLVEMENT

Part of that healthy marketplace, according to Morrison, will be led by millennials.

“Here’s what’s going to drive it: The demographic groups are going to drive it because you have the millennials who make up 100 million people,” he said. “Right now, they’re between the ages of 8 and 33. In the next 10 years, they’ll be growing into their high-earning years. That’s what’s going to drive growth.”

And record numbers of buying power will mean products will need to be heat treated, according to Morrison.

“The next eight to 10 years should be pretty healthy for just about anybody, in any industry, because you have a record number of people who will be in their consumable years,” he said. “This means record numbers of houses, cars, cellphones, computers, recreational equipment, medical devices — I mean everything should be record numbers because you’ve got a record number of people who are going to be in the economy consuming in the next 10 years.”

INDUSTRY 4.0

To keep disruptions in production at a minimum, Morrison said that businesses will need to take advantage of the growing need for the Internet of Things and other areas of Industry 4.0.

“It’s imperative that companies try and figure out how they can automate as much of their processes as soon as they can,” he said. “Between changes in the marketplace and things like the coronavirus, the more you’re automated, the easier it is to dial back production. When you’ve got 100 employees, it’s much more difficult to dial back than someone with 50 employees or less.”

That’s something Morrison hopes FNA attendees will be able to take away.

“Coming to the show, they’re going to be able to see how they can automate information flows, production flows, and operational flows,” he said. “People who figure out how to implement Industry 4.0 and the Internet of Things into their marketplace and into their operations are going to be the big winners in the next 10 years.”



MORE INFO www.heattreat.net and www.furnacesnorthamerica.com



“Coming to the show, they’re going to be able to see how they can automate information flows, production flows, and operational flows.”

conomic pinch in 2022 or 2023, but he stressed that the current economic environment might be the course correction.

“We feel like this could be that correction happening now,” he said. “We should see a nice eight- to nine-year run in the economy where heat treating’s going to be very prevalent and be very lucrative and a good marketplace. So, we expect the next 10 years to be

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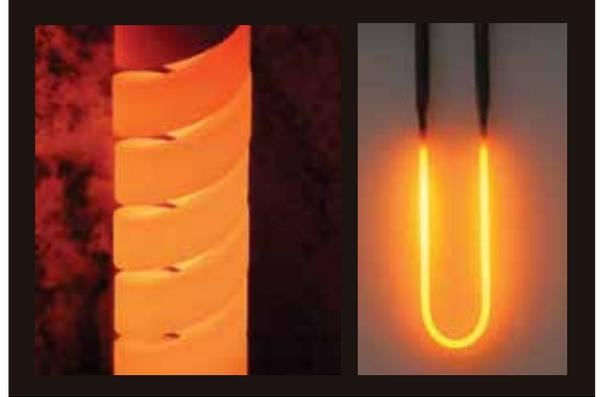
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Q&A /// INTERVIEW WITH AN INDUSTRY INSIDER

MICHAEL HAGER /// BUSINESS UNIT MANAGER /// VERDER SCIENTIFIC/CARBOLITE GERO

“We went back to the drawing board and completely redesigned our entire tube furnace line.”

What’s a typical day like for you at Verder Scientific/Carbolite Gero?

Our office here in North America is primarily focused on sales, technical support, and service for Verder Scientific products. So really, a typical day for the Carbolite Gero team in Pennsylvania is supporting our customers, providing quotations, answering technical questions, and shipping. We provide custom equipment as well, and that offers its own challenges. We ensure that the specifications for these custom units are clear, while ensuring the engineers and end-users are on the same page. We also provide onsite and web-based trainings.

One thing that has always really impressed me about Verder Scientific and Carbolite Gero is how they strive to improve the product and improve the knowledge base about the product, both with end-users and Verder Scientific representatives worldwide.

What role does Verder Scientific play in the heat-treat industry?

Verder Scientific has been around for more than 80 years making furnaces and ovens. We offer everything from small bench top laboratory fan convection ovens, all the way up to large industrial scale vacuum chamber furnaces.

The new Carbolite Gero tube furnaces are offered in a comprehensive range of temperatures and sizes. (Courtesy: Verder Scientific)



Tell us about your new tube furnaces.

We went back to the drawing board and completely redesigned our entire tube furnace line. The tube furnaces have been redesigned from the ground up. The design and engineering teams have really done something special with these and they have completely blown me away. All the tube furnaces now are manufactured with a modular design. We can now provide one kit that can operate horizontally or vertically. All of the furnaces can be removed from the control cabinet and are on a two-meter conduit, so there’s flex-

ibility with regards to mounting. In our previous design, systems had to be specifically configured for the way you wanted it mounted. That is no longer an issue.

All of the systems now have quick disconnect connectors to disconnect the furnace from the control box. The furnace can be disconnected easily without opening the control cabinet. This has some excellent advantages for service.

Increasing the ease of service was also a strong focus of the redesign. There is a singular metal tray inside of the unit and all of the electrical components are actually attached to that tray. By removing several screws, the entirety of the electrical components can be removed from the back of the control box, giving full access. We have also changed the way the thermocouples are oriented inside the system to allow those to be more easily replaceable.

We are also expecting a significant reduction in lead time for these systems as a result of the redesign.

The new Carbolite Gero tube furnaces are offered in a comprehensive range of temperatures and sizes. They range from a 32mm outer diameter, all the way up to 200mm outer diameter and are available in temperatures up to 1,600°C.

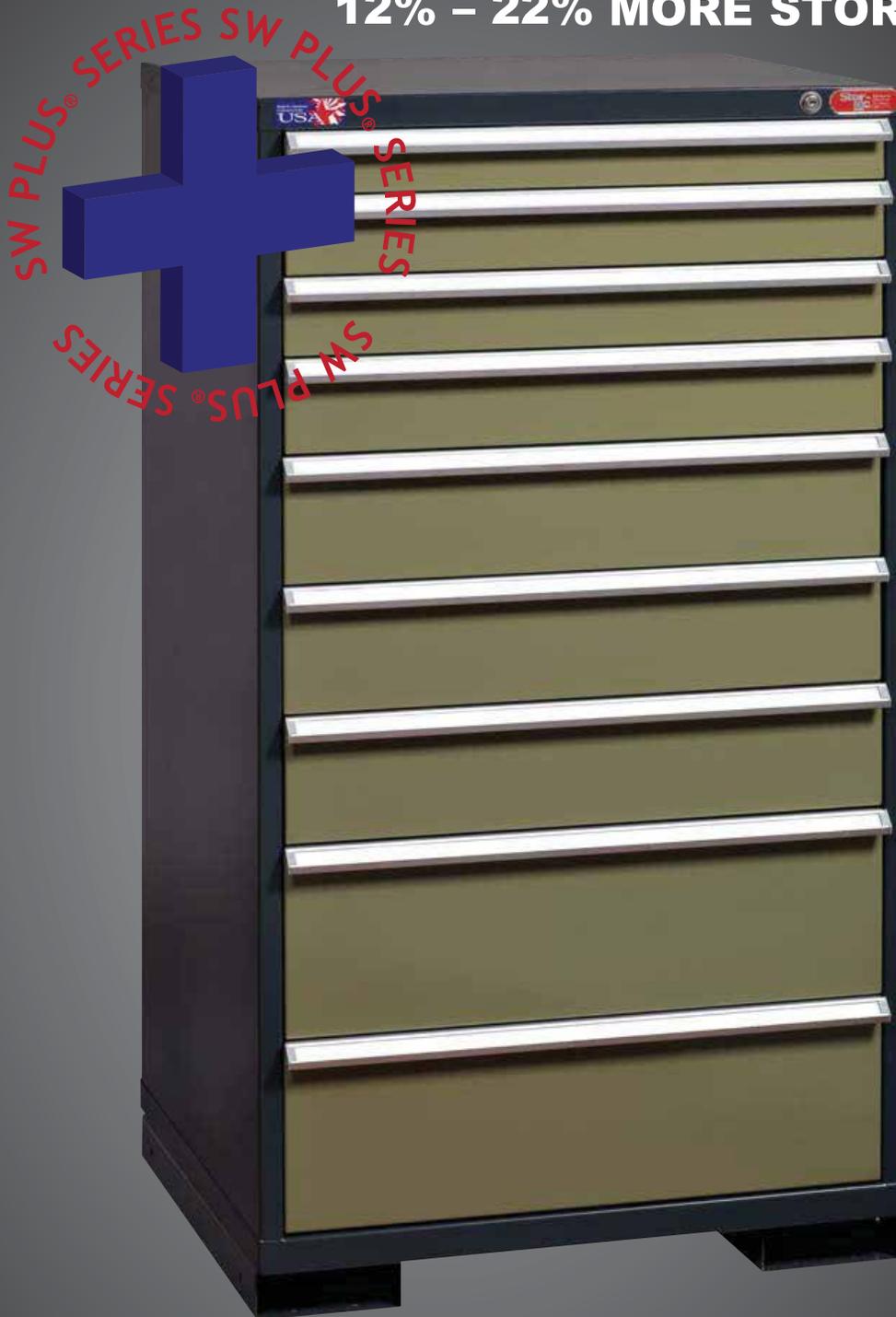
The standard tube furnaces now come standard with Ethernet and a digital programmable controller. We are offering the new CC-T1 on these units. The CC-T1 is a small touch screen controller that has integrated data logging, and a visual map of the temperature profile. We have taken advantage of modern-day electronics to improve functionality and ease of use.

What kind of industry response have you gotten so far?

We’re now quoting the redesign tube furnaces. They’re available for purchase worldwide. The industry response so far has been excellent. We have had positive feedback on the look and on the increased functionality. ♣

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