JULY/AUGUST 2022 | VOL 180 | NO 5

# NATERIALS & PROCESSES

#### ADDITIVE MANUFACTURING DED FOR LARGE NEAR-NET SHAPE PARTS P. 13



Production of Ancient Iron

24

Addressing AM Challenges for Metal Components



*iTSSe* and *HTPro* Newsletters Included in This Issue





#### Sample Preparation Solutions for Metal Additive Manufacturing Materials



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#### **DIRECTED ENERGY DEPOSITION MOVES OUTSIDE THE BOX**

#### Judy Schneider and Paul Gradl

Metal additive manufacturing has steadily progressed over the past decade, with today's directed energy deposition processes enabling rapid builds of large structures in near-net shape—and with minimal machining required to achieve final dimensions.

#### **On the Cover:**

Cladding as a directed energy deposition method. Courtesy of NASA/DM3D.



#### **SUSTAINABILITY**

Researchers look at ways to make plastic in a more sustainable way and also use plastic waste to absorb emissions.



IMAT 2022 SHOW PREVIEW

IMAT—the International Materials Applications & Technologies Conference and Exhibition—and ASM's annual meeting will be held in New Orleans, September 12-15.



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The latest news about ASM members, chapters, events, awards, conferences, affiliates, and other Society activities.

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## MATERIALS & PROCESSES

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### **DISCOVERING SOLUTIONS AT IMAT**



elcome to our IMAT show issue with a focus on additive manufacturing (AM). The technology around AM continues to mature and find new applications. And new solutions are being discovered every day to overcome previous limitations.

One of the main challenges with AM is production time. A standard fused deposition modeling printer averages 100 mm/hour. And we are all familiar with the slow, meditative cadence of a typical AM build. Yet, if printing is

accelerated, vibrations occur, leading to poor quality and even misshapen components. Entire batches may need to be scrapped.

To address this problem, Dr. Chinedum Okwudire, a University of Michigan (U-M) professor and his students launched new software that doubles the typical 3D printing speed. Their software, called FBS for Filtered B Splines, performs this veritable magic by compensating for the usual vibrations caused by acceleration. "Say you want a 3D printer to travel straight, but due to vibration, the motion travels upward. The FBS algorithm tricks the machine by telling it to follow a path downward, and when it tries to follow that path, it travels straight," Okwudire explains.

Another development in the AM sector addresses previous limitations regarding component size. Our lead article shows how directed energy deposition now allows for extremely large structures to be built more rapidly and with minimal machining. The AM space keeps evolving and expanding. What other developments are on the horizon?

To learn about additional AM research, attend IMAT 2022, which will



U-M's high-speed 3D printing. Courtesy of E. Dougherty.

feature three days of programming on additive along with many other materials topics. In line with the conference theme of the Circular Materials Economy, Dr. Mrityunjay Singh, FASM, will deliver a special lecture on Tuesday morning covering how AM is disrupting global supply chains and enabling sustainable materials development. See our IMAT Show Preview on page 27 for details of this September conference being held in New Orleans. To provide more options for our attendees, the event is co-located with the Thermal Spray & Surface Engineering (TSSE) Forum and Exposition. More information on TSSE programming can be found on page 34.

An exciting highlight of ASM's annual event this year is the new Fellows Induction Ceremony on Monday evening, September 12. There you can meet three years' worth of the newest ASM Fellows, from Classes 2020, 2021, and 2022. All ASM members and guests are welcome. This unique, first-time event is in addition to the traditional ASM Awards Dinner. We hope to see many of you at both events.

Like the U-M students who are making a name for themselves with their new software launch, our students and emerging professionals have research of their own to share in New Orleans. Presentation and program opportunities for both sets of these next-gen engineers will be available at IMAT. The Emerging Professionals Committee describes their lineup in the ASM News section of this issue. We look to them—as their careers unfold and mature—to devise the next set of novel solutions to AM and many other materials challenges.

me I'm joanne.miller@asminternational.org

## INTRODUCE DEEP LEARNING AI TO YOUR ADDITIVE MANUFACTURING R&D AND QC

Hybridization of DL and traditional imaging creates advantage.

n today's fast-moving environment, reducing project turnaround time, accelerating research and development, and meeting productivity targets, all while reducing operating cost can be challenging without a smart automated approach. To avoid product recalls, meet increased customer demands, and continually innovate, a thorough investigation of materials' microstructure is key. Defect, inclusion, and grain size analysis are only a few approaches that can give meaningful insights into the quality of products.

Modern research and quality control have the unique challenge of working with real world, imperfect micrographs that require a flexible tool suite. MIPAR Image Analysis combines customized algorithms and powerful deep learning systems to produce technology able to perform sophisticated structure investigation. Whether of titanium, copper, steel, aluminum, or ceramics, MIPAR's software allows for automated micrograph analysis that streamlines data analytics, improves data quality, and offers new layers of information. Automation reduces operator error and improves professional productivity.

A primary challenge in modern R&D and QC processes is that the manufacturing environment is increasingly driven



Advanced automation of complex twinned grain size analysis in aged additively manufactured component.



by stringent efficiency requirements in the name of productivity. Guaranteeing product quality often runs contrary to pushing the bottom line, meaning defect and contaminant analysis must be carried out guickly as well as effectively.

Automation and digital integration are central to the push for greater productivity in manufacturing environments. The concept of automating production typically brings to mind the robotic arms of assembly lines, but manufacturers are just as interested in software solutions that accelerate critical processes throughout the manufacturing pipeline. Deep learning is one such solution.

Did you know that more and more companies are using deep learning to double check the material quality provided by suppliers? Be ahead of your customers by introducing these capabilities in-house. Improve your customer satisfaction, avoid rework, while reducing the operational cost.

#### What is Deep Learning?

The aim of deep learning AI (artificial intelligence) is to teach software to adapt to your own micrographs. It works in the presence of varying contrasts and feature texture, as well as sample preparation

artifacts. As little as four images can be used to train an application specific solution. This can be done with minimum training and no programming expertise.

This technology has had a profound role in developing the latest micrograph analysis solutions in the additive manufacturing (AM) space. Not only does AM imagery suffer from the usual challenges of varying contrast/ lighting, sample prep noise, etc., it offers especially complex microstructural features, often with extremely poor contrast due to the rapid cooling rate and high deformation processes involved in part fabrication. While humans have proven adept at identifying these features by eye, traditional automated software has struggled handsomely.

#### Breakthrough Solutions

MIPAR's unique hybridization between deep learning and traditional image processing has allowed for rapid custom development of breakthrough solutions for automated AM materials analysis and inspection. Examples include melt pool quantification, defect classification, layer thickness profiling, ultrafine phase measurement, complex grain sizing, virtual powder precursor sieve analysis, and part porosity mapping, just to name a few.



For more information on deep learning image analysis solutions for AM, contact MIPAR Software at support@mipar.us / 614.407.4510 / www.mipar.us.

## **RESEARCH TRACKS**



MIT researcher Jaclyn Lunger is detailing the atomic-level reactions behind an eco-friendly way to make metals. Courtesy of Yang Shao-Horn/MIT.

#### **MAKING MAPS FOR METAL ELECTROLYSIS**

A team of researchers from MIT, Cambridge, Mass., and SLAC National Accelerator Laboratory, Menlo Park, Calif., are mapping what occurs at the atomic level during metal electrolysis, a process in which a metal oxide is bombarded with electricity to create pure metal with oxygen as the byproduct. They say their work could lead to more efficient and environmentally friendly processes for producing metals such as lithium, iron, and cobalt. By making maps for a wide range of metals, the scientists not only determined the metals that should be easiest to produce using metal electrolysis, but also identified barriers that hinder the efficient production of others.

The research could also boost development of metal-air batteries such as lithium-air. aluminum-air. and zinc-air batteries. These are similar to the lithium-ion batteries used in today's electric vehicles, and they have the potential to electrify aviation because of their much higher energy densities. Metal-air batteries are not yet on the market due to a variety of problems, including inefficiency. All of the research was conducted using supercomputer simulations that explored different scenarios for the electrolysis of

several metals, each involving different catalysts. The team's new map is essentially a guide for designing the best catalysts for each metal, say researchers. web.mit.edu.

#### NEURAL NETWORK **PREDICTS STEEL** PART LIFETIMES

A team of researchers from Russia, Turkey, Canada, and Italy created an artificial deep neural network that is able to predict the lifetime of a component made of AISI 1045 steel-along with choosing the optimal coating and its thickness. First, the scientists conducted a series of physical experiments on steel parts. Approximately 23% of the data was used to train the neural network while the rest was used for testing and validation of the resulting predictions.

The team tried out several neural networks, with different numbers of inner layers and neurons in each layer. Nearly 99% accuracy was reported for the best neural network's predictions. Nickel, hardened chromium, and the galvanization process were used as protective coatings in the model. Further, the scientists were able to determine the optimal protective coating, which turned out to be a 10-15 µm layer of nickel or zinc. Hardened chromium was found to reduce the fatigue lifetime of steel. The team included researchers from RUDN University, Russia, Karabuk University, Turkey, Ontario Tech University, Canada, and the Polytechnic University of Milan. www.rfbr.ru/rffi/eng.



An international team of scientists is using an artificial deep neural network to predict the stability of steel parts and find the best protective coating.

#### BRIEF

Scientists from Texas A&M University, College Station, and Yonsei University, Seoul, discovered a helicoidal-shaped defect in

## MACHINE LEARNING A



A new machine learning technique could reduce the cost of battery development.

#### PREDICTING BATTERY LIFE WITH MACHINE LEARNING

Researchers at the DOE'S Argonne National Laboratory, Lemont, Ill., are using machine learning to predict the lifetimes of a wide range of battery chemistries. By using experimental data from a set of 300 batteries representing six different chemistries, the team can accurately determine how long various batteries will continue to cycle. The study relied on extensive experimental work done at Argonne on a range of battery cathode materials, especially the lab's patented nickel-manganesecobalt-based cathode. "We had batteries that represented different chemistries, that have different ways that they would degrade and fail," says computational scientist Noah Paulson. "The value of this study is that it gave us signals that are characteristic of how different batteries perform."

Paulson believes the machine learning algorithm could accelerate development and testing of battery materials. "Say you have a new material, and you cycle it a few times. You could use our algorithm to predict its longevity, and then make decisions as to whether you want to continue to cycle it experimentally or not," he says. "One of the things we're able to do is to train the algorithm on a known chemistry and have it make predictions on an unknown chemistry." Further study in this area could potentially guide the future of lithium-ion batteries, adds Paulson. *anl.gov.* 

#### AI ASSISTANCE UP For Debate

A team of researchers from Brookhaven National Laboratory, Upton, N.Y., the University of Liverpool in the U.K., and Ruhr University Bochum in Germany developed a new artificial intelligence (AI) agent called the x-ray crystallography companion agent (XCA)

that assists scientists by classifying x-ray diffraction (XRD) patterns automatically during measurements. XCA uses a collection of individual Als that are trained semiindependently of each other. Each agent has a slightly different weighting within its neural network. When presented with data, each AI "votes" based on its own interpretation and analysis. Once the Als cast their final votes, the XCA approach uses a vote tally to interpret what the most likely atomic structure

is and to suggest how confident the researchers should be of the AI analysis. Essentially, XCA is a group of AIs that debate each other while analyzing live-streaming x-ray data.

Consensus among the ensemble implies confidence in the results because differing viewpoints still result in a common conclusion. However, strong disagreement can suggest that the analysis was poorly posed, and researchers

should reexamine their assumptions. Unlike many other AI approaches in this field, this unique "ensemble voting" approach provides both predictions and uncertainties. In effect, this makes the approach a digital expert in XRD analysis. This approach demonstrates how AI and human researchers can work together to address scientific challenges such as developing new energy technologies and supporting human health. The study found that XCA can classify the materials as effectively as a human expert, but in fractions of a second. *bnl.gov.* 



Al agents observe streaming x-ray data, argue among themselves, and vote to establish both classification and uncertainty in the prediction—offering an educated guess about the atomic structure of the material under analysis. Courtesy of BNL.





New research describes how microscopic crystals grow and change shape in molten metals as they cool. Courtesy of Maksim Gusev.

#### BREAKING GROUND ON STRONGER ALLOYS

In a breakthrough for alloy research, scientists from the U.K.'s University of Birmingham have detailed how microscopic crystals grow and change shape in molten metals as they cool. Their work paves the way for improving the tensile strength of alloys used in casting and welding. The researchers used high-speed synchrotron x-ray tomography to image the changing

#### BRIEFS ·····

**Expanite,** Denmark, achieved ISO 14001 certification, the international standard for implementing an environmental management system to measure and reduce environmental impact. The company produces a technology that prevents wear, galling, and corrosion of components in stainless steel or titanium by hardening the material in a pure gas environment with no toxic waste. *expanite.com.*  crystal structures in molten alloys as they cool.

Researchers say that as aluminumcopper alloy cools, the solidification process starts with the formation of faceted dendrites, which are formed by a laver-by-layer stacking of basic units that are just micrometers in size. These units start out as L-shaped and stack on top of each other like building blocks. As they cool, they change

shape and transform into a U-shape, and finally into a hollowed-out cube, while some of them stack together to form beautiful dendrites.

"The findings from this new study provide a real insight into what happens at a micro-level when an alloy cools and show the shape of the basic building blocks of crystals in molten alloys," lead researcher Biao Cai explains. "Crystal shape determines the strength of the final alloy, and if we can make alloys with finer crystals, we can make stronger alloys." www.birmingham.ac.uk.

#### RECOVERING RARE-EARTH ELEMENTS

Using state-of-the-art spectroscopy methods, researchers at Oak Ridge National Laboratory, Tenn., are gaining a better understanding of chemical

Norman Noble Inc. is building a new 51,000-sq-ft corporate headquarters in Highland Heights, Ohio, to be ready later this year. The company specializes in micromachining Nitinol implants to meet its medical OEM clients' requirements for Nitinolbased products such as structural

heart implants and neurovascular devices. *nnoble.com*.

separations needed to recover rareearth elements and secure critical materials for clean energy technologies. Bastnaesite deposits in the U.S. are rich in rare-earth metals but must be mined and separated from unwanted minerals through chemical processes that are not well understood. Fundamental insights are needed to improve current recovery approaches based largely on trial and error. Greater efficiency offers cost-savings as well as benefits to the environment by decreasing mining and carbon impacts.

According to the scientists, the path forward will require predictive modeling to help discover the best candidates for more efficient separations. *ornl.gov.* 



Researchers shed light on chemical separations to recover rare-earth elements. Courtesy of Ben Doughty/ ORNL, U.S. DOE.

#### RESHAPING POLYMERIC THEORY

A longstanding mystery surrounding a nanoscale structure called a double-gyroid was reported to be solved by polymer scientists at the University



of Massachusetts, Amherst. One of the most desirable shapes for materials scientists, and with a wide range of applications, double gyroids have historically eluded scientists' understanding. This unique structure is comprised of a single layer that twists up into a saddle-shaped laver, which then fits into a cubic box in such a way that its surface area stays as small as possible—that's a gyroid. A double-gyroid forms when a second material, also twisted into a gyroid, fills in the gaps in the first gyroid. Each gyroidal material forms a network of tubes that interpenetrates the other. Together, they form an enormously complex material that is both symmetrical on all sides, like many crystals, yet pervaded by labyrinthine channels, each formed from different molecular units. Because this material is a hybrid of two gyroids, it can be engineered to have contradictory properties.

The research team built upon a previous theoretical model, adding a heavy dose of thermodynamics and a new approach to thinking about the packing problem-or how best to fill a finite container with material-borrowed from computational geometry and known as the medial map. The team's updated theoretical model not only explains the puzzling formation of double-gyroids but holds promise for understanding how the packing problem works in a much broader array of self-assembled superstructures, such as double-diamonds and double-primitives-or even structures that have yet to be discovered. The end goal is to be able to engineer a wide variety of materials that take advantage of the double-gyroid's structure and that can help advance a wide range of technologies from rechargeable batteries to light-reflecting coatings. umass.edu.



In a double-gyroid, two materials (pictured as red and blue) interpenetrate each other. Courtesy of Reddy et al., *Nature Communications*, 2022.

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## TESTING CHARACTERIZATION



A visual comparison of super-resolution microscopy imaging obtained by five trained networks. Image courtesy of *npj Computational Materials.* 

#### STUDYING BATTERIES AT THE NANOSCALE

Researchers at the National Renewable Energy Laboratory (NREL), Golden, Colo., are conducting groundbreaking experiments using x-ray diagnostics techniques to examine the structure of battery materials. There is a common consensus that x-ray imaging techniques hold the key to unlocking critical information about the performance of energy storage systems. With the anticipated addition of a new x-ray nanoscale computed tomography (nano-CT) scanner, NREL researchers will have the technology that enables them to get a clearer picture of energy materials than ever before.

"This scanner expands our capabilities at NREL with a new spatial resolution of 50 nanometers, a limit otherwise only achievable at high-energy synchrotron x-ray facilities," researcher Donal Finegan notes. Significant improvements the to resolution of nano-CT systems open the door to advances in how scientists understand the composition, architecture, and properties of battery materials. As the sample rotates, an x-ray beam creates 3D images with extreme resolution. Given the nondestructive nature of nano-CT. researchers can view changes as they occur in real time to understand

the reactions within a battery during operation or cycling. *nrel.gov.* 

#### BIOCOMPATIBILITY OF TITANIUM

Scientists from the Tokyo Medical and Dental University are studying the source of titanium's biocompatibility when implanted into the body, as with hip replacements and dental implants. Using photoelectrochemical measurement and x-ray photoelectron spectroscopy, they find that its reactivity with the correct ions in the extracellular fluid allows the body to recognize it. This work may lead to longer lasting next-generation medical implants. Despite numerous studies on biological reactions with implanted materials, the reason for the biocompatibility of titanium remains poorly understood.

The research team tested thin disks of titanium in a solution containing ions meant to mimic the

extracellular fluid of the body, as well as in saline. They measured how much photoelectrical current was generated when they exposed the disks to light of various wavelengths. They also performed x-ray photoelectron spectroscopy to characterize the passive films that were naturally present on the surface of the titanium. "The reactivity of titanium with high corrosion resistance, as revealed in this experiment by its electronic band structure, is one of the primary reasons for its excellent biocompatibility among metals," add the scientists. This research may lead to safer and less expensive implants for hip replacements or dental implants, because titanium is relatively rare and expensive. www.tmd.ac.jp/english.



This graph shows the change in open circuit potentials (OCP) of titanium in Hanks and saline for 72 h.

#### **NOVEL ION BINDING**

With the help of a self-built ion microscope, researchers from Germany's 5th Physical Institute of the University of Stuttgart verified a novel

#### **BRIEF**

**Plastometrex,** based in the U.K., received an **Innovate UK SMART** grant along with testing service providers **ROSEN** and **Element Materials Technology,** and the U.K.'s **National Physical laboratory.** Funds will be used to develop new products for nondestructive testing of metal components in the field. The company launched a benchtop testing device in 2020 and plans to release the portable version in 2023. The system will apply to all metallic materials and will be used to test the strength of a variety of critical metal assets. *plastometrex.com*.

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binding mechanism forming a molecule between a tiny, charged particle and a gigantic Rydberg atom. The molecule exhibits a special feature--it consists of a positively charged ion and a neutral atom in a so-called Rydberg state. These Rydberg atoms have grown in size a thousand times compared to typical atoms. As the charge of the ion deforms the Rydberg atom in a very specific way, the bond between the two particles emerges.

To verify and study the molecule, the researchers prepared an ultra-cold rubidium cloud, which was cooled down close to absolute zero at -273°C. In these ultra-cold atomic ensembles, the ionization of single atoms with laser fields prepares the first building block of the molecule-the ion. Additional laser beams excite a second atom into the Rydberg state. The electric field of the ion deforms this gigantic atom. Notably, the deformation can be attractive or repulsive depending on the distance between the two particles, letting the binding partners oscillate around an equilibrium distance and inducing the molecular bond. The distance between the binding partners is unusually large and amounts to about a tenth of the thickness of a human hair.



An open vacuum chamber with the electric field control and the first lens of the ion microscope sitting in the center. Courtesy of Nicolas Zuber.

A special ion microscope made this observation possible. It was developed, built, and commissioned by the researchers at the 5th Physical Institute in close collaboration with the workshops of the University of Stuttgart. In contrast to typical devices working with light, the researcher's special ion

microscope influences the dynamics of charged particles with the help of electrical fields to magnify and image the particles onto a detector. Next, the researchers aim to study dynamical processes within this unusual molecule. www.pi5.uni-stuttgart.de.



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## SUSTAINABILITY



Courtesy of Pixabay/CC0 Public Domain.

#### MAKING PLASTIC MORE SUSTAINABLY

Scientists at Cardiff University, U.K., report a new method of creating cyclohexanone oxime, a precursor to Nylon-6, a common plastic used in the automotive, aerospace, and medical industries. It is estimated that global production of Nylon-6 will reach around 9 million metric tons a year by 2024, prompting scientists to search for greener and more sustainable ways of producing cyclohexanone oxime. Currently, cyclohexanone oxime is produced industrially through a process involving hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), ammonia, and a catalyst called titanosilicate-1 (TS-1). The  $H_2O_2$  used in this chemical process is produced elsewhere and needs to be shipped in before it can be used in the chemical reaction. This is a costly and carbon-intensive process that also necessitates the shipping of highly concentrated H<sub>2</sub>O<sub>2</sub> to the end-user prior to dilution, which effectively

wastes large amounts of energy used during concentration.

Similarly, stabilizing agents often used to increase the shelf-life of  $H_2O_2$ can limit reactor lifetime and often need to be removed before arriving at a final product, leading to further economic and environmental costs. To address this issue, the team devised a method where H<sub>2</sub>O<sub>2</sub> is synthesized in-situ from dilute streams of hydrogen and oxygen, using a catalyst consisting of gold-palladium nanoparticles that are either directly loaded on to the TS-1 or on a secondary carrier. The method can produce yields of cyclohexanone oxime comparable to those seen in current commercial processes while avoiding the major drawbacks associated with commercial H<sub>2</sub>O<sub>2</sub>. *www.cardiff.ac.uk*.

#### PLASTIC WASTE THAT ABSORBS CO<sub>2</sub>

Researchers at Rice University, Houston, led by chemist James Tour, discovered that heating plastic waste in the presence of potassium acetate produced particles with nanometer-scale pores that trap carbon dioxide molecules. According to the team, these particles can be used to remove CO, from flue gas streams. "Point sources of CO, emissions like power plant exhaust stacks can be fitted with this waste-plastic-derived material to remove enormous amounts of CO<sub>2</sub> that would normally fill the atmosphere," explains Tour. "It is a great way to have one problem, plastic waste, address another problem, CO<sub>2</sub> emissions."

To make the material, waste plastic is turned into powder, mixed with potassium acetate, and then heated at 600°C (1112°F) for 45 minutes to optimize the nanoscale pores. The process produces a wax byproduct that can be recycled into detergents or lubricants. Pyrolyzing plastic in the presence of potassium acetate produces porous particles able to hold up to 18% of their own weight in CO<sub>2</sub> at room temperature. The lab estimates the cost of carbon dioxide capture from a point source like post-combustion flue gas would be \$21 a ton-far less expensive than the energy-intensive, amine-based process in common use to pull carbon dioxide from natural gas feeds, which costs \$80-\$160 a ton.

Like amine-based materials, the sorbent can be reused. Additionally, it is expected to have a longer lifetime than liquid amines, cutting downtime due to corrosion and sludge formation. *rice.edu*.



Paul Savas feeds raw plastic into a crusher to prepare it for pyrolysis. Courtesy of Jeff Fitlow.

#### BRIEF

Researchers at the DOE's **National Renewable Energy Laboratory,** Golden, Colo., created a solar cell with a record 39.5% efficiency under 1-sun global illumination. This is reportedly the highest efficiency solar cell of any type measured using standard 1-sun conditions. *nrel.gov.* 

## DIRECTED ENERGY DEPOSITION MOVES OUTION HE BOX

Judy Schneider, FASM,\* University of Alabama in Huntsville Paul Gradl, NASA Marshall Space Flight Center

Metal additive manufacturing has steadily progressed over the past decade, with today's directed energy deposition processes enabling rapid builds of large structures in near-net shape—and with minimal machining required to achieve final dimensions.

\*Member of ASM International

Image courtesy of NASA/RPM Innovations.

etal additive manufacturing (MAM) processes have matured from their early use as rapid prototyping tools to producing today's critical end-use components<sup>[1-4]</sup>. Since the early 2010s, an increasing number of MAM processes have emerged that were initially referred to by various acronyms<sup>[5]</sup>. ASTM Committee F42 on Additive Manufacturing Technologies undertook the task of standardizing the terminology by issuing ASTM Standard F2792-12a in 2012<sup>[6]</sup>. In the most general terms, fusion-based MAM processes are characterized in terms of feedstock and the energy source used to fuse or melt the feedstock into the desired component geometry.

Figure 1 provides an overview of the fusion-based processes in which either a powder or wire feedstock is combined with an energy source that melts the feedstock to either create a new freeform part or add material to an existing part. The two main categories include powder bed fusion (PBF) and directed energy deposition (DED). In PBF, a focused beam is used to trace out the part according to a defined toolpath from a CAD model in a layer-bylayer method using either a laser (L-PBF) or an electron beam (EB-PBF)<sup>[7,8]</sup>. DED can use either a powder feedstock integrated with a laser (LP-DED) or a wire feedstock with either a laser beam (LW-DED), an electric arc (AW-DED), or an electron beam (EBW-DED)<sup>[9-12]</sup>. Other solid state MAM processes exist, but are not the focus of this article.

#### IN THE BOX VS. **OUT OF THE BOX**

The two primary categories of MAM can be thought of as "in the box"



Fig. 1 — Overview of fusion-based metal additive manufacturing processes.

for PBF versus "out of the box" for DED. Although the most highly cited "in the box" metal AM process is L-PBF, the size of the build chamber restricts the final size of the component. To eliminate this size constraint, "out of the box" DED processing has emerged, although some systems may use a large purge chamber to prevent oxidation and issues with reactive alloys. The ability to fabricate outside the box removes size constraints, but tradeoffs between feature and geometric resolution, build and post-processing time, component size, microstructure and resulting properties, and process availability must always be considered. Figure 2a highlights the increased build dimensions made possible by using DED compared to PBF. In contrast, Fig. 2b shows that as build size increases, the deposition rate also increases with a resulting reduction in feature size.

Figure 3 showcases several largescale structures fabricated using MAM DED. A NASA HR-1 alloy channel wall nozzle with integral internal passages for a liquid rocket engine is shown in Fig. 3a, built using LP-DED. An aluminum tank structure built using AW-DED is shown in Fig. 3b.

#### MULTIPLE MATERIAL CHOICES

DED can build using a variety of alloys including those based on nickel, iron, copper, cobalt, titanium, and





aluminum, as well as refractory alloys<sup>[12-14]</sup>. DED also enables deposition of multiple materials within the same setup, providing unique design solutions to optimize component-specific requirements for thermal, electromagnetic, pressure, loading, and dynamic properties.

Deposition rates for DED processes can approach 9 kg/hour depending on component geometry, greatly reducing manufacturing time compared to other methods. In addition, less material is wasted because parts can be built to near-net shape in contrast to the subtractive machining required by forgings.

#### **LARGE-SCALE BUILDS**

The ability to print large-scale structures is enabled by integrating the MAM DED deposition head with either a robotic arm or CNC controller platform, as shown in Figs. 4a and b, respectively.

Directly mounting the deposition head onto the appropriate platform allows operation in an open-air environment using only localized shielding gas to prevent oxidation. Several DED systems that use laser as an energy source are integrated with an enclosed structure to allow for a fully inert environment, necessary for reactive alloys such as titanium. Electron beam (EBW-DED) requires the part to be deposited within a vacuum chamber and is also used for reactive alloys. Regardless of the enclosed or open-air environment, an integral inert purge is typical through the center of the LP-DED nozzle or AW-DED and LW-DED deposition heads.

The various DED processes allow additional degrees of freedom (5+) during deposition as compared to L-PBF, which is a 2+1 axis system. The deposition head can typically move in three axes with some systems allowing for tilt and rotate. Use of a kinematic robotic arm further increases the number



Fig. 3 – Nozzle extensions fabricated using MAM DED illustrate the large-scale format: (a) LP-DED of NASA HR-1, 1.83 m tall x 1.5 m OD with 1 mm wall thickness and internal integral channels; and (b) AW-DED of aluminum tank for a launch vehicle. Courtesy of RPM Innovations Inc./NASA, and Relativity Space, respectively.



(a)

Fig. 4 – Deposition heads can be mounted on either (a) robotic arms or (b) CNC machines. Courtesy of NASA/DM3D Technology, and RPM Innovations Inc./NASA, respectively.

of axes and the size of the component as either the robot or part, or both, can be mounted on a moveable platform. A trunnion table is often integrated within DED platforms adding tilt and rotational axes. These aspects of the MAM



Fig. 5 — Various powder nozzles: (a) coaxial (continuous annulus); (b) multi-jet; and (c) vectored nozzle capable of depositing inside a 57-mm ID to a depth of 610 mm. Courtesy of RPM Innovations Inc.



Fig. 6 – (a) LP-DED system with multiple powder feeders or hoppers<sup>[5]</sup>; (b) coaxial nozzle powder flow path; and (c) vectored nozzle powder flow path.

DED processes allow for complex curvature component shapes to be built with nonsymmetric features such as those in Fig. 4b.

In the powder-based DED processes, various nozzles can be used as illustrated in Fig. 5. They range from a coaxial configuration to multi-jet nozzles to vectored nozzles for internal cladding (Figs. 5a-c). Inert carrier gas is used to propel powder through the various nozzle configurations into the melt pool, creating the deposited bead.

Figure 6a provides a schematic overview of the LP-DED system showing the powder feeders or hoppers. Use of multiple hoppers facilitates the build of bimetallic and functionally graded parts by varying the types of powder as well as the mixture ratios. Powder flow paths are illustrated in Fig. 6b for a coaxial nozzle and Fig. 6c for a vectored nozzle. Research is also being conducted using central wire or powder feed deposition nozzles with an annular laser beam or series of off-axis laser beams around the center axis<sup>[15]</sup>.

Several of the processes that use wire feedstock are illustrated in Fig. 7. For these, the fusion source uses standard welding-based processes including metal inert gas (MIG), gas tungsten arc (TIG), laser beam (LW-DED), and electron beam (EBW-DED). In contrast to fusion welding, the arc and beam sources melt the wire into a free-form structure of the desired part geometry.

Molten pool

Powder passages

Axial nozzle



Fig. 7 – Comparison of various arc-based processes that use wire: (a) metal inert gas (MIG); and (b) gas tungsten arc (TIG)<sup>[16]</sup>.



**Fig. 8** — Deposition of features onto standard bar stock. Use of additive/subtractive hybrid systems enables improvement of surface finishes after deposition. Courtesy of DMG Mori.

Wire-based processes feature the highest deposition rate, allowing large structures to be built rapidly with superior material usage efficiency.

In addition to doing away with size constraints and boosting deposition rates, MAM DED processes offer further advantages. Because the substrate can be either a sacrificial build plate or part of the final component design, complex geometries can be built onto standard wrought shapes as shown in Fig. 8. By adapting the deposition heads to multi-axis robots and CNC systems, features can be locally added to parts creating near-net shapes and significantly reducing final machining. In addition, use of hybrid DED equipment combines the ability to both add and subtract material in one process during component fabrication. Ancillary systems such as process monitoring and feedback loops can also be incorporated onto machine platforms.

#### CONCLUSION

MAM DED offers the ability to rapidly build large structures in near-net shape with minimal machining to final dimensions. Because MAM DED occurs outside of a box to locally deposit the feedstock, it provides more efficient material use. Additional cost savings can be achieved by deposition of complex geometries onto standard wrought product, which can be used as both the build plate and part of the finished component. Localized feature repair is also possible with DED. Use of hybrid additive/subtractive systems allows incorporation of machining tools to achieve design objectives such as dimensional control of internal passages and improved surface finish. ~AM&P

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## ANCIENT IRON PRODUCTION AND PROCESSING IN THE OLD WORLD

## A look at processing and production of iron during the Iron Age, 1200 to 500 B.C., including early unintentional forms of steel.

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previous contribution to AM&P's series on archaeometallurgy presented a brief overview of production and process metallurgy for copper and silver alloys in the Old World<sup>[1]</sup>. Consistent developments of these technologies began around 3500 B.C., with bronzes becoming so important that the period from about 3300 to 1200 B.C. is commonly referred to as the Bronze Age. However, dates vary considerably according to geographical locations, and the Bronze Age is usually subdivided into Early, Intermediate, and Late periods, again with differing dates.

This article provides a broadly similar overview for the Iron Age, which dates from approximately 1200 to 500 B.C. As before, the dates vary according to geographical locations and the relative sophistication of the production and processing techniques. A recent survey of the beginnings of iron in the Near East is given by Erb-Satullo<sup>[2]</sup>. He argues, like others, that ironsmelting technology was derived from the earlier developments of coppersmelting. This is very likely because the technology is complex, with many difficulties to overcome. This has been amply demonstrated by modern-day experimental archaeometallurgy. These difficulties are described in this article.

Before discussing ancient iron production and processing, note that without these and much later developments, beginning in the mid-18th century A.D., the Industrial Revolution would have been more problematic. Although there were many contributing causes of the Industrial Revolution, the development of large-scale iron and steel production, particularly for manufacturing industrial machines and tools, played a major role.

#### **ANCIENT IRON PRODUCTION**

The very beginning of iron smelting is uncertain. From the middle of the 19th century A.D. until the 1970s the concept of bowl furnace smelting was generally favored<sup>[3]</sup>. However, experiments have shown that this method is usually unsuccessful<sup>[4]</sup>, but it is possible to obtain small amounts of partly consolidated iron using high-quality iron ores<sup>[5]</sup>.

Regular iron smelting in the Old World was preceded by about 2000 years of copper and bronze production, by which time shaft furnaces for copper smelting were well-developed<sup>[1]</sup>. Thus, it might seem logical that iron smelting could be done in a similar fashion. However, there were, and are, major differences. Firstly, iron cannot be melted in a shaft furnace, but accumulates as a porous mixture of iron and slag called a "bloom." Secondly, the operating conditions are (much) more complex. A schematic of an iron smelting shaft furnace and the various production stages and their locations is shown in Fig. 1. This schematic is based on experiments with a Hungarian shaft furnace design from the 10th century A.D.<sup>[6]</sup> In the present context this is anachronistic, but the design is generically representative, and the experimental study has the considerable advantage that it is combined with details of the production stages<sup>[6]</sup>.

The schematic in Fig. 1 shows that shaft furnace iron smelting consists of pre-roasting iron ore and its subsequent reduction in a multistage process. The complexity of this furnace reduction would not have been recognized by the ancient ironmasters. Successful smelting also requires<sup>[7,8]</sup> (and would have required) an empirically determined optimum combination of the following parameters: furnace size, tuyère position, forced air volume, the type and size of ore particles, size of the charcoal, and the sequence of adding charcoal and ore<sup>[7,8]</sup>. Much more information about shaft furnace iron smelting is given in Ref. 6-8.

When a bloom is removed from the furnace it is spongy, consisting of a mixture of iron and slag. It is therefore first consolidated by hammering that squeezes out as much slag as possible. If too large to be hammer-forged by hand, the bloom is then split with a maul driven by sledgehammers.



Fig. 1 – Schematic of an iron smelting shaft furnace and details of the production stages. Adapted from Thiele<sup>[6]</sup>.

Thereafter, the single or split pieces of bloom are hand hammer-forged to the required shapes suitable as workpieces for further processing. Examples from experimental archaeometallurgy are shown in Fig. 2. The forging steps usually require intermittent reheating of the bloomery workpieces to maintain sufficient malleability.

Bloomery workpieces, today classified as wrought iron, were basically low-carbon steels (< 0.1 wt% C) and invariably contained slag inclusions that actually improved the malleability<sup>[9]</sup>. Figure 3 shows examples of objects with microstructures essentially the same as bloomery workpieces, namely single phase (ferrite) metal and slag inclusions<sup>[10,11]</sup>. There were numerous other applications, such as nails, hinges, andmuch later-Roman bridge pile-shoes<sup>[12]</sup>. However, wrought iron is relatively soft and unsuitable for taking cutting edges in tools and weapons. In these applications wrought iron was not competitive with hardened (cold-worked) bronzes until the accidental discovery of steel. This discovery and its exploitation started the Iron Age.

#### **DISCOVERY OF STEELS**

The archaeological evidence indicates that steels (iron-carbon alloys) were first produced accidentally owing to incomplete control of the complex bloomery smelting process (Fig. 1) and



**Fig. 2** — Iron bloom processing: (a) slag removal and bloom consolidation, showing pieces of slag surrounding the bloom; (b) a large bloom split using a maul and sledge hammers; (c, d) hammer hand-forging to the required shape. Adapted from various experimental archaeometallurgy sources.

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also subsequent processing using forging hearths. In other words, these primary and secondary processes could, and did, result in harder regions (owing, as we now know, to higher carbon contents) in blooms and subsequent products. Early ironsmiths were able to distinguish these harder regions, adapt thermo-mechanical treatments for producing medium and high-carbon steels, and selectively use these materials for the production of tools and weapons. This involved forge-welding and shaping selected portions of blooms, followed by grinding and polishing. An early example, a sword from the Hittite New Empire (1400 to 1180 B.C.) is shown in Fig. 4<sup>[13]</sup>.

(a)

#### STEEL DEVELOPMENTS IN ANTIQUITY

Since the discovery of steels much attention has been paid to weapons, culminating in high quality allsteel swords, made by empirically controlling their carburization levels, microstructures, and overall chemistry. These controls were exercised by heat treatments in reducing and/or oxidizing environments, and by thermo-mechanical working, including quenching and tempering. This means that steels have been produced intentionally from iron blooms, with varying but generally increasing success, for over 3000 years. Figure 5 schematically represents an intermediate stage from Roman times, when processing developments allowed various types of swords to be manufactured<sup>[14]</sup>.

The long-term success of obtaining steel products from bloomery iron may be attributed to its workability, owing to its generally low carbon content. In turn this meant that smelters empirically favored production conditions yielding low-carbon blooms. However, during the latter part of the 1st century B.C. and into the first and second millennia A.D., the production of crucible steels was developed in India, several central Asian lands and Anatolia. These steels were typically derived from melting wrought iron with charcoal, but other additions such as wood and cast iron were sometimes included. The

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**Fig. 3** — Heavily corroded objects made from bloomery iron: (a) axe fragments, Western Anatolia, Archaic Period<sup>[10]</sup>; (b) spearhead, Northern Iran, Iron Age III<sup>[11]</sup>; (c) etched and (d) unetched images of the corresponding microstructures, showing single phase (ferrite) metal and Si-Fe-Al slag inclusions. These microstructures are representative of worked bloomery iron, with locally varying slag contents and, as shown by the etched sample, non-uniform grain sizes.



**Fig. 4** — (a) Hittite fire-welded iron and steel sword with cast-on bronze hilt; (b, c) etched crosssections of a sample, showing layers with differing carbon contents and stringers of elongated slag inclusions (arrowed). Adapted from Yalçin<sup>[13]</sup>.

resulting ultrahigh-carbon (UHC) steels (having about 1.0 to 2.3 wt% C), though more difficult to work, were used to make heavy duty tools as well as weapons (swords and knives).

In particular, UHC steels were used to make so-called Damascene steel sword blades, renowned for their surface patterns. These patterns were achieved by complex thermo-mechanical forging and are due to the alignment of iron carbides into bands. The iron carbides and their distributions in the UHC steels resulted from solid-state precipitation during thermo-mechanical working. Figure 6 shows two artifacts, one a knife from Central Anatolia<sup>[15]</sup> and the other an ornamental plaque from Iran<sup>[16]</sup>. The corresponding microstructural images show the typical bands

of iron carbides and matrices that result in Damascene surface patterns. Even so, obtaining true Damascene patterning was subject to uncertain $ty^{[16]}$ . In fact, considerable scientific effort and resulting controversy has only recently been resolved to explain the occurrence, or non-occurrence, of Damascene patterning<sup>[17]</sup>. The arrow in Fig. 6b indicates a missing piece owing to post-manufacturing brittle fracture. This breakage and a large brittle crack in an almost identical plaque<sup>[16]</sup> retroactively demonstrate some of the



**Fig. 5** — Schematics of five Roman sword designs. The most common ones were mixtures of iron and steel, while the highest quality were steel laminates hammer-welded together and sometimes carburized. The final heat treatments varied from air-cooled to quenched. Adapted from Williams<sup>[14]</sup>.



**Fig. 6** — Crucible steel objects with Damascene surface patterns: (a) knife blade, Central Anatolia, 13th century A.D.; (b) ornamental pierced plaque, Iran, 17th century A.D.; (c) optical metallography of the etched microstructure of the blade cutting edge; (d) SEM metallography of an etched microstructure for a sample from the plaque. Both microstructures show bands of iron carbides and matrices that give rise to Damascene patterning. However, the matrices are different: in (c) the carbides are dispersed in martensite owing to selective quenching<sup>[15]</sup>; in (d) the carbides are dispersed in a matrix of ferrite, martensite and lamellar pearlite<sup>[16]</sup>. The arrow in (b) indicates a missing piece owing to post-manufacturing brittle fracture (see main text). Figs. 6b and d courtesy of Mohammad Mortazavi.

difficulty of smithing UHC steels.

#### EMPIRICISM AND CORROSION PROTECTION

The empirically based achievements of ironmasters and smiths in ancient and historic times are amazing, both because of the complexity of production and processing, and the fact that scientific explanations began only in the late 19th century A.D.

Not so amazing, but very important, is the problem of protecting archaeological iron and steel objects from continuing damage by corrosion. This is obvious from the objects portrayed in Figs. 3a, 3b, 4a, and 6a. Excellent handbooks for conservation are available<sup>[18-20]</sup>. ~AM&P

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#### **TECHNICAL SPOTLIGHT**

## ADDITIVE MANUFACTURING PRESENTS NEW CHALLENGES FOR METAL COMPONENTS

A look at how companies in regulated industries can test materials to produce higher quality metal components with additive manufacturing.

s one of the fastest growing manufacturing technologies, demand for metal components produced by additive manufacturing (AM) is expanding in nearly every market sector. Industry reports project additive manufacturing with metal powders to grow at a compound annual growth rate (CAGR) of 21% from 2022 to 2028-that's four times faster than growth in conventional metal casting during the same forecast period. Most notable is a sharp increase in AM requirements from highly regulated spaces such as the aerospace, biomedical, and automotive industries. While high barriers to qualification and certification in these industries may have delayed entry of AM components, it has long been clear that the flexibility and efficacy inherent to AM would draw their attention.

#### **PRODUCTION ADVANTAGES**

The marked demand for AM products is largely credited to its production advantages. Chief among them is a high buy-to-fly ratio—that is, the ability to produce parts with minimum material loss during production and finishing. Another is that AM components can be manufactured with a high degree of internal complexity and individualization that would be difficult or inefficient to produce by any subtractive method.

While production advantages have accelerated the maturity of AM technology to date, an entirely different set of advantages is driving new attention and growth to the industry. Supply chain challenges have affected



Mechanical testing of an AM coupon using a video extensometer.

nearly every corner of the market, but they have been particularly pronounced in the sourcing of aerospace, automotive, and medical device components. As the burdens of a long, extended supply chain create headaches for all equipment manufacturers alike, the ability to shorten and simplify that chain through inclusion of AM components becomes increasingly urgent. In this respect, advocates of AM technology have leveraged their compelling capability to reduce manufacturing steps and reshore most if not all operations closer to finishing and assembly.

#### **VARIABILITY CHALLENGES**

However, to be successful, particularly in these highly regulated industries, AM components must not only meet the rigorous qualification and certification requirements of conventional manufacturing. They must also confront new challenges associated with their characteristic layer-by-layer build. Since builds are essentially made up of many incremental small casts from individual melt pools, the challenges faced by a traditional casting are multiplied by each melt pool created in a part's production. The greatest resulting challenge is the increased variability observed in mechanical properties and part-to-part performance.

Mechanical property variability can be directly linked to variability in process, which can occur part-topart, operator-to-operator, machineto-machine, and facility-to-facility. Because mechanical properties are highly sensitive to small microstructure changes, they are naturally impacted by the processes which develop that microstructure. Therefore, it is essential to monitor and control all process variables such as beam power and velocity as well as production parameters

Laver-by-layer build-up of AM metal components presents

opportunities for variability in mechanical properties.

mechanical testing to confirm satisfactory performance

Strength and toughness must be verified through

in service.

such as melt pool size and build temperature. While the need to maintain process control to ensure reliable, repeatable outcomes is not unique to AM, this new technology does introduce a new family of processes to be measured and documented during production. The ASTM F42 technical committee has made significant progress in addressing these new developments and their im-

plications for quality.

Process variability is not the only source of variability in AM manufacturing. Unlike conventional casting, AM components exhibit highly directional properties based on the nature of their layer-by-layer build. Most notable is an expected lower tensile strength in the build direction (Z) as compared to the XY-plane. To qualify AM processes and parts, AM components need not only more data points through testing greater sample sizes, but also require more comprehensive data from each specimen. This requires both a quantitative increase in mechanical tests and a qualitative improvement in data handling, from supporting software architecture to managing the vast data sets generated.

Inherent in the complexity and customized nature of AM component builds lies the major difficulty in part gualification and certification. Well-designed feedback controls that monitor and respond to process parameters ensure dimensional accuracy and surface condition. Load-bearing and stressed components must also be shown to exhibit satisfactory strength. Two AM components with perfectly identical geometries may exhibit wildly different mechanical properties due to their thermal history. The rapid solidification that makes AM builds possible may extend the solubility of unwanted material phases to various degrees. This in turn can negatively impact strength and ductility. Even more detrimental is possible incomplete fusion of internal structures when solidification occurs too rapidly.



Digital image correlation (DIC) is an optical technique that compares images of a tested specimen's surface to generate pictures that can be used to visualize strain and displacement over the full two-dimensional surface of the test specimen.

25

26

Another area of concern with AM is the directionality of heat subtraction from the forming part. Directionality in cooling yields increased directionality in grain growth, and, as a result, increased anisotropy in strength and ductility. Anisotropy in mechanical properties is acceptable when understood and is ideally optimized in design, but it is impossible to understate the importance of testing in the directions that correspond to which way the part will be stressed in service. Periodic checks to verify repeatability are similarly necessary, especially in the high stakes medical device, aerospace structures, and automotive parts industries.

#### DATA MANAGEMENT

While increasing the specimen quantity in a testing regimen is one way to garner more qualifying information, it's also beneficial to apply methods that deliver more data for each specimen tested. Digital image correlation (DIC) is one tool that can be used to identify strain concentrations across



AM testing generates enormous datasets that can be difficult to manage. The workload can be greatly reduced by modern software infrastructure, which enables the quick search, display, and analysis of results over time, across multiple samples and test systems.

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the gauge length, increasing the field of observed behavior from a static test

alone. While also useful in conventional applications, DIC is especially useful in AM part and process qualification where greater variability is expected. The combined effect of increased specimen quantities and increased values being measured may seem daunting for legacy data management systems; however, advancements in data handling have risen to the occasion. DIC in conjunction with data management software significantly reduces the workload of evaluating more locations from more specimens and ultimately enabling greater laboratory throughput.

As additive manufacturing becomes increasingly viable for applications that are highly regulated and require predictable mechanical properties, it's important not to let the inherent challenges of this production method detract from the valuable advantages of the technology. While rapid solidification and directional heat subtraction raise concerns for internal quality, they are also fundamental to this process that enables the engineering of complex, optimized, and individualized geometries. In conjunction with well-designed process monitoring and control, increased testing and modern data management provide the critical infrastructure for gualification and certification of a new generation of AM metal components. ~AM&P

For more information: Dean Lovewell, metals market manager, Instron, 825 University Ave., Norwood, MA 02062, 781.575.5000, dean\_lovewell@instron. com, www.instron.com.



INTERNATIONAL MATERIALS, APPLICATIONS & TECHNOLOGIES



#### SEPTEMBER 12-15, 2022 | NEW ORLEANS, LOUISIANA

#### SHOW PREVIEW

ASM International invites you to participate in its new fall event, IMAT 2022—the International Materials, Applications & Technologies Conference and Exhibition. Meet us in New Orleans from September 12–15 for a comprehensive lineup of technical sessions, high level keynotes, networking events, and an exhibit floor. With a focus on the Circular Materials Economy, the conference combines with a two-day exposition to bring together major OEMs, materials partners, and suppliers to highlight advanced materials processes and applications throughout the materials community.

No other conference integrates applied materials technologies and applications with economics, environmental issues, and enabling digital technologies. With its broad reach, ASM unites different market segments that cross the entire materials world and connects industry, academia, and government to solve global materials challenges. The result is a program rich with top-notch experts brought to you by ASM and its organizing partner, the Association for Materials Protection and Performance. Also, for your comfort, many new safety protocols will be in place to protect the health of our attendees. Join us for this exciting one-of-a-kind materials event!

#### **TECHNICAL PROGRAM**

This new IMAT event features robust programming with over 350 technical presentations in 18 topic areas, with Failure Analysis as one of the largest symposia. Special sessions on corrosion and environmental degradation will be delivered by event partner, Association for Materials Protection and Performance. In addition, multiple panel sessions are planned. For details on session topics and schedule, visit imatevent.org.

The IMAT event is also co-located this year with the Thermal Spray & Surface Engineering Forum and Expo (TSSE). For details on TSSE programming, see page 34 in this issue's *iTSSe* supplement.





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ASM International's main priority is to ensure the safety of our members, speakers, attendees, exhibitors, and staff as we continue to monitor all relevant information on the COVID-19 virus and its impact on hosting events in public spaces. For up-to-date information, visit imatevent.org or contact events@asminternational.org.

#### **KEYNOTE LECTURES**

#### **MONDAY, SEPTEMBER 12**

#### A MORE SUSTAINABLE FUTURE VIA POLYMER CIRCULARITY



9:00–10:00 A.M. DR. KATHRYN BEERS Program Manager, Circular Economy NIST

#### **TUESDAY, SEPTEMBER 13**

#### SUSTAINABLE MATERIALS AND PROCESS STRATEGIES FOR THE AIRCRAFT OF TOMORROW



3:00-4:00 P.M.

**DR. KAY YOUNGDAHL BLOHOWIAK** Senior Technical Fellow Boeing Research & Technology

#### WEDNESDAY, SEPTEMBER 14

INTEGRATED COMPUTATIONAL MATERIALS ENGINEERING IMPACT ON INDUSTRY AND NEW FRONTIERS IN DIGITAL TRANSFORMATION



3:00-4:00 P.M.

**DR. JASON SEBASTIAN** *President QuesTek Innovations LLC* 

#### PANEL DISCUSSION: THE CIRCULAR MATERIALS ECONOMY



10:30 A.M.-12:00 P.M.

Representatives from each of the six ASM Affiliate Societies will gather to share perspectives and insights from

their disciplines on the circular materials economy. Discussion will center on reduce, reuse, and recycle as key components in sustainable materials design, production, maintenance, and management programs.

Subject to change. Speakers current as of July 7.

For event details and to register, visit **imatevent.org** 

#### SPECIAL LECTURES

ALPHA SIGMA MU LECTURE MONDAY, SEPTEMBER 12 | 1:20-2:40 P.M.

INDUSTRY 4.0 AND ICME: THE EVOLUTION AND REVOLUTION OF MATERIALS SCIENCE AND ENGINEERING



**DR. DAVID FURRER, FASM** Senior Fellow, Discipline Lead, Materials & Processes Engineering Pratt & Whitney

ASM EDWARD DEMILLE CAMPBELL MEMORIAL LECTURE 2020 TUESDAY, SEPTEMBER 13 | 9:00-10:00 A.M.

ADDITIVE MANUFACTURING: DISRUPTING GLOBAL SUPPLY CHAINS AND ENABLING SUSTAINABLE DEVELOPMENT



**DR. MRITYUNJAY SINGH, FASM** *Chief Scientist Ohio Aerospace Institute* 

#### ASM EDWARD DEMILLE CAMPBELL MEMORIAL LECTURE 2022

TUESDAY, SEPTEMBER 13 | 10:30-11:30 A.M.

MODERN PHYSICAL METALLURGY: IMPORTANCE, USE OF NEW TOOLS, AND HOW TO FINANCE THE METALLIC MATERIALS ENTERPRISE



**DR. HAMISH L. FRASER, FASM** Ohio Regents Eminent Scholar and Professor Center for the Accelerated Maturation of Materials, The Ohio State University

#### **HENRY CLIFTON SORBY LECTURE 2022**

TUESDAY, SEPTEMBER 13 | 10:50-11:50 A.M.

CHOREOGRAPHY OF ATOMS DURING THE BAINITE TRANSFORMATION

**DR. HARRY BHADESHIA** *Professor of Materials Science and Metallurgy University of Cambridge* 

Subject to change. Speakers current as of July 7.

CIRCULAR MATERIALS ECONOMY INDUSTRY • ACADEMIA • GOVERNMENT

#### **SPECIAL EVENTS**

#### **MONDAY, SEPTEMBER 12**

#### 11:30 A.M.-1:30 P.M.

#### ASM LEADERSHIP AWARDS LUNCHEON

ASM's Chapters and organizational unit awards as well as awards and scholarships of the ASM Materials Education Foundation will be presented. ASM's incoming Committee/ Council chairs will also be recognized for their leadership.

#### 4:00-5:00 P.M.

#### ASM 109<sup>TH</sup> ANNUAL SOCIETY MEETING

The election of Officers for the 2022-2023 term and transaction of other Society business will take place at this meeting. ASM members and guests are welcome.

#### 7:00-10:00 P.M.

#### ASM FELLOWS INDUCTION CEREMONY

We look forward to celebrating the induction of the 2020, 2021, and 2022 FASMs. Join us for this special evening as we recognize the recipients of one of the highest honors in the field of materials and celebrate their achievements. Tickets include cocktails and hors d'oeuvres and can be purchased with IMAT registration.

#### **TUESDAY, SEPTEMBER 13**

#### 7:00-9:00 A.M.

ASM WOMEN IN MATERIALS ENGINEERING BREAKFAST Three leaders will share their stories as part of a panel focused on entrepreneurship, followed by a lively Q&A. This breakfast is a popular annual event and usually sells out.

#### 4:00-5:30 P.M.

#### WELCOME RECEPTION WITH EXHIBITORS

Take the opportunity to network with colleagues, attendees, and exhibitors in a casual setting on the show floor.

#### 7:00-9:30 P.M.

#### **ASM AWARDS DINNER**

Join us in celebrating the accomplishments of ASM's 2020, 2021, and 2022 award recipients. Tickets include the President's Reception following dinner and can be purchased with IMAT registration.

#### **EXHIBITORS**

Airflow Sciences Corporation Allied High Tech Products Ardleigh Minerals, Inc. ASM Materials Education Foundation **B4C** Technologies Bruker Nano Analytics Carl Zeiss Microscopy, LLC CompuTherm ECM USA Inc. Electron Microscopy Sciences Fluxtrol Inc. Fort Wayne Metals GeniCore Goodfellow Corporation IKH USA-INC. Impact Innovations GmbH

Inductotherm Group Industrial Heating Instron JAS Alloy Kymera International LECO Corporation MTS Systems Corporation Nippon Kornmeyer Carbon Group NSL Analytical Services Oseir Ov Park Systems Polycontrols Technologies Inc. Proto Manufacturing Quaker Houghton SECO/VACUUM Technologies Springer

#### WEDNESDAY, SEPTEMBER 14

#### 6:00–9:00 P.M. SOCIAL EVENT: MARDI GRAS WORLD (TICKETED EVENT - \$85)

Join us on Wednesday night for a special networking event at the Grand Oaks Mansion on the grounds of Mardi Gras World. Attendees will enjoy cocktails and local food delicacies in the extravagant gardens and pathways of this Southern Plantation recreation. In addition, guests will be able to tour the famous Mardi Gras World Float Den, full of huge colorful floats that have made an appearance in a past Mardi Gras parade. Tickets includes round trip transportation from the Hilton New Orleans, two drink tickets, reception-style dinner, entertainment. It's also walkable from the convention center!

#### IMS AND FAS GENERAL MEMBERSHIP MEETINGS

ASM affiliate societies, the International Metallographic Society and the Failure Analysis Society, will be conducting their annual business meetings at IMAT. At those gatherings, officers will be elected for the 2022-2023 term and other Society business will be transacted. IMS and FAS members and guests are welcome. *Check imatevent.org for dates and times*.

#### STUDENT AND EMERGING PROFESSIONAL OPPORTUNITIES

Be sure to check out these great programs ASM is organizing for next-gen engineers.

For full event details, visit bit.ly/3u7muUP

- Perspectives for Emerging Materials Professionals (Monday AM & PM sessions)
- Introduction to Failure Analysis (Tuesday AM session)
- Career Connections Program
- Participation Grants
- DomesDay Competition
- Passport Program
- HTS Strong Bar

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Exhibitor list current as of July 7.

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Super Systems

Ted Pella, Inc.

TESCAN USA

Tecnar Spray Sensors

• Fluxtrol Student Research Competition

#### **EXHIBITION HOURS**

TUESDAY, SEPTEMBER 13

9:00 a.m.–5:30 p.m.

Expo Welcome Reception with Exhibitors: 4:00–5:30 p.m.

WEDNESDAY, SEPTEMBER 14

9:00 a.m.-5:00 p.m.

For event details and to register, visit imatevent.org

## IMAT 2022 and THERMAL SPRAY & SURFACE ENGINEERING FORUM – EXHIBITOR SHOWCASE

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#### stackmet.com – Booth 333

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for users who are new to electron microscopy. Schedule an appointment now for a demo. www. thermofisher.com/imat2022



thermofisher.com/materialsscience - Booth 433

#### Additive Manufacturing Processes

#### FORMATS:

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Pages: 475

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**Ceramic Additive Manufacturing Processes** Division Editor: Ming Leu

**Metal Additive Manufacturing Processes** Division Editors: Allison Beese and Howard Kuhn

**Additive Manufacturing of Metals** Division Editor: Howard Kuhn

**Direct Write Processes** Division Editor: Eric MacDonald

#### ASM HANDBOOK, VOLUME 24: ADDITIVE MANUFACTURING PROCESSES

VOLUME EDITORS: DAVID L. BOURELL, WILLIAM FRAZIER, HOWARD KUHN, AND MOHSEN SEIFI

> This valuable addition to the ASM Handbook series was developed to address the current and rapidly expanding importance of additive manufacturing (AM) and provides the latest knowledge in materials, processes, and applications of AM, written by the leading experts in research and industry.

This volume begins with an introduction and history of AM, authored by some of the key participants in that history as they trace the evolution of AM. The complete suite of materials and processes for polymers and ceramics are described in detail in the next two divisions. A division on metal AM methods begins with an in-depth description of the production and characterization of metal powders, a major factor in the success or failure of metal AM processes.

This handbook illustrates the differences in characteristics of metal alloys produced by AM processes in contrast to conventional methods. It also covers direct-write processes, which take advantage of AM techniques to combine materials and devices for multifunctional engineering applications.

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JULY/AUGUST 2022 | VOL. 17 | ISSUE 2



INTERNATIONAL THERMAL SPRAY & SURFACE ENGINEERING

THE OFFICIAL NEWSLETTER OF THE ASM THERMAL SPRAY SOCIETY

# ACHIEVING ZERO DEFECTS IN THERMAL SPRAY

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# SEPTEMBER 13-14, 2022 NEW ORLEANS, LOUISIANA

# SHOW PREVIEW

Join us at the new Thermal Spray & Surface Engineering Forum and Expo (TSSE), which will encompass technical programming from two co-located conferences — the North American Cold Spray Conference (NACSC 2022) and New and Emerging Markets in Thick Films & Coatings: Unconventional Uses of Thermal Spray (NEM-TS 2022). This two-day event will be co-located with IMAT 2022 in New Orleans, September 13-15, giving attendees access to more than 300 additional presentations for a small add-on fee to the TSSE registration.

TSSE Forum & Expo attendees will have the opportunity to learn from leading experts in the thermal spray field and see state-of-the-art products in the thermal spray pavilion on the expo show floor. A special poster session and competition will showcase the work of emerging professionals and students.

# **KEYNOTE LECTURES**



# MITOCHONDRIAL GENERATOR DEVELOPMENT AND PRODUCTION FOR POWERING SENSORS

**Dr. Bertrand Jodoin, FASM** Professor, Department of Mechanical Engineering University of Ottawa



# COLD SPRAY TECHNOLOGY FOR NUCLEAR ENERGY SYSTEMS

**Dr. Kumar Sridharan, FASM** *Professor, College of Engineering University of Wisconsin-Madison* 

# **TECHNICAL PROGRAM TRACKS**

# Track 1: North American Cold Spray Conference

Including: Emerging Applications, Fundamentals, R&D

# Track 2: New and Emerging Markets for Thick Films and Coatings: Unconventional Uses of Thermal Spray

Including: Markets and Applications, Power Generation, Novel Cold Spray Applications, Aerosol Deposition, Corrosion in Nuclear Industry and Biological Applications

# For event details and to register, visit tssforum.org

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# SCHEDULE AT-A-GLANCE

# MONDAY, SEPTEMBER 12

| 7:00 a.m5:00 p.m.       | Conference Registration Open |
|-------------------------|------------------------------|
| TUESDAY, SEP            | TEMBER 13                    |
| 7:00 a.m5:30 p.m.       | Conference Registration Open |
| 8:00-8:45 a.m.          | Keynote Session              |
| 9:00 a.m5:30 p.m.       | Exhibit Hall Open            |
| 9:00-10:00 a.m.         | Breakout Sessions            |
| 10:00–10:30 a.m.        | Break with Exhibitors        |
| 10:30 a.m12:00 p.m.     | Breakout Sessions            |
| 12:30-1:30 p.m.         | Lunch with Exhibitors        |
| 1:30-3:00 p.m.          | Breakout Sessions            |
| 3:00-3:30 p.m.          | Break with Exhibitors        |
| 3:30-4:30 p.m.          | Breakout Sessions            |
| 4:30-5:30 p.m.          | Exhibitor Welcome Reception  |
| WEDNESDAY, SEPTEMBER 14 |                              |
| 7:00 a.m.–5:00 p.m.     | Conference Registration Open |
| 8:00–8:45 a.m.          | Keynote Session              |
| 9:00 a.m.–5:00 p.m.     | Exhibit Hall Open            |

# **EXHIBITION HOURS**

Breakout Sessions

**Breakout Sessions** 

Break with Exhibitors

Lunch with Exhibitors Breakout Sessions

Break with Exhibitors Breakout Sessions

Social Event: Mardi Gras World

**Tuesday, September 13** 9:00 a.m.–5:30 p.m. Welcome Reception with Exhibitors: 4:00 – 5:30 p.m.

# Wednesday, September 14

9:00 a.m.-5:00 p.m.

9:00-10:00 a.m.

10.00-10.30 a m

12:00-1:30 p.m.

1:30-3:00 p.m.

3:00-3:30 p.m.

3:30-4:30 p.m.

6:00-9:00 p.m.

10:30 a.m.-12:00 p.m.

\*Exhibition hours are subject to change. To view full exhibitor list, see page 29 in this issue.



# THE OFFICIAL NEWSLETTER OF THE ASM THERMAL SPRAY SOCIETY

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# EDITORIAL OPPORTUNITIES FOR *iTSSe* IN 2023

The editorial focus for iTSSe in 2023 reflects established applications of thermal spray technology such as power generation and transportation, as well as new applications representing the latest opportunities for coatings and surface engineering.

# April:

Aerospace Industry and Military Applications

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ACHIEVING ZERO DEFECTS IN THERMAL SPRAY: CULTURE, PROCEDURE AND DIGITALIZATION





RECOGNIZING ARTIFACTS IN AIR PLASMA SPRAY THERMAL BARRIER COATING MICROSTRUCTURES



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# **ABOUT THE COVER**

The photo shows a thermal spray cell, encompassing aspects of minimum standards for cleanliness, ergonomics, and layout. Photo courtesy of Turbine Surface Technologies Ltd.

# **EDITORIAL**



# TSS OPEN MIC: SENDING FORTH A TINY RIPPLE OF HOPE

nline working, presentations, and conferences have become part of our daily lives over the past two years, as many of us have been working remotely. Although the pandemic took a toll on all of us, specifically in our thermal spray R&D community, graduate students and post-docs all around the world quickly adapted to the situation and made the best of it. While research lab access has been limited, these early career researchers have been models of resilience and found creative ways to make up for the lack of lab time. Cancellation of the vast majority of international conferences in 2020 was the most detrimental aspect of the pandemic for the development of these young professionals, preventing them from opportunities to get a better feel for the field, meet potential employers, and receive guidance on career options from world experts. However, in the wake of this pandemic disaster, communication platforms became household names within a few months, and some amazing ideas have flourished.

One initiative was started with Prof. Bertrand Jodoin, FASM, (University of Ottawa, Canada) who in May 2020 organized the uOttawa Cold Spray Lab Carte Blanche series, in which members of the thermal spray community were invited to interact informally with his graduate students/ post-docs via webinars. This format aimed to "break the ice" between experienced professionals and the next generation of thermal sprayers. One of the early invited guests was Charles Kay, FASM and past TSS president (Hannecard Roller Coatings - USA). Inspired by this great initiative, Kay realized the amazing opportunity and suggested incorporating it as a TSS program. Jeanelle Harden (ASM Global Conference Manager) promptly went through all the approvals needed, secured sponsors, organized the publicity, and sorted out the technical logistics. And voila, the Thermal Spray Open Mic series of talks was created in October of 2020.

The one-hour virtual TSS Open Mic sessions are geared toward early career researchers. They are free and open to the thermal spray community from all over the world and follow a light and informal format, with Dr. Rogerio Lima, TSS vice president (National Research Council of Canada) as the host. The main objective has been to ensure that young professionals have direct access to thermal spray world leaders, not only to receive science and engineering updates, but more importantly, to learn about their views of our industry and how to better prepare to enter the workforce. The goal is to promote a relaxed discussion and exchange of information at both the technical and non-technical level. Over the months, our speakers shared a great deal of wisdom and professional advice. TSS Open Mic has had a great impact on our community and been a major success. In each talk, we have



Lima

attendees (live!) from more than 30 countries. Many people who were unable to travel had an opportunity to associate a name with a face, seeing amazing thermal spray professionals sharing their knowledge and wisdom.

Inspired by this achievement, Prof. Jodoin had the idea to contact companies to provide funds to sponsor ASM-TSS memberships for young professionals, to get them engaged with our thermal spray community. Thus, the ASM-TSS Student Membership Initiative was created. In 2022, thanks to the sponsors, a total of 45 students and post-docs from 13 different countries were awarded membership. These are their first steps in this professional networking world, and the sky's the limit.

So who is next in line to speak to share ideas and offer mentorship? It is up to us when opportunities arise and how we initiate these ideas to grow the membership. Our common interests drive us together and a long history of remarkable TSS volunteers continue to find new ways to communicate so that all have the opportunity. The TSS Open Mic Series is here for young research professionals and all members of our thermal spray community, with new talks coming for the 2022-23 season. We invite everyone to register for free at www.asminternational.org/ web/tss-open-mic-series where you can watch recordings of past sessions. We hope to "see" you at the next TSS Open Mic.

### **Rogerio S. Lima**

(along with Charles M. Kay, FASM and Prof. Bertrand Jodoin, FASM) ASM Thermal Spray Vice President

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# SOCIETY NEWS

# iTSSe

# **TSS ANNOUNCES THE 2022** HALL OF FAME RECIPIENTS

The ASM Thermal Spray Society (TSS) is pleased to welcome the following 2022 honorees into the TSS Hall of Fame: Heiko Gruner, Neil Matthews, and Subramaniam Rangaswamy.

Dr. Heiko Gruner was born in 1942 in Stuttgart, Germany, and graduated with a Ph.D. in applied physics from the University of Tubingen, Germany, in 1972. He first joined Plasma Technik AG, Wohlen, Switzerland, as general manager VPS (vacuum plasma spray) and was the principal developer of the technology with extensive contribution to the equipment, processes,



Gruner

and applications. As founder and director of Medicoat AG, Gruner's specific goal was to produce coatings for implants using vacuum plasma spraying of porous titanium with hydroxyapatite (Ti/HA) as VPS provided unique advantages to coating properties and introduction. VPS-sprayed implants became the gold standard for cementless surgery allowing millions of patients to profit from larger-lasting implants.

His work in Europe revolved around GTS, the German thermal spray society; and SSB, the Swiss Society of Biocompatible Materials. Most notably, he was engaged in promoting thermal spray to the biomedical community.

Gruner has over fifteen patents submitted, frequently publishes academic papers, and has received several awards including René Wasserman Award, Fraunhofer Institute IPA-Award Die Oberfläche 2014 for Innovative Application of Surface Technologic (Coated Joint Replacement Implant out of Ceramic with VPS-Coatings).

His citation reads: "Heiko Gruner has contributed to sustained innovations in thermal spray through four decades of dedicated research, development, commercialization, and multimillion-dollar worldwide market deployment of vacuum plasma sprayed medical implant coatings."

Neil Matthews is the senior manager for Additive Technologies and is also head of Design Organisation as delegated by the Australian Aviation Safety Authority Defence (DASA). He has been employed in a range of engineering and operations roles before becoming the business lead and technical advisor for Additive Technologies. Matthews has Matthews an aeronautical engineering degree



from RMIT University and a masters of aircraft design from Cranfield University (England). For over 50 years, he has dedicated his career to military aircraft engineering, serving as an Air Force officer before moving into the commercial and military aviation industry.

Since 2004, Matthews has pioneered and revolutionized the use of additive technologies for component repair, in particular supersonic particle deposition (SPD) and more recently laser additive deposition (LAD). He has worked closely with the Australian Department of Defence, as well as Australian and international research and academic institutions such as RMIT University, Swinburne University, Monash University, and the U.S. Army Research Laboratories to develop, certify, and commercialize additive technology repair solutions for the restoration and enhancement of aerospace metal components and structures.

Matthews holds worldwide patents for additive repair technology applications.

Matthews is recognized for "leadership in the adoption of advanced thermal spray manufacturing in the area of aerospace component repair technologies that has led to efficiency and economic outcomes."

Dr. Subramaniam Rangaswamy was born in India and completed his bachelor's degree in metallurgical engineering from the Indian Institute of Technology in Kharagpur before moving to the U.S. for graduate studies. He earned an M.S. and Ph.D. in materials science from SUNY at Stonybrook. At Stonybrook, he was mentored by Prof. Herbert Herman, a pioneer and inaugural ASM TSS-HOF recipient.



Rangaswamy

Rangaswamy joined Metco (a division of Perkin Elmer) in 1980. There he was responsible for the development of several patented advanced materials including ternary TBCs, HT clad self-bonding composites, and microcrystalline alloys. In 1988, he joined Sulzer Plasma Technik in Michigan where he led the development of new high temperature abradable powders and coatings for gas turbine applications. In 1996, he joined Wall Colmonoy Corp. where he was responsible for the development and management of nickel/cobalt base hard facing alloys and braze products.

After returning to Sulzer Metco in 2004, until his retirement in 2018, Rangaswamy managed Oerlikon's thermal spray metals and alloys portfolio. He is inventor/co-inventor of 22 U.S. patents with numerous foreign derivatives for the thermal spray industry.

Rangaswamy is recognized "for exceptional service to the TS industry through innovations in ternary TBCs, HT abradables, self-bonding composites, microcrystalline alloys, and unique attrition milled powder manufacturing processes. Also, for educating, training, and mentoring many TS professionals for nearly 40 years."



# JOURNAL OF THERMAL SPRAY TECHNOLOGY VOLUME 30 BEST PAPER AWARDS

The Journal of Thermal Spray Technology (JTST) is pleased to announce the winners of the JTST Volume 30 Best Paper Awards, as chosen by an international committee of expert judges. The awards were presented during the International Thermal Spray Conference and Exposition in Vienna in May.

The Editorial Committee of the journal believes it is important to evaluate the quality of engineering and scientific contributions published in *JTST* and to provide recognition of excellent work and its publication. Each paper is reviewed and evaluated on its merits for scientific and engineering content, originality, and presentation style. The *JTST* Editorial Board and the ASM Thermal Spray Society Executive Board of Directors extend their congratulations to the winning authors.

# The Journal of Thermal Spray Technology Volume 30 Best Paper Award

"Particle Impact Characteristics Influence on Cold Spray Bonding: Investigation of Interfacial Phenomena for Soft Particles on Hard Substrates" by Dr. Aleksandra Nastic and Prof. Bertrand Jodoin, University of Ottawa Cold Spray Laboratory; and Dr. Jean-Gabriel Legoux, and Dr. Dominique Poirier, National Research Council of Canada.





Jodoin

Nastic

Legoux

Poirier

# SEEKING NOMINATIONS FOR THERMAL SPRAY HALL OF FAME

The Thermal Spray Hall of Fame, established in 1993 by the Thermal Spray Society of ASM International, recognizes and honors outstanding leaders who have made significant contributions to the science, technology, practice, education, management, and advancement of thermal spray. For a copy of the rules, nomination form, and list of previous recipients, visit tss.asminternational.org or contact maryanne.jerson@asminternational.org. **Nominations are due September 30 of this year for recognition in 2023.** 

# The Journal of Thermal Spray Technology Volume 30 Best Paper Honorable Mention (tie)

"A Self-Consistent Scheme for Understanding Particle Impact and Adhesion in the Aerosol Deposition Process" by Mr. Robert Sanders, Dr. Scooter Johnson, Dr. Douglas Schwer, Dr. Eric Patterson, Dr. Heonjune Ryou, and Dr. Edward Gorzkowski, U.S. Naval Research Laboratory.

# The Journal of Thermal Spray Technology Volume 30 Best Paper Honorable Mention (tie)

"Benefits of Hydrogen in a Segmented-Anode Plasma Torch in Suspension Plasma Spraying" by Dr. Alice Dolmaire, Mrs. Enni Hartikainen, Mr. Simon Goutier, Dr. Emilie Bechade, Prof. Michel Vardelle, and Dr. Pierre-Marie Geffroy, University of Limoges; and Dr. Aurélien Joulia, Safran Tech.

# The Journal of Thermal Spray Technology Volume 30 Outstanding Review Paper

"Thermal Spray Copper Alloy Coatings as Potent Biocidal and Virucidal Surfaces" by Prof. Javad Mostaghimi, Dr. Larry Pershin, and Prof. Maurice Ringuette, University of Toronto; Prof. Hamidreza Salimijazi, Isfahan University of Technology; and Prof. Mojgan Nejad, Michigan State University. ~iTSSe

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# ACHIEVING ZERO DEFECTS IN THERMAL SPRAY: CULTURE, PROCEDURE AND DIGITALIZATION

Developing a disciplined approach to standardizing special processes and introducing new technologies is key to eliminating defects in thermal spray applications.

Ann Bolcavage, FASM,\* and Benjamin Lagow, Rolls-Royce Corp., Indianapolis Goetz Feldmann, Rolls-Royce Deutschland Ltd & Co KG, Oberursel, Germany

hermal spray remains the process of choice for the application of protective and functional overlay surface treatments for gas turbine engine components to improve performance and extend serviceable life. Coating selection is an integral part of the design process and coatings can be selected for multiple reasons, including wear reduction, corrosion and oxidation protection, and prevention of oil and air leaks, especially in the flow path where leakage can harm overall performance. The performance and reliability of gas turbine engines depend greatly on the quality of the coatings within, and therefore manufacturing control of processes is of utmost importance to ensure that the resulting coating conforms to the full engineering definition. The cost of poor quality is considerable and as



**Fig. 1** — Thermal spray cell, encompassing aspects of minimum standards for cleanliness, ergonomics, and layout. Courtesy of Turbine Surface Technologies Ltd.

a percentage of sales can be as high as 15%, even for 5-sigma processes.

The recent market challenges and post-pandemic recovery for aerospace will place a renewed emphasis on the drive toward a zero-defect culture within operations and the supply chain. The journey to zero defects can be a particular challenge for thermal spray and other special processes as quality cannot easily be measured in situ. A step change reduction in nonconformance and hidden waste can be achieved by using a disciplined approach to special process standardization and new technology introduction that incorporates:

- 1. Implementation of the right operating environment and culture
- 2. Quality procedures employing a system of standardized defect prevention tools
- 3. Digitalization through application of process sensors and diagnostics to assist in data-driven control solutions

# A CULTURE OF COMMITMENT

The foundation for a zero-defect production culture starts at the thermal spray kit and surrounding shop area. Establishing an operating culture committed to process ownership, high functional standards, and a drive to continuously improve through root cause identification and corrective actions is not an engineering project but a leadership initiative. In other words, the organization must create an environment that is intolerant of nonconformance.

A useful exercise to help put the right operating culture in place is to make a detailed walk of the process and assess the thermal spray cell infrastructure, equipment, and procedures (Fig. 1). Ideally, a collaborative audit should be carried out by a cross-functional team. An honest and evidence-based assessment of criteria incorporating people (training and resources), equipment (asset care, raw materials control), and environment (visual management, cleanliness) should be made against an acceptable minimum standard. Improvement plans with regular reviews can then be put in place to address any criteria below the minimum standard. In many cases, conformance to an established minimum standard can result in immediate returns in the form of reduced noncompliance.

These audits are not intended to replace process specifications, Nadcap check sheets, and supplier maturity assessments. Instead, they are expected to supplement those methods and to create an environment where a cross-functional team feels ownership for the process and for ensuring that it remains under long-term control.

# STANDARDIZED DEFECT PREVENTION TOOLS

Once a minimum standard framework is in place, the next step in the journey to zero defects focuses on defect prevention tools—ones that enable better understanding of the process, which can then be leveraged for better control. Three critical tools—the process flow diagram (PFD), the process failure modes and effects analysis (PFMEA), and the control plan—are described in SAE AS13100 and RM13004. These tools are designed to be used together by iterative interactions among them and in conjunction with other important design and manufacturing quality tools (Fig. 2). When these methods are applied and kept up to date, they become essential knowledge management tools that aid in identifying and correcting quality risks. These tools require an intimate knowledge not only of the thermal spray process, but also of the product and features to be coated.

The PFD is a representation of the required process steps to apply a coating onto a component, and it includes all process steps from receipt of parts into the facility to shipment of the completed product. The PFD must also identify any inputs, resources, and controls, as well as any subcontracted process steps. It must also list the sequence of operations and a detailed description of all steps within a given operation.

In turn, the PFD itself is a key input when developing a process failure modes and effects analysis (PFMEA). The PFMEA focuses on identifying those steps where potential noncompliance can jeopardize product quality. In general, each individual part number will be assigned its own PFMEA because there are typically important differences between processes among different components.

The process of creating a PFMEA consists of the following steps:

- · Identify characteristics the process must achieve
- Identify ways the coating can be applied incorrectly (failure modes)
- Understand potential impact of nonconformance to the business (severity)
- Determine which adverse process condition will lead to nonconformance (cause)
- Identify ways to detect failure mode (controls)
- Calculate a risk priority number for each potential hazard (ranking)

Finally, the control plan describes feature checks and measurements to be performed at each processing step to reduce variation in the process and in its product. It summarizes all defined controls (including asset care, calibration, and maintenance) and is based on the PFMEA results. The control plan is not the same as a work instruction or process routing. Instead, it is ideally designed to work alongside such instructions to help improve long-term quality control.

Once a solid foundation is in place—with the right organizational culture and reliable defect prevention tools—the benefits of digital technologies can be fully realized.

# DIGITALIZATION

The need to understand and reduce or eliminate all possible sources of processes variation is increasingly driving development of novel sensors and improved equipment for thermal spray applications. Closed loop controllers, multi-axis robots, and in-flight particle measurement diagnostic tools are among the most successful examples embraced by the industry.

One area where sensors and digital methods can bring greater process understanding is the quantification of



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Fig. 3 – Schematic illustration of digital control system sensors and linkages for enhanced control.

uncontrolled "noise" within the thermal spray process and its effect on measurable inputs and outputs. Consider the acoustic monitoring system developed by Rolls-Royce Corp. in conjunction with Virginia Tech and the Commonwealth Center for Advanced Manufacturing (CCAM). This technology acquires audible feedback from the thermal spray system and analyzes it in real time. The acoustic monitoring system can accurately detect many different types of events in the thermal spray cell, including high or low gas flow rates, powder line leaks, port and nozzle wear, and pulsing in the powder feed and/or carrier gas. Another sensor has been developed to monitor the spray plume for visual changes, including variations in angle and intensity. Data from each of these sensors can be correlated to variables such as powder and gas feed rate, enabling a better understanding of the state of the thermal spray cell.

Ultimately, the vision is to create a system capable of self-correction within prescribed limits to ensure output quality. By monitoring external attributes such as light and sound, a potential control system has been demonstrated (Fig. 3). Changes detected by acoustic and visual monitoring are fed back to a control algorithm that actively updates the internal processing parameters of the thermal spray cell to compensate for any fluctuations in the system.

### **SUMMARY**

The journey to zero defects is about understanding the sources of variation and reducing or eliminating them from a controlled process. A focus on prevention, robust use of defect reduction tools, and increased use of process diagnostics are more essential than ever for eliminating defects and nonconformance from thermal spray processes. New tools and emerging technologies can be leveraged to create a manufacturing environment where thermal spray rework can be greatly reduced, resulting in cost reductions and shorter lead times for suppliers and OEMs. **~iTSSe** 

**For more information:** Ann Bolcavage, engineering associate fellow – coatings, Rolls-Royce Corp., 420 S. Meridian St., Indianapolis, IN 46225-1103, ann.bolcavage@rolls-royce. com.

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# RECOGNIZING ARTIFACTS IN AIR PLASMA SPRAY THERMAL BARRIER COATING MICROSTRUCTURES

Identifying metallographic TSC artifacts, specifically in air plasma spray zirconia-based thermal barrier coatings.

he microstructure of a thermal spray coating (TSC) is typically a primary indicator of the coating quality, its functional properties, and proper deposition and processing. Due to the nature of the TSC deposition process, the microstructures are not homogeneous, comprising multiple phases and phase boundaries, as well as layers and directionality. As a result, TSC microstructures are prone to interactions with common metallographic procedures that may result in artifacts and misinterpretation of the TSC microstructure.

Many technical papers have been published on the best practices for metallographic preparation of TSCs<sup>[1,2]</sup>. Any of several metallographic preparation methods have been shown to produce a true and reproducible TSC microstructure. For comparative purposes, the TSCs used in this case were deposited simultaneously and polished using variants of ASTM E 1920-03 Method-II<sup>[3]</sup>. Artifacts that result from specific sectioning and mounting practices, as well as from different polishing times, are presented.

# **TYPICAL APS MICROSTRUCTURAL FEATURES**

The air plasma spray (APS) thermal barrier coating (TBC) is typically a bi-layer coating comprised of a softer, relatively ductile, metallic bond coating (BC) and a harder, porous, lower ductility ceramic top coating (TC). In the TC, porosity and cracks are key to its function as a thermal barrier layer in aero and power generation turbine engines. In the BC, features related to cohesion between splats and adhesion to the substrate (porosity, oxidized boundaries, unmelted particles, cracks) are indicators of TBC service life at high temperature.

A simplified drawing of key microstructural TSC features highlights the degree of inhomogeneity present in these coatings (Fig. 1). In addition to three distinct layers that make up a TBC (the substrate alloy, the BC, and the TC), there are other features common to TSCs in general such as voids or pores, oxidized particles in the metallic BC, metallic inclusions in the TC, unmelted or spherical particles, grit particles at the substrate-BC interface, vertical micro-cracks, and horizontal debonding cracks. Some amount of each of these features are allowable in a TBC TSC and are defined in the specific quality requirements for the coating.



Fig. 1 - Key TSC microstructural features<sup>[1]</sup>.

# **TBC METALLOGRAPHIC PROCESS**

Metallographic processing of TBCs follows the same general steps as those of wrought material, that is, sectioning, mounting, planar grinding, polishing, microscopy, and analysis. An exception is that chemical etching of the TSC is not typically done prior to microscopy. As described in other sources, the magnitude and direction of forces during sectioning can damage the TBC significantly, such that subsequent metallographic steps cannot remove the damage. Similarly, mounting and polishing methods also affect loading on internal microstructure interfaces and phase boundaries; consequently, these features can be changed by the method of mounting and polishing that is employed. Accepted metallographic practices for thermal spray coatings are well documented<sup>[1,2]</sup> and specific deviations from accepted methods are used in this article to create artifacts in the TBC



microstructure and ASTM E1920-03 Method- $II^{[3]}$  is the basis of all polishing.

# FACTORS THAT CAUSE TSC ARTIFACTS

**Sectioning** involves applying several forces to a part/ sample, including clamping pressure, blade incursion, bending, friction, shear, and thermal expansion stresses. Specific to as-deposited TSCs that are layered structures and are not metallurgically bonded to the substrate, the saw, blade, or cutoff wheel applies directional forces on the sample. If the blade enters the substrate and exits through the coating, it applies forces that push the coating away from the substrate.

If those forces exceed the local bond strength of the coating, "debonding" can occur. Debonding presents as horizontal cracks or gaps along or near the substrate interface. Also, specific to TBCs, where the TC is a less-ductile material, sectioning into the substrate and exiting the TC surface can also cause horizontal cracking within the TC. The TC cracks may appear discontinuous as the crack propagates in and out of the plane of view of the image. Debonding and TC cracking are possible artifacts of sectioning.

Subsequent grinding and polishing steps may not remove the artifacts of sectioning. A unique artifact of a TBC TC initiates as horizontal cracking due to sectioning and presents in the final polished structure as large, elongated pores or strings of pores that are not randomly distributed. Misinterpretation of this structure characterizes the TC as having greater pore fraction than actual.

Figure 2 shows a TBC microstructure with artifacts from improper sectioning, i.e., at high incursion rate, using limited coolant, and with the blade oriented to cut into the substrate and exit the coating surface. Figure 2a shows several fields of view demonstrating the crack path in the TC and loss of TC that resulted from cracking during sectioning. Figure 2b shows a higher magnification of this sample with the specific artifacts labeled.

For non-porous wrought materials, sectioning is performed prior to mounting. For TBCs, the porosity in the TC is critical to the function of a TBC, and those pores represent internal surfaces that can fracture or yield under applied loads from sectioning and mounting. Because TBCs are porous and easily damaged by the forces involved in sectioning, coatings should be vacuum-infiltrated with epoxy prior to sectioning to support the pore walls during sectioning, thus minimizing the risk of artifacts.

**Mounting** processes can be characterized broadly as hot-mount type and cold-mount type. Among the various mounting materials in either process, cold-mount materials applied using vacuum infiltration provide the lowest risk of artifacts for TBCs. All samples presented in this section were polished using ASTM E 1920-03 Method-II, non-modified. The distinctions of polishing using Method-I or Method-III are not discussed here. Figure 3 highlights common TBC microstructural artifacts caused during mounting. The images show several distinctive artifacts: a) a hot-mount acrylic with artificially large pores in the TC, most elongated or aligned horizontally, which is consistent with horizontal cracking in the TC that becomes emphasized in relief during polishing; b) a hot-mount phenolic with a continuous crack along the TC-BC interface due to elastic-plastic strain, collapse of TC pores under compression, or overall bending forces; c) cold-mount acrylic samples have no horizontal cracking but may have localized large elongated pores near the bottom of the TC





**Fig. 2** — Artifacts in TBC caused by sectioning shown in (a) lower magnification images showing loss of TC by cracking and decohesion during sectioning and (b) high magnification image with arrows indicating additional horizontal cracking in the TC along the substrate interface in the BC. Samples and images provided by FL Institute of Technology, Center for Advanced Coatings, Melbourne, Fla., and Technetics Group, Deland, Fla.

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**Fig. 3** — Artifacts in TBC resulting from mounting methods. Images provided by the Center for Advanced Coatings at Florida Institute of Technology.

because cold-mount acrylic is fast-curing, highly viscous, and may not infiltrate the TC entirely leaving pores unsupported and subject to relief; d) a vacuum-infiltrated epoxy mount has negligible artifacts due to high internal pore support and low applied stresses.

**Grinding and polishing.** Even when proper sectioning and mounting methods are used, minor and localized artifacts will be present near the sectioning plane, and these can be removed by proper grinding and polishing practices. For this reason, the initial planar grinding step is typically longer compared to grinding wrought materials. The grinding abrasive size is also typically finer than those used for wrought materials. One industry practice suggests a removal of 0.060-in. from the mount height during planar grinding of TSCs to ensure artifacts from previous steps have been removed.

Of course, planar grinding uses large abrasive size and the deep abrasion forces can cause artifacts of their own. Subsequent polishing with a series of finer abrasive media can remove the planar grinding artifacts when applied for sufficient time. In this section, all samples were properly sectioned then mounted using vacuum-infiltrated slow-curing epoxy. All were planar ground using 180-grit SiC paper with water coolant, replacing the paper every 30 seconds until each sample was planar. The effect of the supplier of consumables and abrasives is not addressed in this paper, but abrasive quality and polishing surface resiliency may affect artifact formation.

The post-planar polishing steps follow the ASTM E 1920-03 Method-II<sup>[3]</sup>, however, polishing times were adjusted to demonstrate how each step contributes to pore structure stabilization and removal of artifacts from the previous step.

Under all polishing conditions here, the BC appears the same, exhibiting clear splat boundaries, voids, and unmelted particles. The BC is far less sensitive to artifacts than the TC under these polishing conditions, but BCs also have artifacts. The most common artifact in a BC is smearing (plastic deformation) of the metal on the polished plane that obscures these BC features. Smearing can be caused by worn consumables, dull abrasives, or improper polishing surface. Also, the BC is softer than the TC and can exhibit scratches more easily. Scratches in the BC can indicate insufficient polishing time to remove artifacts from the previous step. Scratches may also indicate contaminated or worn polishing paper.

An artifact common to any polishing method is edge-rounding. It is typically identified during the optical microscopy analysis as a field of view (FOV) where all



features within the FOV cannot be brought into focus. The term "edge-rounding" connotes that the edge of the coating at the mount medium interface is not in plane with the rest of the coating. It can occur when the mount medium is softer than the coating and abrades faster than the coating. It can also be caused by highly resilient polishing papers that yield excessively into pores and along edges, increasing the polishing rate in those locations. The addition of  $Al_2O_3$  particles to the cold-mount epoxy is one method to minimize edge rounding by adding hard particles to support the epoxy. **~iTSSe** 

**For more information:** This article is an excerpt of a document prepared by the ASM Thermal Spray Society Accepted Practices Committee. Special thanks are extended to the committee members and the contributors of this paper: Elaine Motyka, Technetics Group, and Frank Accornero,

Florida Institute of Technology, Center for Advanced Manufacturing. The authors may be reached at elaine.motyka@ technetics.com and faccornero@fit.edu.

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# JTST HIGHLIGHTS

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he Journal of Thermal Spray Technology (JTST), the official journal of the ASM Thermal Spray Society, publishes contributions on all aspects—fundamental and practical—of thermal spray science, including processes, feedstock manufacture, testing, and characterization. As the primary vehicle for thermal spray information transfer, its mission is to synergize the rapidly advancing thermal spray industry and related industries by presenting research and development efforts leading to advancements in implementable engineering applications of the technology. Articles from recent issues, as selected by *JTST* Editor-in-Chief Armelle Vardelle, are highlighted here. In addition to the print publication, *JTST* is available online through springer-link.com. For more information, visit asminternational.org/ tss.

# POROUS APS YSZ TBC MANUFACTURED AT HIGH POWDER FEED RATE (100 g/min) AND DEPOSITION EFFICIENCY (70%): MICROSTRUCTURE, BOND STRENGTH, AND THERMAL GRADIENTS

### **Rogerio S. Lima**

There is a strong driving force to improve the production efficiency of thermal barrier coatings (TBCs) manufactured via air plasma spray (APS). To address this need, the highenthalpy APS torch Axial III Plus was employed to successfully manufacture TBCs by spraying a commercial YSZ feedstock at powder feed rate of 100 g/min using an optimized set of N<sub>2</sub>/H<sub>2</sub> spray parameters, which yielded an impressive YSZ deposition efficiency (DE) value of 70%. This exact same set of optimized spray parameters was used to manufacture the same identical YSZ TBC (over ~160  $\mu$ m-thick bond-coated substrates) but at two distinct YSZ thickness levels: (i) ~420  $\mu$ m-thick and (ii) ~930  $\mu$ m-thick. (Fig. 1).



**Fig. 1** — Experimental procedure equipment: the overall view of the NRC's thermal gradient laser rig.

# EFFECT OF METALLIC INTERLAYER HARDNESS ON DEPOSITION CHARACTERISTICS OF COLD-SPRAYED COPPER PARTICLES ON CARBON FIBER-REINFORCED POLYMERS

# Panteha Fallah, Rohan Chakrabarty, Jun Song, André McDonald, and Stephen Yue

Copper (Cu) has been successfully cold spray deposited on carbon fiber-reinforced polymer (CFRP) by a hybrid fabrication process. In this study, the feasibility of Cu coating build-up on a Cu electroplated CFRP under a process with two-step gas pressures was investigated numerically and experimentally. The deformation and deposition behavior of the Cu particles on CFRPs coated with tin (Sn), nickel (Ni), and Cu were studied by comparing the single-particle impact with thick coating fabrication. The deposition efficiency (DE) of the cold-sprayed coatings was measured, and the microstructure and microhardness of the coatings were evaluated. (Fig. 2).



Fig. 2 — SEM image of the feedstock powder Cu.

# PERSPECTIVE: CHALLENGES IN THE AEROSPACE MARKETPLACE AND GROWTH OPPORTUNITIES FOR THERMAL SPRAY

### M.R. Dorfman, G. Dwivedi, C. Dambra, and S. Wilson

The market needs for increased engine efficiency and environmentally friendly solutions remain the key drivers for the aerospace industry. These efficiency gains will be achieved by meeting the challenges of higher engine operating temperatures, weight reduction, and novel surface solutions for increased component longevity. A critical question to address is if the thermal spray (TS) industry can continue to meet the challenges and demands seen by the airlines and the engine manufacturers. In addition to non-aerospace influences, the COVID-19 pandemic has dramatically affected the landscape of industry growth, not only directly on airlines but also on the associated supply chain. This article reviews this market, its suppliers, and identifies the challenges and opportunities for future growth. (Fig. 3).



**Fig. 3** — Schematic of jet engine highlighting areas that require critical coating solutions.

# THERMAL SPRAY COATNGS FOR PROTECTION AGAINST MICROBIOLOGICALLY INDUCED CORROSION: RECENT ADVANCES AND FUTURE PERSPECTIVES Jianxin Wen and Hua Li

Corrosion has been persisting as the most severe concern for steel structures in the marine environment. Due to the widespread occurrence of biofouling, apart from the electrochemical corrosion, microbiologically induced corrosion (MIC) is an important factor that triggers the deterioration of marine steel infrastructures. Traditional anticorrosion coatings usually lack the antifouling function, attachment, and colonization of marine microorganisms, therefore, in most cases, accelerate existing corrosion damage. Anticorrosive coatings fabricated by thermal spray has been extensively applied for marine corrosion prevention, yet the anti-MIC coatings deposited by thermal spray technical route remain elusive. Developing thermal-sprayed coatings with dual anticorrosion and antifouling performances is key to combating MIC.

# AN OVERVIEW OF THERMALLY SPRAYED Fe-Cr-Nb-B METALLIC GLASS COATINGS: FROM THE ALLOY DEVELOPMENT TO THE COATING'S PERFORMANCE AGAINST CORROSION AND WEAR

### Moreira Jorge Jr. and Walter José Botta

Fe-based bulk metallic glass (BMG) presents unique tribological and electrochemical properties. Given the inherent brittle nature and dimensional limitations of Fe-based BMGs, technological and scientific efforts are focused on their use for surface engineering solutions. Fe-based BMG coatings are promising to protect steel components operating in a wide array of hostile environments, with encouraging resistance against corrosion and wear. This article summarizes the progress of Fe-Cr-Nb-B glassy coatings in terms of alloy design, glass-forming ability, crystallization, powder production, thermally sprayed coatings, and how the microstructural features dictate the basket of properties. The strategy for selecting the alloy composition with high glass-forming ability is discussed based on thermodynamic calculations. (Fig. 4).



**Fig. 4** — View of nozzle upon atomizing and the spherical particles produced.



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PRACTICAL PLASMA NITRIDING FOR 17-4 PH STAINLESS STEEL

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BY JON L. DOSSETT

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# PRACTICAL ASPECTS OF PLASMA NITRIDING KINETICS FOR 17-4 PH STAINLESS STEEL

*E. Rolinski, A. Springer, and M. Woods* Plasma nitriding is very effective in removing the passive layer of chromium oxide formed naturally on the surface of stainless steel, but controlling the kinetics is key for optimal results.



# TECHNIQUES FOR Determining retained Austenite

### Thomas Wingens

Accurate measurement of retained austenite levels is important in the development and control of a heat treatment process.

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# EDITORIAL OPPORTUNITIES FOR *HTPro* IN 2022

The editorial focus for *HTPro* in 2022 reflects some key technology areas wherein opportunities exist to lower manufacturing and processing costs, reduce energy consumption, and improve performance of heat treated components through continual research and development.

**November/December** Atmosphere & Vacuum Heat Treating

To contribute an article to an upcoming issue, contact Vicki Burt at vicki.burt@asminternational.org.

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# **DEPARTMENTS**

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# **ABOUT THE COVER**

Plasma nitriding, also referred to as ion nitriding, eliminates chromium oxides on the surface of stainless steels. Courtesy of Advanced Heat Treat Corp.

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# GROWING DIVERSITY IN THE HEAT TREAT INDUSTRY

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Brilliance can come from anywhere. We as an industry are missing valuable opportunities for technological breakthroughs and material innovation. We see high turnover, struggle with retention, employees who lack a sense of belonging and community in the workplace, and all of this is stifling creativity. Increasing diversity, building equity, and including people from



Frame

a greater variety of backgrounds and life experiences will make our industry more dynamic, innovative, and successful. You've heard of DEI (diversity, equity, and inclusion). ASM International and its affiliate societies focus considerable effort in this direction. Diversity relates to people, backgrounds, perspectives, education, ability, and interests. Diversity in genders, race, ethnicity, nationality, age? Yes, I mean all of this.

Before the pandemic, in 2017, 25% of first year college students in STEM were women, but over 32% of those women switched out of STEM programs prior to graduation<sup>[1]</sup>. In the workforce in 2017, only 13% of engineers were women. This is a sad starting point, and while we still do not know the full impact of COVID-19 on women in STEM, we do know it is significant<sup>[2,3]</sup>. Women in STEM were more negatively impacted by the pandemic than any of us expected. In fact, the impact of COVID-19 on women and black, indigenous, and people of color is so profound that the National Academies and National Science Foundation have each focused considerable effort on addressing longterm repercussions of the pandemic on women in STEM<sup>[4-6]</sup>.

Data on equity specifically in heat treating are quite limited, but the ways we celebrate some of our rising stars serves as a litmus test. *Heat Treat Today* runs an annual "40 under 40" award list, and the 2021 lineup was 15% women<sup>[7]</sup>. This puts us on track with pre-pandemic women in engineering demographics as a whole, which is good, but 15% is still far too low. How can we do better?

**Plant the seeds.** Start by reaching out to younger audiences. Team up with local schools, Girl Scout troops, or the ASM Education Foundation; encourage your business to host a student tour of the factory or go visit your child's classroom. It doesn't take much to get sixth graders interested in heat treating. I regularly show up at my children's schools with a package of piano wire, a blow torch, safety glasses, some needle nose pliers, and a bucket of water. Plant those seeds of interest in metastable phase transformations and processing-property relationships that are fundamental to heat treating. **Grow.** Next, focus on mentorship and encouragement. Provide students with research opportunities, internships with built-in mentoring, and travel grants for conferences. ASM and HTS do a lot on this front, and we always need support to do even more. Ask your company to sponsor an award at a conference, donate \$500 for a student travel grant, or sponsor a senior design project.

At the career level, encouragement looks a little different. Cast a broad net when hiring a new team member. Reach out to historically black colleges and universities and Hispanic-serving institutions, work with ASM at conferences to attract new talent, and write job descriptions to ensure a wide pool of applicants. A commonly repeated statistic from Sheryl Sandberg's book "Lean In," is that women tend to only apply for jobs when they meet 100% of the qualifications, but men tend to apply for jobs when they meet at least 60% of the qualifications. Admittedly, the accuracy of this statistic is debated, but research by LinkedIn and others have corroborated the trend<sup>[8,9]</sup>: equally qualified women apply for fewer jobs than their male counterparts. Knowing this, we can rewrite job descriptions to attract more women applicants. Move "required" qualifications into a "nice to have" category, use inclusive language and less masculine wording, and mention com-



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# **GUEST EDITORIAL**

mitments to mentoring new hires as a benefit of working at the company.

**Nurture and Support.** Finally, support and maintain the diverse team you build. Making employees feel welcome can be as simple as having safety gear and uniforms that actually fit the women on your team, being forthcoming about maternity leave policies, being flexible with shifting workday hours, or even just stopping by and asking the new hire how she is doing. Other efforts are more involved: promote diversity in leadership levels, ensure your employees know the organization's DEI goals, provide effective training on how to be an ally, how to use inclusive language, and how to be a mentor. Each of us can learn more about DEI and provide better support.

Who can promote a diverse future for Heat Treating? Well yes, each of us must contribute to creating an inclusive and welcoming environment, but we know there are some with more influence than others. The Monty comes out with a list of the most influential people in the North American Heat Treating Industry each year<sup>[10]</sup>. None of the people on this list are women, and very few are people of color. This means that advocating for women in heat treating cannot just be the job of the women already in heat treating. We need support and action from all levels (especially from those in top positions in the field) and from all genders. If you are in position of power (a senior fellow, a department head, manager or director, an HR manager, or maybe you are a president, CEO, or CTO), I urge you to leverage that position to change your organization policies and practices to promote DEI at your workplace. Emulate the best practices, create new opportunities, and help us to build a more diverse future for heat treating. Together we can push heat treating to lead the charge for diversity, equity, and inclusion in STEM and usher in a new era of innovation and success to our field.

### Lesley D. Frame

Assistant Professor, University of Connecticut, Storrs President, Heat Treating Society





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# HEAT TREATING SOCIETY NEWS

# ADVANCED MATERIALS & PROCESSES | JULY/AUGUST 2022

# **HTS HONORS PRIME CONTRIBUTOR WINNERS**



In 2017, ASM's Heat Treating Society R&D Committee established a new recognition for the best papers in the heat treat industry each year. To be considered for the ASM HTS Prime Contributor recognition, papers must appear in either the *HTPro* newsletter or be

published in ASM's Heat Treat conference proceedings. All manuscripts from industrial companies (not universities or national labs) are automatically considered on an annual basis. Papers are judged on several criteria, including production readiness, breadth of potential applications, and writing clarity, among other factors.

The aim of the honor is twofold—to recognize outstanding industry-focused manuscripts and to encourage submissions to both *HTPro* and the Heat Treat conference, which takes place every other year. This year's winners include the following authors and their companies:

- Kyozo Arimoto of Arimotech Ltd., Japan, for his paper, "A Brief Review on Validation for Heat Treatment Simulation" published in Heat Treat 2021: Proceedings from the 31st Heat Treating Society Conference.
- Thomas Wingens of Wingens LLC International Industry Consultancy for his paper, "Retained Austenite Benefits or Avoidance Requires Dependable Determination" published in Heat Treat 2021: Proceedings from the 31st Heat Treating Society Conference. See page 10 for an excerpt of this paper.

Winners receive a plaque and a digital logo for use in promoting their awards. For more information, contact Vicki Burt at vicki.burt@asminternational.org.





Kyozo Arimoto with his Prime Contributor award.

Thomas Wingens shows his Prime Contributor award.

# HEAT TREATING PROGRAMMING AT IMAT 2022

IMAT is ASM's annual meeting and industry-focused conference and exposition. This year's theme is: The Circular Materials Economy. Held in New Orleans from Sept. 12-15, the IMAT program has focused efforts on materials, applications, and technologies that involve industrial processes and economic activities that are restorative or regenerative by design. The show includes extensive heat treating programs, listed below. We hope you will join your colleagues at the show. Visit the registration page at imatevent.org for more information.

# **Technical Programming**

- Monday, Sept. 12, 10:30–11:30 a.m. Distortion Control and Residual Stress Session, chaired by **Lesley Frame**, University of Connecticut
- Monday, Sept. 12, 1:00–2:00 p.m. Surface Heat Treatment Session, chaired by Olga Rowan, Caterpillar Inc.
- Wednesday, Sept. 14, 3:00-4:00 p.m. "The Circular • Materials Economy" Panel Session: A circular materials economy reduces material use, redesigns materials to be less resource intensive, and recaptures "waste" as a resource to manufacture new materials and products. It encompasses traditional efficient and lean practices as well as the now commonplace trifecta of "reduce, reuse, recycle" as key components of sustainable materials design, production, maintenance, and management programs. Because of the importance of reducing environmental impacts and preserving natural resources to the global health and well-being of current and future generations, the focus for IMAT 2022 has been chosen around this topic. Given the interdisciplinary nature of materials, the panel will include representatives from each of the six ASM International affiliate societies. The HTS representative serving on the panel will be **Robert Madeira**, Inductotherm Group.

# **Student Activities Organized by HTS**

**Fluxtrol Academic Research Competition.** The goal of this competition is to encourage the participation of young scientists in the ASM Heat Treating Society, and to provide attractive offers and opportunities in the worldwide thermal processing community. All students will participate in the poster competition on Tuesday. Selected finalists will then participate in the oral presentation phase on Wednesday. **Robert Goldstein,** Fluxtrol, is the competition chairperson.



**HTS Strong Bar Competition.** Student teams will heat treat steel bar to achieve the highest combination of bending strength and bend deflection. Sponsored by Inductotherm Group and Instron, this is a two-phase competition starting with poster judging on Tuesday, Sept. 13, and bend testing on Wednesday, Sept. 13. **Robert Cryderman**, retired, Colorado School of Mines, is the competition chairperson.



### Additional Competitions/Awards

The Prof. Valentin S. Nemkov Academic Research Award is Fluxtrol Inc.'s annual recognition program for post-doctoral researchers, lecturers, and professors (academic researchers) that is presented annually at either IMAT or the ASM Heat Treating Society Conference & Expo for the researcher that had the greatest impact on the event in the area of thermal processing of materials.

The next award will be given at IMAT 2022. Candidates are required to be present and the winner will be announced at the event. **Robert Goldstein,** Fluxtrol, is the award chairperson.



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FEATURE

# PRACTICAL ASPECTS OF PLASMA NITRIDING KINETICS FOR 17-4 PH STAINLESS STEEL

Plasma nitriding is very effective in removing the passive layer of chromium oxide formed naturally on the surface of stainless steel, but controlling the kinetics is key for optimal results.

> **E. Rolinski,\* A. Springer,\* and M. Woods** Advanced Heat Treat Corp., Monroe, Michigan

he nitriding of stainless steels is a valuable process, as when it is done correctly, it can greatly increase both the surface mechanical properties as well as corrosion resistance. However, the nitriding of stainless steels requires special care due to the problems relating to activation of the surface of the components when conventional methods such as gas and salt bath nitriding are used<sup>[1-11]</sup>. Hydrogen chloride admixture is often used as a surface activator<sup>[10]</sup> in these atmospheres. This may have side effects on the equipment. Therefore, in many situations the preferred method for nitriding stainless steels (SS) is plasma nitriding. The most common questions asked by engineers about nitriding are those related to kinetics of the process and properties of the layers. Plasma nitriding, also referred to as ion nitriding, is very efficient in eliminating native chromium oxides present on the SS surface. However, kinetics of the process must be under control to achieve the desired results.

To demonstrate some of those challenges, a series of ion nitriding tests was performed using sandblasted samples of 17-4 PH steel. The samples were treated at a temperature range of 482° to 557°C (900° to 1034°F) with a nitriding time up to 132 hours and constant gas composition at constant processing pressure<sup>[1]</sup>. Changes in the core hardness of the nitrided samples were analyzed. The results show that microhardness stayed at a high level



**Fig. 1**—Hardness profile in the ion nitrided 17-4 PH sample. Nitriding parameters: temperature 513°C and soak time 15 hrs. Total case depth was 0.0735 mm and the etched zone measured 0.063 mm. The following curve fit equation was used: [LgstcDoseRsp] y=a+b/(1+x/c)<sup>d</sup>,  $r^2$  = 0.998, confidence interval = 95%.

in the nitride layer and then fell abruptly at the interface layer/substrate<sup>[1]</sup>. Nano-hardness measurements were performed on the treated samples and the hardness, as well as the Young's modulus of the nitrided layer were determined<sup>[12]</sup>. Glow discharge spectroscopy was performed on the near-surface regions of the sample and an elevated level of oxygen was found in the distance close to the surface. Case depth was determined via metallographic examination, using Marble's and Nital etching, as well as via microhardness curve determination. A three-dimensional relationship was developed for the mathematical prediction of the case depth dependent on processing time and temperature based off of this data.

# **EXPERIMENT**

The 17-4 PH 1.25-in. diameter bars were aged at 593°C (1100°F) at a general heat-treating facility. Incoming hardness of the steel was 371 HV1, determined by an average



**Fig. 2**—View of an individual indentation after slight etching from the traverse of Fig. 1.



of five readings. Oxides were present on the surface of the samples after the treatment. Therefore, all the samples were sand blasted 5 minutes before each nitriding test. The plasma nitriding was carried out in a cold-wall vessel with a very good leak rate. Plasma density was in the range of 2 W/cm<sup>2</sup>. The typical leak rate was found to be: 0.52 torr·in<sup>3</sup>/ sec or 0.000012 ( $1.2 \times 10^{-5}$ ) mbar·L/s.

The processing pressure was arbitrarily selected at 3.2 mbar (2.4 torr). A gas concentration of 10% nitrogen and 90% hydrogen was used to avoid formation of the excessive compound zone or carbonitrides network. Ramping rates were the same for all samples, about 27°C/hr for the last ramp, prior to soaking.

Processing temperatures were selected to allow covering the full range of interest for the case depth and for the temperature range typically applied for processing various industrial parts. The aim was to quickly calculate the required processing time for the specified case depth and



**Fig. 3**—Photomicrograph of the case formed in the 17-4 PH sample. Nitriding parameters: temperature 513°C and soak time 15 hrs. Observed at 400x, etched with a) Marble's b) and Nital. Note a sharp drop of hardness at the transition zone (the case/matrix).

temperature using interpolation rather than extrapolation method for future runs.

FEATURE

# **CASE DEPTH DETERMINATION**

The case depth was initially analytically determined from the microhardness traverse with the TableCurve2D software using the common definition of the total case depth as the core hardness + 50 HK0.05. A typical hardness curve using the above method is shown in Fig. 1.

It is also worth mentioning that the core hardness of the sample nitrided at 557°C dropped from the initial 371 HV1 to 310 HV1. This demonstrates that severe over-aging processes took place in the steel during this short exposure of the sample at such a high nitriding temperature. Based on the spread of the core hardness data, it cannot be excluded that the samples were not from the same heat-treating batch. This however, has a secondary meaning to the total case depth formation in the steel.

As can be seen from the microhardness curve, it is extremely difficult to precisely measure hardness in the transition zone between the case and the matrix because of the abrupt change of the steel properties in this zone (Fig. 2). Therefore, measurements of the case depth in this steel may be more accurate using optical methods. For this study, it was possible and appropriate to measure the case in the samples etched with Marbles and Nital reagents, as shown in Fig. 3.

# NANO HARDNESS MEASUREMENTS

Due to customer interest in mechanical properties, such as Young's modulus and hardness of the nitrided layer in 17-4 PH steel, nanoindentation and GDS studies were performed at an outside source<sup>[12]</sup>. These tests were performed on a flat sample prepared in a special way by polishing prior to nitriding for the purpose of the test. The results of this study are presented in Fig. 4. As can be seen from the graphs, Young's modulus, as well as the nano hardness of the layer, is lower near the surface of the sample than at the depth of 240 nm (0.24  $\mu$ m), where these values seem to stabilize. Nano hardness in the plateau is about 18,000 MPa, which is equivalent to about 1835 HV. This value is quite high; however, it has to be treated as the true hardness value, more precisely determined than the Knoop value of about 1200 to 1300 HK0.1; a typical result of microhardness testing, which usually is not free of cracking at such high hardness level.

# GLOW DISCHARGE SPECTROSCOPY (GDS) MEASUREMENTS

As can be seen from the distribution of the elements in the near-surface region in Fig. 5, the sample is enriched

# FIPRO FEATURE

in nitrogen to an estimated 18 at.% in its peak value at a depth of approximately 0.33 to 0.5 µm. It stays at a level of about 8 to 9 % through the 12 µm of the tested thickness of the case depth. It should also be noted that the oxygen level near surface exceeds 20 at.% and diminishes rapidly in the first 0.5 µm. Elevated carbon and hydrogen could also be detected in the first 0.5 µm of the nitrided layer. Presence of oxygen in the near-surface area of the 17-4 PH steel is nothing new. This steel in the un-nitrided condition adsorbs significant amount of oxygen on the surface and this is typical for all other types of stainless steels in the passive condition<sup>[5,6]</sup>. However, because oxygen is not accurately determined via GDS, its content at the surface has to be treated as of qualitative nature only. More accurate readings are at deeper locations. Oxygen concentration in the un-nitrided sample showed a similar trend. This data should also be treated as of the qualitative nature only.

What is interesting here is that the presence of oxygen in the steel in these experiments reaches more significant



**Fig. 4**—Young's modulus (green line), force penetration depth (blue) and nano hardness (purple) of the near-surface area of the 17-4 PH sample, ion nitrided at 513°C for 15 hrs. X scale represents distance from the surface in nm<sup>[12]</sup>.



**Fig. 5** —GDS analysis of the near-surface area of the 17-4 PH sample ion nitrided at 513°C for 15 hours, showing nitrogen, oxygen, and carbon.



**Fig. 6** — Total case depth in the 17-4 PH samples nitrided at various temperatures and various length of time in the two different views. Case determined "as etched with Marbles" depth. Coefficient of coordination  $r^2 = 0.993$ . The fit equation is:  $lnz = -1.4321+0.5251\cdot lnx-724691/y^2$ , (mm).

depth than in the naturally passivated steel, suggesting that this gas is still present in the chamber when the sample achieves final temperature. Therefore, nitrogen and oxygen as well as carbon and hydrogen diffuse into the steel together at the same time although maximum ultimate depth is reached only by nitrogen.

# **KINETICS**

3D graphs of case depth as a function of both time and temperature were created. Figure 6 illustrates the complex character of the relationship. However, when temperature is treated as constant, the relationship has a parabolic character. When time is treated as a constant, the relationship has an exponential character, as clearly seen in Fig. 6.

# **CONCLUSIONS**

These studies demonstrated how to overcome the main issues related to obtaining a nitride layer of a specific depth in 17-4 PH stainless steel using the plasma method. Knowing the identified relations will lead to higher reproducibility in future runs of nitride 17-4 PH components. This study also lays the groundwork to examine other processing parameters such as pressure and gas composition. To further understand the effectiveness of nitride 17-4 PH in field applications, studies into both corrosion resistance and wear could be investigated<sup>[11,13]</sup>. A study into how different alloys perform under time vs. temperature studies could lead to a better understanding of alloying elements and their effects on nitriding results. Processing has to be done in the vacuum system with exceptionally good leak rate and well controlled temperature of the treated objects. ~HTPro





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# **TECHNIQUES FOR DETERMINING RETAINED AUSTENITE**

Accurate measurement of retained austenite levels is important in the development and control of a heat treatment process.

# Thomas Wingens\*

Wingens LLC, Sewickley, Pennsylvania

Retained austenite is that fraction of austenite which remains untransformed at the end of the hardening process. Retained austenite is considered detrimental or undesirable in most cases but there are certain applications where some amount of retained austenite is considered as desirable.

Retained austenite strongly influences the properties of steel. The fatigue life, ductility, toughness, hardness, yield strength, and machinability all depend on austenite content. Accurate measurement of the volume percent of retained austenite is of critical importance to the optimization of heat treatment procedures. Austenite, being an unstable phase at room temperature, will transform to martensite during use, causing brittleness and an increase in volume potentially leading to failure of critical components.

However, the accurate measurement in manufactured steels remains a challenge as commonly used visual metallurgical sample investigations are subjective and mostly provide a very false reading, magnetic measurements need part specific calibration, and electron back scattering (EBSD) measurements require expensive equipment, intensive sample preparations, and long measurement times.

New developments in x-ray equipment provide measurements in minutes and can also compensate for the influences of carbides in high carbon steels or texture orientations in rolled sheet metals.

# **RETAINED AUSTENITE CREATION**

Hardening of steels requires heating to an austenitic phase and quenching to room temperature to produce a hard martensitic phase. Austenite is an FCC phase that is stable above a temperature of 727°C. Due to incomplete transformation during quenching some austenite is retained at room temperature. Retained austenite can dramatically decrease the mechanical properties of the steel. Properties such as fatigue strength, toughness, hardness, yield strength, and machinability can be influenced by retained austenite.

Austenite can transform in service as a result of thermal cycles, plastic deformation, or shock. Shot peening, for example, will transform the austenite on the surface of gear teeth. Exposure to extreme cold renders the austenite increasingly unstable as the temperature diminishes. The transformation of austenite to ferrite involves a nominal 4% volume increase. A linear dimensional increase on the order of the cube root of that would lead to seizure and excessive interference in precision gearing and bearings. Accurate measurement of the retained austenite levels is important in the development and control of a heat treatment process.

Austenite transforms to martensite between the  $M_s$  and  $M_f$  temperatures However, this transformation never goes to completion for carbon contents higher than 0.25 wt%, i.e., 100% martensite (Fig. 1).

The  $M_s$  and  $M_f$  temperatures are lowered by most alloying elements and an increasing austenitizing temperature but mostly by increasing the carbon content as seen in Fig. 2.

Higher austenitizing temperature brings more carbon and alloying elements into solution in austenite. Also, this increased temperature results in more thermal stresses on quenching, which oppose martensitic transformation, which is to say, both factors increase retained austenite.



**Fig. 1** — Martensite start ( $M_s$ ) and finish ( $M_f$ ) temperatures as a function of carbon content for plain carbon steels<sup>[1]</sup>.



**Fig. 2** — Retained austenite as a function of carbon content for plain carbon steels quenched to room temperature<sup>[1]</sup>.

The deformation of austenite above a temperature called  $M_d$  (higher than  $M_s$ ), lowers  $M_s$  temperature resulting in increase of retained austenite. This untransformed austenite is called retained austenite. For example, steels with carbon less than 0.25%, when quenched to room temperature have little retained austenite, because room temperature is below the  $M_r$  at such carbon contents in steels.

# WHY MEASURE RETAINED AUSTENITE?

Retained austenite is the result of an unfinished martensite transformation after quenching of high carbon steel after austenitizing. This results in:

**Lower hardness:** Austenite being a softer phase, if RA% is high, one will observe soft spots. The soft spots are detrimental in applications such as cutting tools where wear resistance is one of the most important factors affecting the service performance and life.

**Volume change:** The transformation of austenite to martensite is accompanied by increase in volume. Therefore, retained austenite, when it transforms to martensite during the service life of the component, would affect the dimensional stability of the component. This is detrimental in applications such as bearings and gauges where dimensional stability is extremely important.

**Premature failure:** The fraction of austenite retained (untransformed) at the end of the hardening process is likely to transform to martensite during subsequent tempering or when the hardened part undergoes stress and strain in service. This newly transformed martensite will remain untempered and cause brittleness, which can cause premature failure of the component in service. Due to this reason, retained austenite is detrimental in applications such as tools and dies, where high impact loading is an essential service condition.

HIPRO

**Increased fatigue resistance:** Finely dispersed retained austenite resists the propagation of fatigue cracks and improves rolling contact fatigue (RCF) stress. Therefore, some amount of retained austenite is considered beneficial in certain applications. One such application is bearings working with contaminated lubricants, such as railway bearings. In some cases, bearing components made of through hardening steels like SAE 52100 are carbonitrided because the carbonitriding process gives higher surface hardness and increases wear resistance and it also promotes retained austenite<sup>[2]</sup>.

# MEASURING RETAINED AUSTENITE

The volume fractions of phases in materials are typically evaluated by optical microscopy, magnetic analysis, and x-ray diffractometry. Among these methods, the x-ray diffraction method is one of the most efficient means<sup>[3]</sup>.

The accuracy of the techniques used to identify and measure austenite in steel decreases significantly with decreasing amounts of austenite. As RA fractions become small, the morphology makes measurement difficult, for instance, thin RA films between martensite plates or laths<sup>[4]</sup>. The most common measurement techniques are: visually with a microscope, magnetic (martensite is ferro magnetic and austenite is paramagnetic), electron back-scatter diffraction (EBSD), and x-ray diffraction (XRD) measuring different lattice structure of the martensite (BCT) and the retained austenite (FCC).

Microscope. Measuring retained austenite with a light microscope is performed on a metallographic prepared (cut, ground, high polished) and etched microsample typically at 500x magnification. The proper segmentation of the sample out of the part or gear is very crucial to receive a representative quantity of the measured area. The mechanical force and heat introduction during segmentation must be minimized to avoid the transformation of austenite. The removal of surface layer during grinding and polishing of the microsample may also change the evaluation plane. The right etchant must be selected for the material (3 to 5% Nitric acid in alcohol is commonly used) and the etching time and intensity can vary the contrast of the structure, in a way, that untempered (tetragonal) martensite plates cannot be differentiated from the residual unetched retained austenite.

Figure 3 shows plate martensite next to retained austenite in a 1.31% C, 0.18% Si, 0.2% Mn steel after 950°C austenitizing and water quenching. In the as-quenched condition, the martensite is light and the retained austen-



Fig. 3 — Light untempered (left) and tempered (right) plate; martensite next to retained austenite $^{[5]}$ .



Fig. 4 – Schematic of the magneto-inductive measuring method<sup>(6)</sup>.

ite darker as shown in Fig. 3a. After additional tempering at 150°C, the martensite appears darker than the retained austenite due to the carbide precipitation<sup>[5]</sup>.

**Magnetic.** As illustrated in Fig. 4, the sample is magnetized to saturation and the saturation polarization is measured. The difference between measured and theoretical saturation, the retained austenite content, can be calculated:  $V_{RA}$  = 100Vol.% – $V_{M}$ %.

**Electron backscatter diffraction (EBSD).** EBSD is a destructive technique that measures small volumes. The sample is (monochromatic) scanned in a scanning elec-

tron microscope and moved. The results are somewhat similar to XRD with much higher efforts on preparation, operator, and equipment. An example of retained austenite detected by EBSD is shown in Fig. 5.

**X-ray diffraction (XRD),** illustrated in Fig. 6, is considered the most accurate method of determining the amount of retained austenite in steels. It is a nondestructive analytical technique used to identify and quantify phases in a material. Every crystalline phase produces a characteristic diffraction pattern (e.g., fingerprint).

The volume fraction can be determined by the x-ray diffraction since x-ray diffraction intensities are directly proportional to the volume of the phase considered.

The most severe shortcoming with this method is the problem of calibration due to orientation, because all materials have a preferred orientation



Fig. 5 — EBSD maps showing the effects of strain on retained austenite (a) 0%/6.3% RA; (b) 5%/6.1%RA; (c) 10%/5.4%RA; (d) 20% 3.9% RA<sup>[6]</sup>; and (e) pipe 4.8% RA.

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HIPRO

to some extent. The problem of quantitative phase analysis in textured material is one of obtaining the integrated intensity averaged over all orientations of the specimen with respect to the x-ray beam. Methods of averaging involve randomizing the intensities by mathematical or mechanical means. Several methods of averaging the intensities have been proposed for the quantitative phase analysis of textured materials<sup>[3]</sup>.

Any electromagnetic radiation interacts with the material through either absorption and energizing the material system and expelling neutrons from atoms; or diffusion, during which the radiation is diffused by the matter and the electromagnetic waves associated with it change direction of propagation. This change can be accompanied by energy exchanges between photons and matter.

The technique of x-ray diffraction is based on coher-



Fig. 6 — Scattering schematics.



Fig. 7 — Schematics and rotating the sample while measuring with x-ray.

ent elastic scattering: the macroscopic phenomenon of diffraction arises from the coherent sum of all the electromagnetic waves diffused by the atoms found along the same family of reticular planes. To manifest itself, it necessarily requires the presence of a reticular order, as found in crystals or in crystalline materials.

The incoming beam (upper left in Fig. 7) scatters, re-radiating a small portion of its intensity as spherical wave. If this happens symmetrically at a discrete distance *d*, the waves are in synchrony (constructive mode) only in the direction where their path-length difference  $2d\sin\theta$  is equal to an integer multiple of the radiation wavelength  $\lambda$ . This creates a diffracted beam at an angle measuring 20 producing a figure called diffraction pattern that can be collected and represented as follows in function of the detecting method.

In addition to phase analysis, x-ray diffraction can also be used to analyze microstructural features such as texture, residual stress, and grain size. Texture produces systematic deviations of peak intensity from the characteristic diffraction pattern of a phase. The intensity deviation can be used to quantify the fraction of grains in a certain orientation by tilting and rotating the sample in the diffractometer as shown in Fig. 7.

With the x-ray measurement, the diffraction plane is crucial: only the grain in the lattice  $n_{hkl}$  are registered. Therefore, the sample has to be turned and tilted and is measuring the intensity at multiple angle positions.

# **CARBIDE CORRECTION**

Carbide presence can have an influence on the determination of RA%Vol. The influence is given by the presence of overlapped or adjacent carbide peaks that can interfere and alter the intensity of the ferrite or austenite peaks. Standard algorithms based on the evaluation of the region of interest, can lead to an erratic evaluation of the RA%Vol since it can be totally or partially considered in the peak integration.

Using the full profile approach, it is possible to consider the influence of this peak and do not consider it, as well as all the other peaks of carbides that can interfere.

# **ORIENTATION**

Measurements of retained austenite using x-ray diffraction are often employed despite the caveat that these methods only apply to uniform (random) texture distributions. Due to the strong crystallographic texture caused by deformation during processing, these assumptions

are typically not valid for rolled sheet steel (such as transformation induced plasticity "TRIP" steels). There is data to indicate that the transformation will not be distributed evenly as a function of orientation, but particular stress states will cause some orientations to transform at a higher rate than other orientations. A technique using complete pole figure averaging using neutron diffraction was developed to measure the retained austenite in textured TRIP steels as well as provide an estimate on the uncertainty in the phase fraction<sup>[7]</sup>.

# **CONCLUSIONS**

Retained austenite plays a significant role in the performance of heat-treated steel components. Data indicates even very small amounts of RA may be influential. The ideal RA depends on the alloy and its application. As a result, appropriate measurement techniques and process controls need to be implemented to deliver targeted performance.

Light microscopy provides qualitative assertion with a subjective quantitative assumption, however, retained austenite contents below 15% are hard to determine. Magnet inductive measurement is a volume measurement and needs to be calibrated to specific materials, heat treat, and geometries. The obtained results are usually higher than with XRD, which has no ability to do profile measurements. Results from EBSD measurement are somewhat similar to XRD with much higher requirements for preparation, operator, and equipment.

XRD measurement is unproblematic with untextured (isotropic) homogeneous steels. The presence of additional phases and reflections due to grain size, carbides, or texture can cause disturbances and variances. However, the newest generation XRD machines can compensate for these obstacles and provide precise, repeatable, and fast measurements at a very competitive cost.

### Acknowledgments

The author would like to thank the companies Vericheck LLC, Bethel Park, Pa., and GNR Analytical instruments, Italy, for the support of this work. **Note:** This is an excerpt from the HTS Prime Industry Contributor award winning article, Retained Austenite Benefits or Avoidance Requires Dependable Determination, which can be accessed here https://doi.org/10.31399/asm. cp.ht2021p0212. ~**HTPro** 

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ASMNEWS



# ASM Announces 2022 Award Program Honorees

he ASM International Board of Trustees has named award program honorees for 2022. The awards program recognizes achievements of members of the materials science and engineering community. Awards will be presented at ASM's Awards Dinner on Tuesday, September 13, during IMAT 2022 in New Orleans. Tickets for the Awards Dinner can be ordered by using the IMAT registration form. We also look forward to recognizing the 2020 and 2021 Award Recipients who will be in attendance. Those interested in purchasing a table may contact Christine Hoover, administrator, Awards Program, ASM International, Materials Park, Ohio 44073-0002; 440.338.5444; or email christine. hoover@asminternational.org.

### **Honorary Membership**



**Prof. Christopher C. Berndt, FASM, TSS-HoF,** professor, Swinburne University of Technology, Australia, will receive this year's award "for demonstrated professional commitment and lifelong learning, as well as being a mentor and exemplar, to early career engineers and scientists." Honorary Membership in the Society was established in 1919 to recognize distinguished service to the materials science

Berndt

and engineering profession, service in areas of ASM strategic plan initiatives, and the progress of mankind.

# Gold Medal



# Prof. Glenn S. Daehn, FASM, pro-

fessor and Mars G. Fontana Chair, The Ohio State University, Columbus, will receive this year's award "for creative and impactful development and dissemination of materials science and technology, including impulse welding, joining and forming, and deployment of materials science in numerous K-12 classrooms." The medal was estab-

Daehn

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lished in 1943 to recognize outstanding knowledge and great versatility in the application of science to the field of materials science and engineering, as well as exceptional ability in the diagnosis and solution of diversified materials problems.

# **Engineering Materials Achievement Award**

Dr. Dawn R. White, senior staff scientist, Oak Ridge National Laboratory, Tenn.; Mr. Matt Short, director of engineering and business development, ToolTex Inc., Grove City, Ohio; Mr. Mark Norfolk, president, Fabrisonic LLC, Columbus, Ohio; and Dr. Karl F. Graff,





Short



EWI (retired), professor emeritus, The Ohio State University, Columbus, will receive this year's award for the "development and commercialization of the ultrasonic additive manufacturing process."

Norfolk

White

Established in 1969, this award recognizes an outstanding achievement in materials or materials systems relating to the application of knowledge of materials to an engineering structure or to the design and manufacture of a product.

Graff

# Albert Sauveur Achievement Award

**Dr. Mark F. Horstemeyer, FASM,** dean, School of Engineering, Liberty University, Lynchburg, Va., will receive this year's award "for being a pioneer in Integrated Computational Materials Engineering (ICME) where he has contributed in multiscale materials modeling and simu-

# HIGHLIGHTS 2022 AWARD PROGRAM HONOREES



lating the process-structure-propertyperformance sequence in creating optimized structural components." Established in 1934 in honor of a distinguished teacher, metallographer, and metallurgist, the award recognizes pioneering materials science and engineering achievements that have stimulated organized work along similar lines to such an extent that a marked

Horstemeyer

basic advance was made in the knowledge of materials science and engineering.

### William Hunt Eisenman Award



**Dr. Valery Rudnev, FASM,** director, science and technology, Inductoheat Inc., Madison Heights, Mich., will receive this year's award "for dedicated service to the global materials science community, leadership, development and promotion of induction heating and heat treating technologies and novel technologies." The award was established in 1960, in memory of a

Rudnev

founding member of ASM, and its first and only secretary for 40 years. It recognizes unusual achievements in industry in the practical application of materials science and engineering through production or engineering use.

### J. Willard Gibbs Phase Equilibria Award



Prof. Kallarackel T. Jacob, pro-

fessor, department of materials engineering, Indian Institute of Science, Bangalore, India, will receive this year's award "for outstanding leadership in research and education in the area of thermodynamics and phase equilibria of metal and ceramic systems, encompassing innovation in experimental techniques and applications to materials processing." The award honors J.

Jacob

Willard Gibbs, one of America's greatest theoretical scientists. In addition to many other contributions, Gibbs laid the thermodynamic foundations of phase equilibria theory with his brilliant essay "On the Equilibrium of Heterogeneous Substances," published in 1876 and 1878 in the *Transactions* of the Connecticut Academy.

### Allan Ray Putnam Service Award

**Dr. Guiru Nash Liu, FASM,** senior experimental metallurgist, Progress Rail Inc., LaGrange, Ill., will receive this year's award "for exemplary and continuous volunteerism, stewardship, and contributions to the Chicago Regional Chapter, ASM International, and Material Advantage."



Established in 1988, the award recognizes the exemplary efforts of various outstanding members of ASM International on behalf of the Society to further its objectives and goals. The purpose of this award is to recognize those individuals whose contributions have been especially noteworthy and to whom the Society owes a particularly great debt of appreciation.

Liu

# Albert Easton White Distinguished Teacher Award



**Prof. John J. Lewandowski, FASM,** Arthur P. Armington Professor of Engineering II, department of materials science and engineering, Case Western Reserve University, Cleveland, will receive this year's award "for outstanding contributions to teaching and research in materials science and engineering. Under his tutelage, his students have performed world-class research in the mechanical behavior

Lewandowski

of intermetallics, metallic glasses, and high-performance structural materials." The award was established in 1960 in memory of an outstanding teacher and research engineer, who was a founding member and president of ASM in 1921. It recognizes unusually long and devoted service in teaching, as well as significant accomplishments in materials science and engineering and an unusual ability to inspire and impart enthusiasm to students.

### Silver Medal Award



Astarita



Flury

**Dr. Antonello Astarita,** associate professor, University of Naples, Italy, will receive this year's award "for consistent dedication to the science of manufacturing and for his contribution in the sharing of knowledge worldwide and in the mentoring of future generations of engineers."

**Ms. Margaret Bush Flury,** principal materials engineer, failure analysis, Medtronic, Fridley, Minn., will receive this year's award "for passion for failure analysis, and using it to improve medical devices, patient lives, and to help grow others in the failure analysis and materials science communities during her 21-year ASM membership."

# 2022 AWARD PROGRAM HONOREES HIGHLIGHTS

Established in 2010, the honor of Silver Medal of the Society recognizes members who are in mid-career positions (typically 5 to 15 years of experience), for distinguished contributions in the field of materials science and engineering, and the Society. The purpose of this award is to recognize leadership at an early stage and encourage individuals to grow, nurture, and further contribute to the growth of the profession, as well as the Society.

### **Bronze Medal Award**



**Dr. Abdallah Elsayed,** assistant professor, School of Engineering, University of Guelph, Ontario, Canada, will receive this year's award "for technical contributions to light metals development in transport and related fields as well as engaging students, professionals and retirees through ASM service activities."

Elsayed



**Dr. Sumedh Gostu,** senior research scientist, Air Liquide, Newark, Del., will receive this year's award "for groundbreaking and novel contributions in the chemical processing and extractive metallurgy of primary raw materials and secondary metallurgical waste materials and enduring contributions to the ASM Brandywine Valley Chapter and the International Hybrid Chapter Task Force; devising network-

Gostu

ing strategies to increase emerging professional involvement with ASM."

Established in 2014, the honor recognizes ASM members who are in early-career positions, typically 0 to 10 years of experience, for significant contributions in the field of materials science and engineering through technical content and service to ASM and the materials science profession.

# Bradley Stoughton Award for Young Teachers



**Dr. Ashwin Shahani,** assistant professor, materials science and engineering, University of Michigan, Ann Arbor, will receive this year's award "for creativity and extraordinary effort in teaching undergraduate and graduate students, for providing innovative programming in materials science and engineering to underrepresented

youth." This award, accompanied by

Shahani

\$3000, was established in 1952 in memory of an outstanding teacher in metallurgy and dean of engineering who was pres-

ident of ASM in 1942. The award recognizes young teachers of materials science, materials engineering, and design and processing, by rewarding them for their ability to impart knowledge and enthusiasm to students. The recipient must be 35 years of age or younger by May 15 of the year in which the award is made.

### Henry Marion Howe Medal

Xiaoyan Wang, Yuanfei Han, Xin Su, Guangfa Huang, and Weijie Lu, will receive this year's award for their paper entitled "The Formation of {10-12} Deformation Twin in Hybrid TiB-TiC Reinforced Titanium Matrix Composites," published in *Met. Trans. A*, Vol. 52, Issue 1. The award was established in 1923 in memory of a distinguished teacher, metallurgist, and consultant, to honor the author(s) whose paper was selected as the best of those published in a specific volume of *Metallurgical and Materials Transactions*.







Wang

Su



Han

# ASM Historical Landmark Designation

**Ford Rouge Complex,** Dearborn, Mich., has been selected as a 2022 Historical Landmark, and the citation reads, "For being the most successful full-scale vertically integrated manufacturing facility for almost a century, that has transformed raw materials that enable sustainable manufacturing and assembly processes to yield high-volume, technologically complex vehicles for the masses."



# HIGHLIGHTS 2022 CLASS OF FELLOWS

National Research Council of Canada, Sussex Drive Laboratories, Ontario, has been selected as a 2022 Historical Landmark, and the citation reads, "Created in 1930, this site is known as the birthplace of Canada's scientific activities from foundational research in physics and chemistry to groundbreaking innovations in materials science, engineering, and beyond."



In 1969, the ASM Historical Landmarks Designation was established to permanently identify the many sites and events that have played a prominent part in the discovery, development, and growth of metals and metalworking. In 1987, the scope of this award broadened to include all engineered materials.

# **ASM Student Paper Contest**



**Mr. Bernoulli Andilab,** Ph.D. student, Ryerson University, Toronto, is recognized for his paper entitled "Characterization of a Cast AI-Cu Alloy for Automotive Cylinder Head Applications" published in the *J. Mat. Eng. Perform.*, February 2022.

The contest was established in

1985 as a mechanism for student par-

Andilab

Anomal ticipation in Society affairs. The award recognizes the best technical paper with a graduate or undergraduate student as first author that is published in an ASM-sponsored publication during the year.

# Emerging Professionals Achievement Award



**Dr. Caitlin E. Walde,** principal materials engineer, Solvus Global, Ashland, Mass., will receive this year's award "for her leadership in WPI's Material Advantage Chapter to her current role as chair of the Boston Chapter, Caitlin continues to champion ASM and materials science by fostering student/ industry partnerships." Established in 2010, the award recognizes and honors extraordinary ASM volunteers who are less senior individuals, i.e., 0-5 years of experience post-graduation, who have made a significant impact on ASM International through devoted service and dedication to the future of the Society.

# Canada Council M. Brian Ives Lecturer



**Dr. Frank Cheng,** professor, University of Calgary, Alberta, Canada, will be this year's lecturer. The Canada Council of ASM International annually selects an individual who has made distinguished and significant contributions to the Canadian materials community and designates that person as the M. Brian Ives Lecturer. This individual makes a technical presentation to

Cheng

each of the eight Canadian Chapters of ASM International and serves as a de facto ambassador for both ASM International and ASM Canada Council.

# ASM ANNOUNCES 2022 CLASS OF FELLOWS

In 1969, ASM established the Fellow of the Society honor to provide recognition to members for their distinguished contributions to materials science and engineering and to develop a broadly based forum of technical and professional leaders to serve as advisors to the society. ASM will recognize the 2022 Class of Fellows as well as the Class of 2021 and 2020 at the Fellows Induction Ceremony during IMAT 2022 on Monday, September 12, in New Orleans. Tickets can be purchased with IMAT registration.

Following are the members recognized by their colleagues for 2022. Additional Fellows may be elected to this distinguished body in subsequent years. The solicited guidance, which the Fellows will provide, will enhance the capability of ASM as a technical community of materials science and engineering in the years ahead.



### Dr. Othmane Benafan, FASM

Materials Research Engineer National Aeronautics and Space Administration (NASA) Cleveland

For exceptional scientific contributions to the field of shape memory alloys research and technology.

Walde
# 2022 CLASS OF FELLOWS HIGHLIGHTS



**Prof. Nick Birbilis, FASM** Professor

College of Engineering and Computer Science

The Australian National University Acton

For the development of new corrosion-resistant magnesium alloys and fundamental studies on the micro-

structure-processing-property relationships in lightweight alloys.



Dr. Carelyn E. Campbell, FASM

Supervisory Metallurgist National Institute of Standards and Technology Gaithersburg, Md.

For significant contributions, leadership, and education in data driven computational materials science and engineering design.



Dr. Qing Chen, FASM

Chief Scientific Officer Thermo-Calc Software AB Solna, Sweden For the development of key CALPHAD modeling methods, and their implementation into commercial software modules and databases.



Dr. Ryan Evans, FASM

Director-Research and Development The Timken Company North Canton, Ohio

For significant contributions in the development and commercialization of thin-film coatings and surface-engineering solutions to improve the performance of tribological contacts.



**Prof. Nikhil Gupta, FASM** Professor Mechanical and Aerospace Engineering New York University Brooklyn, N.Y.

For pioneering contributions to the science and technology of lightweight polymer and metal matrix composites,

and exceptional dedication to the education of the public for scientific discoveries.



# Prof. Rainer J. Hebert, FASM

Professor Department of Materials Science and Engineering University of Connecticut Storrs

For contributions to additive manufacturing involving metal powder behavior and melt pool solidification, and for

developing advanced alloys for applications in aerospace and undersea vehicle technologies.



Prof. Bertrand Jodoin, FASM

Professor Department of Materials Science and Engineering University of Ottawa Canada

For significant contributions to the understanding and development of the cold spray process and cold spray additive manufacturing.



# Prof. Hideyuki Kanematsu, FASM

Specially Appointed Professor, FIMF National Institute of Technology (KOSEN) Suzuka College Japan

For outstanding contributions to the field of elucidation of corrosion/degradation behaviors and surface characteristics for a variety of materials.



### Prof. Jian Luo, FASM

Professor Department of NanoEngineering University of California, San Diego

La Jolla

For pioneering work in developing grain boundary phase diagrams; uncovering the mechanisms of grain boundary embrittlement, activated

sintering, and ultra-fast sintering, and advancing high-entropy materials.

# HIGHLIGHTS 2022 CLASS OF FELLOWS



Mr. Paul K. Mason, FASM President Thermo-Calc Software Inc. McMurrav. Pa. For outstanding contributions to the

development, integration, and support of broad-based computational tools and data infrastructure, and for exceptional leadership in the use of

these tools to advance the field of materials science and engineering.



# Prof. Douglas M. Matson, FASM Professor

Mechanical Engineering Tufts University Medford, Mass.

For significant contributions in microgravity rapid solidification and thermophysical property measurement and for mentorship of students to

develop sustainable engineering projects for disadvantaged communities.



### Prof. Todd A. Palmer, FASM

Professor

Department of Engineering Science and **Mechanics** 

Pennsylvania State University University Park

For significant contributions to the advancement of processingmicrostructure-property correlations through impactful theoretical and

experimental investigations of high-energy beam processing of important alloys.



### Mr. Luc Pouliot, FASM

Chief Operating Officer & Chief Technology Officer/Co-Owner Polycontrols Technologies Inc. Quebec, Canada

For the development and worldwide deployment of industrial online process control sensors yielding disruptive technology changes that contributed significantly to broadening the use of

thermal spray in different industrial sectors.



# **Prof. Timothy J. Rupert, FASM**

Professor Department of Materials Science and Engineering

University of California, Irvine

For significant contributions in the understanding of structure-property relations of nanocrystalline metals and alloys, addressing the limitations of the material class, and enabling the wide-

spread usage of these advanced materials.



### Dr. Adrian S. Sabau, FASM

Senior Research Staff Member Oak Ridge National Laboratory Computational Sciences & Engineering Division Tenn.

For unique applications of computational fluid dynamics and advanced modeling to materials science and engineering, specifically related to sur-

face treatments, solidification, and other key areas in processing and performance.



# Dr. Narasi Sridhar, FASM

Chief Executive Officer MC Consult LLC Temecula, Calif.

For sustained contributions to the fundamental understanding of localized corrosion, the development of corrosion resistant alloys, and the mitigation of risk associated with corrosive systems.



# Dr. Franco Stellari, FASM

Yorktown Heights, N.Y.

For sustained and outstanding contributions to the development and application of novel techniques to the testing, fault diagnosis, and failure analysis of semiconductor devices.

# NOMINATIONS SOUGHT **HIGHLIGHTS**



**Dr. Michael D. Uchic, FASM** Principal Materials Research Engineer Air Force Research Laboratory Wright-Patterson AFB, Ohio For groundbreaking experimental

research on micro deformation and 3D characterization of materials combined with visionary contributions to understanding of microscale metal plasticity.



Prof. Jian Wang, FASM Professor Department of Mechanical and Materials Engineering University of Nebraska-Lincoln For significant contributions in the fundamental understanding of defects-microstructures-properties relations of metals and alloys, especially for the interface strengthening and deforma-

**Dr. Julio C. Villafuerte, FASM** Corporate Technology Strategist Centerline Windsor Ltd. Ontario, Canada

For the development and successful commercialization of low-pressure cold spray technology and for leadership in R&D for developing application solutions for industry.



Dr. Cyril L. Williams, FASM Senior Research Engineer U.S. Army Research Laboratory Aberdeen Proving Ground, Md. For distinguished contributions to the

development of structure-property relationships and the understanding of dynamic deformation under extreme conditions and spall failure of single

crystals, polycrystalline, nanocrystalline, and fine-grained alloys.

tion twinning utilizing atomistic simulation, crystallographic

analysis and modeling, and experimental characterization.

# Nominations Sought for 2023 ASM/TMS Distinguished Lectureship in Materials & Society

Nominations are currently being taken for the ASM/ TMS Distinguished Lectureship in Materials & Society. The lecture was established in 1971 and is jointly sponsored by The Minerals, Metals & Materials Society (TMS) and ASM International. The topic of the lecture shall fall within these objectives:

- To clarify the role of materials science and engineering in technology and in society in its broadest sense.
- To present an evaluation of progress made in developing new technology for the ever-changing needs of technology and society.
- To define new frontiers for materials science and engineering.

Qualifications of the lecturer include:

- A person experienced in national or industrial policymaking in the field of materials science and engineering.
- An eminent individual who has an overview of technology and society in which technology and society are affected by development in materials science and engineering.

• A person associated with government, industry, research, or education.

Nominations may be proposed by any member of either Society. **Submit your nominations by September 1 for consideration.** Recommendations should be submitted to the headquarters of either Society.

View sample forms, rules, and past recipients at asminternational.org/membership/awards/nominate. To nominate someone for any of these awards, contact christine. hoover@asminternational.org for a unique nomination link. You may also contact Deborah Hixon at TMS Headquarters, hixon@tms.org.

# Official ASM Annual Society Meeting Notice

The Annual Society Meeting of members of ASM International will be held on:

# Monday, September 12 - 4:00 - 5:00 p.m.

The purpose of the ASM Annual Society Meeting is the election of officers for the 2022-2023 term and transaction of other Society business.

# HIGHLIGHTS FROM THE PRESIDENT'S DESK

# FROM THE PRESIDENT'S DESK

# Aligning ASM's

# Constitution, Governance, Board Policies, Strategy and Annual Operating Plan for the Next Decade of Opportunity



At the annual meeting on September 13, 2021, the ASM membership approved the Constitution and Bylaws of ASM International, as amended and



restated on April 22, 2021. Rules for Government of ASM International as amended and restated by the Board of Trustees were also approved on September 13, 2021. These documents bring the 2004 revision of the ASM Constitution into compliance with the current State of Ohio regulations and ensure internal consistency of requirements, and completeness of definitions and statements for critical governance issues.

Our year-long, deep dive into governance issues highlighted the critical need to review ASM's board policies to distinguish policies (determined by the board) from procedures (led by the staff) and to align both with ASM's current practice, strategic plans, and the annual operating plan. Consequently, we have embarked on an ambitious plan, in partnership with ASM Executive Director Sandy Robert, the ASM staff, and volunteers from ASM's Committees, Affiliate Societies, chapters, and the membership, to review 76 documents and propose a revised set of policies with their accompanying procedures for approval at the board meeting during IMAT, or soon thereafter.

To date, ASM's 16 classes of policy areas have already been screened for redundancies, duplications, consolidations, revisions, updates, and documents to be sunset. In the next couple of months, teams of board members, staff, and volunteers will iterate proposed revisions for their respective areas and report back to the entire group with their recommendations. The streamlined set of policies with accompanying procedures will then be reviewed by general counsel and proposed modifications will be incorporated, as appropriate. The compiled documents will then be presented to the board for final discussion and approval. From this process, we aim to align our Constitution, Rules for Government, and board policies with the prioritized actions in the ASM Strategic Plan and the Annual Operating Plan and budget led by Ms. Robert and her staff. Our intent is to bring efficiencies into ASM operations, make strategic investments in both immediate and long-term revenue generating activities for our Society, advance our members, and empower our staff leadership as they develop their areas of expertise in collaboration with the board and ASM volunteers. I wish to thank the entire team for the time they are investing in bringing ASM to leadership preeminence in the global materials community.

Meanwhile, it will soon be time for us all to meet in person at IMAT 2022 in New Orleans, for the first time in two years. We have many exciting programs, activities, and celebrations planned. I look forward to seeing you all at our reunion!

ASM President, Judith A. Todd, FASM judith.todd@asminternational.org

# EXECUTIVE DIRECTOR CORNER

# The Role of Governance and Strategic Planning in Designing our Future

In my last column, I shared with you how I am developing a deeper understanding of our organization through meetings, travel, and financial review. Building on this initial orientation, I have familiarized myself with key activities in our governance and strategic planning cycle. If you haven't volunteered with ASM International or other professional societies, the notion



Robert

of governance and strategic planning may be unfamiliar concepts. To those of us who serve as chief staff executives of associations, they are familiar and essential tools for collaborating with volunteer leadership to design our future. First, let's consider governance. Associations are mission-driven organizations, generally governed by a volunteer board. At ASM International, our Board of Trustees exercises this responsibility, providing oversight for the organization according to our Constitution and Bylaws, and Rules for Government. The officers and board work closely with the executive director to set strategic direction, to provide required resources, and to make key decisions that staff operationalize to meet the needs of our members and partners.

Many of my duties as executive director relate to supporting ASM International's governance activities, and particularly those relating to the work of the board. Since late spring, I have developed a clearer understanding of our

# EXECUTIVE DIRECTOR CORNER **HIGHLIGHTS**

nominating process, awards, and board policy structure. As president, Judith Todd and the other officers and I met to discuss ASM International's board policies, and we agreed that the sheer scope of our policy manual merited a deliberate review, with an eye to streamlining the document. Judith provides an excellent overview of our deliberations and process in her column for this issue. While board and staff members are investing tremendous time into board policy this year, we jointly believe that it will provide clearer definition of the leadership, fiduciary, legal, and ethical requirements required to ensure successful organizational oversight.

Strategic planning is another key responsibility of the board, which we undertake annually at ASM International, led this year by Vice President David Williams. David framed this year's strategy work as bringing a deeper, more precise definition of our strategic initiatives in the current 2020-2025 strategic plan. Our process this year focused on leveraging the expertise of a wide range of our volunteer leaders through a leadership questionnaire, development of an external market outlook brief by our chief sales and marketing officer Ryan Milosh, and a series of interviews. In this way, we connected the internal expertise of about 70 key volunteers to external industry trends affecting materials science and manufacturing, to decide on key priorities for the organization over the coming year. Facilitator David Gammel of McKinley Advisors guided the board through the full-day session, which we will be refining through the fall with the board to shape next year's operating plan.



ASM President Judith Todd greets staff member Ray Fryan at the Dome on June 28.

Another step in our annual governance calendar will take place at IMAT 2022, September 12-15 in New Orleans, during our Annual Society Meeting (Monday, September 12, 4:00-5:00 p.m.) to elect officers for the 2022-2023 term, and to discuss Society business. What a welcome change it will be to greet you in person and share our ongoing progress in creating a more resilient organization that builds on over 100 years of research and discovery!

Wishing you an enjoyable summer and sending all best wishes from the Dome.

Sandy Robert, CAE Executive Director, ASM International sandy.robert@asminternational.org



Navin Manjooran, Amber Black, David Scannapieco, Sandy Robert, and John Kuli with results of their brainstorming session.



Nicole Hudak, André McDonald, David Williams, Elizabeth Hoffman, and Ryan Milosh in a strategic planning breakout session.



# International Metallographic Contest at IMAT

### Deadline: September 2

The International Metallographic Contest (IMC), an annual contest cosponsored by the International Metallographic Society (IMS) and ASM International to advance the science of microstructural analysis, will be held at IMAT 2022 in New Orleans, September 12-15. Six different classes of competition—including a new video class—cover all fields of optical and electron microscopy:

Class 1: Light Microscopy—All Materials

Class 2: Electron Microscopy—All Materials

**Class 3:** Student Entries—All Materials (Undergraduate Students Only)

Class 4: Artistic Microscopy (Color)—All Materials Class 5: Artistic Microscopy (Black & White)—All Materials

**Class 6:** Video Entry—Topic of Choice involving defined problem (Undergraduate Students Only)

Best-In-Show receives the most prestigious award available in the field of metallography, the Jacquet-Lucas Award, which includes a cash prize of \$3000. For a complete description of the rules, tips for creating a winning entry, and judging guidelines, visit asminternational.org/web/ims/ membership/imc or contact IMC chair, Ellen Rabenberg, at ellen.m.rabenberg@nasa.gov.

# FROM THE FOUNDATION

### Thankful for Volunteers

This has been an exciting year as we finally returned to in-person programming for the first time in three years! Our volunteers worked tirelessly to help meet the challenge of organizing teacher and student camps across the country and making our programs a success. We couldn't be more thankful for their continuous dedication to our organization and mission.



Wilson

As executive director of the ASM Materials Education Foundation Board of Trustees, one of my many roles is talking with ASM members about the ASM Materials Education Foundation's mission and work. Through these conversations, I have found many ASM members are surprised to learn that the Foundation is a separate entity from ASM International. The ASM Foundation has its own budget, board of trustees, and bylaws and is a separate 501(c)(3) non-profit organization. But that doesn't mean we do it all on our own.

The Foundation counts on the support of you, our ASM members, to increase outreach efforts and continue to provide best-in-class resources. We also need your help to further the mission of the ASM Materials Education Foundation. We ask you to join us as we fulfill our mission today.

The ASM Foundation's priorities include increasing the number of students entering the materials science and engineering field and in STEM fields in general. These priorities can be achieved through increasing the number of participants in our ASM Materials Camp for teachers and students and expanding our outreach efforts to help individuals learn more about materials science and engineering. Your support is needed to help carry out these goals. It takes all of us working together to provide essential resources for the next generation of professionals in the field. As we adjust to our new normal, now is the time to accelerate the growth of these proven programs and inspire thousands more young people to explore materials science and consider engineering and technical fields.

On behalf of the staff and board of the ASM Materials Education Foundation, we look forward to the accomplishments we will achieve together through this partnership. As ASM members, you understand the urgent need to increase the number of students entering materials science and the excitement of teaching students through the ASM Materials Camp for students. Working with teachers through our ASM Materials Camp for teachers allows our Master Teachers to share hands-on, minds-on experiments and activities that can reach exponentially more students to engage their interest in STEM, as well as entering the materials science and engineering field.

The partnership between ASM members and the ASM Materials Education Foundation is critical to the Foundation's success. Thank you for all your work to make the 2022 programs happen. We are grateful for your continued support.

Carrie Wilson Executive Director ASM Materials Education Foundation

# EMERGING PROFESSIONALS HIGHLIGHTS

# EMERGING PROFESSIONALS

### **EPC Symposium at IMAT**

After years of inability to meet in person due to the global pandemic, ASM is hosting the International Materials, Applications & Technologies (IMAT) conference and exhibition. This year, IMAT is co-located with the Thermal Spray and Surface Engineering Forum and Expo and will be held in New Orleans, September 12-15. IMAT 2022 will feature technical sessions organized by the ASM Programming Committees, AeroMat Committee, Inclusion, Diversity, Equity, and Awareness Committee, Emerging Professionals Committee (EPC), as well as other ASM affiliated societies in addition to informational courses, keynotes, networking events, and an exhibit floor.

The EPC symposium for IMAT 2022 titled "Perspectives for Emerging Materials Professionals" consists of six invited speakers at different points in their careers from academia, industry, and national laboratories. The speakers are asked to share their life stories about navigating the world of materials science and engineering and provide insight to the emerging professionals regarding their own career paths. The symposium ends with a panel discussion where the speakers collectively answer questions from the audience.

For the emerging professional, IMAT 2022 will provide the opportunity to keep up to date with the field of materials science and engineering in both the technical aspects and networking. It is a great chance to get introduced to new topics, learn more about what your peers are working on, and meet people who will become future collaborators, mentors, and friends. For students, IMAT 2022 is a glimpse into the materials world beyond the degree. Talking to conference attendees from multiple universities, companies, and institutions offers key insights into the bright future that materials science professions can hold. For everyone else, IMAT 2022 is a wonderful occasion to remain current and connected to colleagues who they haven't been able to see in person for years.

To stay up to date on the latest news about IMAT 2022, visit the IMAT website (https://www.asminternational.org/ web/imat-2022/home) or follow ASM International on social media.

If interested in the EPC or helping with future emerging professional programming or projects, contact Drew Fleming at drew.fleming@asminternational.org.

# VOLUNTEERISM COMMITTEE

# Profile of a Volunteer

Bernoulli Andilab, Ph.D. student in mechanical engineering, materials, metallurgy, and metal casting, Ryerson University, Toronto

Andilab

ing background, Bernoulli Andilab grew up wanting to learn how things work. As an undergraduate student in mechanical engineering at Ryerson University, he landed a

Inspired by his dad's engineer-

job as a research assistant in the Centre for Near-net-shape Processing of Materials lab and got hooked on metal casting research. He went on to pursue his master's and is now doing Ph.D. research on developing an aluminum alloy with optimal strength and thermal properties for automotive and aerospace applications.

Andilab first heard about ASM from his research supervisor, Dr. Comondore (Ravi) Ravindran, FASM, a longtime member and volunteer. Andilab became a student board member of the affiliate International Metallographic Society (IMS), and together with friends helped start the school's first Material Advantage student chapter. He serves on the membership subcommittee, setting up guest speakers to talk about their fields and careers.

For Andilab, the added perk of IMS was meeting industry professionals and learning from their experience. He also appreciates the online community, ASM Connect. "You can type in a technical or research question and there's always someone who can answer. I look at it every night."

He's improved his communication skills with leaders in their fields, including people from Europe, the U.S., and Asia. "Being tasked with a project, like setting up virtual career talks, really improved my leadership and soft skills, as I'm not the most outgoing person. I could just sit in the lab doing research, not getting involved or talking to professionals in the industry. But I'd be missing out if I was just crunching numbers and doing experiments."

Once he completes his Ph.D., Andilab plans to explore options in R&D, including the consulting side of forensic engineering and failure analysis.

"I encourage other ASM members to get involved; don't shy away from it. Once you are doing a role, you're more engaged. You never know if you don't try. You can serve others, but it's also good for your own personal development."

# HIGHLIGHTS CHAPTERS IN THE NEWS

# THE FACE OF MATERIALS ENGINEERING

This profile series features members from around the world at all stages in their careers. Here we speak with **Amy Elliott,** 3D printing scientist at Oak Ridge National Laboratory, Knoxville, Tenn.



# What part of your job do you like most?

I love the part of my job where I Elliott

get to mentor younger staff. I love see-

ing the lightbulbs go on in their eyes when we have meaningful discussions.

### What is your greatest professional achievement?

I would have to say I am most proud of all the patents and licenses I've gotten over my short career. I feel truly lucky to be involved on so many high-performing teams that develop useful technology. I've also been fortunate to be honored with a life-sized, 3D-printed statue of myself, which was displayed along with 120 other statues of women in STEM at the Smithsonian Castle in D.C. earlier this year.

### What is your engineering background?

I have a B.S. and Ph.D. in mechanical engineering from Tennessee Technological University and Virginia Tech, respectively.

### What attracted you to engineering?

I love being able to problem-solve with machines. Building a machine to basically "do my bidding" is one of the biggest thrills out there!

# Did you ever consider doing something else with your life besides engineering?

I am interested in producing kids STEM shows. I think that would be a lot of fun! I have some experience being on TV with the shows *The Big Brain Theory* and *Outrageous Acts of Science*, so I feel I could navigate that world.

### Best career advice, given or received:

My best advice is to work hard and be nice! And help others when you can.

### Last podcast listened to?

The Sound of Science podcast from Oak Ridge National Laboratory.

### **Favorite motto:**

When you work hard, you'll be prepared when opportunities come your way.

# Tell us about your involvement with ASM International, why are you a member?

I have always thoroughly enjoyed attending local chapter meetings, getting to know my fellow materials enthusiasts, and hearing about exceptional work being done locally in the materials space. The ASM community has been one of the warmest, most welcoming professional societies I have experienced.

Do you know someone who should be featured in an upcoming Face of Materials Engineering profile? Contact Vicki Burt at vicki.burt@asminternational.org.

# CHAPTERS IN THE NEWS

### Mohawk Valley Tours Trenton Technology

Members of the ASM Mohawk Valley Chapter toured Trenton Technology's manufacturing facility in Utica, N.Y., on May 17. During the tour, Ed Wheeler, director of manufacturing engineering, explained how the company manufactures printed circuit assemblies for commercial, government, and defense contractors. With 100% U.S.-based design, engineering, manufacturing, and technical support, combined with a long-term business strategy, Trenton has not been seriously affected by recent supply chain disruptions. Based on its advanced engineering and impressive facility, Chapter representatives called the company a "hidden gem of technology" in the Mohawk Valley.



The ASM Mohawk Valley Chapter toured Utica's Trenton Technology in May.



Congratulations to these ASM Chapters celebrating milestones of serving local members!

Mohawk Valley—75 Years Purdue—75 years Thank you for your commitment! We look forward to celebrating your future success!

# MEMBERS IN THE NEWS **HIGHLIGHTS**

# 77

# Los Angeles Hosts Student Night

Jeanette Kwan and Angela Sifuentes were each awarded \$2500 by the ASM Los Angeles Chapter. Checks were presented at the Chapter's annual Student Night held on May 25, the first in-person Student Night since 2019. Pictured from left are Dana Shatts, Chapter historian and scholarship committee member; Jeanette Kwan, scholarship recipient; Angela Sifuentes, scholarship recipient; and Kentaro Lunn, Chapter webmaster and meeting chair.



# Hartford Hears About **TBCs**

On May 18, the ASM Hartford Chapter featured a technical talk on the "Challenge of CMAS Attack on TBCs and Strategy for Novel TBC Development," by Dr. Chin Ma of Curtiss-Wright Corp.



### Ма

# MEMBERS IN THE NEWS

# Williams Visits Materials Camp

ASM senior vice president David Williams, FASM, was on hand to support the final day of this summer's Columbus ASM Materials Camp for Teachers. Williams is also an ASM Materials Education Foundation board member. The camp was held at Tolles Career & Technical Center in Plain City, Ohio, June 27-July 1. The photo shows enthusiastic attendees and Master Teachers (including Glenn Daehn, Todd Bolenbaugh, and Caryn Jackson), along with Williams.



# Past Presidents Meet in Vienna

Jack G. Simon, FASM, and Hans H. Portisch, FASM, both past presidents of ASM International, had a chance to get reacquainted this past June on European soil. The last time they were together was in 1989 when they attended one of Austria's famous evenings of waltz dancing with their wives and the larger Portisch family. Simon served as ASM president in 1994 and Portisch in 1999.



From left: Jack and Bette Simon along with Christiana and Hans Portisch in Vienna on June 12.

# Radzilowski Credited by **Nobel Prize Winner**

Ronald H. Radzilowski, FASM, was surprised to be credited during a May 23 talk by Stan Whittingham, 2019 Nobel Laureate in Chemistry. Whittingham was giving the Van Vlack Lectureship at the University of Michigan on the topic of climate change abatement and referenced Radzilowski's published work on ionic transport in beta alumina. The two worked on the solid-state electrolyte battery in 1966-1970 while Whittingham was at Stanford University and Radzilowski was at the Ford Motor Co. Scientific Research Labs in Dearborn, Mich. After a 53-year hiatus, the two met in person at the Van Vlack Lecture and subsequent awards dinner.



Stan Whittingham, Ron Radzilowski, and Chris Radzilowski at the Van Vlack Awards dinner at the University of Michigan Art Museum in May.

# HIGHLIGHTS IN MEMORIAM

### Kanematsu Publishes COVID-19 Science

Hideyuki Kanematsu, FASM, specially appointed professor at the National Institute of Technology, Suzuka College, Japan, just released a new book. "Studies to Combat COVID-19 using Science and Engineering" was published by SpringerLink. The book provides ongoing, lead-



Kanematsu

ing-edge research related to various aspects of COVID-19 and includes important developments from the viewpoint of materials science and engineering applications. Kanematsu is a member of the 2022 Class of ASM Fellows, the AM&P Editorial Committee, and the International Materials Review Committee.

# IN MEMORIAM



Hahn

**George Thomas Hahn, FASM,** 92, passed away on June 7. He was born in Vienna in 1930. When he was eight, his family left Austria for New York City to escape Hitler's persecution. Hahn was a professor emeritus of mechanical engineering and materials science at Vanderbilt University, Nashville, Tenn., where he served for 18 years with distinction until his retirement in 1998. Prior to that, he worked for 19 years at Battelle Laboratories in Columbus, Ohio, where in his last position he was the division chief and manager of the Metal Science Section. He published approximately 200 papers in the fields of fracture mechanics, rolling contact, and riveted connections and was listed in "Who's Who in Engineering Academia." Hahn earned his bachelor's in mechanical engineering from New York University in 1952, then served in the U.S. Air Force from 1953 to 1955 as a project engineer at Wright-Patterson Air Force Base in Dayton, Ohio. After serving, he earned a master of science degree in metallurgical engineering from Columbia University in 1955 and a doctorate of science in metallurgy from MIT in 1959. Hahn was the ASM Edward DeMille Campbell Memorial Lecturer in 1981.

Word has been received at ASM Headquarters of the death of **Anthony C. Demos,** 94, of Bryn Mawr, Pa. He formed the Chemalloy Company in 1960 and led it until his retirement in 2016. Demos was a member of the Philadelphia Chapter.

**Ray W. Fenn, FASM,** of Vancouver, Wash., also passed away in the last year. He had been a member of the ASM Santa Clara Valley Chapter and devoted his career to the aerospace industry working for Lockheed Martin.

In addition, the Society was notified of the passing of **William Henry**, 90, of Perkasie, Pa. Henry was a member of the ASM Philadelphia Chapter and the Failure Analysis Society.

# MATERIALS & PROCESSES **EDITORIAL PREVIEW**

# SEPTEMBER 2022

# **Nondestructive Testing and Failure Analysis**

# **Highlighting:**

- Mechanisms of Metallurgical Failure
- Terahertz Wave Diagnostics
- ISTFA Show Preview

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**Advertising closes August 8** 

# **OCTOBER 2022**

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- Ceramics and 3D Printing
- Combining AM and Metal Injection Molding

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# ROPRNSHOP



3D-printed glass lattices, displayed in front of a U.S. penny for scale. Courtesy of Joseph Toombs.

# **REVISED PROCESS PRINTS GLASS WITH FINE FEATURES**

A combined team from UC Berkeley and the Albert Ludwig University of Freiburg, Germany, have improved upon a 3D-printing process developed three years ago-computed axial lithography (CAL)-to print much finer features and to print in glass. They dubbed this new system "micro-CAL." The new method prints glass microstructures faster and produces objects with higher optical quality, design flexibility, and strength.

The CAL process is different from other industrial 3D-printing manufacturing processes that build up objects from thin layers of material. CAL 3D prints the entire object simultaneously. Researchers use a laser to project patterns of light into a rotating volume of light-sensitive material, building up a 3D light dose that then solidifies in the desired shape. The layer-less nature of the CAL process enables smooth surfaces and complex geometries. "With micro-CAL, we can print objects in polymers with features down to about 20 millionths of a meter, or about a guarter of a human hair's breadth," says Hayden Taylor, principal investigator and professor of mechanical the binder, then the researchers heat the printed object to remove the binder and fuse the particles together into a solid object of pure glass.

Berkeley.

To print

glass, Taylor and his

research team col-

laborated with scien-

tists from the Albert

Ludwig University of

Freiburg, who have

developed a special

resin material con-

taining nanoparticles of glass surrounded

by a light-sensitive

binder liquid. Digital

light projections from

the printer solidify

the

"The key enabler here is that the binder has a refractive index that is virtually identical to that of the glass, so that light passes through the material with virtually no scattering," says Taylor. berkeley.edu.

# **FINDING DEFECTS IN LPBF** METAL WITH ULTRASOUND

Researchers from Lawrence Livermore National Laboratory (LLNL) are using laser-based ultrasound to reveal surface and sub-surface defects in laser powder bed fusion (LPBF) metal 3D printing. The all-optical ultrasound technique on-demand perform can characterization of melt tracks and detect formation.

"The demonstrated laser-based ultrasound, surface acoustic wave (SAW) system showed excellent sensitivity to surface and near-surface features, including breaks in the LPBF melt line, metal surface splatter, and subsurface

air voids," says LLNL engineer and prinengineering at UC cipal investigator David Stobbe.

> Surface acoustic waves have historically been used to characterize surface and near-surface features such as cracks, pits, and welds in engineering materials, and are used in geology for detecting subterranean features such as caves. Due to their surface and near-surface sensitivity, SAWs are well-suited for characterizing melt lines in LPBF printing, according to researchers.

To test this potential, the LLNL team carried out experiments by producing laser melted lines using a fiber laser directed into a vacuum chamber and produced samples of titanium alloy for analysis with 100, 150, and 350 W powered lasers. Next, they developed a method for producing and detecting surface acoustic waves, using a pulsed laser to generate ultrasound and measured the displacement with a photorefractive laser interferometer.

"A system like this may find use for rapidly qualifying new LPBF machines and in-service machines after changes to metal powder feedstock or modifications to the melt laser power or scan speed," says Stobbe. Ilnl.gov.



Surface acoustic waves generated by laser-based ultrasound could find defects in LPBF metal 3D printing. Courtesy of David Stobbe/LLNL.

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# What if the materials data you need doesn't exist?

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Linear expansion vs temperature for Ti-6Al-4V



Calculated latent heat compared to handbook values for a specific 316L stainless steel chemistry



Calculated electrical resistivity of aluminum alloy 7075

# Gain insight into materials processing



Time temperature precipitation of M<sub>23</sub>C<sub>6</sub> in 308 stainless steel

# Solidification

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Solidus variation within Alloy 718 specification (Gaussian, n=1000)

# Diffusion



Carbon diffusion profile near surface during carburization of a martensitic stainless steel

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