

Technologies and Processes for the Advancement of Materials

Thermal processing

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INDUCTION HEATING / QUENCHING

INDUCTIVE HEATING & FLOW CHEMISTRY

A PERFECT SYNERGY OF EMERGING ENABLING TECHNOLOGIES

ALSO INSIDE ///

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INDUCTIVE HEATING AND FLOW CHEMISTRY: A PERFECT SYNERGY OF EMERGING ENABLING TECHNOLOGIES

Inductive heating and flow chemistry are an ideal combination for performing continuously operated high-temperature and high-pressure syntheses.

USING POLYMER QUENCHANTS IN AN INTEGRAL QUENCH FURNACE

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BRIAN KELLY

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FROM THE EDITOR ///



We take your message farther than anyone

If the global climate has taught us anything, it's shown us that the industry is made up of an amazing network of companies that are changing and adapting in order to keep those furnaces and ovens hot and running.

Was it easy? The answer to that would be an emphatic "no." But it's been a necessary task that has proven the industry's mettle — or should that be metal?

Thermal Processing wants you to know that we are here for you, and in ways that continue to make us your No. 1 source for heat-treating news and information on a variety of platforms.

What do I mean by that?

Thermal Processing is the only heat-treat magazine that presents this information in print as well as online.

What does that mean for you?

It means your information — whether it's an eye-catching advertisement or an intelligently written article presented by an industry expert — is not only visible and available on the internet through our website and social media, but it also enjoys a long shelf life as a physical printed vehicle in offices and homes around the country.

That's good news for your audience in search of the very services and products that you can provide every day.

With that in mind, I hope you find the articles in our July issue of interest.

Our July issue takes a deep dive into induction hardening and quenching.

On the subject of quenching, prolific contributor D. Scott MacKenzie shares his amazing insights on using polymer quenchants in an integral quench furnace.

Also, in our Focus section, experts from the Institute of Organic Chemistry in Hannover, Germany, take a deep dive into how inductive heating and flow chemistry are an ideal combination for performing continuously operated high-temperature and high-pressure syntheses.

In addition to those main articles, this issue also offers up a look at how advanced scanning acoustic microscopy can validate the integrity of diffusion bonds, an essential joining method for achieving a high-purity interface between two similar and dissimilar bonds.

That's just a taste of what July's issue has in store for you.

Thermal Processing is here to serve you. With that in mind, if you have any suggestions or would like to contribute, please contact me. I'm always looking for exciting articles to share.

Stay cool out there, and, as always, thanks for reading!

KENNETH CARTER, EDITOR

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Akademi Metalurji, based in Turkey, upgraded their heat-treatment facility with a turnkey nitriding installation provided by Nitrex. (Courtesy: Nitrex)

Akademi Metalurji upgrades heat-treat facility with Nitrex

Akademi Metalurji, a full-service commercial heat-treating solutions provider located in Turkey, has recently upgraded its heat-treatment facility with a turnkey nitriding installation provided by Nitrex. The project includes a mid-sized pit-type furnace, advanced controls, three process technologies (Nitreg[®] controlled nitriding, Nitreg[®]-C controlled nitrocarburizing, and ONC[®] post-oxidation), along with accelerated cooling. The latter is add-on equipment that will help the customer reduce cycle times, optimize production

between batches, and run more cycles.

The decision to invest in a new nitriding system was driven by the company's objective to overcome efficiency and quality challenges faced with their previous furnace at the Gebze facility. The prior system consumed excessive amounts of process gases and yielded inconsistent nitriding results. With the addition of a Nitrex NX-1015 pit furnace, Akademi Metalurji can now save on process gases and production time, while also expanding their heat-treatment capabilities to accommodate wider-dimensioned parts. The nitriding furnace offers an effective work zone of 39" diameter X 59" high (1,000 x 1,500 mm) and can handle a load of up to 4,400 pounds (2,000 kg). The supplied library of Nitreg-based recipes is tailored to

meet different application requirements, resulting in a hardened surface that is highly wear-resistant. For applications such as machinery components, tooling, dies, and molds, where Akademi Metalurji specializes, Nitreg delivers improved tooling performance, ensuring longer service life and higher throughput. Ultimately, this leads to tool cost savings for their customers.

"Although there were other contenders for this project, our solution and proven references within the local market were instrumental in choosing Nitrex," said Marcin Stoklosa, product manager at Nitrex. "While we were priced higher, the guarantee of consistent nitriding results, savings in process gases, shortened production times, and accelerated return on investment all helped to secure this order."

"This Nitrex system is not only compact but also adaptable, making it a perfect fit for any facility," said Utku Inan, the local Nitrex representative who coordinated the sales and commissioning process. "We extend our sincerest gratitude to Akademi Metalurji for entrusting us with their heat-treatment capital needs and development project. Today, we proudly welcome a new member to the Nitrex family, and we look forward to many years of collaboration and shared success."

The system was successfully installed at the Gebze facility, located southeast of Istanbul, and commenced operations in April 2023. Since then, it has been running smoothly, delivering excellent results.

MORE INFO www.nitrex.com

Blue M ships mechanical convection oven

Blue M, a global manufacturer of industrial and laboratory ovens, has shipped a mechanical convection oven to a medical products manufacturer.



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.



The Blue M oven is designed with an advanced PLC/HMI system. The system features a large display which makes it easier for the operator to navigate through the menus. (Courtesy: Blue M)

This oven has a temperature range of 15°C above ambient to 350°C and interior chamber dimensions of 48" W x 24" D x 36" H. The unit is designed with silicone-free construction, so the silicone parts in the oven have been replaced with materials that reduce contamination inside a cleanroom. Seven 304 stainless steel slotted shelves were included and provide excellent resistance to high-temperature operation.

This unit features an industrial uninterruptible power supply (UPS) system wired to the oven's control circuit. In the event of a power loss the UPS system will supply power to the control circuit only for approximately 15 minutes. Since the main temperature controller stays on during a power loss event with a UPS, any recorded temperature data will help identify quality issues with that batch of product. A safety door switch and buzzer are also installed on the control panel which automatically shuts off the oven blower system and heating elements when the door is opened and sounds the buzzer. This feature allows the operator to load the oven without hot air blowing on them.

The special PLC and HMI control system installed on this unit are considered top of the line and offers the customer advanced control capabilities. The PLC uses analog and digital input/output modules for monitoring process inputs and for the control of external devices as directed through the OIT. The large color touchscreen is programmed with a multitude of user-friendly menus and functions. The operator can easily use these menus to set up and tune the heat-control

system, access maintenance and security functions, and graph temperature trends. The system also includes on-board Ethernet and USB connectivity.

"This Blue M oven is designed with an advanced PLC/HMI system," said Jonathan Young, Blue M product manager. "The system features a large display which makes it easier for the operator to navigate through the menus."

Unique features of this Blue M mechanical convection oven include:

- » Silicone-free construction.
- » Uninterrupted power supply system.
- » Advanced PLC/HMI system with Ethernet connectivity.
- » Space-saving design that saves on facility floorspace.
- » Airflow switch shuts off heating elements if blower system fails.
- » Over-temperature protection.
- » UL508A certified control panel.
- » Swivel casters with leveling feet.

MORE INFO www.bluem.com

Wisconsin Oven ships horizontal quench system

Wisconsin Oven shipped one electrically heated horizontal quench system to a manufacturer of products for the automotive industry. This system will be used for the solution treatment of aluminum.

HOT.



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The maximum temperature for this Wisconsin Oven horizontal quench furnace is 1,200°F and the system has the capacity to heat 450 pounds of aluminum parts and the steel basket within 60 minutes. (Courtesy: Wisconsin Oven)

The maximum temperature for this horizontal quench furnace is 1,200°F and interior chamber dimensions are 2' wide x 2' high x 2' long. The system has the capacity to heat 450 pounds of aluminum parts and the steel basket within 60 minutes. Temperature uniformity of +/- 10°F at set points 850°F and 1,050°F was verified with a nine-point profile test prior to shipment.

This horizontal quench system provides a 15-second quench delay from when the door begins to open until the load is fully submerged in the tank. The quench is manual, where the operator pulls the load out of the oven and onto the quench elevator using a hook.

The quench delay time is recorded and enforced using sensors to detect the start and end of the quench operation, and to stop the process if the quench delay time expires.

"This horizontal quench system was designed with a manual quench which is a cost-effective option and still ensures there are no improperly processed parts," said Doug Christiansen, senior application engineer. "The system also features foldable unload wheel rails to save on floor space."

Unique features of this furnace include:

- » Watlow F4T digital temperature controller with Ethernet capabilities.

- » High limit controller for product over-temperature protection.

- » A pneumatically actuated quench tank lift.

- » Foldable unload wheel rails as a space-saving feature.

- » Remote push-button control of the quench tank lift.

- » Combination style airflow to maximize heating rates.

- » Guaranteed heat-up of parts in 60 minutes or less.

- » Vertical lift pneumatically operated door.

MORE INFO www.wisoven.com

CCAI FEF awards 2023 student scholarships

The Chemical Coaters Association International Finishing Education Foundation (CCAI FEF) announced this year's scholarship recipients.

Since 1992, CCAI has awarded scholarship money from the CCAI Matt Heuert Scholarship Fund to students enrolled in

programs that could lead to a career in coatings and finishing. The scholarships are being awarded through the CCAI FEF, a 501 (c)(3) tax-exempt charitable organization. Applications from qualified students were reviewed by the CCAI FEF Board of Directors, who determined this year's scholarship winners.

CCAI FEF awarded a total of \$13,000 in scholarships to seven deserving students.

"This year's scholarship recipients include high school seniors along with undergraduate students. We are excited to support the academic endeavors of these seven promising students. They are well deserving of the scholarships and have bright futures ahead of them," said Sheila LaMothe, CCAI FEF executive director.



In addition to cash awards sent to the student's school account, each scholarship recipient receives a one-year student membership in CCAI, which allows them to gain significant exposure

to the industry and benefit from the multitude of resources available through the association and its regional chapters across the country.

The Board of Directors selected Jordan Clabaugh, Iowa State University, as the recipient of the Jim Gallagher Scholarship, awarded to the highest scoring applicant.

CCAI and CCAI FEF extend sincere congratulations to all the recipients of a 2023 Matt Heuert Scholarship:

- » Scott Andraea, University of Wisconsin – Stout.

- » Jordan Clabaugh, Iowa State University.

- » Madeline Kehoe, Michigan Technological University.

- » Megan Kokesh, University of Wisconsin – Stout.

- » Daniel Knecht, University of Cincinnati.

- » Mary Schilling, University of Wisconsin.

- » Brooke Strege, University of Wisconsin – Madison.

CCAI FEF accepts scholarship applications each year beginning in January.

The Chemical Coaters Association International (CCAI) is a technical and professional organization that provides information and training on surface coating technologies. CCAI works to raise the standards of finishing operations through educational meetings and seminars, training manuals, educational videos, and outreach programs with schools, colleges, and universities.

The Chemical Coatings Association International Finishing Education Foundation (CCAI FEF) is a 501(c)(3) tax-exempt charitable organization founded in November 2018 to aid in the development and delivery of high-impact educational and training opportunities that support the advancement of the industrial finishing and coatings industry.

MORE INFO www.ccaiweb.com
www.finishingfoundation.org

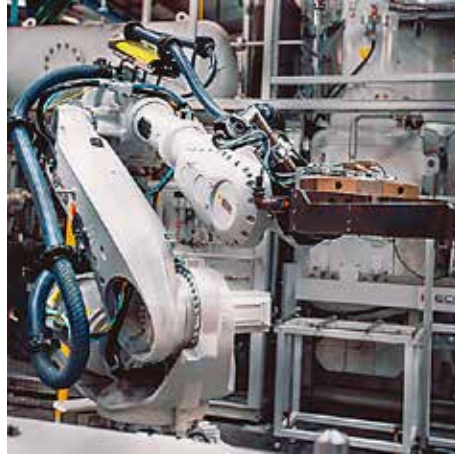
ECM meets heat-treat demands with robotics system

ECM USA has partnered with global manufacturer SEW-EURODRIVE to commission a modular NANO vacuum furnace system completely integrated with advanced automation for their Lyman, South Carolina, facility. It is the third ECM NANO system with integrated robotics and advanced capabilities for the U.S. heat-treat market within the last two years.

The six-chamber, 20 bar quench NANO vacuum furnace system provides maximum flexibility and integration using the addition of 16 tempering positions, advanced solvent-based washer (both oil- and water-based contaminants), and robotic workload assembly/disassembly.

Dunnage management is also provided and fully automated within the robotics configuration. The system was specifically designed to run multiple materials (including carburized grades and tool steels) and has the modular flexibility to adapt to an increase in production for various load scenarios and processes.

SEW-EURODRIVE MOVITRANS® (SEW-EURODRIVE's patented inductive energy power transfer supply system) will also be incorporated within ECM's vacuum furnace transfer system.



ECM USA and SEW-EURODRIVE have commissioned the third ECM NANO system with integrated robotics and advanced capabilities for the U.S. heat-treat market within the last two years. (Courtesy: EMC USA)

As the industry demand for automated solutions grows, ECM meets new heat-treatment production expectations with innovative technology, highly efficient equipment, and superior service.

MORE INFO www.ecm-usa.com

Tenova, POSCO work toward a decarbonized future

Tenova, a leading developer and provider of sustainable solutions for the green transition of the metals industry, will supply an electric arc furnace (EAF) equipped with Consteel® and electromagnetic stirrer Consterr® to POSCO for its Gwangyang plant in South Korea.

The South Korean steelmaker, the sixth largest worldwide with about 43Mt of steel produced in 2022, has a record of pursuing the decarbonization of its high-quality steel products, which mostly include grades traditionally produced via the integral cycle only, such as interstitial-free grades for the automotive industries and the much-coveted electric steel grades required, for instance, by all providers of electrical mobility and green-power generation solutions.

A key part of this transformation toward sustainability consists in the gradual conversion from the BF-BOF route toward electric

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steelmaking based on EAF. After a two-year process of co-engineering and competitive comparison, POSCO has selected Tenova's equipment: a full-platform EAF capable of tapping 280t of liquid steel, equipped with the continuous scrap charging system Consteel, and the electromagnetic stirring system Consterrer — jointly patented by Tenova and ABB.

The new EAF will be uniquely designed to match the needs of quality, productivity, and efficiency that such an experienced steel-maker demands. It will be equipped with a full set of robotic applications and enhanced safety solutions such as the Safe+ EAF water leakage detection system to dispel any concerns about EAF safety and user-friendliness some BOF users might still have.

"Besides requiring a radical re-thinking of the production process, an EAF of this size and power is an entirely new machine for most integral steelmakers and may understandably scare away engineers who are accustomed to the relative quietness of most BOF converters halls," said Paolo Stagnoli, sales director in Tenova for EAF&LF. "As a member of Tenova, I find profoundly gratifying that such a top-class steelmaker recognized our unique experience in large EAFs and understood the value of our patented technology. We are committed to become a long-standing partner of POSCO as we build together a new environmentally friendly and record-breaking furnace."

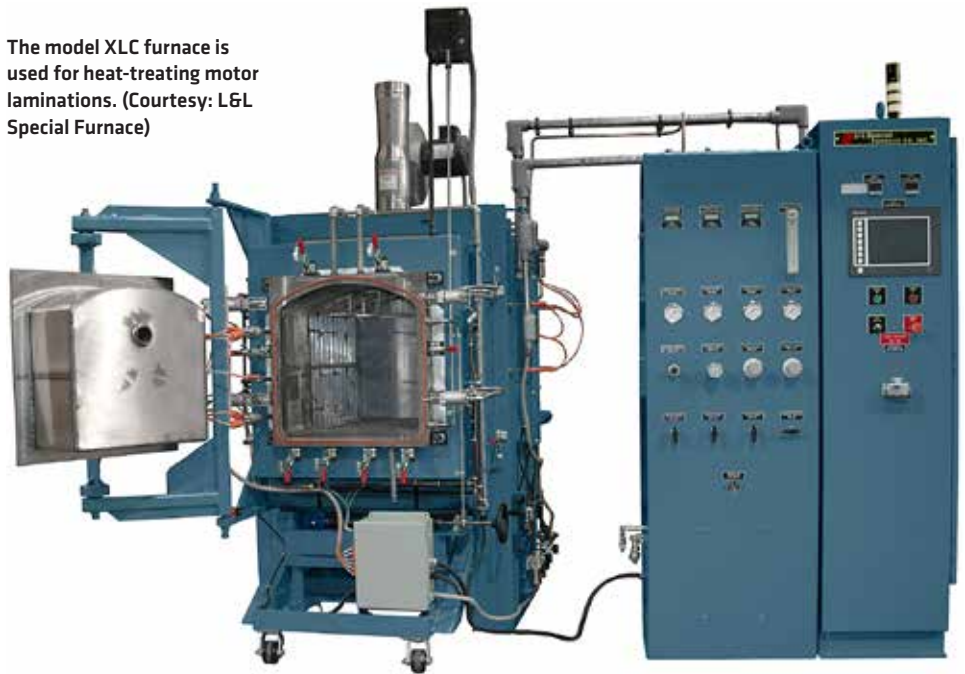
The new EAF, whose product will be merged with the stream of liquid iron produced by the existing blast furnaces, will be installed in a dedicated new section of the Gwangyang plant and is scheduled to enter production by the end of 2025.

MORE INFO www.tenova.com

L&L ships retort furnace to motor manufacturer

L&L Special Furnace has shipped a model XLC3348 retort furnace with an Inconel 602CA alloy retort to a leading manufacturer of motor laminations located in the Midwestern United States. The furnace serves a primary role in the manufacturing of motor laminations in an atmosphere of up to 100 percent

The model XLC furnace is used for heat-treating motor laminations. (Courtesy: L&L Special Furnace)



hydrogen. The laminations are deployed for motors in various aerospace, military, automotive, medical, and industrial fields.

The model XLC3348 has an effective work zone of 23" X 23" X 36" and uniformity of $\pm 15^\circ\text{F}$ above $1,200^\circ\text{F}$ after optimal tuning and balancing of any gradients using digital biasing. The control system includes one control loop along with six zones of heating volume that can be adjusted to achieve the required temperature gradients.

The furnace is equipped with a controlled cooling system, which is critical to the motor lamination process. L&L also offers retort furnaces with removable retorts to meet the most stringent cooldown requirements.

The model XLC3348 satisfies all requirements for AMS2750F class 3 uniformity and type B instrumentation. The furnace features a water-cooled, front flange and door plug cart for easy handling of the door with guides. Included is a complete gas-mixing panel with independent hydrogen, nitrogen, and emergency nitrogen lines. The process gas lines are required copper refrigeration lines along with stainless steel to ensure a very low dew point in the process gasses, which is critical to the process.

A Honeywell HC900 hybrid PLC-process controller with CR10 operator interface is included and clearly displays operating state, alarm conditions, and programming aspects of the process logic parameters. This is completely set up, configured, and logic-tested

prior to shipment.

The control panel is a floor-standing NEMA12 enclosure with fused disconnect and all required fusing and wiring. The electrical system is designed in compliance with NFPA86 guidelines for safety and includes all required electrical components, placard and paint schemes.

All L&L furnaces can be configured with various options and be specifically tailored to meet your thermal needs. We also offer furnaces equipped with pyrometry packages to meet the latest revision of ASM2750.

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

MORE INFO www.lfurnace.com

Lindberg/MPH ships mesh belt conveyor furnace

Lindberg/MPH announced the shipment of an electrically heated, full muffle, mesh belt conveyor furnace. This furnace is designed for treating pressed powdered material.

The furnace is designed for applications with a maximum process temperature of

1,000° C (1,832° F) that use a nitrogen or clean dry filtered air process atmosphere. The unit is configured with nine temperature control zones and includes a water-cooling section. The conveyor system features a 14-inch-wide mesh belt with belt stop alarm to provide an audible and visual indication in case of a conveyor stoppage.

There are purge chambers at the entrance and exit of this conveyor furnace. In these chambers, purge gas flows from perforated plenum chambers above and below the belt and exhaust stacks with adjustable butterfly valves are located at the inner ends of each chamber. Load and unload tables at the entrance and exit of the conveyor provide ease of product transfer for the operator.

The furnace zone temperatures are controlled by Eurotherm controllers. These controllers have advanced PID control with variable overshoot inhibition. A hi-limit Eurotherm controller guards against over-temperature conditions in each zone of temperature control. A visual and audible alert

and removal of power to the heating element occur in the event the monitor temperature exceeds a desired set point.

“This furnace utilizes nine high temperature zones which provides the customer the flexibility for time/temperature profile adjustments to develop and meet their specific process requirements,” said Bill St. Thomas, business development manager.

Unique features of this mesh belt conveyor furnace include:

» Entrance and exit purge chambers.

» Furnace muffle.

» Eurotherm Model 3216 temperature controller.

» Eurotherm Model 3216i hi-limit controller.

» Solid state, closed loop, belt drive control.

» Zero-Fired solid state relay heating circuit power controllers.

» Platinel II thermocouples.

MORE INFO www.lindbergmph.com

Seco/Warwick adds EV/CAB for European manufacturers

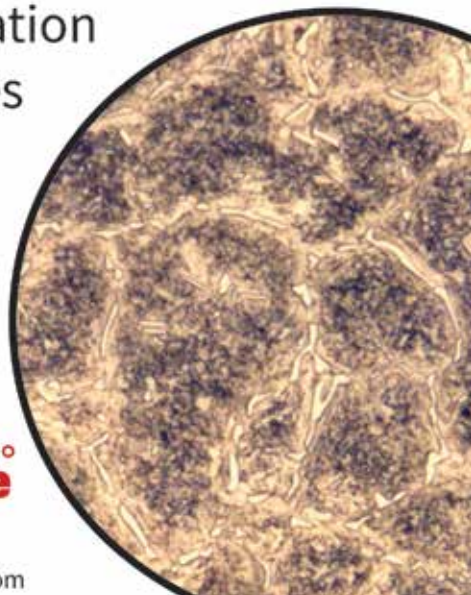
Seco/Warwick, a leader of the CAB technology, will provide long-term partners from Poland and the Czech Republic with further EV/CAB lines for electric vehicle battery coolers' protective atmosphere brazing. The solutions ordered are the third and eighth lines delivered to these manufacturers, respectively.

EV/CAB continuous lines are products dedicated to the automotive sector. They are used for battery coolers' protective atmosphere brazing. EV/CAB solutions work for heat exchangers' leading manufacturers and are used for mass production of large-size car battery coolers.

Both partners have been cooperating with the Seco/Warwick Group for many years. The Czech manufacturer has just

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ordered the eighth CAB line, and the third line will be delivered to the Polish manufacturer. Subsequent orders for the same solutions are the highest form of the delivered products' quality recognition as well as general satisfaction with the cooperation. Seco/Warwick takes pride in long-term cooperation and customers who return with further needs. Partnership and mutual trust are the foundation of more than 20 years of cooperation with the automotive industry.

"The orders we are talking about come from our long-term partners, who in total have several Seco/Warwick brand solutions," said Piotr Skarbiński, vice president of the Aluminum and CAB products segment at the Seco/Warwick Group. "Globally, the demand for electric cars and thus for battery coolers is increasing. Because our furnaces have been used by radiator manufacturers for years, they can be sure of their quality and reliability. Wanting to have repeatable production, they are looking for the best solutions in the field of protective atmosphere brazing, and

we try to effectively meet their needs."

The production of battery coolers for electric cars is growing at a rapid pace, and the demand for electric vehicles is increasing all over the world. Due to their size, this type of exchangers cannot be brazed in a standard furnace; you need a special product, which is EV/CAB. EV/CAB is a complete line adapted to the production of large-sized heat exchangers. Thanks to continuous operation, it guarantees an uninterrupted production process and plants' high efficiency. Its greatest advantage is the perfect temperature uniformity over the entire production line width, thanks to which perfect and repeatable elements are obtained, which is the key to success in mass production.

According to the European Automobile Manufacturers' Association (ACEA), in the first three quarters of 2022, approximately 717,000 new, totally electric passenger cars (BEV, battery electric vehicle) were registered in the European Union Member States. This is as much as 26 percent more than in

the same period of 2021. Including plug-in hybrids (PHEV), sales of electric cars for the first three quarters of 2022 amounted to over 1.2 million units, and the year-on-year increase was almost 8 percent.

MORE INFO www.secowarwick.com

Ecoclean to bring parts cleaning know-how to FABTECH

Parts cleaning has become a quality critical manufacturing step in all industry sectors. Thus, companies have to take great effort to reproducibly meet even more demanding particulate and surface tension cleanliness specifications. Ecoclean is answering to these requirements with future-oriented parts cleaning solutions. Information on the various machines for solvent and water-based cleaning for the general industries

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will be provided at FABTECH in Chicago on September 11-14.

Due to rising demands on component quality, new manufacturing technologies, modified processes and/or materials, and digital transformation as well as stricter energy efficiency and climate protection targets, parts manufacturers are confronted with new challenges. Even more stringent specifications on particulate and surface tension residual contaminations have to be fulfilled in a stable manner. Ecoclean and UCM — the division of the SBS Ecoclean Group specializing in high-precision and ultra-fine cleaning — are responding to these demands with optimally adapted solutions and innovative developments. At FABTECH, Ecoclean will be providing information on a broad range of future-oriented solutions for the general industries.

As a full-range supplier, Ecoclean offers industrial parts cleaning solutions for every cleaning task in the general industries. Adapted to the specific requirements and

applications (e.g. manufacturing technology and level, type of contamination, cleanliness demands, throughput, weight), different systems are available for batch cleaning with water-based media and environmentally compatible solvents.

For solvent-based cleaning with hydrocarbons and modified alcohols (semi-polar solvents), the product portfolio comprises of fully enclosed machines carrying the fluid in a closed circuit. All solvent-based cleaning systems come equipped with an integrated distillation system and filtration systems for continuous automatic reconditioning of the solvent. As a result, operator exposure to the solvent is virtually eliminated and an unchanging cleaning quality and long solvent life is ensured.

Water-based cleaning with alkaline, neutral, and acidic media is the technology most frequently employed in the industry. True to the principle of “like dissolves like,” it enters the field whenever water-based (polar) contamination — e.g., coolant and lubricant

emulsions, polishing pastes, particles, abrasive materials, salts or fingerprints — need to be removed. For water-based part cleaning processes, Ecoclean’s product program includes an extensive range of both standard cleaning systems and customer-specific designs.

In addition to the machine information, Ecoclean will provide information on solutions for industry-specific requirements, including the diverse tasks in electromobility, medical engineering, as well as maintenance and remanufacturing. Forward-looking service solutions are another topic at Ecoclean’s Booth #D 40562. These include an iOS and Android compatible service app for maintenance and repair requirements, tailored service and maintenance concepts, developments regarding the digitization of cleaning processes, options for modernizing and adapting systems, as well as training programs for customer employees. ♣

MORE INFO www.ecoclean-group.net



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| Flow Meters | Waukee |
| General | Control panel mounted to generator frame |



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INTERNATIONAL FEDERATION OF HEAT TREATMENT AND SURFACE ENGINEERING

Get ready for Heat Treat 2023

Heat Treat 2023, October 17-19 in Detroit, Michigan, will co-located with IMAT 2023 and the Motion+Power Technology Expo and cover many topics of interest. This is the 32nd ASM Heat Treating Society Conference and Exhibition.

At the present time, there are about 125 papers from international heat-treating professionals.

It is also co-located with ASM's annual meeting, "International Materials, Applications and Technologies (IMAT)" Conference & Expo. It will provide heat-treat attendees with access to 100 materials-related exhibitors and more than 400 additional technical presentations and workshops. Additionally, with the event being co-located with the Motion + Power Technology Expo 2023, attendees will have access to an additional 300 exhibitors.

There are numerous student/emerging professionals initiatives, including free college student registration, Fluxtrol Student Research Competition, and the new ASM Heat Treating Society Strong Bar Student Competition. This is an opportunity for young professionals and students to meet international heat-treating experts.

The technical program is available at www.asminternational.org/heat-treat/technical.

28TH IFHTSE CONGRESS

November 13-16, 2023 | Yokohama, Japan

The 28th IFHTSE Congress is sponsored by the Japanese Society for Heat Treatment. This wide-ranging conference offers participants the opportunity to network and hear papers on a variety of topics, including thermal processing of steel, surface-hardening additive manufacturing, and modeling and simulation of industrial processes.

WHAT HAPPENED AT EUROPEAN HEAT TREATING CONGRESS?

About 160 attendees and about 30 booths were at the European Heat Treating Congress, in Genova, Italy, May 29-31. Conference paper presentations on a wide variety of subjects were well attended.

Perfect weather made the event special with the reception dinner



Heat Treat 2023 will be October 17-19 in Detroit, Michigan, and co-located with IMAT 2023 and the Motion+Power Technology Expo.

at the Genova Aquarium, the largest aquarium in Europe.

SPOTLIGHT ON MEMBERS

IFHTSE is a federation of organizations not individuals. There are three groups of members: scientific or technical societies and associations, universities and registered research institutes, and companies.

Swiss Association for Heat Treatment

The Swiss Association for Heat Treatment "SVW" has existed since 1953 and offers comprehensive training, conferences, and other events to keep its members up-to-date with the latest in heat treatment, coatings, and joining technology.

Bernard Kuntzmann, Listemann AG, is the president. Contact him at bkuntzmann@haerten.ch.

IFHTSE FELLOW VALERY RUDNEV WINS ASM WILLIAM HUNT MEDAL

Dr. Valery Rudnev was selected to receive the ASM William Hunt



Stéphane Andrietti, Transvalor France, presents his paper “Benefits of FEM modeling for efficient and sustainable heat treatments at the European Heat Treating Congress.”

award “for dedicated service to the global materials science community, leadership, development, and promotion of induction-heating



Dr. Valery Rudnev

and heat-treating technologies and novel technologies.” The award was established by ASM International in 1960 in recognition of unusual achievements in industry in the practical application of materials science and engineering through production or engineering use.

Rudnev was director Science & Technology of Inductoheat Inc., an Inductotherm Group Company. He is considered by many as one of the leading global figures in the induction-

heating and heat-treating industry. He joined Inductotherm Group in 1993 and is widely known as “Professor Induction.”

At the 23rd IFHTSE Congress 2016 in Savanna, Georgia, Rudnev was appointed IFHTSE fellow for “his preeminence in induction heat treating and modeling of the induction heat treating process.”

IFHTSE sincerely congratulates Rudnev with this prestigious award.

IFHTSE 202X EVENTS

OCTOBER 17-19, 2023

Heat Treat 2023

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

NOVEMBER 13-16, 2023

28th IFHTSE Congress

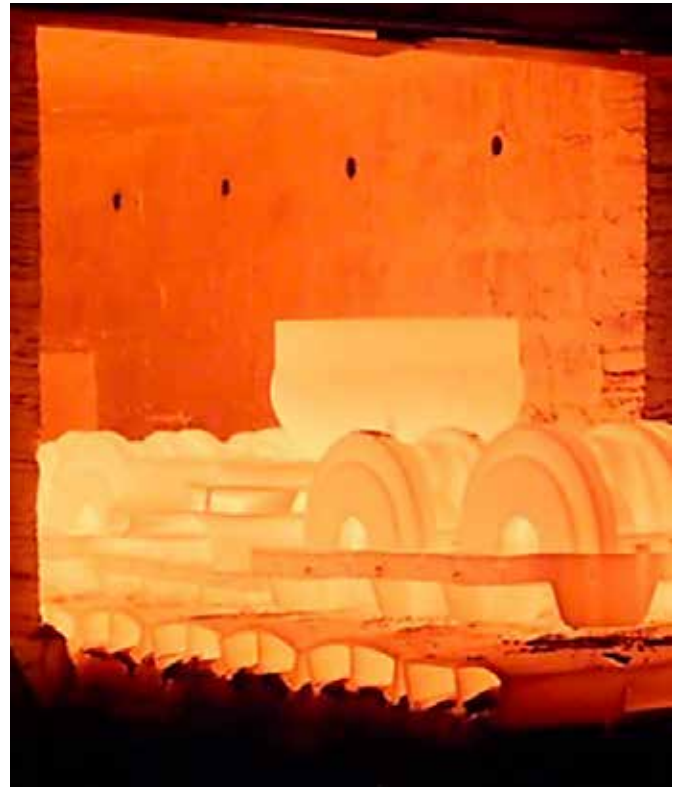
Yokohama, Japan

SEPTEMBER 30-OCTOBER 3, 2024

29th IFHTSE Congress

Cleveland, Ohio | with IMAT and ASM's annual meeting

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



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INDUSTRIAL HEATING EQUIPMENT ASSOCIATION

IHEA announces 2023-2024 board of directors and officers



The 2023-24 IHEA Board of Directors: Back row, left to right; Michael Stowe, John Stanley, Doug Glenn, Jeff Rafter, Scott Bishop, Gary Berwick and Bob Fincken. Front row, left to right; Ben Gasbarre, Jason Safarz, Helen Tuttle, Brian Kelly, John Podach and Jeff Valuck. Not pictured: Alberto Cantu.

The Industrial Heating Equipment Association (IHEA) recently announced its 2023-2024 board of directors and executive officers. Taking over as president is Brian Kelly of Honeywell Thermal Solutions; vice-president is Jeff Rafter of Selas Heat Technology Co. LLC.; and treasurer is Gary Berwick of Dry Coolers, Inc. Jeff Valuck of Surface Combustion assumes the past-president position.

IHEA President Brian Kelly has been an active IHEA member for more than 20 years and has served as the Education Committee and Combustion Seminar chair for many of those years. When asked what goals he has for IHEA, Kelly said, “I plan to focus on further defining IHEA’s sustainability/decarbonization vision and solidify a roadmap that puts IHEA in the forefront of emerging trends that are impacting our industry. In addition, I plan to push to finalize the Advanced Online Industrial Process Heating course to offer

more educational opportunities for people in the thermal processing industry.”

IHEA also welcomes a new face to the board of directors, Helen Tuttle. Currently serving as vice president of finance, Tuttle has been with WS Thermal Process Technology Inc. since 2000.

WS Thermal Process Technology Inc. is the North American sales and service office for WS Warmeprozess-technik GmbH, selling the WS line of high temperature, highly efficient, low emissions burner systems for industrial furnaces. Over the past several years, Tuttle has been involved with IHEA’s Education Committee and said she looks forward to working more on knowledge base initiatives. She is a graduate of the University of Toledo with personal interests that include traveling with her husband and enjoying the outdoors. IHEA is happy to have Tuttle’s involvement as the first woman to serve on

the IHEA board of directors.

To complete the slate of IHEA's board of directors for 2023-2024, the following members continue their service: Scott Bishop, Southern Company; Alberto Cantu, Nutec Bickley; Bob Fincken, Super Systems, Inc.; Ben Gasbarre, Gasbarre Thermal Processing Systems; Doug Glenn, Heat Treat Today; John Podach, Fostoria Infrared; Jason Safarz, Karl Dungs, Inc.; John Stanley, Karl Dungs, Inc.; and Michael Stowe, Advanced Energy.



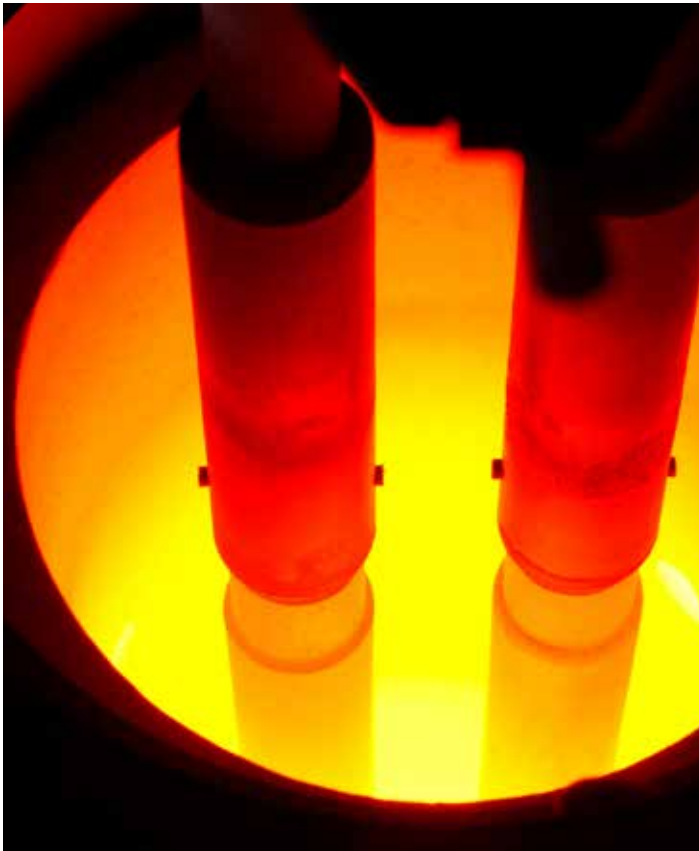
IHEA's newest board member, Helen Tuttle of WS Thermal Process Technology Inc.

In addition, IHEA is equally pleased to note the dedication and service of all members who serve on IHEA committees and divisions. Special appreciation goes to our committee chairpersons: Sustainability Committee led by Jeff Rafter, Selas Heat Technology Co. LLC; Safety Standards and Codes Committee led by Jason Safarz, Karl Dungs, Inc.; Education Committee led by Brian Kelly, Honeywell Thermal Solutions; Marketing Communication & Membership Committee led by Erik Klingerman, Industrial Heating

magazine. The Infrared Division is chaired by Marty Sawyer, Trimac Industrial Systems; and the Induction Division is chaired by Michael Stowe, Advanced Energy.

Established in 1929 to meet the need for effective group action in promoting the interests of industrial furnace manufacturers, IHEA has expanded and currently includes designers and manufacturers of all types of industrial heat-processing equipment used for the melting, refining and heat processing of ferrous and nonferrous metals and certain nonmetallic materials and heat-treatment of products made from them.

For information on how to join IHEA, go to www.ihea.org.



IHEA CALENDAR OF EVENTS

JULY 20

Sustainability & Decarbonization Webinar Series - DOE Tools and Programs for GHG Reduction

There are many options available to help determine carbon emissions for equipment, processes, sites, and organizations. This presentation will review some of these available tools and how to apply them to different situations.

AUGUST 21

Fundamentals of Industrial Process Heating

6 week online course | \$775 IHEA members / \$950 non-members

This course is designed to give the student a fundamental understanding of the mechanisms of heat transfer within an industrial furnace and the associated losses and the operation of a heating source either as fuel combustion or electricity.

AUGUST 24

Sustainability & Decarbonization Webinar Series - Ongoing Sustainability: Industry Best Practices

Carbon reduction is not a project, it is a process, and must be ongoing. Earlier sessions will help you determine your carbon footprint and understand ways to track and impact your carbon footprint. In this presentation, we will review methods and programs to ensure the continual improvement of your carbon reduction efforts.

OCTOBER 31

Safety Standards & Codes Seminar

Cincinnati, Ohio | \$800 IHEA members / \$975 non-members

This seminar is designed for individuals involved in the design, manufacture, service or operation of ovens, furnaces, kilns, dryers, thermal oxidizers and a wide range of industrial applications. It is intended to help the attendee become better acquainted with the newly updated NFPA 86 - Standard for Ovens & Furnaces.

OCTOBER 31

IHEA's Annual Combustion Seminar

Cincinnati, Ohio | \$800 IHEA members / \$975 non-members

Long the industry premier seminar for industrial process heating professionals, this two-day event offers attendees the chance to learn the latest in combustion technology and visit with industry suppliers during a tabletop exhibition the first day. The IHEA Combustion Seminar is designed for persons responsible for the operation, design, selection and/or maintenance of fuel-fired industrial process furnaces and ovens. Seminar speakers are industry leaders in the combustion industry. Presentations will be non-commercial and promote the technology overall, not a specific product or company.

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

INDUSTRIAL HEATING EQUIPMENT ASSOCIATION

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Liquid carburizing, or cyaniding, is a cost-effective hardening solution for gears but may not always be the best choice.

Heat treatment techniques overview

EDITOR'S NOTE » This is the third in a five-part series.

In this third segment of my series on heat-treating techniques, I will discuss the pros and cons of gas liquid carburizing, also known as cyanide carburizing (cyaniding).

THE BASICS OF THE PROCESS

These processes produce a hard, thin layer between 0.25 and 0.75 mm (0.01 and 0.03 inches) that is similar to or harder than the one produced by carburizing. Typically, the process can be completed in 20 to 30 minutes compared to the several hours common with the case carburizing process, thus neither the internal and/or surface stresses will be relieved. Heat-treat processes that require the surface and/or body of the part to be heated above approximately 1,100°C will relieve these stresses. If these internal and/or surface stresses are not uniform, the relief of these stresses will be non-uniform, and heat treatment distortion will occur. Even if the base material is very uniform in both internal stresses (stress relieved and annealed, etc.), the process of cutting the gashes to form the gear teeth induces non-uniform stresses in the surface of the part from the shearing operation of cutting. Unfortunately, the major drawback of cyaniding is that cyanide salts are poisonous.

The difference between liquid carburizing and gas carburizing is that in gas carburizing, carbon is generated by disassociate butane, methane, propane, or natural gas. In liquid carburizing, carbon is leached out from a molten salt composed mainly of sodium cyanide (NaCN) and barium chloride (BaCl₂). In pack carburizing, carbon monoxide is driven out of coke or hardwood charcoal and then disassociated. Liquid carburizing may be distinguished from cyaniding (which is performed in a bath containing a higher percentage of cyanide) by the character and composition of the case produced. Cases produced by liquid carburizing are lower in nitrogen and higher in carbon than cases produced by cyaniding.

LIQUID CARBURIZING

The liquid carburizing process facilitates generation of a hard, thin case on the exterior of metal parts with a high basic (internal) carbon content. It usually requires comparatively little time to complete. Liquid carburizing proves very efficient for strengthening the surfaces of small and medium-sized parts; manufacturers can automate this process easily. This is a process used for case (surface) hardening steel

or iron parts. The parts are held at a temperature above Ac1 in a molten salt bath. In the supercooled austenite transformation curve of steel, Ac1 represents the critical temperature at which pearlite transforms to austenite during heating; Ac3 represents the final critical temperature at which free ferrite is completely transformed into austenite during heating. Once the temperature of the part has sufficiently stabilized, the process then infuses carbon and nitrogen, or carbon alone, into the surface of the metal part. Most liquid carburizing baths contain cyanide, which introduces both carbon and nitrogen into the case.

Liquid carburizing is carried out by placing the component in



Figure 1: An example of a simple carburizing box of heat resisting stainless steel. [1]

a salt bath at a temperature of 845 to 955 °C. The salt is usually a cyanide-chloride-carbonate mixture and is highly toxic. The cyanide salt introduces a small amount of nitrogen into the surface which further improves its hardness. Although this is the fastest carburizing process, it is suitable only for small batch sizes. Liquid or cyanide carburizing is a case-hardening process that is fast and efficient; it is mainly used on low-carbon steels.

It is a method of case hardening involving the diffusion of carbon and nitrogen into the surface layer of steel in cyanide-salt baths at temperatures of 820 to 860°C, defined as medium-temperature cyaniding, or 930 to 950°C for high-temperature cyaniding. Its principal purpose is to increase the hardness, wear resistance, and fatigue limit of steel products.

During cyaniding, the cyanide salts are oxidized with the liberation of atomic carbon and nitrogen, which diffuse into the steel. In medium-temperature cyaniding, the cyanide layer formed (containing 0.6 to 0.7 percent carbon and 0.8 to 1.2 percent nitrogen) has a

thickness of 0.15 to 0.6 mm, while in high-temperature cyaniding (a method often used instead of carburizing), the cyanide layer, containing 0.8–1.2 percent carbon and 0.2–0.3 percent nitrogen, has a thickness of 0.5 to 2 mm. After cyaniding, a product undergoes hardening and low-temperature tempering.

Liquid carburizing is carried out in a container filled with molten salt, such as sodium cyanide. This bath is heated by electrical immersion elements or by a gas burner, and stirring is done to ensure uniform temperature. This process gives a thin, hardened layer up to 0.08 mm thick. Parts that are to be case-hardened are dipped into a liquid bath solution containing calcium cyanide and polymerized hydro-cyanide acid or sodium or potassium cyanide along with some salt. The bath temperature is kept between 815 to 900 °C. The furnace is usually built around a carbon steel case or heat-treatment tray, which may be fired by oil, gas, or electrically. If only selected portions of the components are to be carburized, then the remaining portions are covered by copper plating. There are some advantages of the liquid bath carburizing process as a greater depth of penetration is possible.

CYANIDING OR PACK CYANIDE OR PACK CARBURIZING

Cyaniding or pack cyanide carburizing is used to case harden steel. It generates a very thin but hard outer case. Cyaniding is a case-hardening process in which both carbon and nitrogen in form of cyaniding salt are added to the surface of parts machined from low- to medium-carbon steels. Sodium cyanide or potassium cyanide may be used as the hardening medium, which is a process of superficial case hardening. It combines the absorption of carbon and nitrogen to obtain surface hardness. The components to be case hardened are immersed in a bath containing fused sodium cyanide salts kept at 800 to 850 °C. The component is then quenched in bath or water. This method is effective for increasing the fatigue limit of medium- and small-sized parts such as gears, spindle, shaft, etc.

Cyanide hardening has some advantages and some disadvantages over the case carburizing or nitriding process methods. The cyaniding process also provides a bright finish on the part. More importantly, distortion can be manageable and / or avoided and the effective fatigue limit can be increased. However, as mentioned earlier, the main disadvantage of this process is that it is costly and uses highly toxic agents in the process, which compares less favorably to other processes of case hardening.

Cyaniding or pack cyanide carburizing has the highest production rate. In this process, components are packed in an environment with high carbon content. The key difference between cyaniding and carbonitriding is that cyaniding uses sodium cyanide liquid, whereas the carbonitriding process uses a gaseous atmosphere consisting of ammonia and hydrocarbons. Moreover, cyaniding involves temperatures around 855 to 955 °C. Despite this increased complexity, gas carburizing has become the most effective and widely used method for carburizing steel parts in large quantities. The process provides a number of advantages, such as (but not limited to) high process speed and ease of control, low cost of equipment and production space, the possibility of precluding grain regeneration owing to the short duration of the cyaniding process, uniformity of heating of the part, high flexibility of the process (since parts requiring different depths of hardness may be treated in the same bath), and the possibility of direct quenching from the salt bath.

Metals to be carburized such as low carbon steel are placed in cast iron or steel heat-treatment trays containing a material rich in carbon such as charcoal, crushed bones, potassium ferro-cyanide, or charred leather (in high-performance gear applications, we tend to use a more controlled carbonizing material). Such containers or trays

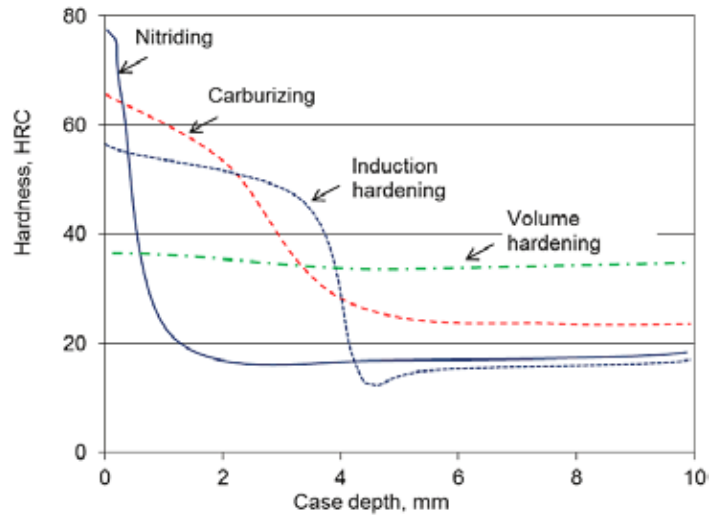


Figure 2: Effective hardness as a function case depth (as an example).

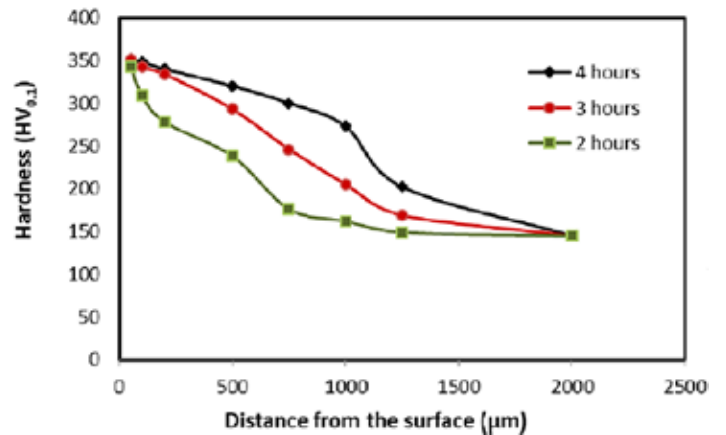


Figure 3: Pack carburization of mild steel, using charcoal as carburizer.

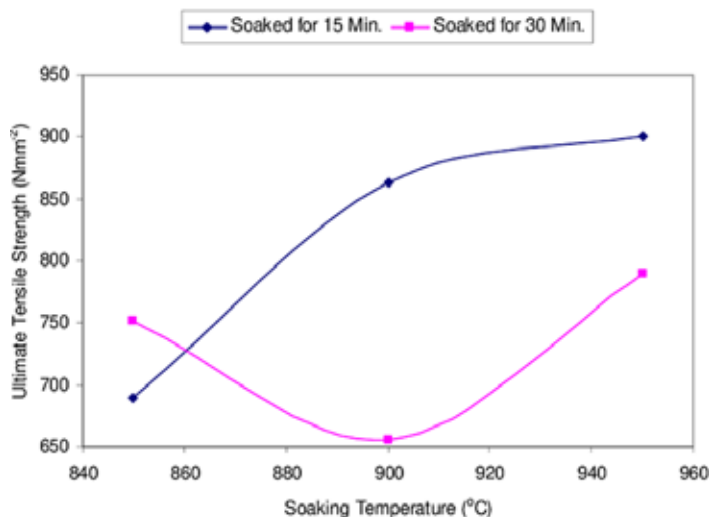


Figure 4: Pack carburization of mild steel, using pulverized bone as carburizer.

are made of heat-resisting steel (or other materials) which are then closed and sealed. The parts, carbon material, and trays are heated to a temperature of 900 to 950°C according to the type of steel being treated. The carbon enters the metal to form a solid solution with iron and converts the outer surface into high carbon steel. Consequently, pack-hardened steel pieces have carbon content up to 0.85 percent in their outer case. After this treatment, the carburized parts are cooled in boxes. Only plain carbon steel is carburized in this process for hardening the outer skin and refining the structure of the core to make it soft and tough. Small gears are case hardened by this process for which they are enclosed in the cast iron or steel box containing a material rich in carbon, such as small piece of charcoal, and then heated to a temperature slightly above the critical range. Depth of hardness from 0.8-1.6 mm is attained in three to four hours. The gears are then allowed to cool slowly within the box and then removed. The second stage consists of reheating the gears (so obtained) to about 900°C and then quenching in oil so that its structure is refined, brittleness removed, and the core becomes soft and tough. The metal is then reheated to about 700°C and quenched in water so that the outer surface of gear, which had been rendered soft during the preceding operation, is again hardened.

The difference between liquid carburizing and cyaniding is that carburizing offers advantages because it supplies case hardening for the exterior surfaces of low carbon steel and iron alloys. Metal parts which undergo heat treatment and carburizing tend to resist abrasions more effectively.

Carbonitriding is similar to cyaniding except that the heat treatment is conducted in a gaseous atmosphere of ammonia and hydrocarbons instead of sodium cyanide. If the part is to be quenched, it is heated to 775 to 885°C (1,427 to 1,625°F). If the processing plan for this particular part does not include quenching, then the part is heated to only 649 to 788°C (1,200 to 1,450°F).

THE DIFFERENCE BETWEEN LIQUID CARBURIZING AND CYANIDING OR PACK CYANIDE

Cyaniding and carbonitriding are two forms of case hardening processes that are useful in getting a hard surface on metal. The key difference between cyaniding and carbonitriding is that cyaniding uses sodium cyanide liquid, whereas the carbonitriding process, as noted above, uses a gaseous atmosphere consisting of ammonia and hydrocarbons. Moreover, cyaniding involves temperatures around 871 to 954°C. In carbonitriding, if the part is to be quenched, the temperature of the bath is maintained between 445 to 885°C. If the part is not going to be quenched, then the metal surface temperature would be maintained between 649 to 788°C.

Another use of pack cyanide or cyaniding is in metal finishing, widely used in electroplating industries in order to assure the high quality of the finished products. Consequently, it is a common contaminant in different industrial effluents including metal processing, gold mining, and plastics, which generally co-exist with other toxic substances such as heavy metals.

The disadvantages of cyaniding are its high cost and the toxicity of the cyanide salts, the latter necessitating the adoption of special measures to protect workers and the environment.

To briefly summarize the differences between a carburizing heat treatment and nitriding:

» Carburizing is a heat-treatment process that diffuses carbon into the surface of a metal to create a hardened surface, whereas nitriding is a heat-treating process that diffuses nitrogen into the surface of a metal to create a hardened surface.

» Carburizing uses a carbonaceous environment; nitriding uses nitrogen instead of carbon.



Carburizing and nitriding are two types of surface hardening processes that are used to make a steel surface hard while the core remains soft.

» Carburizing is done at very high temperatures; nitriding can be done at low temperatures.

» In carburizing, carbon is diffused onto the surface of the metal alloy. In nitriding, nitrogen is diffused onto the surface of the metal alloy.

Carburizing and nitriding are two types of surface hardening processes that are used to make a steel surface hard while the core remains soft. Reiterating, the main difference between carburizing and nitriding is that in carburizing, carbon is diffused to the steel surface whereas, in nitriding process, nitrogen is diffused to the steel surface.

COMPLEMENTARY PROCESSES

Ferritic nitrocarburizing (FNC). This is a surface engineering process for the heat treatment of steels and cast irons performed within a temperature band 550 to 740°C (1,020 to 1,350 F). All heat treatment is done in a gaseous environment, rich in nitrogen. The objective is to develop an iron nitride surface layer of approximately 5 to 50 (mm). This is in turn supported by a nitrogen-rich diffusion zone in the underlying material. The ferritic nitrocarburizing diffuses mostly nitrogen and some carbon into the case of a workpiece below the critical temperature, approximately 650°C (1,202°F), which is under the critical temperature of the microstructure of the material, which does not convert to an austenitic phase but stays in the ferritic phase, which is why it is called ferritic nitro-carburization.

The FNC process result provides for a high surface hardness of the compound layer with a surface microporosity, which enhances lubricant retainment (which in turn delivers excellent wear-resistant function). The specifics of the developed surface layer provide for high resistance to indentation of plastic deformation; thus, it provides excellent resistance to surface distress and cumulative damage.

The FNC process is done at temperatures below that wherein internal and surface stresses are relieved during heat treatment. Thus, this process does not cause distortion common to the amount caused by processes such as case carburizing. To be clear, FNC does not generate a thick (relative) layer as does case carburizing, which affords

parts treated via case carburizing the ability to sustain more surface distress and the follow-on cumulative damage and degradation of the contact surface (e.g. micro-progressing to pitting and subsequent spalling).

Nitriding: This is another case-hardening process wherein nitrogen is used to saturate the surface of a part and hold it for prolonged periods of time, generally at temperature from 480 to 650°C in an atmosphere of ammonia gas (NH₃). The nitrogen from the ammonia gas enters into / onto the surface of the steel and forms nitrides that impart extreme hardness to surface. Nitriding is a case-hardening process in which nitrogen instead of carbon is added to the outer skin of the steel. This is a heat-treating process that diffuses nitrogen onto (with only a slight infusion layer into the part, on the order of a few ten-thousands of an inch) the surface of a metal to create a hardened surface. The nitriding process uses nitrogen and heat. This is commonly used for fuel injection pumps. In this method, nitrogen is diffused to the steel surface instead of carbon. Nitriding can be done at lower temperatures than carburizing. This process is used for those alloys which are susceptible to the formation of chemical nitrides.

The part to be nitrided is placed in a container (usually made of high nickel chromium steel) and with inlet and outlet tubes through which ammonia gas is circulated. Ammonia gas is used as a nitrogen-producing component. The alloy steel containing Cr, Ni, Al, Mo, V and other nitrile alloys are widely used for this process. Plain carbon steels are seldom nitrided. Nitride-forming elements must be present for this method to work; these elements include chromium, molybdenum, and aluminum. The advantage of this process is that it causes little distortion, so the part can be case-hardened after being quenched, tempered,

and machined. No quenching is typically done after nitriding.

Nitriding is a better case-hardening process than carburizing or cyaniding, because in this method, nitrogen is diffused to the steel surface instead of carbon. Nitriding can be done at lower temperatures than carburizing. The diffusion of nitrogen gas normally occurs at low temperatures, and hardening occurs without quenching. Only the surface is hardened, the core remains at the same basic properties and hardness. ♨

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Calculating the amount of decarburization present after heat treatment can be used to sort out practical heat-treating problems.

Calculating decarburization

In this column, I will be talking about decarburization of steel and how to calculate its depth.

INTRODUCTION

Decarburization of steel is the depletion of carbon from the surface of steel. Carbon at the surface of the steel reacts with the oxygen in the furnace atmosphere and creates CO/CO₂. In an oxygen-rich atmosphere, CO₂ would be formed. The oxygen also reacts with the iron in the steel to form an oxide scale. At typical heat-treating temperatures, the predominant oxide present would be FeO. An example of decarburization is shown in Figure 1.

CALCULATION OF DECARBURIZATION

As decarburization proceeds, more and more carbon diffuses to the surface, down the concentration gradient. Since this is a diffusion problem, and exactly the opposite of carburization, the same principles apply. In this case, the case depth would be the depth of decarburization, and the surface carbon potential would be 0 %C.

The case depth is a function of time, temperature, and chemistry; of the process; and the available carbon (carbon potential) at the surface of the steel. This follows Fick's Second Law of Diffusion for the concentration of a diffusing species as a function of time, *t*, and position *C(x,t)*:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2}$$

The solution to this differential equation is:

$$\frac{C(x,t) - C_0}{C_s - C_0} = 1 - \operatorname{erf} \left[\frac{x}{2\sqrt{Dt}} \right]$$

where *C(x,t)* is the concentration of carbon (in the case of carburizing) at position *x* at time *t* (s). *C*₀ is the initial carbon content in the steel, *C*_s is the carbon potential of the atmosphere at the surface of the part, and *D* is the diffusion coefficient of carbon in austenite. The function *erf(z)* is called the error function and is often encountered in diffusion and heat-transfer calculations. The diffusion of carbon in austenite is [1]:

$$D_{C(\gamma-Fe)} = 0.162 * \exp \left[\frac{-137,800}{RT} \right]$$

where *D* is in cm²/s, *R* = 8.31 J/mol·°K. Using this method, the depth of decarburization can be calculated.

Using the data in Table 1, decarburization was calculated, then compared to data generated by JMatPro [2]. The resulting comparison is shown in Figure 2.

While the effects of alloying elements can affect the predicted diffusion coefficient, the hand-calculated and predicted profiles match very well. At least within the constraints of this calculation,

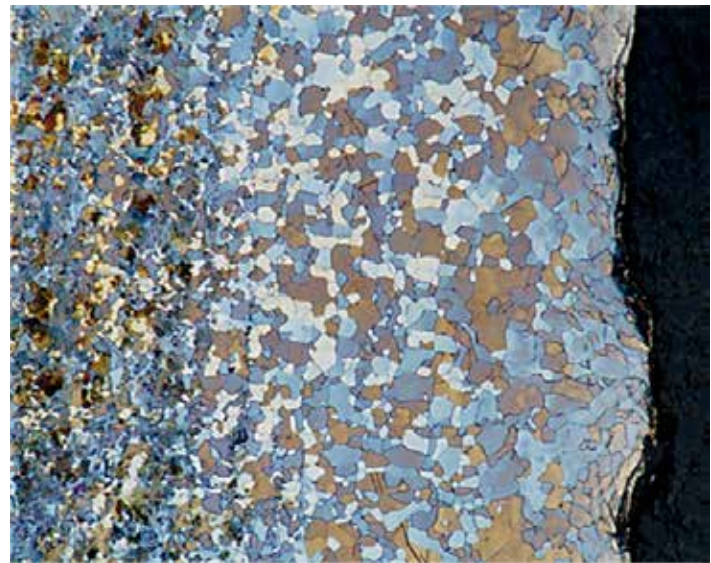


Figure 1: Bright-field image of decarburized and fully annealed SAE 4140 steel, etched with Beraha's sulfamic acid reagent. 100X.

| Item | Units | Value |
|----------------|--------------------|--------------------------|
| T | °F | 1600 |
| T | °K | 1144.1 |
| R | J/mol·°K | 8.31 |
| D | cm ² /s | 8.222 x 10 ⁻⁸ |
| C _s | % | 0 |
| C ₀ | % | 0.5 |
| t | s | 3600 |

Table 1: Data used to calculate comparison decarburization profiles in a SAE 1050 steel.

the amount of decarburization can be calculated from application of Fick's Second Law of Diffusion.

EXAMPLE

Recently, a customer complained of slow hardness in their as-quenched parts. Using a medium carbon steel (SAE 5140), the parts are forged, allowed to air cool, then reheated to 850°C, held at temperature for 150 minutes, then quenched in a polymer quenchant. The parts are heat treated in air with no protective atmosphere during forging or heat treatment. After quenching, the surface is ground (with coolant), and 200 μm of the surface is removed. The hardness is then measured using a 3,000 kg Brinell hardness tester. A minimum as-quenched hardness of 470 BHN is expected. A hardness of 350 BHN has been observed.

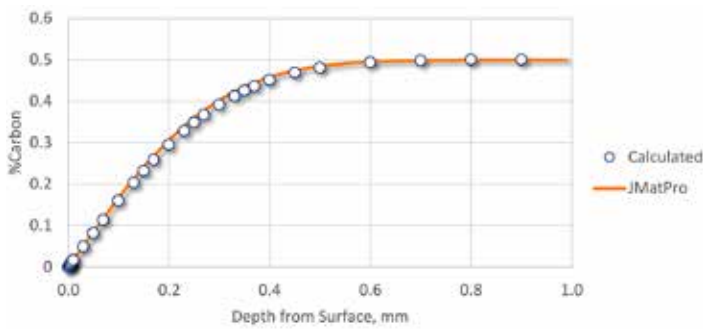


Figure 2: Comparison of hand-calculated values of decarburization in SAE 1050 steel, compared to that calculated by JMatPro, heat treated at 1,600°F (871.1°C) for one hour.

The quenching operation is specifically designed for this operation and has adequate agitation. All pumps and agitators are working properly. The quenchant is a PVP-type quenchant, and examination of the concentration, physical properties, and the cooling curve match very well with a new quenchant. There was no apparent contamination apparent visually, or by FTIR. The quenchant condition looked to be in good condition.

Because the steel wasn't processed using a protective atmosphere during heat treatment or forging, decarburization could explain the low as-quenched hardness. A calculation was performed using the methodology above, and the extent of decarburization determined as a function of surface depth. The results are shown in Figure 3.

The depth of decarburization was determined to be nearly 0.8 mm deep (800 μ m), or four times the amount of material removed. The carbon level at 200 μ m would be approximately 0.2 percent. Assuming that the transformation to 95 percent martensite occurred, the resulting Brinell hardness would be approximately 370 BHN. This hardness is much lower than the desired as-quenched hardness but is close to the measured as-quenched hardness.

As a result of this analysis, recommendations to the customer included improving surface preparation by grinding 0.8 mm from the surface prior to taking the Brinell hardness.

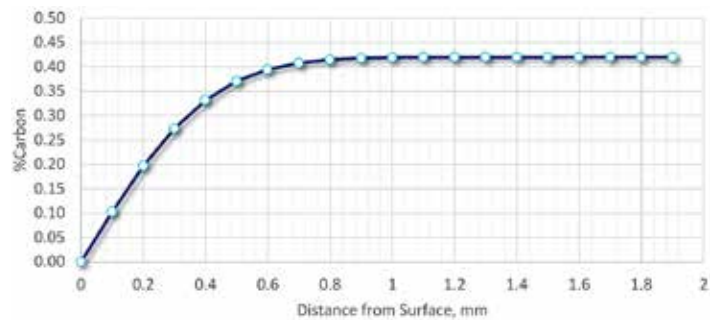


Figure 3: Calculated decarburization depth of an SAE 5140 steel, heat treated in air at 850°C for 150 minutes.

CONCLUSIONS

In this short column, I have shown the methodology to calculate the amount of decarburization present after heat treatment. I have also shown an example of how these calculations can be used to sort out practical heat-treating problems on the shop floor.

Should you have any questions regarding this column, or have any suggestions for future articles, please contact the editor or writer. ✉

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ISSUE FOCUS ///

INDUCTION HEATING / QUENCHING

INDUCTIVE HEATING AND FLOW CHEMISTRY

***A PERFECT SYNERGY OF
EMERGING ENABLING
TECHNOLOGIES***

Inductive heating and flow chemistry are an ideal combination for performing continuously operated high-temperature and high-pressure syntheses.

By CONRAD KUHWALD, SIBEL TÜRKHAN, and ANDREAS KIRSCHNING

Inductive heating has developed into a powerful and rapid indirect heating technique used in various fields of chemistry but also in medicine. Traditionally, inductive heating is used in industry, e.g., for heating large metallic objects including bending, bonding, and welding pipes. In addition, inductive heating has emerged as a partner for flow chemistry, both of which are enabling technologies for organic synthesis. This article reviews the combination of flow chemistry and inductive heating in industrial settings as well as academic research and demonstrates that the two technologies ideally complement each other.

INTRODUCTION

Several decades ago, inductive heating was introduced as an indirect technique in various applications, including industrial manufacturing, synthetic chemistry [1-3], and medicine (Figure 1) [4-7]. Compared to microwave heating [8-9], the other major indirect heating technique, inductive heating has several advantages. Technically, the system is composed of an inductive coil and an alternating current (AC) generator. The material to be heated is usually in the interior of the coil or in its vicinity, so the heat is not generated by convection across a surface. Compared to heating under microwave irradiation, the system does not need to be encased for safety reasons. Inductive heating of materials is extremely fast with the best determined power transfer value of all heating technologies [10]. It has therefore found wide industrial application for heating large metallic objects and workpieces. It is used for bending tubes, bonding, welding, sintering, and annealing of metals and alloys [11]. In addition to steel and alloys, glass or silicon are also heated or melted under inductive heating conditions [12]. In the last decade, inductive heating has also been used for bonding, heating rubber, deforming plastic, or shrinking workpieces [13-15]. These materials are not conductive like steel, copper, or alloys, so another mechanism must take hold to introduce heat. This is often achieved by embedding small superparamagnetic ferromagnetic or ferrimagnetic nanoparticles into these materials. For this purpose, superparamagnetic iron oxide nanoparticles (SPION) are most commonly used, of which the main forms are magnetite (Fe_3O_4) and its oxidized form maghemite ($\gamma\text{-Fe}_2\text{O}_3$). Although cobalt and nickel are also highly magnetic materials, they are less common due to their inherent toxicity and ease of oxidation.

Another branch of research involving SPIONs focuses on developments in nanoscience, nanomedicine, and nanoparticle-assisted imaging, diagnosis, and drug delivery [4-7], an area that is not covered in this article.

1 THEORETICAL BACKGROUND OF ELECTROMAGNETIC INDUCTION

To better understand the mechanisms of inductive heating, some basic physical principles are first explained. This type of heating depends on various structural, morphological, chemical, and physical properties of the materials to be heated. When a suitable receiver

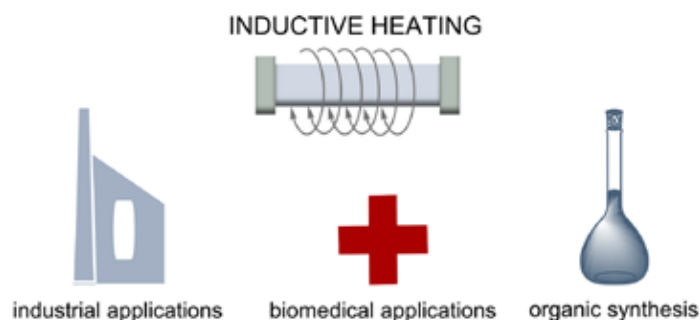


Figure 1: Inductive heating, a powerful tool in industry and the life sciences.

is placed in an alternating electromagnetic field, this energy is converted into heat, apart from minor losses due to convection, conduction, and thermal radiation. This conversion of energy into heat takes place according to three different principles, which depend on the properties of the material.

1.1 Hysteresis loop

The orbital motion and electron spin profile of a material determine its magnetic properties. Ferromagnetic (FM) materials have unpaired electron spins that couple in space and provide a strong magnetic force. However, ferromagnetic materials consist of multiple domains. In a magnetic field, electron spins align within a domain, but commonly not in all domains. As such, ferromagnetic materials consume the energy to grow domains in the direction of the field. However, the multidomain state becomes energetically unfavorable once the material under consideration reaches a certain size such that the energy required to form a domain wall is higher than the energy required to maintain the magnetostatic energy of a single domain. In ferromagnetic single-domain materials, the spins align in the same direction and act as a giant magnetic moment [16-17]. The coupling of these spins to the crystal lattice is called hysteresis. When a magnetic field is applied, the electromagnetic energy of the atoms is transferred to the lattice in the form of heat, which is undesirable for many applications of magnetic materials. This process is therefore referred to as magnetic loss. The amount of energy loss per cycle of magnetic field generation is interpreted as the magnetization of a material in a hysteresis loop, which is defined as magnetic hysteresis loss (Figure 2). It is characterized by three parameters: 1) the saturation magnetization (M_s), at which the material reaches its maximum in the magnetic field, 2) the remanent magnetization (M_r), which is retained by the material when the magnetic field is removed, and 3) the coercivity (H_c), which is the magnetic field required to demagnetize the sample and determines the heat release to the surrounding media. These three parameters are critical to the heat release of magnetic nanoparticles and can vary for different particle types. Coercivity is an inherent property of magnetic nanoparticles that reaches a maximum at a critical diameter of magnetic nanoparticles from multidomain to

single domain structures. In hysteresis curves, the area between M_r and H_c is correlated with the energy absorbed per mass. For ferromagnetic materials, the area indicates their magnetic heating mechanism depends on hysteresis losses.

1.2 Néel relaxation

Magnetic particles consisting of a single domain have a remanent magnetization (M_r) of zero; therefore, they lack the hysteresis contribution in the heating process (Figure 2). Their mechanism of electromagnetic energy dissipation is described by the Néel relaxation mechanism [18]. This phenomenon is called superparamagnetism (SPM) and occurs with decreasing particle size when reaching the nanoscale. The energy barrier of such superparamagnetic nanoparticles to reverse magnetization is directly related to magnetic anisotropy and particle volume [19-20]. In external magnetic fields, these spins rotate in the direction of the magnetic field direction, and the axis of magnetic moment fluctuates along the magnetocrystalline anisotropy axis. Néel has described the relationship between the relaxation time of the thermal fluctuations of the magnetic moments of the individual domains and the uniaxial anisotropy. In Néel relaxation, the energy barrier for the remanence of magnetization decreases with smaller particle volume. The Néel relaxation process can be observed in dry, powdered single-domain nanoparticles or in immobilized nanoparticles, e.g., when embedded in tumor tissue.

1.3 Joule effect

Eddy currents or Foucault currents are generated by an oscillating electromagnetic field that penetrates the resistance of a magnetically conducting receiver and releases energy through the Joule effect [21-22]. The heating power in eddy currents is directly correlated with the square of the applied frequency and field amplitude. In contrast to the two previous effects, the distribution of current density is not homogeneous when a conductor is introduced into an oscillating electric current. It decreases exponentially starting from the surface with increasing distance, e.g., into the depth of the material. The fact the heat is not evenly distributed, but is mainly located on the surface, is called the skin-depth effect. It should be noted this effect decreases significantly with increasing frequency. Two further parameters to be considered for inductively heated materials are the Curie temperature (TC) and the blocking temperature (TB). They mark the phase transition from ferromagnetic to paramagnetic and from ferromagnetic to superparamagnetic materials, respectively. These values represent the thermal limit up to which the materials can be inductively heated, since above this point, they lose their permanent magnetism [23].

2 INDUCTIVE HEATING IN INDUSTRIAL APPLICATIONS UNDER FLOW CONDITIONS

2.1 General remarks

Energy efficiency is one of the most important cost factors in industrial processes, especially for high-temperature reactions in fixed-bed reactors. In general, conductive particles or (superpara)magnetic nanoparticles are suitable as fixed-bed materials for heat generation by applying an external oscillating electromagnetic field. Due to the high specific surface area of these bulk materials, rapid heat transfer by radio frequency (RF) heating is possible (Figure 3). RF-induced heating offers several advantages for use in high-temperature reactions. Advantageously, the heat is generated directly within the reactors,

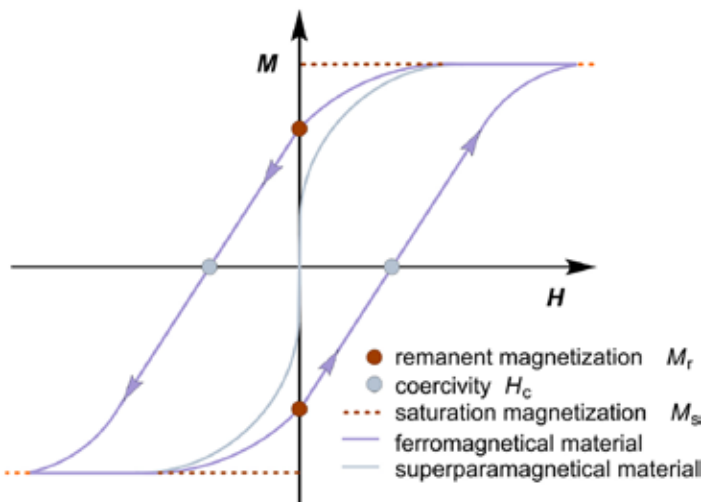


Figure 2: Electric displacement field of a ferromagnetic and superparamagnetic material.

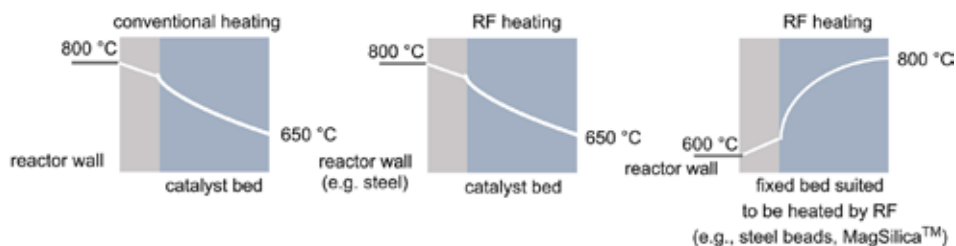
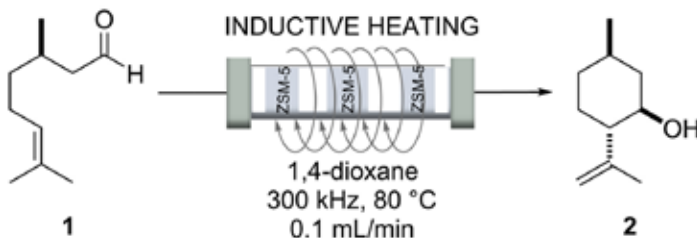


Figure 3: Temperature profiles of reactors heated conventionally and by RF heating (Figure 3 redrawn from [24]).



Scheme 1: Continuous flow synthesis of isopulegol (2) from citronellal (1).

bypassing the problem of thermal gradients. Another advantage of generating heat directly at or near the catalyst surface arises from the potential for hot spots to form, which can substantially exceed the volume temperature of the surrounding reaction medium and lead to significant accelerations of chemical reactions. It is also important that the reactor wall is not exposed to the high temperatures in this process, which has safety implications. Finally, the desired temperature is reached more quickly compared to convective heating and better temperature control can be ensured, e.g., by IR pyrometers.

2.2 Application of trickle bed reactor systems for isopulegol production

Berenguer-Murcia et al. [25] developed a near isothermal micro trickle bed reactor operated by radiofrequency (RF; 300 kHz) heating of nickel ferrite particles (110 μm) deposited in the fixed bed. To achieve near-isothermal conditions at a reactor length of 50 mm, at least three heating zones were set up. The fixed bed was composed of alternating catalyst and heating zones. The heating zones consisted of a mixture of nickel ferrite particles and glass spheres with a particle size of 110 μm . Conventionally heated trickle bed reactors using externally

located heating devices suffer from uneven temperature distribution in the reactor bed and the formation of hot spots that can lead to rapid deactivation of the catalyst.

The authors selected the synthesis of isopulegol (2) from citronellal (1) as test reaction (Scheme 1). Thus, citronellal (1) was cyclized to isopulegol in the heating zone. This was achieved at 80°C in 1,4-dioxane using a Zeolite-encapsulated magnetic nickel ferrite nanoparticles (NiFe₂O₄@TiO₂@ZSM-5) catalyst, an aluminosilicate zeolite, which gave best results due to its high Brønsted acidity [26]. Using inductive heating resulted in a highly improved catalytic system that showed long-term stability. This example is of relevance for the fragrance and flavor industries, as isopulegol (2) can be transformed into menthol in one step by catalytic hydrogenation.

2.3 Dry and steam methane reforming

The commencement of the energy transformation is associated with the search for alternative and more environmentally friendly energy sources [27]. The dry reforming of methane is a particularly interesting process in this context (Scheme 2, Reaction 1). A second variant is the steam methane reforming process. Since complex solids such as wood, sewage sludge, or municipal waste cannot be evaporated, they are reformed using supercritical water on a heterogeneous catalyst at 250-300 bar, 400-550 °C, and a large excess of water [28]. The former process is the preferred route for large-scale production of syngas from biogas [29], while the latter is the main catalytic route [30].

The intrinsic problem with these processes is the extremely high temperature required, typically above 700°C (ambient pressure)



Scheme 2: Dry (Reaction 1) and steam (Reaction 2) methane reforming.



Scheme 3: Calcination and RF heating.

[27]. Since most such processes do not operate at ambient pressure, much higher temperatures of about 950°C are actually required. At these high temperatures, the selectivity of the process is a challenge. Possible side reactions such as hydrogenation of CO and CO₂, decomposition of CH₄, and the Boudouard reaction lead to the formation of elemental carbon [31]. Another problem is the Curie temperature (T_C) associated with the material. Depending on the composition of the stainless steel, this is about 750°C [32]. In 2017, Mortensen's team performed the reaction for the first time with inductively heated Ni-Co NP alloys deposited on a magnesium aluminate (MgAl₂O₄) spinel [33]. The alloy prepared specifically for this case contained 12.6 wt % Ni and 9.0 wt % Co with a Curie temperature higher than 800°C. By using a high Co content (T_C = 1115 °C), they were able to maintain ferromagnetic properties even at very high temperatures. Since the addition of nickel further catalyzes this reaction, complete conversions with low-carbon formation could be obtained at low flow rates. At higher flow rates, reaction kinetics was the limiting factor. Later, it was found that, by doping the alloy with small amounts of copper, almost complete conversion (95%) could be achieved at lower electromagnetic fields and higher flow rates (Q = 152 NL/h) [34]. Not only was less carbon formation observed with this new material, but also little to no reduction in catalytic activity. Although this is not yet the most

efficient process that could be used on a large scale, it is a remarkable application for inductive heating.

2.4 CO₂ storage and release under RF heating

The climatic changes are associated, among other factors, with increasing emissions of CO₂ into the atmosphere. The group of Rebrov et al. investigated the storage of CO₂ in CaO via the calcination process using inductive heating (Scheme 3) [35].

The cycle can be divided into two individual processes: First, the carbonation step, in which the CaO absorbs the CO₂ to form the calcium carbonate at 650-680°C, and second, the subsequent calcination step, in which the CaO-based sorbent is sintered at temperatures of 850-950°C to release the captured CO₂ and form CaO again. The inductive method is a very easy to implement and cost-effective system that can be installed in production plants. A higher desorption rate (15.4%) and a lower degree of sintering of the sorbent were observed with the IH method compared to conventional heating methods, coupled with shorter cycle and start-up times. Rebrov et al. also suggested the system can be used during periods of low power consumption to reduce the load on the electrical system.

2.5 Preparation of hydrocarbons (the Fischer-Tropsch process)

Monodisperse Fe@FeCo core shell nanoparticles as well as Fe(0) nanoparticles with a Ru(0) layer exhibit large heating capability when exposed to an external oscillating electromagnetic field. These particles combine magnetic and surface catalytic properties and thus

have been employed in the Fischer-Tropsch process. The heating performance is characterized by the specific absorption rate (SAR) of the material. The tested materials showed high SAR values when brought into an electromagnetic field of 50 mT at a frequency of 54 kHz. Under these conditions, the particles were able to catalyze the hydrogenation of

CO. The presence of ruthenium increased the catalytic activity and allowed the catalytic process to be carried out at lower reaction temperatures, which was explained by the fact that the surface temperature of the nanoparticles was in fact significantly higher than 200°C. It was also not necessary to implement an additional heating device for the outer reactor wall. Thus, this process represents a promising example of “cold magnetic catalysis” as it is termed in the article [36].

2.6 Methane production (“Sabatier” process)

One of the largest challenges for sustainable power generation is to create a system in which all energy sources can be converted into each other efficiently and according to demand. In the so-called “power to gas” (PtG) technology, an efficient catalytic approach has been missing to date [37-38]. A very challenging catalytic process, which is gaining importance in this context, is the Sabatier reaction.

In this reaction, CO₂ and H₂ are converted to CH₄ (Scheme 4). This process can be used as a catalytic cycle in combination with electrolysis of water to produce the hydrogen exactly when the demand requires it. The reactions are very powerful and can represent an important access to synthetic fuels. This is important not only in the context of energy storage, CO₂ reduction, and climate change prevention, but also because they provide a country-independent source of energy. However, for use in a continuous flow system, the catalyst must have extremely high SAR and catalytic activity. Recently, a series of promising systems have been produced for this purpose. These include (Fe_{2.2}C) NPs [39], (ICNPs@Ni; 29 wt % Ni), and (ICNPs@Ru 1 wt % Ru) on a silica-alumina support (SiRAl₂O₃). The Ru nanoparticles far

outperformed the previously known catalysts. A methane yield of 93% with complete selectivity could be achieved with high flow rates (125 mL/min; reactor dimensions according to SI: 2 cm diameter and 1 cm height of catalyst filling) in an external electromagnetic field of 28 mT. Heating can be problematic when exothermic reactions are performed. To prevent catalyst deactivation, the group of Giambastiani et al. developed homogeneously sized Ni nanoparticles (4 ± 1 nm) decorated on an oxidized carbon felt (OCF) matrix [40]. A laser pyrometer was used to measure the temperature of the catalyst bed in the quartz reactor. The inductor was controlled by a proportional-integral-derivative (PID) controller, which regulates the temperature. This feedback loop allowed the temperature of the catalyst bed to be fine-tuned and adjusted in real time to suit the conditions.

2.7 Biofuel production

The increasing demand for renewable energy sources goes hand-in-hand with the sustainable and efficient use of naturally occurring waste. Two factors that mainly affect the conversion rate of biodiesel are the use of catalysts and the heating process chosen. Inductive heating is applied to improve the pyrolysis of bio-oils, a process used to obtain high-quality biofuels. Inductive heating is advantageous in this process because a rapid and uniform heating of the biomass and catalyst is important for product quality. Compared to a conventional heating process, the authors found a higher quality of bio-oil with a higher yield of aromatic hydrocarbons and a lower oxygen content is obtained in a process with RF heating [41].

A potential energy source for biofuel production is napier grass. The group of Lin investigated the yield and pyrolytic products of HF heating in the pyrolysis of napier grass (Scheme 5) [42]. The yield of liquid products increased with heating rates up to $150^\circ\text{C}/\text{min}$. Pine sawdust and its major components, lignin and cellulose, were pyrolyzed by RF heating at high temperatures ranging from 500°C to 700°C . The authors found higher temperature resulted in higher gas yield and lower liquid yield. The results could be relevant to the forestry and paper industries, which produce large amounts of lignin as a byproduct. The authors compared the fast pyrolysis of poplar wood and switchgrass by RF heating. The highest yield of bio-oil was obtained for switchgrass at a pyrolysis temperature as low as 450°C .

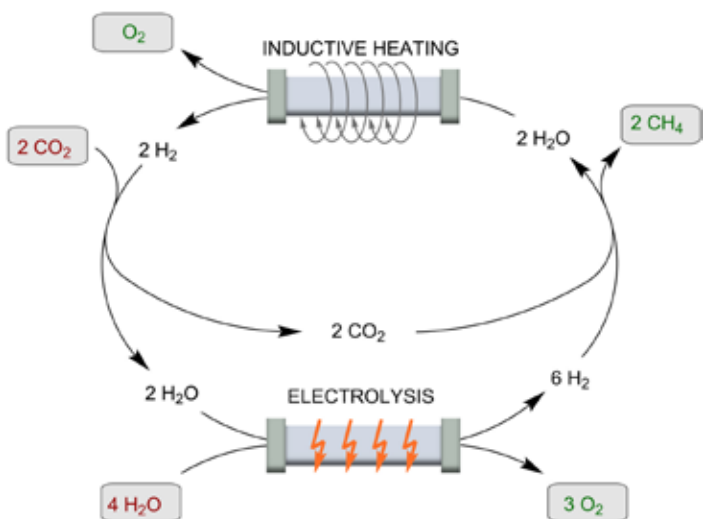
2.8 Water electrolysis

Electrolysis of water as a power-to-hydrogen (PtH) concept is not new, but has become one of the most important topics being discussed today. Due to the energy of hydrogen-hydrogen bonding, water electrolysis enables chemical storage of renewable electricity. Chatenet, Carrey, and co-workers showed the electrocatalytic reaction of hydrogen formation from water can be improved by using RF heating (Scheme 6) [43]. Thus, nickel-coated iron carbide NP (FeC-Ni) was developed to drastically reduce the overpotential at $20 \text{ mA}/\text{cm}^2$ ($\approx 200 \text{ mV}$ for OER). This kinetic enhancement corresponds to a temperature increase of 200°C , although the actual temperature only increased by 5°C . The authors suggested the use of RF heating may allow water splitting near the equilibrium voltage at room temperature. Although it was expected that the magnetic field applied by inductive heating would disturb the flowing current, mainly positive effects were observed.

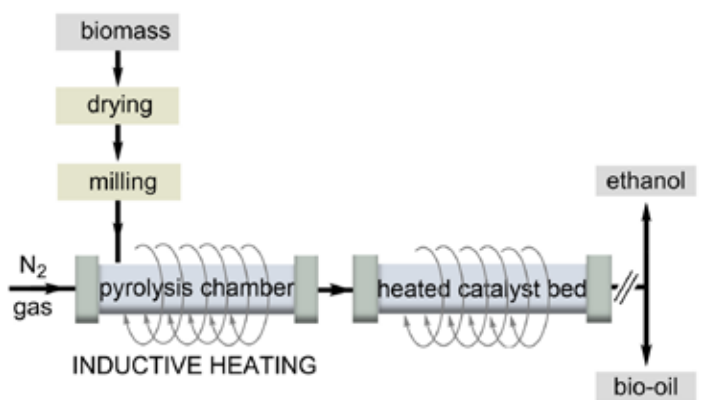
3 MICRO- AND MESOFLOW TECHNOLOGY AND INDIRECT HEATING

3.1 Microwave-accelerated reactions under flow conditions

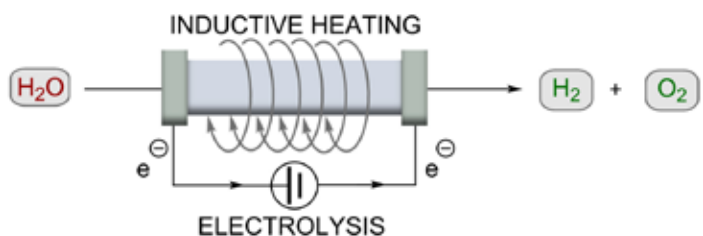
Reactions that take 20 minutes or longer under classical batch conditions can be accelerated considerably under continuous flow condi-



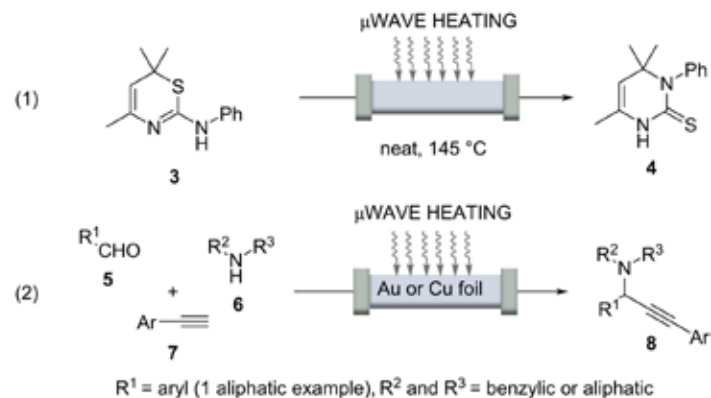
Scheme 4: The continuously operated "Sabatier" process.



Scheme 5: Biofuel production from biomass using inductive heating for pyrolysis.



Scheme 6: Water electrolysis using an inductively heated electrolysis cell.



Scheme 7: Dimroth rearrangement (Reaction 1) and three-component reaction (Reaction 2) to propargyl amines 8 under continuous flow conditions with microwave assistance.

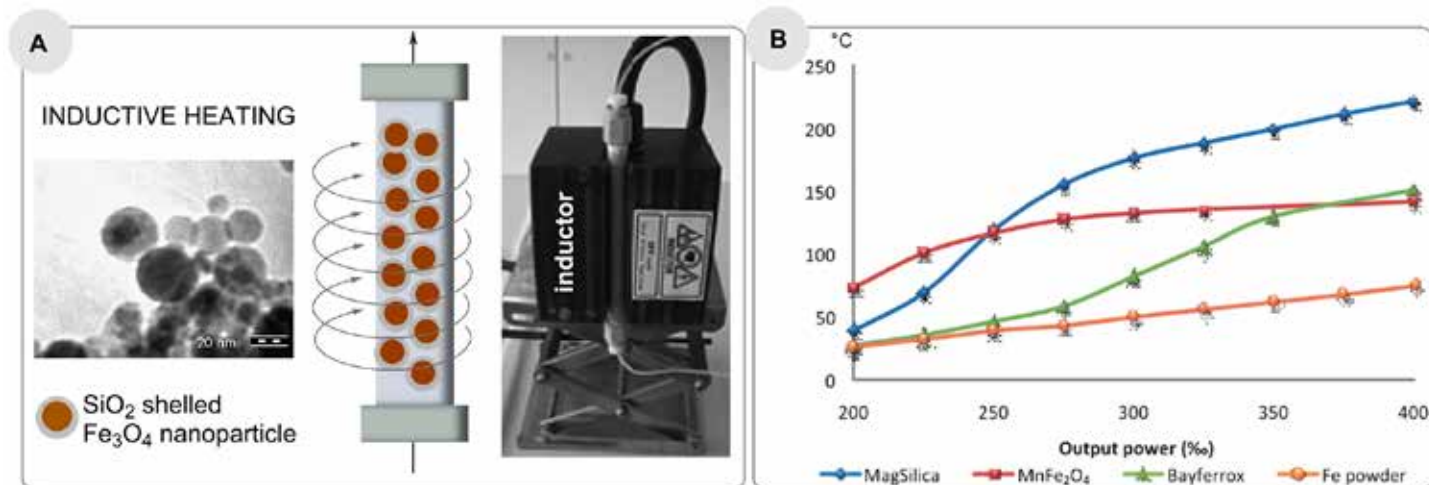
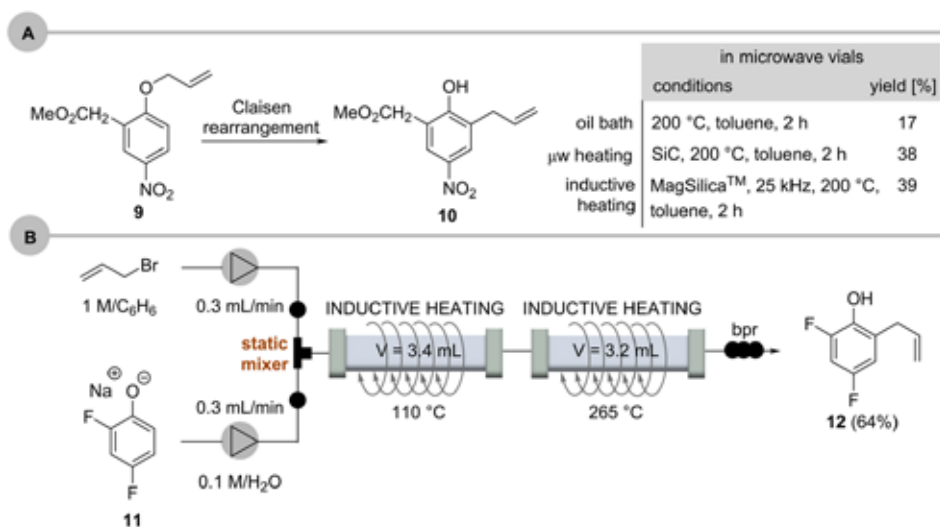


Figure 4: a) Flow reactor filled with magnetic nanostructured particles (MagSilica™) and packed bed reactor embedded in inductor (right); b) heating profile of different materials exposed to an electromagnetic field. Heating profiles of MagSilica™, MnFe₂O₄, Bayferrox™, and iron powder.



Scheme 8: Claisen rearrangement in flow: a) comparison between conventional heating (external oil bath), microwave irradiation, and inductive heating; and b) coupled flow-through protocol consisting of O-allylation and Claisen rearrangement for the continuous synthesis of 2-allyl-4,6-difluorophenol (SiC = silicon carbide) [52].

tions by rapid heating, because flow chemistry usually involves the use of pressure-stable reactors, which leads to shortened residence times. Inductive heating, in addition to microwave irradiation [44–46] heating, can serve as an indirect and rapid heating technology that, when combined with pressure-resistant microstructured flow reactors, enables “flash” heating so that even supercritical conditions can be achieved. In this context, Poliakoff and co-workers used supercritical water to perform several industrially relevant and continuously guided conversions, using microwave irradiation as an indirect heating method [47]. Other examples in which the two enabling technologies microwave and flow were combined are the Dimroth rearrangement exemplified for the conversion of 1,3-thiazine 3 to the corresponding 3-substituted hydroxypyrimidine 4 (Scheme 7, reaction 1) [48].

A noteworthy example was recently published by Organ and co-workers [49]. A three-component reaction of an aldehyde 5, a secondary amine 6, and a terminal alkyne 7, afforded arylpropargylamines 8 in up to 84% yield under flow conditions (Scheme 7, Reaction 2). Microwave irradiation interacted with a thin foil of Cu or Au that served as a catalyst inside the glass capillary. The work must be highlighted in that the actual temperature of the glass/metal surface could

be determined locally using a high-resolution IR camera. It was found to be 950°C and not 185°C of the reaction mixture itself. These studies are noteworthy because it can be assumed that the temperatures determined by Organ locally on metal surfaces can also be transferred to inductively heated materials including superparamagnetic nanoparticles.

3.2 Reactions under flow conditions accelerated by inductive heating

3.2.1 First steps and comparison with other techniques

As mentioned in the introduction, various physical phenomena enable rapid heating of inductive materials such as steel or copper, or fixed-bed materials composed of steel beads as well as superparamagnetic nanoparticles in an oscillating electromagnetic field. Kirschning and co-workers introduced nano-

structured particles based on Fe₂O₃/Fe₃O₄ coated with silicon dioxide (core-shell nanostructured particles), called MagSilica™ to be used as fixed-bed materials in many different continuous flow processes (Figure 4a) [50].

These materials are excited very rapidly in a medium frequency (25 kHz) electromagnetic field, heating reaction mixtures in packed bed reactors to temperatures up to 250°C, which was measured at the reactor outlet (Figure 4b).

The heat is generated only at the surface of the iron oxide nanoparticles (eddy currents) and this is dissipated to the surrounding environment, which is why the bulk temperature must be much lower than the surface temperature of the nanostructured particles (Figure 4b).

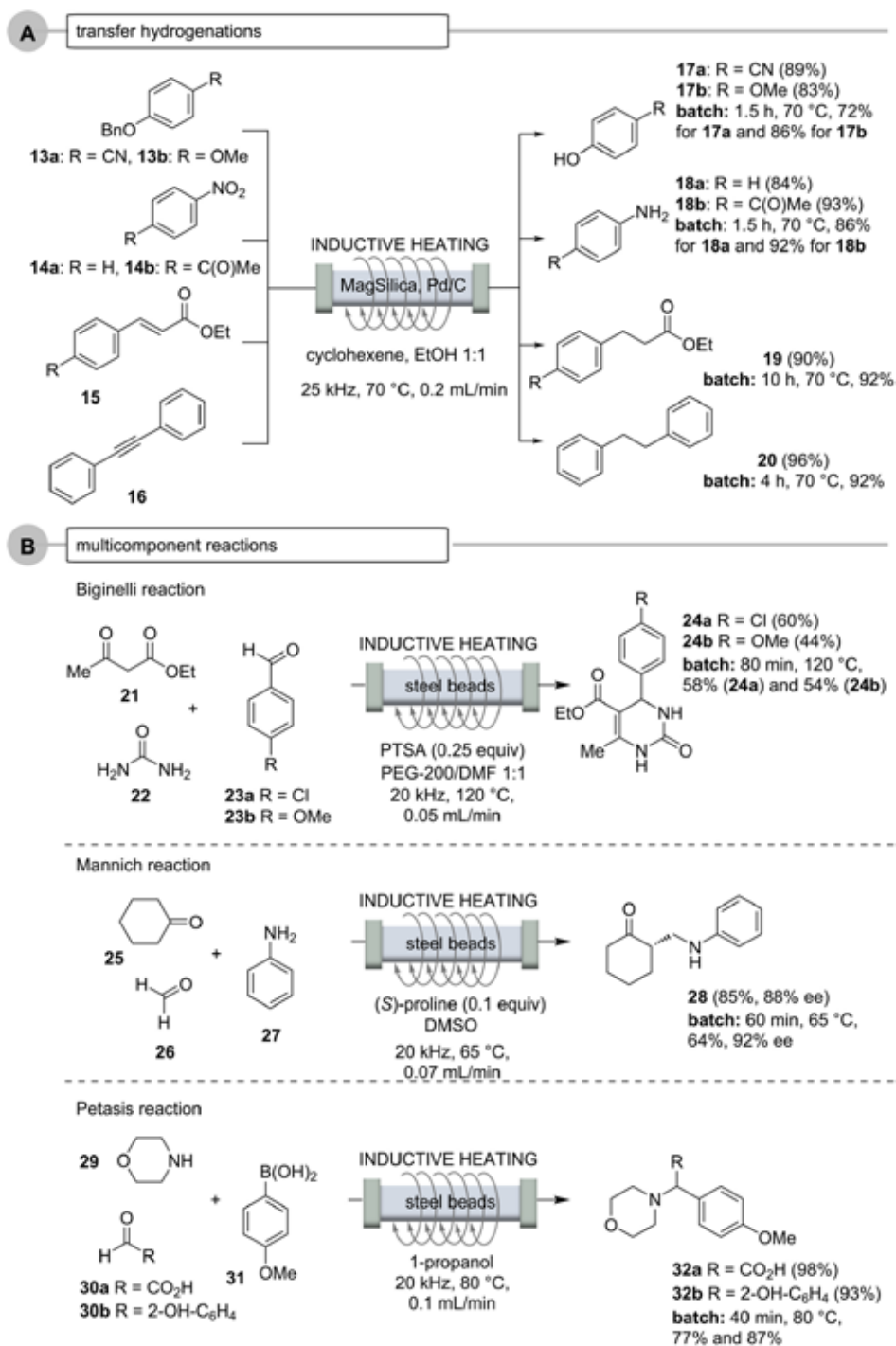
The Claisen rearrangement of the electron-deficient aryl allyl ether 9 was chosen to compare the versatility and performance of inductive heating with conventional and microwave heating (Scheme 8a) [50]. The effectiveness of inductive heating is clearly comparable to microwave-induced heating. In continuation of these studies, a two-step sequence was developed that showed Claisen rearrangements can be accelerated in water as solvent (Scheme 8b) [51]. The phenolate salt 11 was mixed with allyl bromide in a static mixer and inductively heated to 110°C at 110–160 bar to form the O-allyl

phenol which was heated in a second reactor to 265°C where the Claisen rearrangement under near-critical conditions occurred to yield 2-allyl-4,6-difluorophenol (**12**) in 64% yield. In this example, the two reactors made of steel were heated directly by the external electromagnetic field.

Other comparisons include the Pd-mediated transfer hydrogenations using ethanol in cyclohexene (Scheme 9, Case A), multicomponent reactions (Scheme 9, Case B), pericyclic reactions (Scheme 10, Case A) and Pd-catalyzed reactions (Scheme 10, Case B) [53]. Noteworthy, packed bed fillings used for the transfer hydrogenations (Pd) are reusable for several reductions without the need to adjust the overall reaction conditions (flow rate and residence time, temperature, etc.).

Multicomponent reactions (MCRs) are of particular interest in the field of flow chemistry because this enabling technique can be easily automated. Thus, protocols can be iteratively repeated by simply changing building blocks so compound libraries can be quickly accessed [54-55]. The formation of heterocycles traditionally often requires very harsh conditions, so high pressure and high temperature can greatly accelerate many of these transformations. This indeed is the domain of flow chemistry. The Biginelli [56-57], Mannich [58] and Petasis [55,59-61] reactions are typical representative examples, and these have been transferred into flow protocols (Scheme 9B). Steel beads serve as fix-bed materials to inductively heat glass reactors. For the Biginelli reaction, solubility of the starting urea (**22**) as well as the products **24a** and **24b** were an issue that was overcome by using a solvent mixture of PEG/DMF 1:1. Also the proline-catalyzed asymmetric Mannich reaction was achieved with cyclohexanone (**25**), formaldehyde (**26**), and aniline (**27**) and 10 mol % of the organocatalyst to yield β -aminoketone **28** in 85% yield (88% ee), in less than 1 hour. Although a significantly higher yield was achieved compared to the batch experiment, a slight reduction in enantioselectivity was observed. The Petasis or Petasis boron-Mannich (PBM) reaction of glyoxalic acid (**30a**) or salicylic aldehyde (**30b**), with morpholine (**29**) and *p*-methoxyphenylboronic acid (**31**) furnished α -aminocarboxylic acid **32a** and phenol **32b** in excellent yield (98% and 93%), again much higher than the yields found for the batch protocol (77% and 87%).

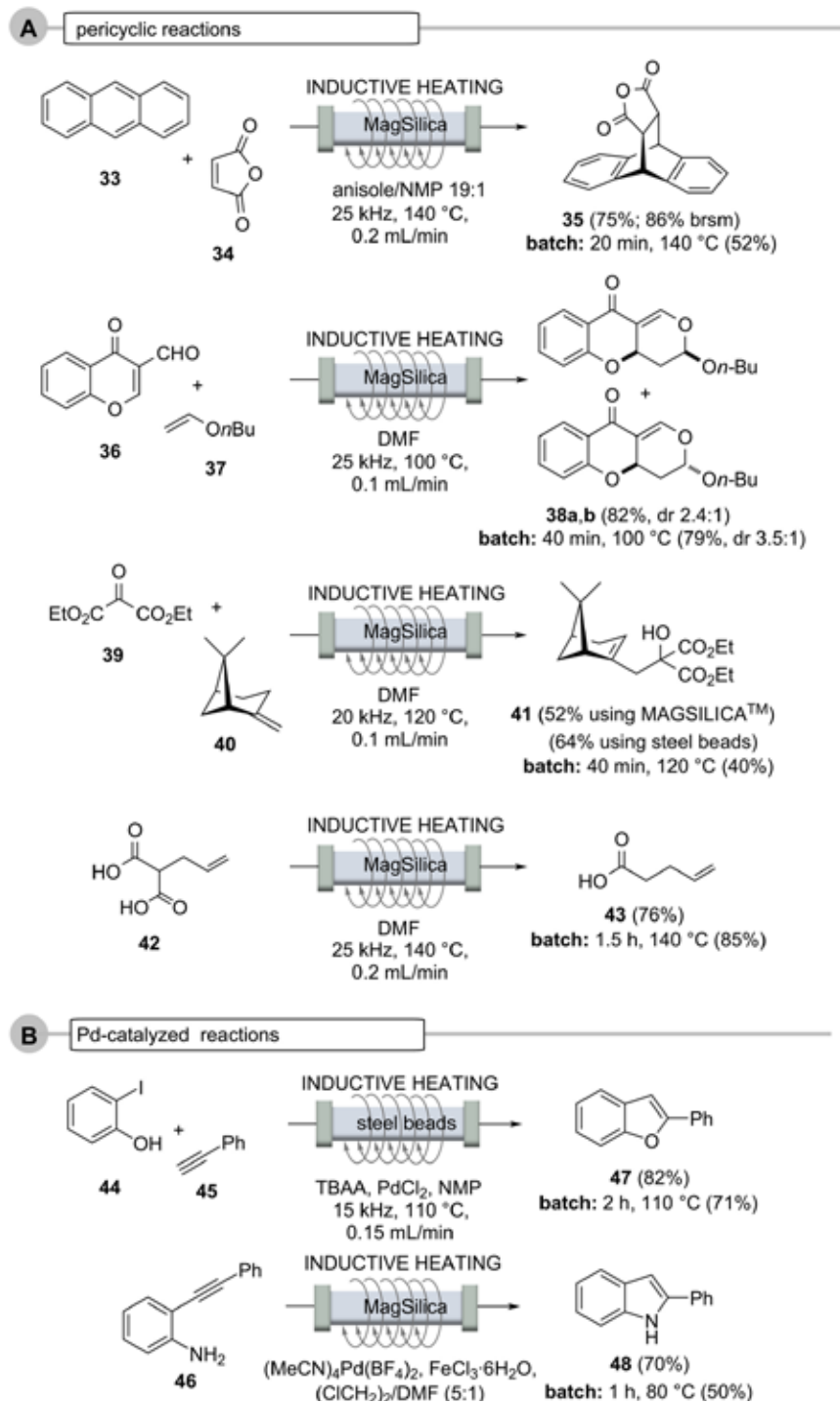
Pericyclic reactions such as the Diels-Alder and hetero-Diels-Alder cycloadditions, the Alder-En reaction, as well as the decarboxylation of α -alkylated malonic acids, are also suitable for flow protocols in combination with inductive heating (Scheme 10, Case A) [53]. The yields, but especially the residence times of the reactions, outperformed those of the analogous experiments carried out under batch conditions by far [62-64].



Scheme 9: Continuous flow reactions and comparison with batch reaction (oil bath). a) Pd-catalyzed transfer hydrogenations using ethanol in cyclohexene [53], b) multicomponent reactions.

The heating method was also successfully tested on various thermally conducted pericyclic reactions (Scheme 10, Case A), such as in (hetero)-Diels-Alder reactions (anthracene (**33**) and maleic anhydride (**34**) to the cycloaddition adduct **35** and chromene carbaldehyde **36** and enol ether **37** to the diastereomeric pyrano-chromenes **38**), Alder-En reactions (oxomalonate diethyl ester (**39**) and β -pinene (**40**) to give the α -pinene derivative **41**), and the thermal decarboxylation of the malonic acid derivative **42** to give pent-4-enecarboxylic acid (**43**). In many cases, the flow protocol provided improved yields compared to the corresponding batch syntheses.

Palladium-catalyzed cross-coupling reactions require higher temperatures and thus can be realized in an inductively heated flow



Scheme 10: Continuous flow reactions and comparison with batch reaction (oil bath). a) pericyclic reactions and b) Pd-catalyzed reactions.

system [65-71]. This is exemplified for the tandem synthesis of benzofuran 47 and phenylindole 48 (Scheme 10, Case B) starting from phenol 44 and aniline derivative 46, respectively. The latter reaction was carried out in a glass reactor filled with MagSilica™ [53].

3.2.2 Using chemically active fixed-beds (stoichiometric reagents)

Flow chemistry can be advantageously combined with the use of chemically active fixed-bed materials, especially heterogeneous catalysts. Here, too, the reactor material can either be heated directly by induction or there are additives in the fixed-bed material that interact with the oscillating electromagnetic field. Recently, Rebrov

and co-workers disclosed the direct amide formation of a carboxylic acid and aniline using high energy ball milling to prepare the sulfated TiO_2 (50 wt %)/ NiFe_2O_4 (50 wt %) catalyst that serves as fixed-bed material (Scheme 11, Case A) [72-73]. The reaction was carried out at 150 °C and an internal pressure of 7 bar. Remarkably, the process could be operated for 15 hours with a slight decrease of efficiency. Importantly, the catalyst activity can completely be restored when heating the packed bed to 400 °C exposed to an air flow. The importance of this study is the fact that no activating agents are required, and water is the only byproduct.

Organometallic chemistry and heating are not among the most intuitively sensible combinations. However, prior to the formation of an organometallic species, e.g., from the metals magnesium or zinc, (thermal) activation is required. This was demonstrated for the Reformatsky reaction (Scheme 11, Case B) [53,74], in which zinc powder was mixed with MagSilica™ and positioned inside the flow reactor. For example, 2-bromopropanoic acid ethyl ester (52) and acetophenone 51 were reacted in a heated fixed-bed reactor with the mediation of zinc to give the Reformatsky product 53, and, as commonly observed, in significantly improved yields compared to the corresponding batch processes.

Oxidations, especially metal oxide-based variants, are among the most frequently performed chemical reactions. Interesting examples are MagTrieve™, which contains CrO_2 and nickel peroxide (NiO_2). Both were mixed with MagSilica™ and used as fixed-bed materials (Scheme 11, case C) [75].

At this point, it is important to note that CrO_2 , despite its paramagnetic properties, does not heat up in an oscillating electromagnetic field because it does not exhibit conductive properties, so it had to be mixed with MagSilica™. Several oxidations were performed, including those of anthracene (33), propargyl alcohol 55 and testosterone (57), which proceeded smoothly with 80%, 93%, and 95% yields, respectively, in a fraction of the time required for the corresponding batch processes. In a simplified purification

protocol, potential metal impurities were then removed using a magnet. This approach could facilitate the use of metal oxides in industry for a broader range of oxidative applications. NiO_2 , on the other hand, was used to achieve the dehydrogenation of amines (to nitriles) and to perform the α,β dehydrogenation of ketones 61.

3.2.3 Using chemically active fixed beds (catalysts)

Copper metal in the form of wires or turnings can also be inductively heated when placed inside flow reactors (Scheme 12, Case A). There, it performs a second role by also becoming a source for a copper catalyst, either by being released into solution or by acting as a surface-active species capable of promoting “click” reactions between alkynes and

azides [76–81]. The process can be coupled with in situ generation of the azide from the corresponding bromide. The 1,2,3-triazoles are formed in up to 99% yield and in less than 10 minutes residence time, which includes azide formation prior to the cycloaddition step. Interestingly, this process could not be successfully repeated under conventional batch conditions. Organ's findings [49] suggest the inductive heating technique creates local hot spots, either on the copper surface due to skin depth effects or alternatively in copper nanoparticles released into solution, likely leading to a dramatic acceleration of the cycloaddition reaction.

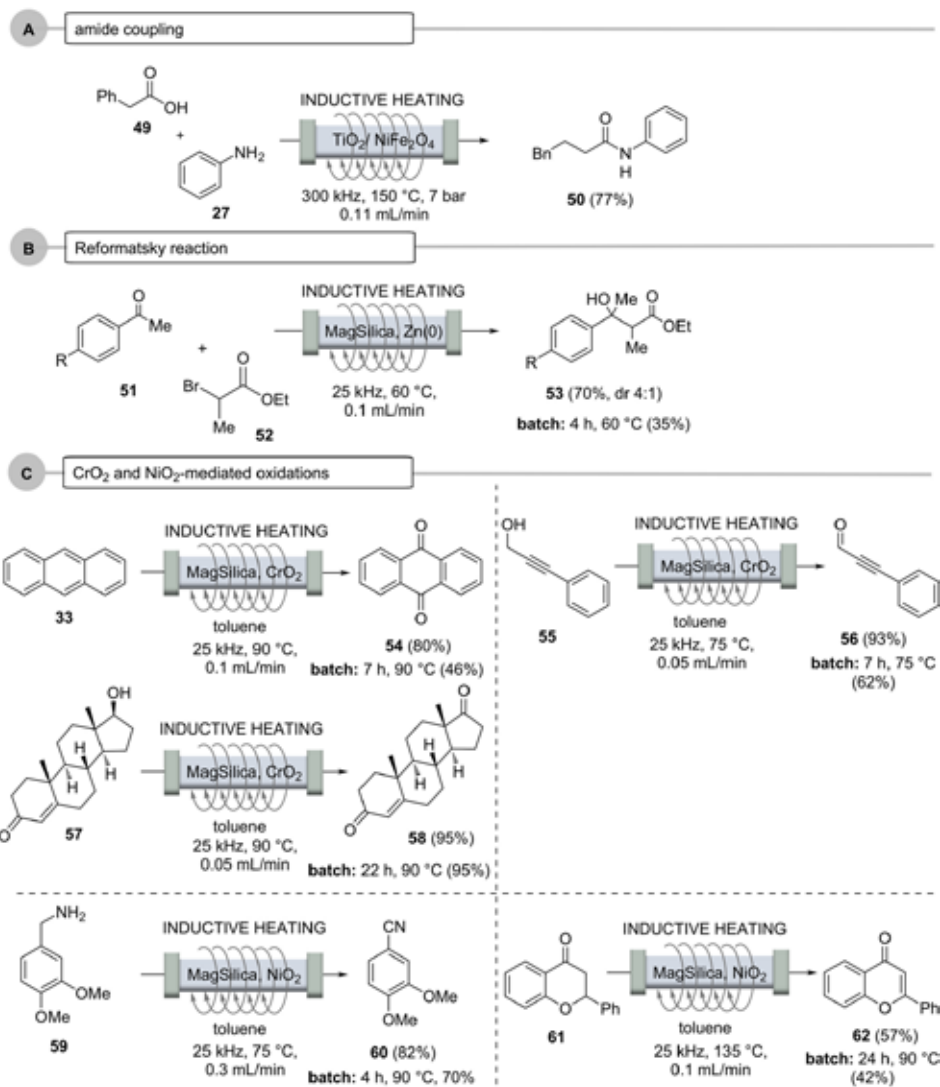
Reactions with soluble metal complexes or metal nanoparticles, used in transition-metal catalysis, are often avoided, especially if the metal contamination in the product exceeds certain limits. This is particularly true for the pharmaceutical industry. A continuous flow protocol for oxidations of alcohols to aldehydes or ketones using gold nanoparticles in the presence of oxygen gas or atmospheric air was achieved by modifying the silica shell of nanostructured MagSilica™ with gold nanoparticles (Scheme 12, case B). After heating these modified SPIONs in an electromagnetic field, a continuous process could be established by oxidation with molecular oxygen introduced into the reaction stream via a tube-in-tube membrane reactor, a process that should be very attractive for industrial applications, as oxygen or air act as cheap and environmentally friendly oxidants [82].

An interesting combination of SPIONs and transition-metal catalysis opens up when both concepts are combined architecture-wise [83]. For instance, catalytically active metal nanoparticles, e.g., consisting of Pd(0), can be deposited on the silicate surface of MagSilica™, so the required heat for Pd(0)-mediated catalysis can be generated directly by the functionalized nanostructured particles (Scheme 12, case C) [50].

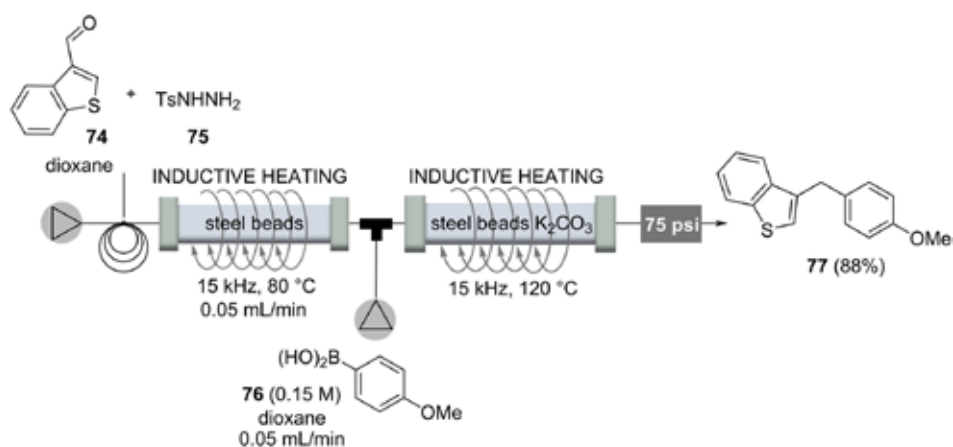
This was achieved by reductive precipitation of Pd(0) nanoparticles from ammonium-bound tetrachloropalladate [84–85], which showed good catalytic activity in various cross-coupling reactions under flow conditions. In these reactions, the leaching of palladium was as low as 34 ppm for Suzuki–Miyaura reactions and 100 ppm for Heck reactions. Importantly, the functionalized nanoparticles could be reused several times without observing a decrease in catalytic activity.

3.2.4 Multistep processes

The inductive heating technology has also been used in multistep processes targeting drugs or important molecules in the fragrance industry.

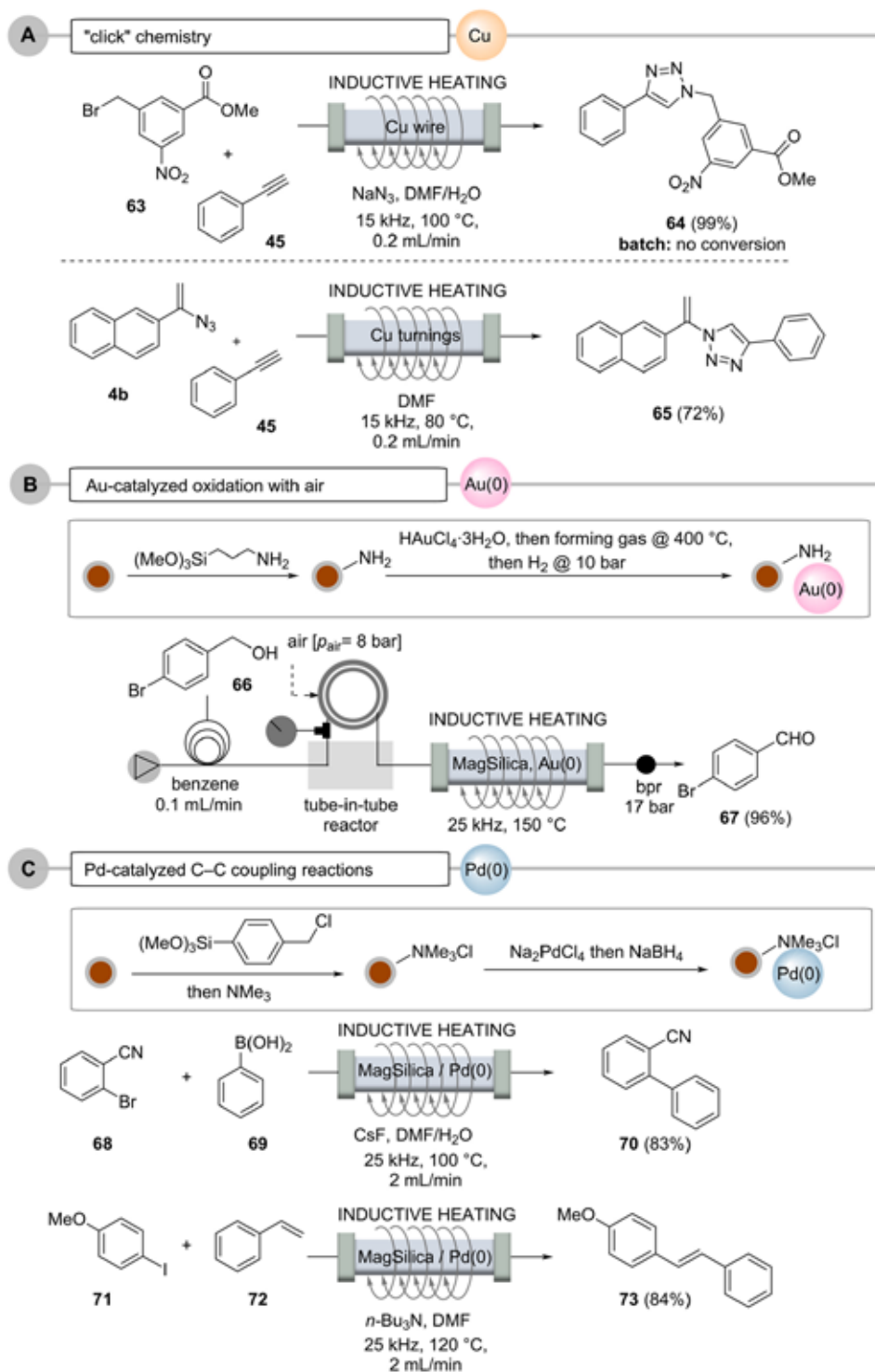


Scheme 11: Reactions under flow conditions using inductively heated fixed-bed materials serving as stoichiometric reagents.



Scheme 13: Two step flow protocol for the preparation of 1,1'-diaryllkanes **77** from ketones and aldehydes **74**, respectively, and boronic acids **76**.

The first example deals with a metal-free carbon-carbon-bond formation process between tosylhydrazones generated from the corresponding aldehydes **74** and boronic acids **76**, yielding a reduced arylation product **77** [86–87]. Mechanistically, either diazo or carbene intermediates can be proposed, as Barluenga has outlined, and migration of the aryl group leads to an alkylboronic acid, which is



Scheme 12: Reactions under flow conditions using inductively heated fixed-bed materials serving as catalysts: a) with copper metal, b) with Au-doped MagSilica™, and c) with Pd-doped MagSilica™.

hydrolyzed by protodeboronation, yielding the arylation product 77. A two-step flow protocol began with the carbonyl compounds (e.g., 74), and the first flow step yielding tosylhydrazones that were transferred directly to the second reactor to be coupled with boronic acids (Scheme 13). Both steps required heating, which was performed by electromagnetic induction of a fixed-bed material based on steel beads. A continuous two-step flow process over a period of almost two days yielded the arylation product in 88% yield, demonstrating the robustness of the process [87].

The use of water as a green solvent is a greatly increasing field of

research. But besides its reduced environmental footprint, water features unique physicochemical properties at supercritical conditions. The five-step synthesis of a typical antipsychotic drug iloperidone (80) is an impressive example of how supercritical water can be used as a privileged solvent in organic transformations (Scheme 14) [88]. Because of space limitations, we here only highlight the last of five steps, which all involve inductive heating between 110–180 °C and four out of five steps are performed in supercritical water. Phenol 79 and the N-alkylated product 78 were mixed and pumped through a 1/8" stainless steel reactor, heated to 180 °C at 4.5 MPa for 7.5 minutes. These conditions allowed to suppress the decomposition of the N-alkylation product 78 by using a 1/8"-reactor. The subsequent purification was realized by a clever catch-and-release protocol based on a silica column, yielding iloperidone (80, 67%).

The tricyclic antidepressant hydrochloride of amitriptyline (84) was the target of a multistep continuous flow protocol in which, for one reaction step, inductive heating was used to achieve water elimination triggered exclusively under thermal conditions [54]. The flow process started with a multilithiation sequence that included a carboxylation and a Parham cyclization and hence a Grignard alkylation of ketone 82 using reagent 81. The resulting alcohol 83 was subjected to thermolysis that led to water elimination. This step proceeded in just 30 seconds by employing the inductive heating technique. The crude elimination product was then mixed with a 1 M HCl solution in iPrOH that initiated crystallization of the hydrochloride salt of amitriptyline (84) (Scheme 15).

Iso E Super® (88) [89] is one of the most successful synthetic fragrances ever developed [90]. It is a component of a variety of perfumes with varying ratios and is the first example of a single ingredient sold as perfume in the fragrance industry. Structurally, it is related to natural terpenes. Starting from myrcene (85), an inductively heated process was developed, initiated with a Diels-Alder cycloaddition that furnished ambrelux (87) (Scheme 16). This was cyclized under acidic conditions with Amberlyst 15™ ion exchange resin embedded inside the flow reactor. However,

successful conversion to an industrial process was hindered by the fact that polymerization of the starting material myrcene (85) could not be suppressed, leading to fouling of the catalyst and consequently to inactivation. The polymerization could be suppressed by preloading the reactor with the vinyl methyl ketone 86 before starting the process. Nevertheless, it could not be sustained over a longer period of time. By splitting the process into two independent operations, a yield of 56% (87 + 88) was obtained for the Diels-Alder cycloaddition, and suppressed polymerization at room temperature. This mixture was then converted in a second step and an Amberlyst 15™-catalyzed cyclization at 60 °C

gave **88** with a selectivity of 95%. Reactions such as polymerizations that inhibit the catalyst are side reactions that are very difficult to control, but this example sheds light on an often-overlooked limitation of flow processes.

Musk-like fragrances occupy a special position among perfumes. An illustrative multistep protocol with practical relevance to the fragrance and flavor industry is a three-step flow-through protocol leading to macrocycles with musk-like olfactory properties, which was realized under extreme conditions. These include the use of safety-hazardous reaction mixtures, the handling of explosive intermediates, and their pyrolysis at high temperatures (Scheme 17) [93]. Cyclohexanone (**25**) was mixed with conc. formic acid, and a mixture of H₂O₂ (30%)/HNO₃ (65%) in a PTFE reactor at rt. This led to the formation of the cyclic triperoxide **91** in 48% (isolated) yield. Interestingly, the equilibrium favors the formation of the trimer **91** over the corresponding dimeric diperoxide. The reaction mixture was then transferred to a continuous phase separator equipped with a semipermeable membrane, from where the organic phase was transferred to a stainless-steel loop reactor. Here, the macrocyclic triperoxide **91** was subjected to pyrolysis at 270°C. This was done by inductive heating and the residence time was only 12 minutes. The Macrolide® **89** was obtained in 14% together with the aliphatic macrocyclic **90**, the latter can be oxidatively converted into the corresponding ketone, which is of practical importance in the fragrance industry. It is clear that this process could not be established as a batch protocol due to the hazardous conditions.

CONCLUSION

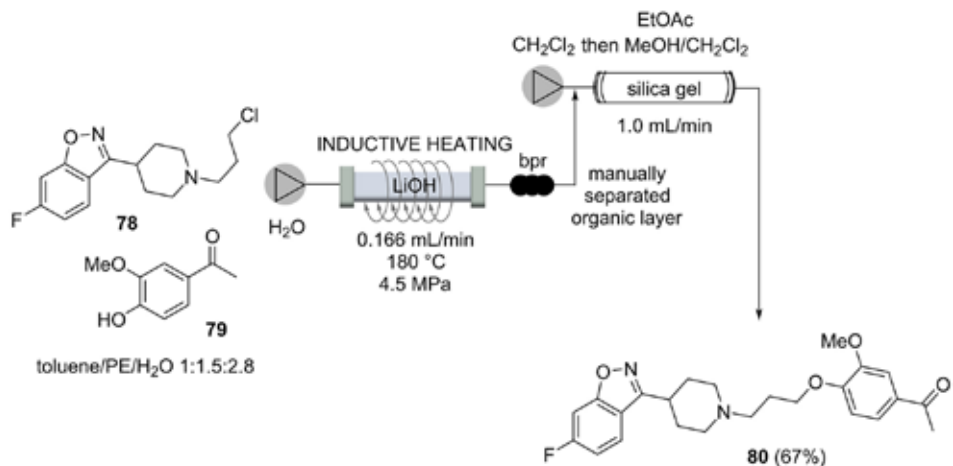
Inductive heating and flow chemistry are an ideal combination for performing continuously operated high-temperature and high-pressure syntheses. The technical setup is quite simple compared to corresponding microwave devices, the heating process is very efficient, and energetically extremely favorable. Remarkable examples in the field of fundamental chemical processes in a world that requires new solutions for energy supply show the power of inductive heating. In addition, academic examples draw attention to the use of continuously operated chemical processes with induction heating for the fields of bulk chemical production and the fragrance and flavor industries, as well as, eventually, the pharmaceutical industry. The authors are certain that this combination of enabling technologies holds great future opportunities.

ACKNOWLEDGMENTS

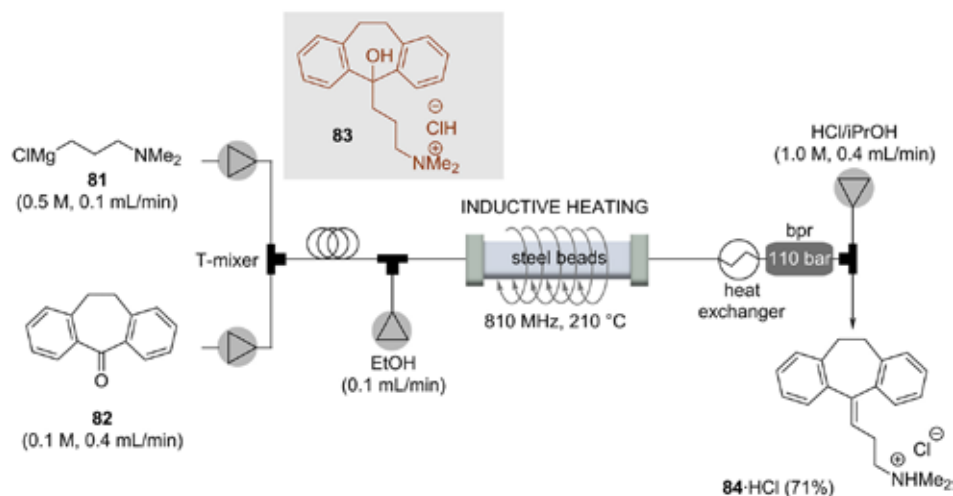
The authors wish to thank Dr. J. Panten (Symrise AG, Holzminden, Germany) and Dr. H. Herzog (EVONIK Degussa GmbH, Essen, Germany) for supporting their work on flow.

NOTES

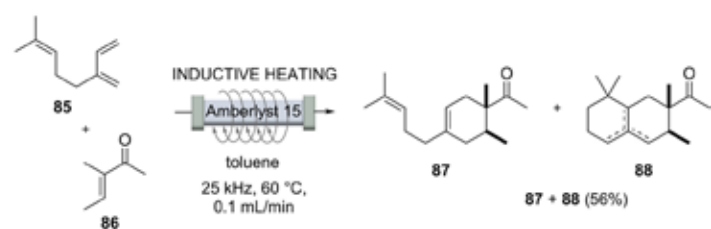
This article is part of the thematic issue "Platform and enabling technologies in organic synthesis." 



Scheme 14: O-Alkylation, the last step in the multistep flow synthesis of loperidone (**80**) accompanied with a “catch and release” purification protocol.



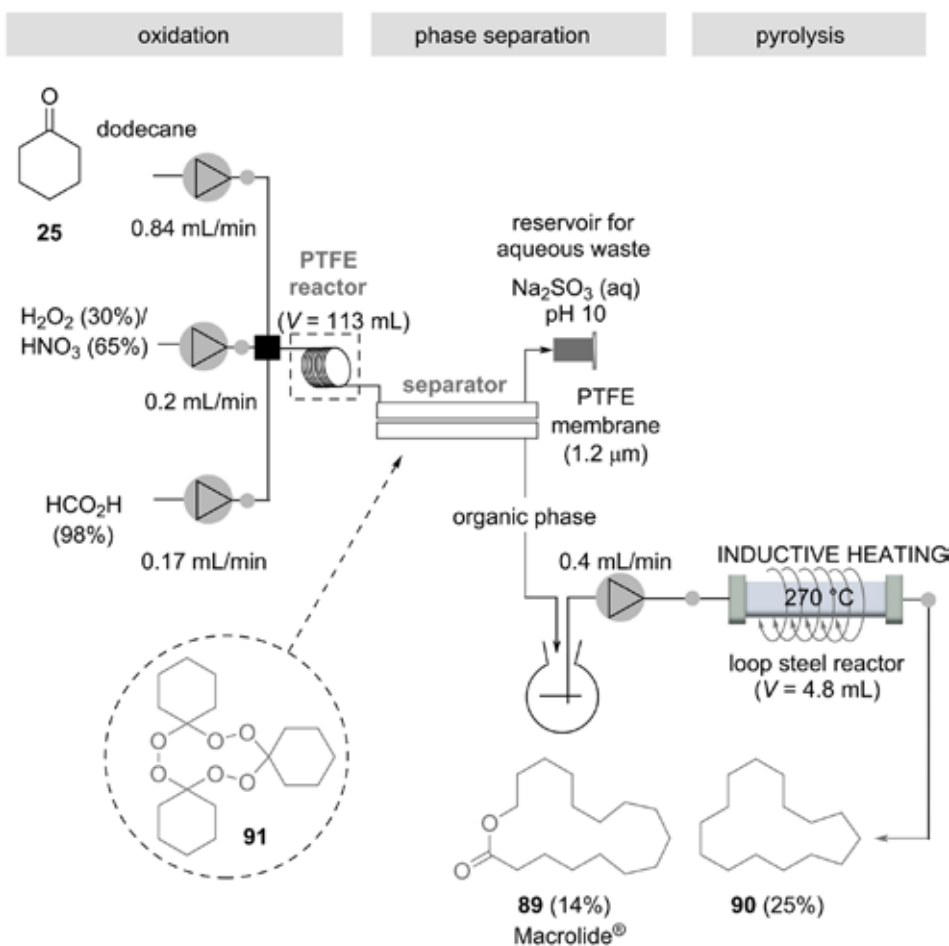
Scheme 15: Continuous two-step flow process consisting of Grignard reaction followed by water elimination being the last steps of a multistep flow synthesis of the hydrochloride salt of amitriptyline **84**.



Scheme 16: Inductively heated continuous flow protocol for the synthesis of Iso E Super (**88**) [91–92].

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Scheme 17: Three-step continuous flow synthesis of macrocycles 89 and 90 with musk-like olfactive properties.

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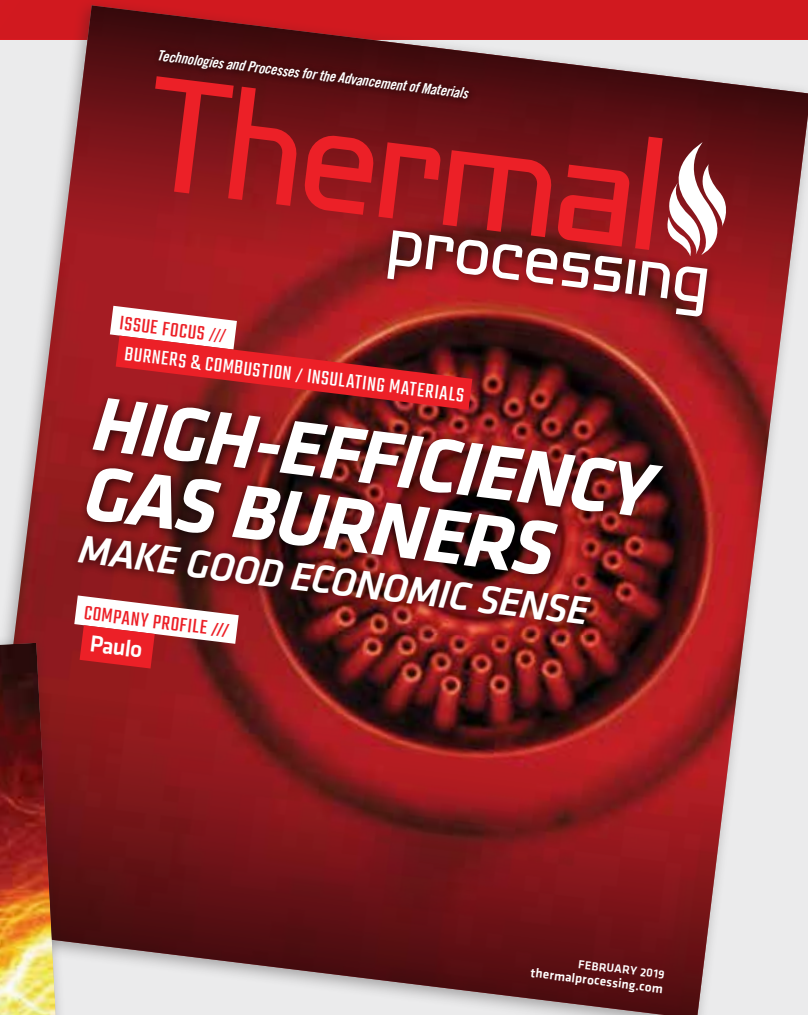
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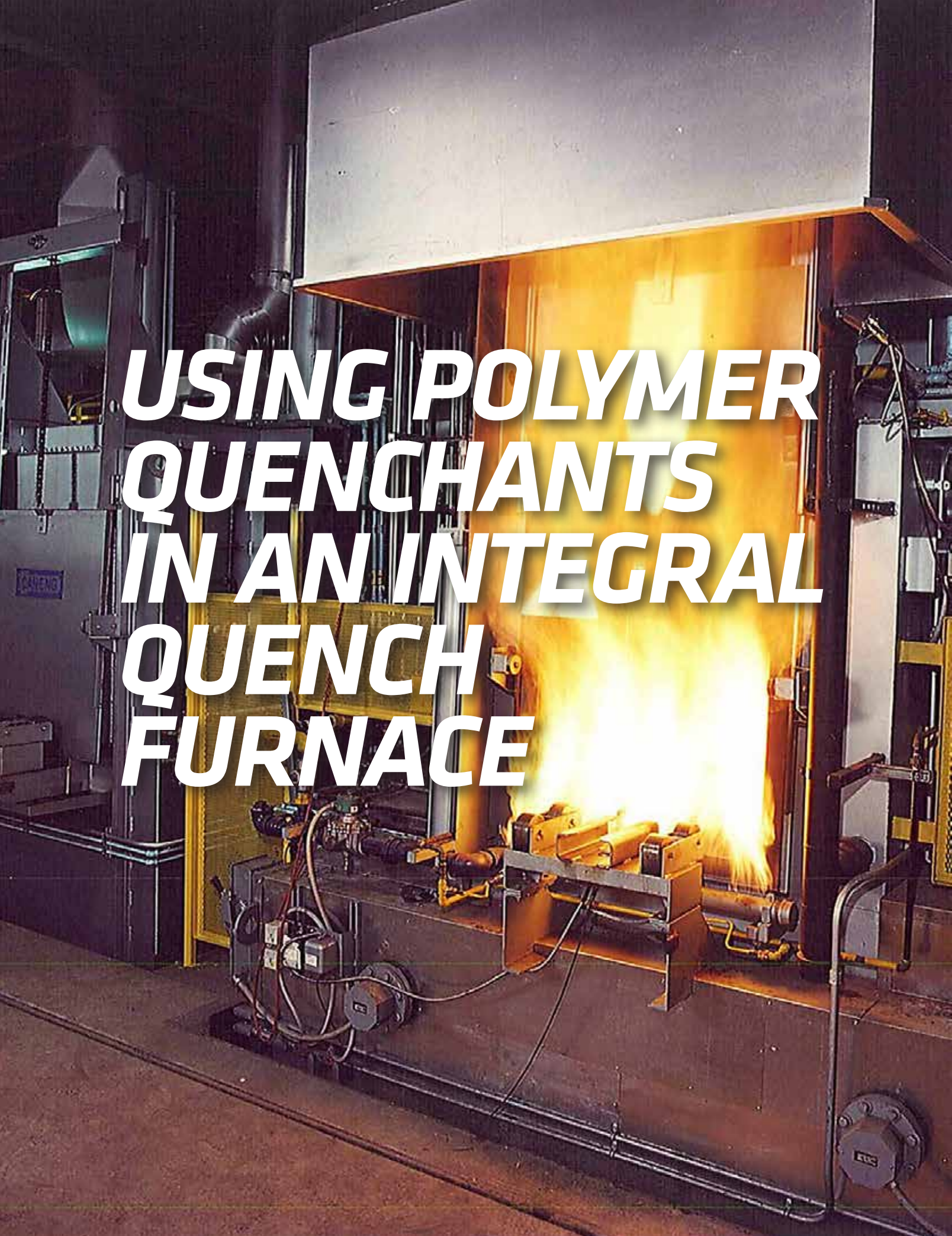
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***USING POLYMER
QUENCHANTS
IN AN INTEGRAL
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While this is more environmentally friendly, safer, and the in-tank cost is lower than oil, there are drawbacks that nullify the benefits of its use in a sealed quench furnace.

By D. SCOTT MACKENZIE

There have recently been several inquiries regarding the use of a polymer quenchant in a sealed or integral quench furnace. There are several drivers for this — cost, for example. While the polymer quenchants have a higher-per-gallon cost than oil quenchants, the in-use concentration of polymer quenchants is much less than an oil. There are also environmental issues. Polymer quenchants do not give off as much smoke as oil, and disposal is easier. The use of a polymer quenchant in a sealed quench furnace can be done, but it requires some special design elements. (Figure 1)

First, there is the hot zone. The inner door needs to be completely sealed to prevent water vapor from infiltrating the hot zone and increasing the chance for creating a decarburizing atmosphere. It is possible to use a much greater volume of atmosphere in the hot zone, creating a higher partial pressure of atmosphere in the hot zone. As far as I know, most manufacturers prefer to seal the inner door to prevent water vapor entering the hot zone.

GAS INTRODUCTION

Since the hot zone is sealed, it is often necessary to introduce nitrogen or other inert gas into the vestibule. This prevents the parts from oxidizing during part transfer into the quench and helps purge oxygen from the vestibule. Using an endothermic atmosphere can be done, but there is a much greater chance of explosion since the vestibule is below 760°C. Nitrogen is probably the easiest solution.

Lastly, there is the quench tank. When quenching in oil, the allowed maximum peak operating temperature during quenching is approximately 50°F (28°C) below the flash-point temperature. There is an additional 50°F (28°C) as a safety factor for hung loads, partially submerged loads, stuck elevator, etc. Using the “one pound per gallon of oil” rule of thumb, the temperature will rise about 70°F (39°C). This also determines the maximum operating temperature of the oil. For an oil with a flash point of 350°F (177°C), the maximum peak temperature during quenching would be 250°F (121°C) from subtracting the regulatory required 28°C, and the additional 28°C safety factor (177°C flash point — 28°C regulatory difference — 28°C additional safety factor = 121°C). Since the maximum peak temperature during quenching is governed by the “one kilogram to eight liters” (one pound per gallon) rule, or a 70°F or a 39°C temperature rise, the maximum operating temperature of the oil is 82°C and the maximum peak temperature during quenching is 121°C.

POLYMER QUENCHANTS

For polymer quenchants, the design rules are completely different and are based on different criteria. There are also different criteria based on different types of polymers. Looking at PAG-type polymers, these polymers have a distinct cloud temperature and are inversely soluble. The cloud temperature is where the polymer precipitates from the water. Inversely soluble means that the solubility of the polymer decreases as the temperature increases. Depending on the

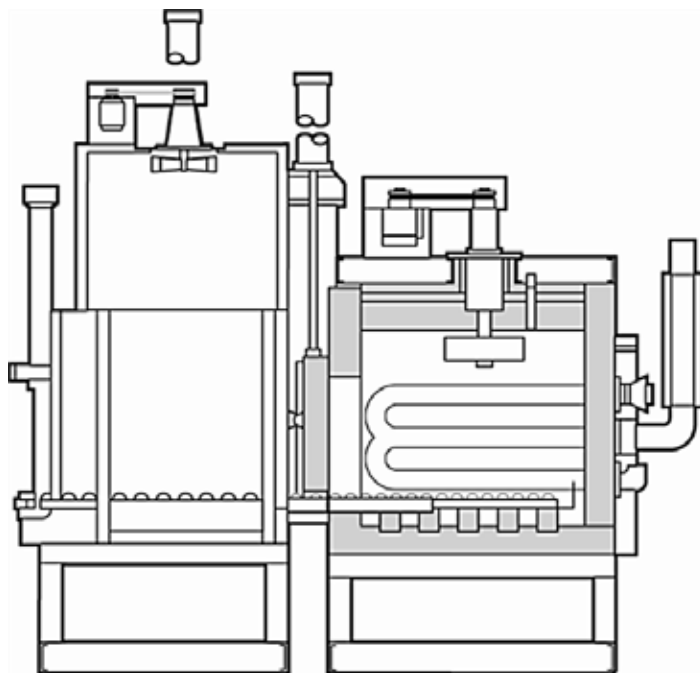


Figure 1: Schematic of a typical integral, or sealed, quench furnace. The hot zone is to the right and the quench vestibule is to the left, with the inner door separating the hot zone and quench tank. (Courtesy: Surface Combustion Corporation, Maumee, Ohio).

PAG polymer, the cloud temperature can range from 57°C to 82°C. Using the higher cloud temperature of 82°C (most common PAG type polymers), this establishes the “do not go past temperature” like flash point. However, there is no safety hazard associated with this temperature, just an extremely high drag-out of the polymer, a sticky mess, and excessive oxidation of the product. Therefore, you need some sort of safety factor for the temperature rise. Most people limit the peak temperature during quenching to a maximum temperature of 60°C (140°F). This temperature is a good compromise between excessive drag-out and oxidation of the polymer.

Other polymers have different criteria. For PVP-type polymers, there is no distinct cloud point, because it is above the boiling temperature of water. It is also inversely soluble. However, the maximum temperature is higher. Some people use 65°C-70°C as the maximum peak temperature during quenching, while others use a lower temperature. Also remember that the ability of the polymer to extract heat decreases as the temperature increases. As temperature decreases, the quench rate slows down. Because of this, often a much lower concentration of polymer is used if a higher allowable temperature rise is permitted. In continuous-type furnaces, where there is a quench chute, roughly half the concentration or less is needed because of the local temperature rise in the quench chute. However, use of a polymer quenchant in a continuous furnace has its own issues.



Polymer quenchants are more sensitive to agitation rates than oils. In general, polymers will require more agitation than oils. The amount is hard to quantify. Uniformity is the biggest issue.

This allowable temperature rise establishes the necessary quench tank size for a given workload size. In general, it takes about twice the volume in polymer as the volume of oil necessary. In other words, if a given sealed quench furnace can handle 1,000 kilograms of product using oil, the same furnace would have a load reduction of 50 percent, or about 500 kilograms, for the same size quench tank.

Additionally, polymer quenchants are more sensitive to agitation rates than oils. In general, polymers will require more agitation than oils. The amount is hard to quantify. Uniformity is the biggest issue.

QUENCH COOLING

Lastly, cooling of the quench is important for the next load. For oil quenchants, the quenchant is easily cooled with an air-oil heat exchanger, since the operating temperature is quite a bit higher than the ambient temperature. For polymer quenchants, since the operating temperature is near ambient temperature, the efficiency using an air-to-polymer heat exchanger is quite low. Additional cooling using a cooling tower and chilled water is necessary. This creates additional cost for installation.

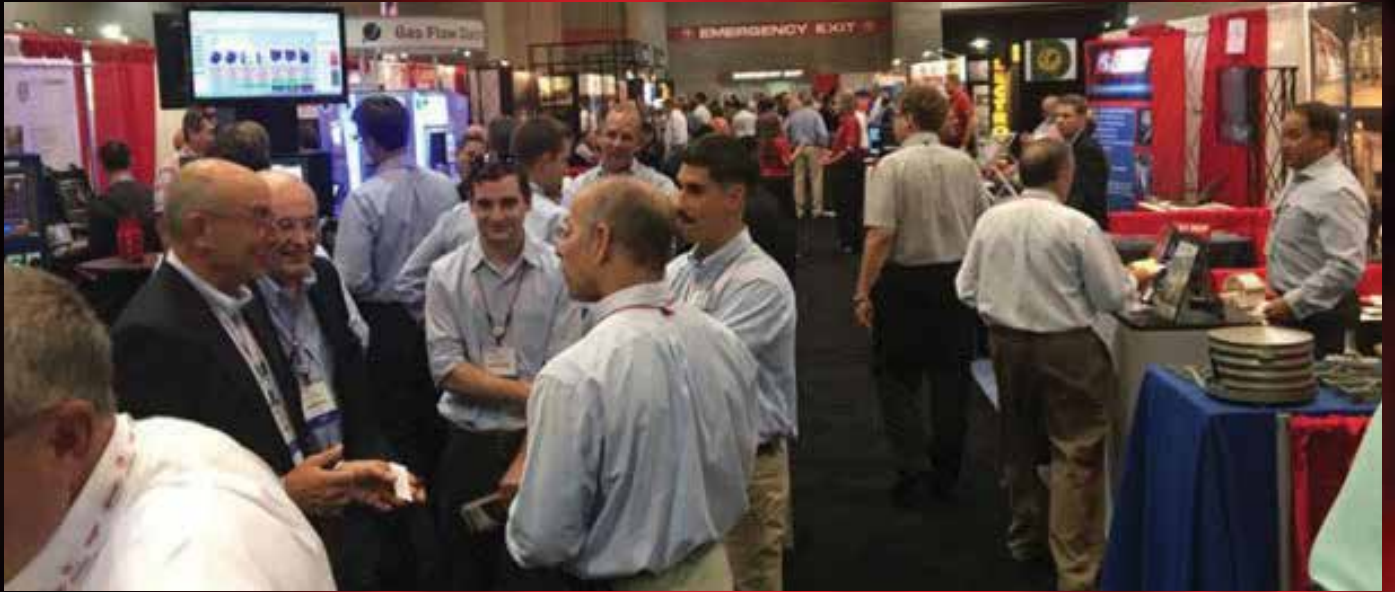
While the use of a polymer quenchant is much more environmentally friendly, safer, and the in-tank cost is much lower than oil, the reduced workload size; the necessity of chilled water to cool the quenchant; and the cost of modifying the furnace to be compatible with polymer quenchants, tend to nullify the benefits of the use of polymer quenchants in a sealed quench furnace. It can be done, and has been done successfully, but you must understand the limitations. 🔥



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HEAT TREAT EVENTS 2023



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ADVANCED SAM VALIDATES INTEGRITY OF DIFFUSION BONDS

When industrial manufacturers take advantage of the higher level of failure detection and analysis, the production yield and overall reliability of products improves significantly. (Courtesy: OKOS)

For bond quality testing, scanning acoustic microscopy offers far greater accuracy and reliability than traditional helium leak testing.

By DEL WILLIAMS

Industries with demanding metal-bonding applications are increasingly relying on diffusion bonding, an essential joining method for achieving a high-purity interface between two similar or even dissimilar metals. The process involves applying high temperature and pressure to metals mated together in a hot press, which causes the atoms on solid metallic surfaces to intersperse and bond.

For similar materials, bond strength approaches base material. For dissimilar materials, it is a function of the intermetallic compounds formed, the thickness of the intermetallic zone, and microscopic anomalies — such as voids — at the interface. To ensure the quality of the interface, materials engineers must analyze samples to validate the quality of the bond. Metrology systems are used to characterize the interface and provide a more quantitative analysis.

If either side of a welded or brazed joined assembly can be pressurized, a cursory helium leak check may be used. However, this method is more of an art and a science because it is highly dependent on user configuration and often produces inconsistent results. Also, the helium leak check cannot pinpoint the exact location of a leak: It only provides pass/fail results. Better technology and methods exist today.

SCANNING ACOUSTIC MICROSCOPY

Due to the imprecision of helium leak testing, experts in diffusion bonding are choosing to analyze samples through a much more advanced, data-driven inspection technique: scanning acoustic microscopy (SAM).

SAM is a non-invasive, non-destructive ultrasonic testing method. The testing is already the industry standard for 100 percent inspection of semiconductor components to identify defects such as voids, cracks, and the delamination of different layers within microelectronic devices. Now, the same rigor of quality testing and failure analysis is being applied to validate the integrity of diffusion-bonded metals.

“Unlike helium-leak testing, SAM will not only indicate when there is a defect in bonding, but also indicate the location and size; it is essentially the equivalent of an X-ray inside the part, so it is a much more comprehensive test method to ensure quality,” said Hari Polu, president of OKOS, a Virginia-based manufacturer of SAM and industrial ultrasonic non-destructive (NDT) systems. OKOS is a wholly owned subsidiary of PVA TePla AG, Germany, and offers both manual and automated inspection systems for flat panels, thin plates, circular discs, sputtering targets, and special alloys.

BENEFITS OF DIFFUSION BONDING AND SAM

Today, diffusion bonding offers substantial benefits to various industries, such as the ability to bond dissimilar metals and high-value alloys. The technology also enables advantageous applications for conformal cooling, which is used in injection molding and die casting.

The importance of designing a dissimilar metal joint often lies in a desire to expose the correct metal surface to specific environmental conditions where a single alloy may not perform as well. Another reason is to introduce material systems that are lighter in weight or provide a level of corrosion resistance that can only be achieved by “packaging” dissimilar metals.

Commercial processes of interest are titanium-to-iron-nickel alloys, titanium-alloys-to-stainless-steel, copper-to-steel, and even



Companies like OKOS offer a range of SAM products from compact, tabletop units to fully automated production line systems. (Courtesy: OKOS)

some aluminum-to-metal applications. The process also enables coupling between different alloys in the same material group, such as mild steel, tool steel, and Al MMC. As a result, this technology has attracted the interest of design engineers in the semiconductor, aerospace, and energy industries.

POTENTIAL APPLICATIONS

Diffusion bonding also has tremendous potential applications for conformal cooling applications. The concept is to bond layers in which cooling channel geometry is optimized to dissipate heat. Various geometries and various materials benefit from such an approach. Depending on the size of the commercial press, layers



For bond quality testing, scanning acoustic microscopy offers far greater accuracy and reliability than traditional helium leak testing. (Courtesy: OKOS)

can be bonded up to a stack height of 600mm.

Another application related to conformal cooling is for plastic injection molds. Conformal cooling channels are an engineered solution to facilitate the requirement for rapid cooling. Dies are typically made in two-layer designs of tool steel and material such as stainless steel (STAVAX).

For validating the integrity of diffusion bonds, advanced, phased array scanning acoustic microscopy works by directing focused sound from a transducer at a small point on a target object. The sound hitting the object is either scattered, absorbed, reflected, or transmitted. By detecting the direction of scattered pulses as well as the “time of flight,” the presence of a boundary or object can be determined as well as its distance.

To produce an image, samples are scanned point-by-point and line-by-line. Scanning modes range from single-layer views to tray scans and cross-sections. Multi-layer scans can include up to 50 independent layers. Depth-specific information can be extracted and applied to create two- and three-dimensional images without the need for time-consuming tomographic scan procedures and more costly X-rays. The images are then analyzed to detect and characterize flaws such as the size and location of any disbonding at the interface layer of diffusion bonded metal parts.

“OKOS has leveraged the lessons and the tight specifications from the semiconductor world and adapted our SAM scanning systems for various form factors to provide unique solutions for metals and alloys in the industrial markets,” Polu said. “With this type of testing, we can inspect materials at a level one to two orders of magnitude better than helium-leak detection to discover disbonding flaws and defects [at the interface] as small as 50-microns that were previously undetected.”

Companies like OKOS offer a range of SAM products from compact, tabletop units to fully automated production line systems.

When high throughput is required for 100 percent inspection, ultra-fast single or dual gantry scanning systems are used along with 128 sensors for phased-array scanning. Multiple transducers can also be used to simultaneously scan for higher throughput.

“In tests with a Fortune 500 multinational company on a sample

of aluminum and steel, the equipment was able to scan the material in three minutes. Before, it took them 40 minutes to do one part,” Polu said.

As important as the physical and mechanical aspects of conducting a scan, the software is critical to improving the resolution and analyzing the information to produce detailed scans. Multi-axis scan options enable A, B, and C-scans, contour following, off-line analysis, and virtual rescanning for metals and alloys, which result in highly accurate internal and external inspection for disbonding defects via the inspection software.

Various software modes can be simple and user friendly, advanced for detailed analysis, or automated for production scanning. An off-line analysis mode is also available for virtual scanning.

“OKOS decided early on to deliver a software-driven, ecosystem-based solution,” Polu said. The company’s ODIS Acoustic Microscopy software supports a wide range of transducer frequencies from 2.25 to 230 MHz.

Polu estimated the software-driven model enables them to drive down the costs of SAM testing while delivering the same quality of inspection results. As a result, this type of equipment is well within reach of even modest testing labs, R&D centers, and material research groups.

“Every company will eventually move toward this level of diffusion bonding metal part inspection and quality control because the level of detection is far superior to helium leak detection,” Polu said. “The increased accuracy, reliability, and comprehensiveness as well as decreased cost and time involved are driving the change.”

When industrial manufacturers take advantage of the higher level of failure detection and analysis, the production yield and overall reliability of products improve significantly. Projects are expedited and potential points of failure are eliminated in the field. ♨



ABOUT THE AUTHOR

Del Williams is a technical writer based in Torrance, California. OKOS is a wholly owned subsidiary of PVA TePla AG, Germany. For more information, go to www.okos.com.

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The screenshot shows a web browser displaying the Thermal Processing storefront for Solar Atmospheres - Eastern PA. The page features the Thermal Processing logo, a navigation menu, and a prominent banner for a 'NOW MONTHLY' subscription to the 'Thermal Processing' magazine for free. The main content area includes the Solar Atmospheres logo, contact information (1969 Clearview Road, Souderton, PA 18964), a company video titled '48 Foot Vacuum', and a Facebook follow button. The 'About Solar Atmospheres' section describes the company as a commercial heat treating provider with over 60 furnaces. A 'Related Articles' section lists 'Specialty Steel Treating Inc.' and 'Introduction Hardening is the ability of steel to partially or completely transform from...'. A 'Tweets' section shows a tweet from Solar Atmospheres Heat Treating (@SolarATM) about a newly designed Vacuum Oil Quench (VOQ) furnace.

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“I want to further define IHEA’s Sustainability/Decarbonization vision and solidify a roadmap for our membership.”

The Industrial Heating Equipment Association (IHEA) recently announced its 2023-2024 board of directors and executive officers. Taking over as president is Brian Kelly of Honeywell Thermal Solutions. Kelly recently shared with Thermal Processing his expertise in heat treating and what he plans to do as IHEA president.

How long have you been involved in the industrial-heating industry?

I started at the Hauck Manufacturing Company in December of 1995.

What are your areas of expertise?

I am presently the applications engineering manager at Honeywell Thermal Solutions (HTS), which is a position that is perfect for me. My areas of expertise would be working with customers to define their thermal needs and then designing a thermal system to achieve their goals. This can cover areas such as temperature uniformity, fuel efficiency, emissions, and, most recently, fuel flexibility and carbon reduction.

What is a typical workday for you at Honeywell?

In meetings and on-call with salespeople, engineering, channel partners, and customers on thermal-system and burner needs and how HTS can help them reach their goals or solve issues they have or are currently experiencing.

How long have you been a member of IHEA?

I attended the IHEA Combustion Seminar as a student a few years after joining Hauck, so I would say that I have been active in IHEA for well over 20 years after being encouraged to become involved by long-time mentors Mike Shay and Jack Marino.

On what IHEA committees/divisions have you served?

When I started to come to the IHEA meetings, I joined the Education Committee and, for quite a few years, have served as the Education Committee chairperson. I also chair the Annual Combustion seminar with the great help of the IHEA staff.

What are your goals for the association during your presidency?

I want to further define IHEA’s Sustainability/Decarbonization vision and solidify a roadmap for our membership as well as finalize the On-line Advanced Industrial Process Heating course to offer more educational opportunities for people in the thermal processing industry.

Anything else you’d like to share?

I am an avid Jeepster, so as many times a year as I can, I enjoy taking my Jeep to various off-road parks in Texas and doing some serious wheeling in the great and rocky terrain of Texas. I hope to get to Moab on one of these trips someday. I also enjoy hunting, golf, reading, movies, and traveling. 🏹

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MORE INFO www.ihea.org





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