NOVEMBER/DECEMBER 2016 | VOL 174 | NO 10

ADVANCED MATERIALS & PROCESSES

AN ASM INTERNATIONAL PUBLICATION

MATERIALS TESTING/CHARACTERIZATION

CORRELATIVE MICROSCOPY OF NEUTRON-IRRADIATED MATERIALS

P.16

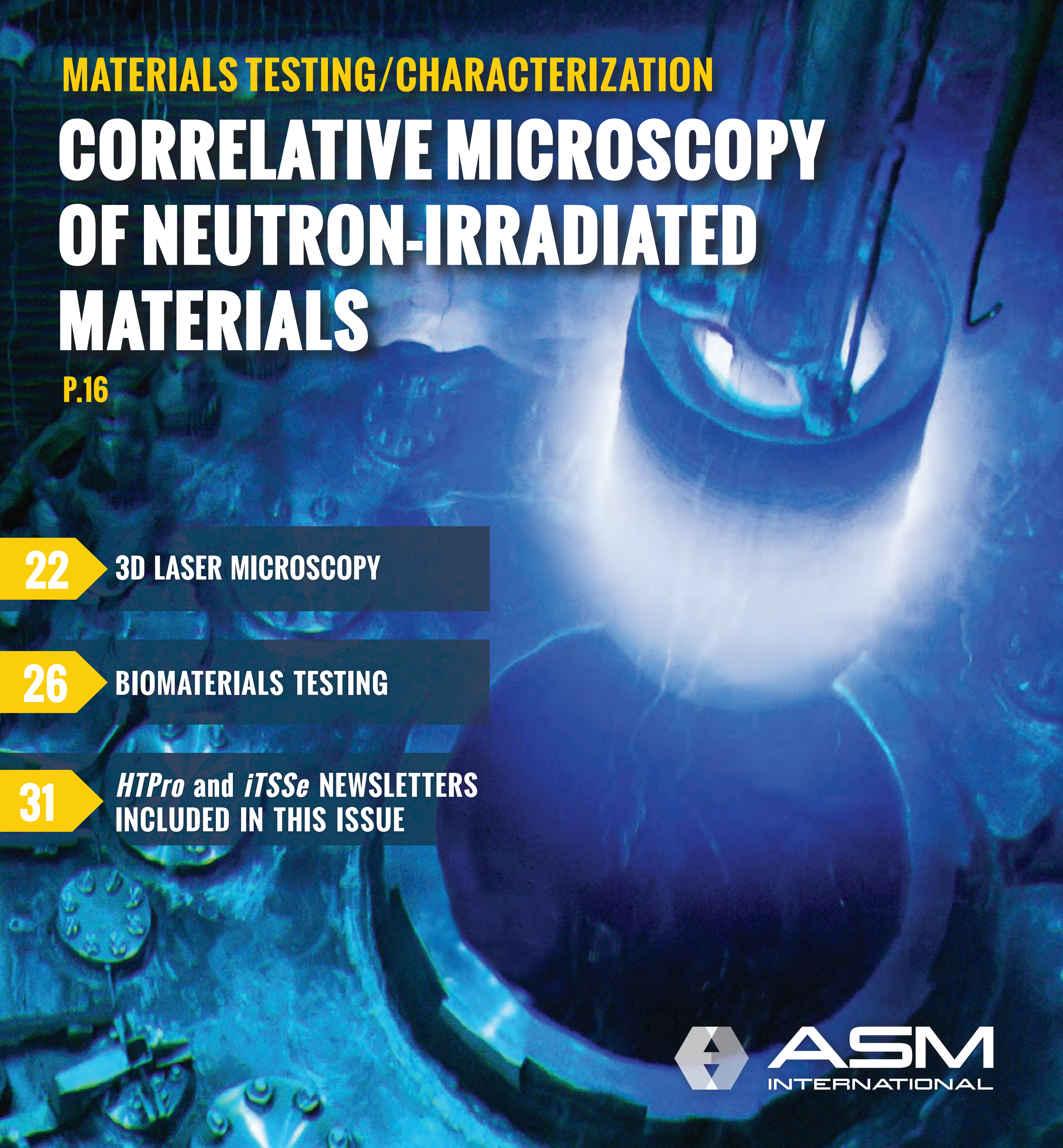
ASM INTERNATIONAL


ALLIED
HIGH TECH PRODUCTS, INC.
NEW!
PowerCut 10x™
Abrasive Cut-Off Saw
NEW for 2017!
NEW!
TechPress 3™
Mounting
Press
3 NEW Machines
Software & Design Updates
Sturdy RIM Covers
Powerful New Motors
CE Certification
NEW!
E-Prep 4™
Polisher
Celebrating Over 33 Years In Business
33
years
2376 E. Pacifica Place
Rancho Dominguez, CA 90220
310.635.2466 Worldwide
800.675.1118 Canada / US
info@alliedhightech.com

NOVEMBER/DECEMBER 2016 | VOL 174 | NO 10

ADVANCED MATERIALS & PROCESSES

AN ASM INTERNATIONAL PUBLICATION



MATERIALS TESTING/CHARACTERIZATION

CORRELATIVE MICROSCOPY OF NEUTRON-IRRADIATED MATERIALS

P.16

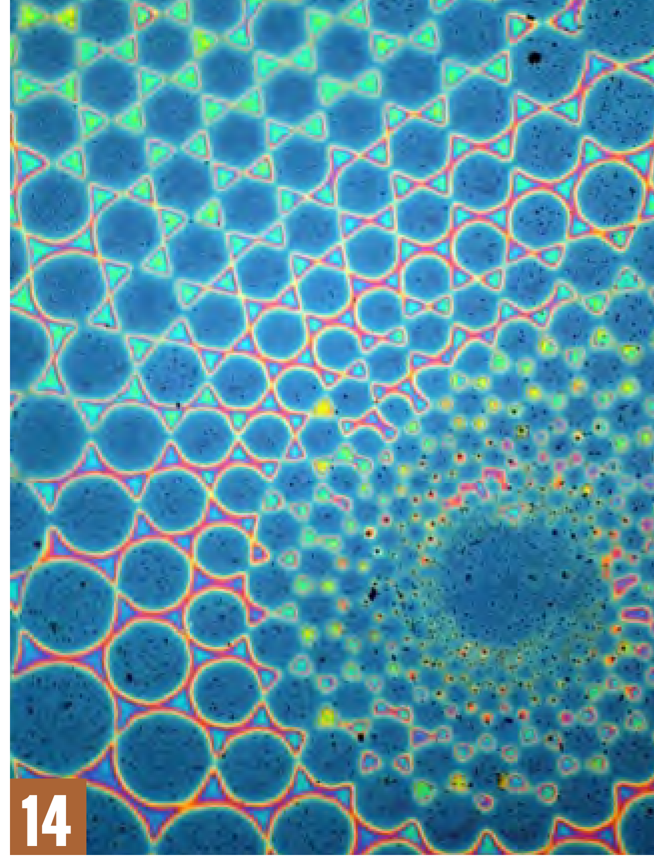
ASM INTERNATIONAL

On The Cover:

High Flux Isotope Reactor core during a routine refueling operation. Courtesy of Oak Ridge National Laboratory, US Dept. of Energy.

ADVANCED MATERIALS & PROCESSES
NOVEMBER/DECEMBER 2016 | VOL 174 | NO 10

FEATURES



14

31 49

TRENDS

INDUSTRY NEWS

DEPARTMENTS

Advanced Materials & Processes (ISSN 0882-7958, USPS 762080) is published monthly, except bimonthly July/August and November/December, by ASM International, 9639 Kinsman Road, Materials Park, OH 44073-0002; tel: 440.338.5151; fax: 440.338.4634. Periodicals postage paid at Novelty, Ohio, and additional mailing offices. Vol. 174, No.10, NOVEMBER/DECEMBER 2016. Distributed at no charge to ASM members in the United States, Canada, and Mexico. International members can pay a $30 per year surcharge to receive printed issues. Subscriptions: $475. Single copies: $51. POSTMASTER: Send 3579 forms to ASM International, Materials Park, OH 44073-0002. Change of address: Request for change should include old address of the subscriber. Missing numbers due to "change of address" cannot be replaced. Claims for nondelivery must be made within 60 days of issue. Canada Post Publications Mail Agreement No. 40732105. Return undeliverable Canadian addresses to: 700 Dowd Ave., Elizabeth, NJ 07201. Printed by Publishers Press Inc., Shepherdsville, Ky.

Check out the Digital Edition online at asminternational.org/news/magazines/am-p

ADVANCED MATERIALS & PROCESSES

ASM International
9639 Kinsman Road, Materials Park, OH 44073
Tel: 440.338.5151 • Fax: 440.338.4634

Frances Richards, *Editor-in-Chief*
frances.richards@asminternational.org

Julie Lucko, *Editor*
julie.lucko@asminternational.org

Ed Kubel and Erika Steinberg, *Contributing Editors*

Jim Pallotta, *Creative Director*
jim.pallotta@asminternational.org

Kate Fornadel, *Layout and Design*

Kelly Sukol, *Production Manager*
kelly.sukol@asminternational.org

Press Release Editor
magazines@asminternational.org

THOUGHTFUL LECTURES OFFER INTRIGUE AND INSPIRATION

It is almost impossible to believe we are wrapping up the final edition of *AM&P* for 2016. With this in mind, it seems that the annual Materials Science & Technology (MS&T) conference occurs at an ideal time—during October, while we still have a chance to think about the year to date and before we are distracted by the joys and challenges of the holiday season. The "big picture" lectures at MS&T continue to amaze and inspire, and this year is no exception.

Rapid changes in society and commerce were important themes of several talks at MS&T, in addition to global engineering challenges. As usual, the Alpha Sigma Mu lecture was outstanding with Al Romig, FASM, speaking about the "14 grand challenges for engineering in the 21st century" as determined by the National Academy of Engineering (NAE). Romig, executive officer of this academy, outlined the mega-engineering problems of our day. All revolve around themes of "continuation of life on the planet, and making our world more sustainable, safe, healthy, and joyful." For example, goals include making solar energy economical, engineering better medicines, providing energy from fusion, preventing nuclear terror, and engineering the tools of scientific discovery. Romig highlighted the importance of the Grand Challenge scholar program to prepare future talent to solve these problems. For more information, visit engineeringchallenges.org.

Signage in front of the Salt Lake City Convention Center.

Speaking about other massive changes in how business is conducted today was Diran Apelian, FASM, of Worcester Polytechnic Institute. In a lively panel discussion on collaborative research programs, Apelian began his talk by posing four intriguing questions and providing the answers after the audience guessed: What company is the world's largest taxi company? (Uber, but they own no vehicles.) What company is the world's most popular media owner? (Facebook, but they create no content.) Who is the world's most valuable retailer? (Alibaba, but they have no inventory.) Who is the world's largest accomodation provider? (Airbnb, but they own no real estate.) Apelian asserted that the most important assets of these companies are knowledge and people, which is the same for every organization—including universities. He went on to make some excellent observations about what makes a successful collaboration.

The plenary session was also intriguing and thought provoking. Julie Christodoulou, FASM, provided a fascinating history of microscopy and characterization tools beginning with Pliny the Elder and continuing through today's technological advancements such as dynamic 3D digital modeling and precession electron diffraction. Her main point was that with today's wealth of sophisticated tools, scientists and engineers must choose projects carefully to use the powerful equipment at their disposal and move progress forward. David Matlock, FASM, then gave an interesting talk on fatigue performance of steel and concluded that a great need still exists to teach "good, common sense engineering."

In other news, we wish you all a happy and healthy holiday season! See you in 2017.

F. Richards

MARKET SPOTLIGHT

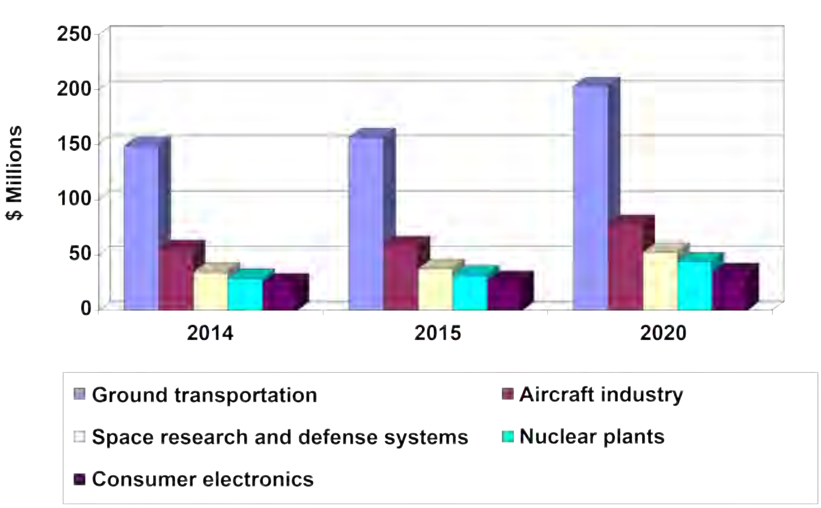
METAL MATRIX COMPOSITES MARKET HOLDS PROMISE

According to a new report from BCC Research, Wellesley, Mass., interest in metal matrix composites (MMCs) remains high due to properties such as greater stiffness, better wear resistance, lower density, and other benefits compared to standard materials. Despite these advantages, however, the market remains modest compared with its potential, according to analysts. MMCs combine high strength and stiffness, low weight, corrosion resistance, and in some cases special electrical properties. This combination makes them very attractive for aircraft and aerospace structural parts among other applications. The most common MMCs are aluminum, magnesium, and copper/titanium alloys as a matrix, with aluminum oxide and silicon carbide as reinforcements.

The global MMC market is forecast to reach $431.1 million by 2020, up from $326.9 million in 2015, reflecting a five-year compound annual growth rate (CAGR) of 5.7%. The largest application market, ground transportation, should reach $203.2 million by 2020 from $156.9 million in 2015, at a CAGR of 5.3%. The space research and defense systems market should reach $52.5 million by 2020 from $37.6 million in 2015, at a five-year CAGR of 6.9%. The space and defense systems, nuclear energy, and aircraft industry MMC markets should experience the strongest growth. Geographically, the most rapid consumption will occur in China and other East Asian nations as a sizeable share of many key end-use market industries migrate to China and elsewhere.

Reducing the cost of manufacturing MMC components would aid use in cost-sensitive industries such as transportation. Both private sector and public initiatives seek to promote MMC use in non-transportation and non-aerospace applications. Some technological and economic barriers have been overcome, although not at the pace most industry participants prefer. High production costs, availability, informational, and technical issues continue to bar market expansion. For more information on *Metal Matrix Composites: The Global Market,* visit bccresearch.com.

Global MMCs Market by Industry, 2014-2020 ($ Millions)



Source: BCC Research

FEEDBACK

SERIOUSLY SCARY

Thank you for your astute insight on artificial intelligence (AI), "Serious Matters," October issue. Back in the 1960s, the movie "2001: A Space Odyssey" profoundly affected my thinking on computers. An astronaut at the end of an interstellar voyage finds himself the lone survivor on board his spacecraft. Other astronauts are found dead in their cryogenic beds. Dave, the live astronaut, discovers that the computer, HAL 9000, has taken over the ship. Soon he is in back of the computer's power supplies disabling it. As he begins shutting it down, the computer speaks to him. "Dave? ... I wouldn't do that if I were you Dave." This is the signature line of the movie. I sure hope a takeover by AI wouldn't get that close before we recognized what could be done about it.

Dana Wilson

SERIOUSLY SKEPTICAL

I'm a serious skeptic about AI. This might be something to be used in smartphones and computers, but can you imagine a machine being able to decide what makes for a good technical publication like *AM&P?* A machine that can decide what a guy like me wants to see? NOT. I think your job is more than safe.

In other news, have you been following the driverless car thing? One of the ladies in our group has placed an order for one of the TBD Tesla cars that will have that technology. She loves the idea. Granted, there are some bad drivers out there, but I don't see this as an improvement. And I definitely put it in the "what could possibly go wrong" category. Two words that should strike fear into the hearts of even the most enthusiastic proponents of driverless cars? Software updates.

T.R.

We welcome all comments and suggestions. Send letters to frances.richards@asminternational.org.

OMG!
OUTRAGEOUS MATERIALS GOODNESS

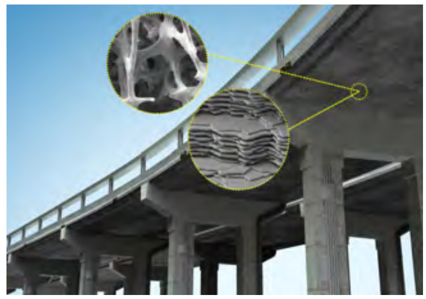
The Mackenzie FX1 is the first double-handed salmon rod to use graphene.

GRAPHENE GOES FISHING

World champion angler Scott Mackenzie and NASA engineer Gary Savage say they have made the first fishing rod using graphene, a double-handed salmon rod called the Mackenzie FX1. The fast recovery is said to make it easier to cast further with little effort. Blanks in the rod are built entirely in the UK using a unique process: A high-pressure auto clave, a machine normally used to make parts for auto racing and aerospace, removes more air than the traditional rod making process, creating a much stronger blank. This means the FX1 is extremely strong, durable, and won't soften or lose performance over time.

"We have taken the best of everything we have learned in Formula One to create the best fly rod ever made. We had an incredibly exciting opportunity to take the art of salmon fishing to a whole new level by harnessing graphene in the right way before anyone else. The rod is a game changer for both experts and less experienced anglers," says Savage. *mackenzieflyfishing.com.*

CONCRETE GETS A MAKEOVER



MIT researchers are seeking to redesign concrete by following nature's blueprints.

Researchers at Massachusetts Institute of Technology, Cambridge, are seeking to redesign concrete by following nature's blueprints. The team contrasted cement paste—concrete's binding ingredient—with the structure and properties of natural materials such as bones, shells, and deep-sea sponges. As researchers observed, these biological materials are exceptionally strong and durable, thanks in part to their precise assembly of structures at multiple length scales, from the molecular to the macro level.

The team, led by professor Oral Buyukozturk, proposed a new bioinspired, bottom-up approach for designing cement paste. "These materials are assembled in a fascinating fashion, with simple constituents arranging in complex geometric configurations that are beautiful to observe. We want to see what kinds of micromechanisms exist within them that provide such superior properties and how we can adopt a similar building-block-based approach for concrete. If we can replace cement, partially or totally, with some other materials that may be readily and amply available in nature, we can meet our objectives for sustainability," Buyukozturk says. *For more information: Oral Buyukozturk, obuyuk@mit.edu, web.mit.edu.*

OLLI TAKES YOU FOR A RIDE

A new self-driving vehicle—a 3D-printed minibus named Olli, capable of carrying 12 people—was unveiled by Arizona-based startup Local Motors outside Washington, D.C. Olli was designed as an on-demand transportation solution that passengers can summon with a mobile app, like Uber. And it can be printed to specification in *micro factories* in a matter of hours.

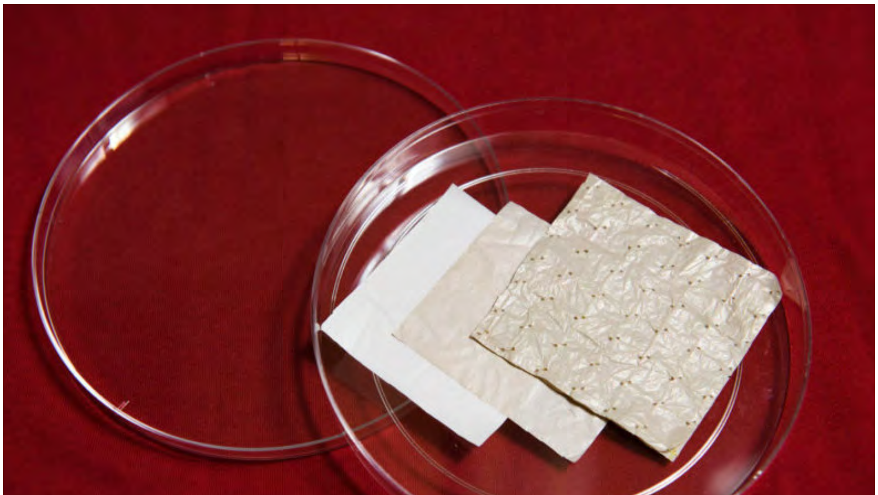
Driving is controlled by a system developed by Local Motors with several software and tech partners. IBM is not doing the driving but is providing the user interface so passengers can have "conversations" with Olli. Using natural language recognition can help create a relationship between the passenger and the vehicle, says IBM's Bret Greenstein. "A vehicle that understands human language, where you can walk in and say, 'I'd like to get to work,' that lets you as a passenger relax and enjoy your journey," he explains.

The vehicle relies on more than 30 sensors and streams of data from IBM's cloud. With the Watson software, passengers can ask how the vehicle works, where they are going, and why Olli is making certain driving decisions. And it can answer the dreaded question, "Are we there yet?" It also can offer recommendations for popular restaurants or historical sites based on user preferences. *localmotors.com.*

Olli, an autonomous shuttle, at the National Harbor in Maryland.

Are you working with or have you discovered a material or its properties that exhibit OMG - Outrageous Materials Goodness?
Send your submissions to Julie Lucko at julie.lucko@asminternational.org.

METALS | POLYMERS | CERAMICS



Stanford researchers modified a sheet of polyethylene with a series of chemical treatments to make a cooling fabric. Courtesy of L.A. Cicero.

ORNL process helps recover magnets from used computer hard drives.

PLASTIC CLOTHING COOLS SKIN

Researchers at Stanford University, Calif., engineered a low-cost plastic material that could become the basis for clothing that cools the wearer, reducing the need for air conditioning. The plastic-based textile, if woven into clothing, could cool a person's body far more efficiently than the natural or synthetic fabrics worn today.

The material works by allowing the body to discharge heat in two ways that would make the wearer feel nearly 4°F cooler than if they wore cotton clothing. It also allows the heat that a person's body emits as infrared radiation to pass through the plastic textile. To develop the new textile, researchers blended nanotechnology, photonics, and chemistry to give polyethylene a number of characteristics desirable in clothing material—it allows thermal radiation, air, and water vapor to pass through, and it is opaque to visible light. *stanford.edu.*

RECOVERING RARE EARTH MAGNETS

The DOE's Oak Ridge National Laboratory (ORNL), Tenn., and Momentum Technologies, Dallas, signed a nonexclusive licensing agreement for an ORNL process designed to recover rare earth magnets from used computer hard drives. The patent-pending process developed as part of DOE's Critical Materials Institute is designed to economically recover large amounts of magnets made using neodymium—a rare earth element that is mined outside the U.S. The permanent magnets are used in everything from computer hard drives and cell phones to clean energy technologies such as electric vehicles and wind turbines.

ORNL's highly automated process for recovering magnets employs a unique system to sort and align hard drives on a conveyer for processing. The method uses a mapping station with barcode scanning and a coordinate measuring machine to populate a database of each make of hard drive so they may be positioned for correct robotic disassembly. The process is designed

BRIEFS

Metaldyne Performance Group Inc. (MPG), Southfield, Mich., acquired **Brillion Iron Works Inc.,** Brillion, Wis., which specializes in the casting design and production of gray, ductile, and austempered ductile iron products. MPG manufactures gray and ductile iron castings and machines iron, aluminum, and steel components for the transportation and industrial markets. *mpgdriven.com.*

The Steel Market Development Institute, Detroit, released its *2016 Steel Industry Technology Roadmap for Automotive,* which looks at advanced high-strength steel (AHSS) material and manufacturing technologies related to design, fuel economy, strength, durability, environmental performance, value, and how AHSS meets those requirements now and in the future. It also identifies AHSS solutions and recommends areas for future research. *autosteel.org.*

to recover the magnets, their permalloy brackets, circuit boards, aluminum, and steel, while automatically destroying data storage media to ensure security.

The magnets may then be directly reused by hard drive manufacturers or in motor assemblies, used in other applications through resizing or reshaping, or processed back to rare earth metal. The recycling method can be adapted to target other consumer goods containing rare earth magnets, such as used electric motors, appliances, and heating and air conditioning systems. *ornl.gov, cmi.ameslab.gov.*

ETCHING METAL CREATES BETTER BONDS

Researchers at Kiel University, Germany, changed the surface properties of metals without affecting their mechanical stability or physical characteristics. The method is based on an electrochemical etching process, in which the uppermost layer of a metal is roughened on a micrometer scale in a tightly controlled manner. Through this *nanoscale-sculpturing* process, metals such as aluminum, titanium, or zinc can permanently be joined with nearly all other materials, become water-repellent, or improve their biocompatibility.

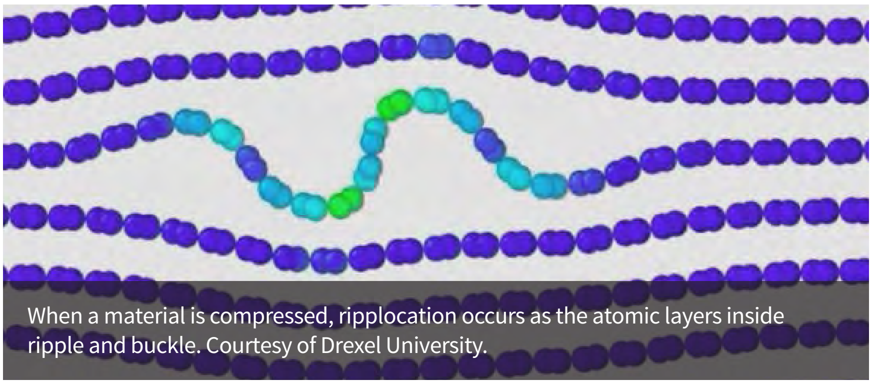
The surface of a metal is converted into a semiconductor, which can be chemically etched and specifically modified as desired. The unique etching process does not damage the metals and does not affect their stability. "In this way, we can permanently connect metals which could previously not be directly joined, such as copper and aluminum," says researcher Jürgen Carstensen.

Through the etching process, a 3D structure with tiny hooks is created. If a bonding polymer is applied between

The surface of an aluminum strip was treated with an electrochemical etching process and permanently bonded with thermoplastic by heating. Courtesy of Julia Siekmann/Kiel University.

two treated metals, the surfaces interlock with each other in all directions like a 3D puzzle. "These 3D puzzle connections are practically unbreakable. In our experiments, it was usually the metal or polymer that broke, but not the connection itself," Carstensen notes. *www.uni-kiel.de/index.shtml.*

TESTING | CHARACTERIZATION



When a material is compressed, ripplocation occurs as the atomic layers inside ripple and buckle. Courtesy of Drexel University.

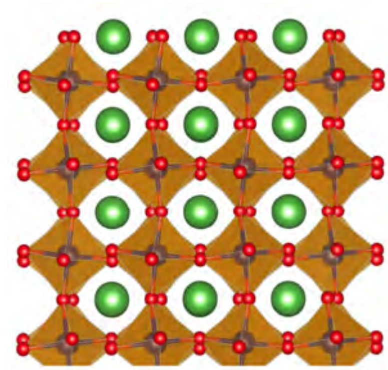
LAYERED MATERIALS BUCKLE UNDER PRESSURE

Researchers at Drexel University, Philadelphia, observed a new type of structural deformation mechanism in bulk materials called *ripplocation*—the rippling and buckling of interior atomic layers when the material is compressed. This evidence supplants the previously held dislocation theory of deformation in these materials, which says that when the planes of layered solid materials are loaded and unloaded edge-on they will either bounce back to their original form—in an elastic material—or be permanently indented. In contrast, ripplocation describes the material returning to its original form while dissipating considerable amounts of energy.

The team ran atomistic simulations on a bulk sample of graphite. By constraining the edges of the sample while compressing the material, they observed the nucleation and motion of a multitude of ripplocations that self-assembled into kink boundaries. Then researchers examined samples of a layered ceramic known as a MAX phase, in which the layers were loaded with a spherical indenter. High-resolution transmission electron microscope images of the defects show they were not dislocations but were consistent with ripplocation. The finding sheds new light on the behavior of most layered materials, from a sliver of graphite to geologic formations. *drexel.edu.*

MATERIAL INTERFACES GET A CLOSE LOOK

Together with an international team, physicists at Würzburg University, Germany, demonstrated a new method to analyze transition-metal oxide interfaces and to model their properties. Studying the charge characteristics of these interfaces has typically been challenging due to their varied behaviors and the scale over which their properties change, often just a few atomic spacings. The new approach uses analysis software based on resonant x-ray reflectometry, which exploits x-ray light created at a synchrotron, with atomic-scale resolution of less than one.



Film of lanthanum cobalt oxide shows a sequence of positively and negatively charged atomic layers. Without electronic reconstruction, an enormous electrostatic field would form between the layers. Courtesy of J.E. Hamann-Borrero and Vladimir Hinkov.

BRIEFS

Thermo Fisher Scientific Inc., Waltham, Mass., completed its acquisition of **FEI Company Inc.,** Hillsboro, Ore., for approximately $4.2 billion following receipt of all required regulatory approvals. The business will become part of Thermo Fisher's analytical instruments segment. *corporate.thermofisher.com.*

ThermoFisher SCIENTIFIC

A new specification for 3D-printed stainless steel alloys will soon be published by **ASTM International,** West Conshohocken, Pa., for parts made of 316L (UNS 31603) stainless steel. *Specification for Additive Manufacturing Stainless Steel Alloy (UNS S31603) with Powder Bed Fusion—F3184M*—describes chemical and mechanical requirements, among others. *astm.org.*

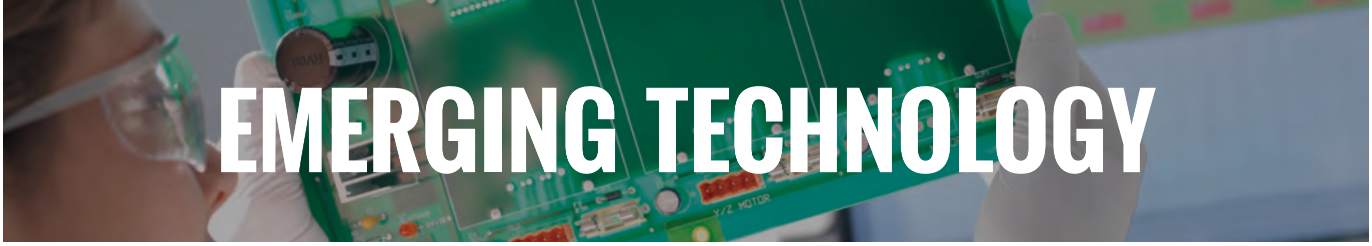
The Science and Technology Center on Real-Time Functional Imaging, a collaboration between scientists from **University of California** (Los Angeles and Berkeley) and the **University of Colorado Boulder,** will receive $24 million from the **National Science Foundation** over a five-year period, with the possibility of an additional five-year extension. The center will focus on advancing real-time imaging by combining and improving single imaging methods such as optical, x-ray, nano-probe, and electron microscopy. *ucla.edu, berkeley.edu, colorado.edu.*

Physicists applied the technique to thin films of lanthanum cobalt oxide—a sequence of positively and negatively charged atomic layers, stacked on a 15-nm-thin film. In these materials, enormous electrostatic fields form between layers. To avoid this high field energy cost, nature rearranges the charges, either through a process that preserves film face smoothness—called electronic reconstruction—or a process that results in a corrugated surface, which hinders devices based on film interfaces, especially when material properties change on an atomic scale. The resonant x-ray reflectometry method reveals the promising news that electronic reconstruction occurs at transition-metal oxide interfaces. This method could be applied to other microscopic properties of these materials as well—such as the electronic occupation of atomic orbitals and spin orientation—and lead to their use in applications such as lossless computer memory and ultrafast processors. *www.uni-wuerzburg.de/en.*

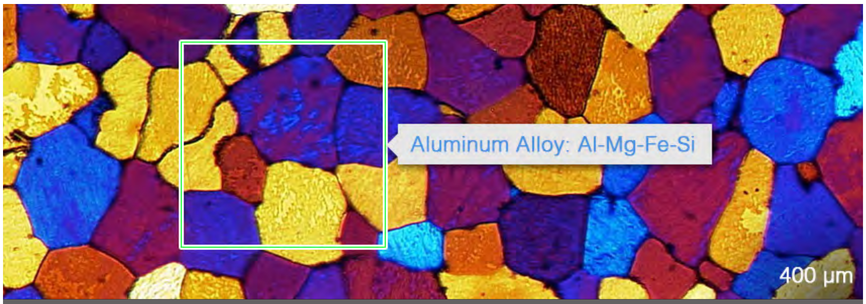
NEW NIST GUIDE BREAKS ONTO SCENE

The National Institute of Standards and Technology (NIST), Gaithersburg, Md., released a new, expanded edition of the NIST Recommended Practice Guide, "Fractography of Ceramics and Glasses" (NIST Special Publication 960-16e2). First issued in 2007, the guide was developed to help engineers and scientists analyze fracture patterns in ceramics and glasses used in consumer goods, building materials, and medical devices and implants. This edition, with nearly 1000 images—300 of which are new—is 15% larger than its predecessor and includes updates such as descriptions of recent documentary standards, material on new microscopy techniques, and an expanded chapter on quantitative fracture analysis. Additionally, case studies help readers see how the guide is used in practice. While hard copies are available upon request, the guide may be accessed free of charge online. *nvlpubs.nist.gov/nistpubs/specialpublications/NIST.SP.960-16e2.pdf.*

Fracture of a sapphire dome. Courtesy of NIST.



EMERGING TECHNOLOGY



Google-like algorithms are now being used to categorize micrographs.

MACHINE LEARNING LEARNS MATERIALS

In a field that has historically relied on human experts to identify research images by hand, a materials science professor at Carnegie Mellon University, Pittsburgh, developed a system that uses machine learning algorithms to search, sort, classify, and identify visual data sets in materials science. Professor Elizabeth Holm is using the algorithms—familiar to Google image search users—to automatically recognize and categorize images of microstructures. Because the algorithms are designed to improve as they encounter more data, they are especially suited to the vast image libraries amassed in materials science.

Holm's machine learning system could find application in research, industry, publishing, and academia. For example, it could be used to search scientific journal archives to determine if a photo was previously published, or automatically categorize visual data for industries or research labs. "Big companies can have archives of more than 600,000 research images," explains Holm. "No one wants to look through those, but they want to use that data to better understand their products." The next step is application in metal 3D printing. Holm and her team are compiling a database of experimental and simulated metal powder micrographs in order to better understand what types of raw materials are most appropriate for additive manufacturing processes. *cmu.edu.*

INSTABILITY PREDICTION PROVEN AFTER LONG STRETCH

Researchers at the Harvard John A. Paulson School of Engineering and Applied Sciences (SEAS), Cambridge, Mass., demonstrated for the first time that a decades-old theory proves true: With the right amount of tensile force, a thick cube of soft material will suddenly deform into a thin, flat plate. In 1948, applied mathematician Ronald Rivlin predicted the behavior, but materials scientists had been unable to prove the theory experimentally—until now. The Harvard team triggered this instability in a centimeters-thick elastomer block, deforming it into a flat surface. To do so, they stretched a thick, soft elastomer made of silicon rubber at each corner. Under biaxial tension, the sample's center deformed, breaking the geometrical symmetry and becoming suddenly flat.

"This research widens the design space for new architected materials that use instabilities to change or enhance their functionality," says Johannes T.B. Overvelde, former graduate student at SEAS. "With this instability, we can create materials that can suddenly switch between behaviors by using simple triggers to change their geometry." *harvard.edu.*

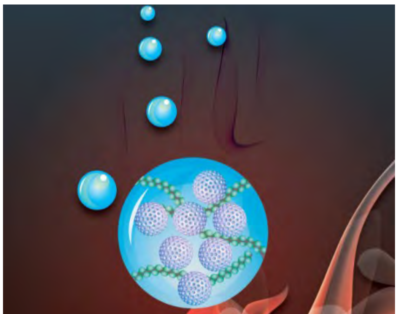
Under biaxial tension, the elastomer sample's center deforms, breaking the symmetry and becoming flat. Courtesy of Harvard SEAS.

BRIEF

Construction is underway on the new Materials Design Laboratory (MDL) at the DOE's **Argonne National Laboratory,** Lemont, Ill. The facility will complete Argonne's Energy Quad, a group of four adjoining buildings designed to maximize collaboration between energy and materials scientists. Encompassing roughly 115,000 sq. ft. of laboratory and office space, including 10,000 sq. ft. of lab space for radiological research, the MDL is on track to receive LEED Gold certification. The lab will house microstructure research and materials testing under extreme conditions. *anl.gov.*

The new Materials Design Laboratory at Argonne National Laboratory broke ground in September.

PROCESS TECHNOLOGY



Artistic interpretation of cold sintering of ceramic particles (white) and polymer strands (green) using low heat to evaporate added water molecules (blue). Courtesy of Jennifer M. McCann/MRI.

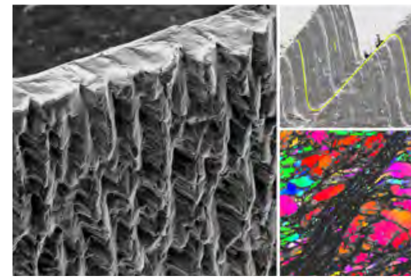
COLD SINTERING PROCESS MIXES THINGS UP

Researchers at Penn State, University Park, Pa., developed a new method for producing compounds of previously incompatible materials that could also lower energy costs for many types of manufacturing. The low temperature method, called cold sintering process (CSP), uses significantly less energy than traditional methods, and can densify some materials to over 95% of their theoretical density in 15 minutes. For example, researchers used CSP to co-sinter ceramic and thermoplastic polymer composites to high density at 120°C in 15-60 minutes.

In CSP, water or acid is applied to ceramic powder, causing particle surfaces to partially dissolve and produce a liquid phase at particle-particle interfaces. With temperature and pressure, the solid particles begin to densify and clusters of atoms or ions leave the interfaces. This aids diffusion, minimizes surface free energy, and allows particles to pack together. The key is a precise combination of moisture, pressure, heat, and time required to capture the reaction rates so the material fully crystallizes and achieves very high density. The team has verified 50 unique combinations so far, including formulas for ceramic-ceramic and ceramic-nanoparticle composites, ceramic-metals, and ceramic-polymers. Applications include thermal insulation, biomedical implants, and electronic components. *psu.edu.*

SHEAR GENIUS

An international team led by researchers at Purdue University, West Lafayette, Ind., discovered microscopic details of the process of shear banding and developed a simple method to control the defect in metals manufacturing. The team studied the displacement profiles created when metal was etched with tiny marks, then processed. The resulting groove-like "micromarkers" revealed large serrated deformations that, surprisingly, resemble liquid flowing past an interface. Turning to mathematical equations typically used to analyze viscous fluid flow, the team determined that shear bands form in two phases. The first occurs within just a few microseconds of cutting when a weak interface is created in the metal. In the second, more gradual phase, metal slides along the weak interface then shears, producing the wave shape.



Shear band formation is a long-standing problem in metals manufacturing. At left, the phenomenon appears as a wavy structure in a titanium strip. Top right, a "micromarker" was traced across a shear band (yellow). Bottom right, an electron backscatter image reveals the band as a severely deformed, dark region about 4 μm thick. Courtesy of Purdue.

To suppress shear banding, researchers added a wedge-shaped die opposite the cutting tool, channeling the chip from the machined metal between the two tools. The die constrains the metal, forcing it to deform more uniformly and eliminating the sliding phase. *purdue.edu.*

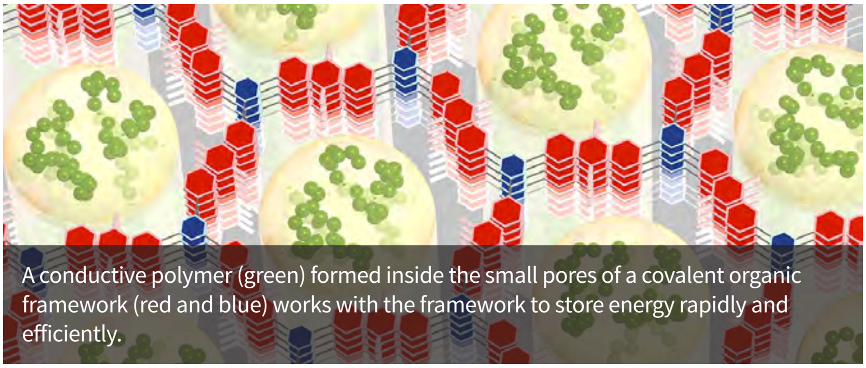
BRIEFS

Norsk Titanium US Inc., Plattsburgh, N.Y., a subsidiary of **Norsk Titanium AS,** Norway, will open a 70,000-sq-ft production and training facility later this year. The Plattsburgh Demonstration and Qualification Center will incorporate Norsk's "ultra lean cell," featuring a 120-ft production line that turns CAD files into finished aerospace parts and allows for 3D printing, heat treatment, nondestructive testing, and final machining in less than 40 hours. *norsktitanium.com.*

NORSK TITANIUM

Sandia National Laboratories, Albuquerque, N.M., and **The University of Akron,** Ohio, approved a master research agreement to collaborate on additive manufacturing and advanced materials research. Areas of interest include the roles of adhesion, friction, and wear in coatings, lubricants, and adhesives, as well as the use of carbon nanotube-based coatings to reduce drag, inhibit ice formation, and provide better heat transfer. In addition, biomimicry will be studied in the context of advanced adhesives for engineering and biological applications. *sandia.gov, uakron.edu.*

ENERGY TRENDS



A conductive polymer (green) formed inside the small pores of a covalent organic framework (red and blue) works with the framework to store energy rapidly and efficiently.

ENERGY STORAGE MODEL COULD BE GAME CHANGER

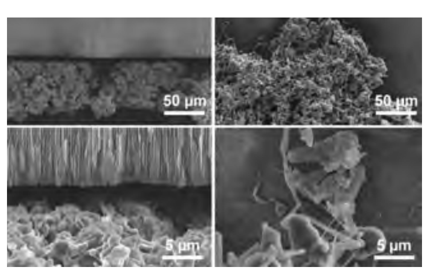
A team from Cornell University, Ithaca, N.Y., developed a way to combine the large energy-storage capacity of batteries with the superior charge-discharge rate of supercapacitors to come up with a powerful electrical energy storage device. The technology—based on a covalent organic framework (COF) infused with an electronically conducting polymer thin film—could benefit numerous industries such as automotive by speeding up the charging process, extending single-charge range, and even incorporating the device into the body of the car itself.

The research centers on COFs—porous crystalline structures that can be fashioned into lightweight building blocks with a variety of applications. Conventional COFs have limited potential for electrical energy storage due to their poor conductivity. The group developed a way to overcome this limitation by electropolymerizing a thin film of poly (3,4-ethylenedioxythiophene) known as PEDOT (an electronically conducting polymer) within the pores of the COF, which is grown on a conducting substrate. The resulting COF film exhibits a 10-fold higher current response compared with unmodified COF films, as well as stable charge-discharge for at least 10,000 cycles. *For more information: Hector Abruña, hda1@cornell.edu, www.cornell.edu.*

METAL HAS MULTIPLE PERSONALITIES

Battery researchers have been focusing on lithium metal electrodes as leading contenders for improving the amount of energy that batteries can store without increasing their weight. But lithium in this metallic form has a problem that has stymied much of this research effort: As batteries are charged, fingerlike lithium deposits form on the metal surface, which can hamper performance and lead to short circuits that damage or disable the battery.

Now, a team of researchers at Massachusetts Institute of Technology, Cambridge, says it has carried out the most detailed analysis yet of how these deposits form, and reports that there are two different mechanisms at work. While both forms of deposits are composed of lithium filaments, the way they grow depends on the applied current. Clustered, "mossy" deposits, which form at low rates, grow from their roots and are relatively easy to control. The much sparser and rapidly advancing "dendritic" projections grow only at their tips. The dendritic type, researchers say, are harder to deal with and are responsible for most of the problems. *For more information: Martin Z. Bazant, balkwill@mit.edu, web.mit.edu.*



On right, SEM images show two types of lithium deposits, the bulky, mossy type (top), which grows from its base, and the needlelike dendritic type (bottom), which grows from the tips. At left, SEM images show the effect of a blocking layer of ceramic material that limits growth of mossy deposits.

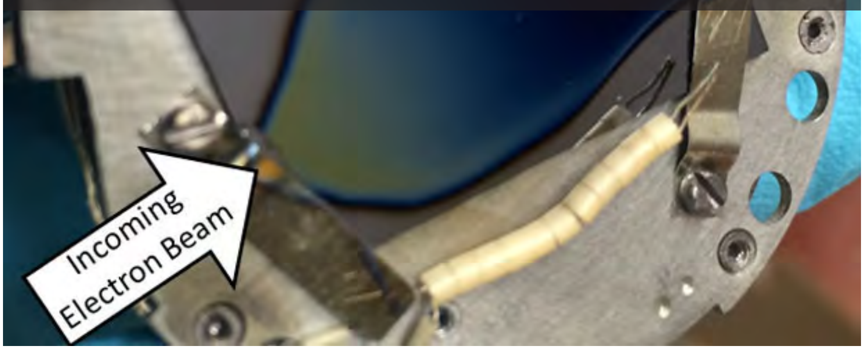
FUEL CELL MEMBRANE IS JUST RIGHT

Fuel cells provide power without pollutants. However, membranes in automobile fuel cells often work at temperatures either too hot or too cold to be maximally effective. Now a polyphenyline membrane patented by Sandia National Laboratories, Albuquerque, N.M., seems to work just right, says chemist Cy Fujimoto.

The membrane, which operates over a wide temperature range, lasts three times longer than comparable commercial products, according to Fujimoto and his colleagues. Their ammonium ion-pair fuel cell—created at Los Alamos National Laboratory—combines phosphates with the Sandia-patented membrane. The ammonium-biphosphate ion pairs exhibit stable performance over a wide range of temperatures from 176°-320°F, respond well to changes in humidity, and last three times longer than most commercial PEM fuel cell membranes. *sandia.gov.*

Cy Fujimoto, right, and Michael Hibbs demonstrate the clarity of their new fuel cell membranes. Courtesy of Randy Montoya.

SURFACE ENGINEERING



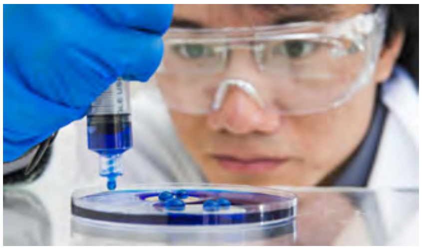
Gallium nitride film is deposited on silicon substrate at 27°C (80°F) using an innovative process for depositing super thin films. Courtesy of University of Colorado Boulder.

ROOM-TEMPERATURE ATOMIC LAYER DEPOSITION

Researchers at the University of Colorado Boulder developed a new approach for synthesizing ultrathin materials at room temperature—a breakthrough over industrial approaches that require temperatures of 800°C or more. The advance opens a path to creating a host of previously unattainable thin-film microelectronics, whose production by conventional methods has been impossible because many components lose their critical functions when subjected to high temperatures.

The new method is known as electron-enhanced atomic layer deposition and was recently developed as part of DARPA's Local Control of Materials Synthesis program. The team demonstrated room-temperature deposition of silicon and gallium nitride as well as the ability to controllably etch specific materials, leading to precise spatial control in 3D. *colorado.edu.*

SPRAY-ON WATER PROOFING



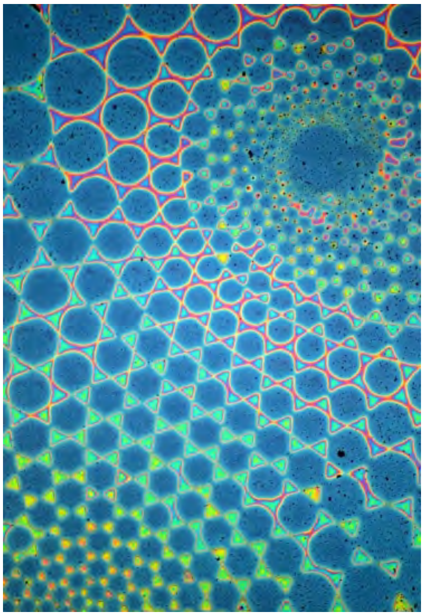
Scientists at ANU developed a new spray-on, water-repellant material. Courtesy of Stuart Hay/ANU.

Scientists at The Australian National University (ANU) developed a new spray-on material with a remarkable ability to repel water. The coating could eventually be used to waterproof mobile phones, prevent ice from forming on airplanes, and protect boat hulls from corroding. The team combined two plastics, one tough and one flexible. "It's like two interwoven fishing nets, made of different materials," says Ph.D. student William Wong. The superhydrophobic coating is also transparent and extremely resistant to ultraviolet radiation. *www.anu.edu.au.*

CREATING POLYMER BRUSHES FOR ENGINEERED SURFACES

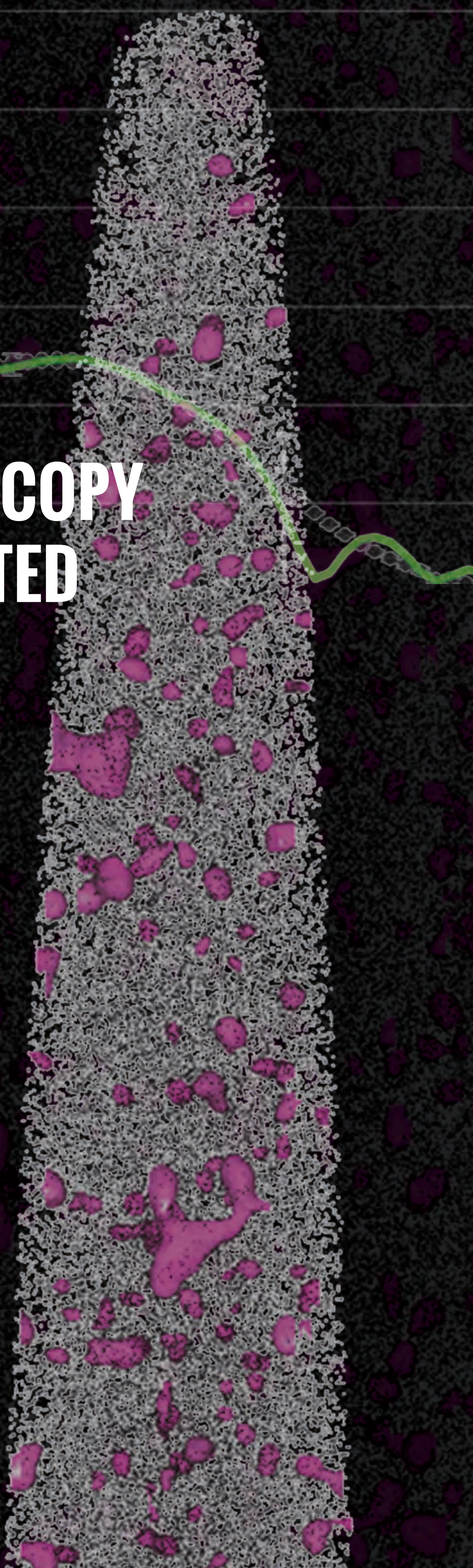
At the University of California, Santa Barbara, researchers are looking to greatly improve on polymer brush creation with a method of micron-scale surface chemical patterning that not only decreases the time and money spent in manufacturing, but also adds versatility to designs. Their method, called "sequential stop-flow photopatterning," features a new platform for functionalizing and engineering surfaces with patterned polymer brushes.

To create the brushes, a substrate (with initiating molecules deposited) is enclosed in a stop-flow cell and a solution streamed in. Irradiation with light can then initiate the reaction. A separate photomask is positioned over the top of the cell, allowing only some light-activated growth. Next, the light is turned off, the first solution is drained from the cell, and a second one is flowed in to functionalize the polymers. Because neither the mask nor the substrate has been moved, only the molecules that have been exposed to light are grown and functionalized. These basic steps may be repeated with variations in the reactants, light source, substrate positions, or photomask to create polymer brush patterns in a single continuous process. *For more information: Christian Pester, pester@mrl.ucsb.edu, www.ucsb.edu.*



The stop-flow photopatterning method enables intricate polymer brush patterns. Courtesy of Christian Pester.

CORRELATIVE MICROSCOPY OF NEUTRON-IRRADIATED MATERIALS



Development of new, radiation-tolerant materials that maintain the structural integrity and safety margins over the course of a nuclear power reactor's service life requires the ability to predict degradation phenomena.

Samuel A. Briggs and Kumar Sridharan, FASM,**
University of Wisconsin, Madison

Kevin G. Field, Oak Ridge National Laboratory, Tenn.

**Member of ASM International*

A nuclear reactor core is an incredibly demanding environment that presents several unique challenges for materials performance. Materials in modern light water reactor (LWR) cores must survive several decades in high-temperature (300-350°C) aqueous corrosion conditions while being subject to large amounts of high-energy neutron irradiation. Next-generation reactor designs seek to use more corrosive coolants (e.g., molten salts) and even greater temperatures and neutron doses. The high amounts of disorder and unique crystallographic defects and microchemical segregation effects induced by radiation inevitably lead to property degradation of materials. Maintaining structural integrity and safety margins over the course of the reactor's service life thus necessitates the ability to understand and predict these degradation phenomena in order to develop new, radiation-tolerant materials that can maintain the required performance in these extreme conditions.

Historically, performing the neutron radiation damage characterization necessary to assess radiation tolerance and performance has faced two primary challenges. First, preventing undue radiation exposure and contamination is problematic, as neutron absorption tends to generate radioactive isotopes in a material, which can make materials handling hazardous without specialized equipment and facilities. As such, the required sample preparation for post-irradiation examination (PIE) often needs to be performed in shielded hot cells and is limited to functions that can be performed therein. Practically speaking, this often precludes access to advanced microstructural characterization tools such as transmission electron microscopes (TEMs) and makes even basic characterization procedures much more costly. Second, neutron irradiation experiments typically have a long turnaround time, as achieving the doses necessary to produce end-of-life microstructure and mechanical properties in a material requires that specimens be exposed in a commercial reactor core for a length of time equivalent to the expected service lifetime. As a result, researchers in this field often use high-energy ion beam accelerators in attempts to emulate neutron damage processes at a much faster rate without inducing radioactivity in specimens. Although these methods provide valuable insight into a material's irradiation response, they are not completely predictive due to differences in the mechanism in which charged particles and neutrons impart energy to the crystal structure[1].

Fortunately, modern PIE experiments can make use of specialized high-flux test reactors and dedicated characterization facilities in order to circumvent some of these issues. The Advanced Test Reactor (ATR) at Idaho National Laboratory (INL) and the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL) are examples of two specialized nuclear reactor designs in the U.S. capable of inducing neutron damage structures at much greater rates compared to commercial LWRs. Further, access to dedicated, radiation-friendly characterization laboratories available through user facilities such as the Nuclear Science User Facilities (NSUF, atrnsuf.inl.gov) can provide the resources and personnel necessary to limit occupational exposure and prevent the spread of radioactive contamination.

An important tool within these user facilities and radiological characterization laboratories is the focused ion beam (FIB), used to prepare specimens for both TEM and atom probe tomography (APT) characterization. Sample preparation through FIB techniques has been particularly impactful in working with radioactive materials due to the significantly smaller working sample volumes leading to increased scientific yield gained from individual experiments. Using carefully performed FIB lift-out techniques, a highly radioactive bulk specimen can be reduced to such a minute volume that it no longer poses a significant exposure risk. As even a single bulk sample can have significant associated cost after irradiation in a reactor like HFIR, extracting the maximum amount of information from these samples is crucially important. As FIB can produce targeted, site-specific analysis and allows for multiple TEM and APT specimens to be prepared from a single bulk sample, it is extremely useful for maximizing the scientific and monetary value of a single bulk irradiated sample.

Some examples of radiation-induced microstructural defects being studied using these techniques include dislocation loops, cavities, gas bubbles, segregation, and precipitates. Characterization of these nanoscale features necessitates advanced microscopy equipment and techniques that often require special considerations when performing data collection or analysis for radioactive materials. This article illustrates the use of these advanced techniques to characterize precipitates in neutron-irradiated Fe-Cr-Al alloys at ORNL, which have extensively utilized the aforementioned research reactors and user facility characterization laboratories. However, the techniques described here can be readily extended to other materials as well.

CHARACTERIZATION OF IRRADIATED Fe-Cr-Al

Fe-Cr-Al alloys are being considered as a possible replacement for Zr-based fuel cladding materials currently used in commercial LWRs for increasing safety margins and enhancing accident tolerance as part of the DOE's Advanced Fuels Campaign[2]. Similar to other high-Cr ferritic alloys, Fe-Cr-Al alloys have excellent high temperature aqueous corrosion and radiation-induced swelling resistance[3], but are susceptible to radiation-induced hardening and embrittlement due to the precipitation of nanoscale Cr-rich α' phase precipitate particles at temperatures below 475°C. Though the kinetics for formation of this phase at LWR-relevant temperatures are slow, this process has been shown to be accelerated by neutron-induced radiation damage[4]. An understanding of how the formation of this phase is affected by composition and how the precipitate microstructure evolves with radiation dose is essential in order to mitigate the embrittlement response in designing a nuclear-grade Fe-Cr-Al alloy for LWR cladding applications.

To this end, neutron irradiation and PIE analysis studies were performed on four Fe-Cr-Al model alloys with nominal compositions ranging from 10-18 wt% Cr and 2.9-4.8 wt% Al. Alloy compositions, as determined by inductively coupled plasma optical emission spectroscopy (ICP-OES), are shown in Table 1. These materials were machined into SS-J2 sub-sized tensile specimens[5] and irradiated in HFIR to various nominal damage doses up to 13.8 displacements per atom (dpa) at a target temperature of 320°C, corresponding to a maximum exposure time of approximately 4900 hrs. Dpa commonly describes radiation damage and is defined as the average number of times an atom is displaced from a lattice site for a given fluence of energetic particles. Dpa is calculated based on the fluence and energy spectrum of incident particles for a given material. The specimens in the 13.8 dpa condition are expected to provide a reasonable approximation of material properties and microstructure at the end of the typical LWR fuel cladding lifetime. Details of final irradiation conditions are shown in Table 2.

Following irradiation, an assessment of tensile behavior was performed using room temperature and elevated temperature tensile testing with subsequent scanning electron microscopy (SEM) fracture surface analysis in-cell at the Irradiated Materials Examination and Testing (IMET) Hot Cell Facility at ORNL. Fractographs for a low- and high-dose condition of the Fe-18Cr-2.9Al following room temperature tensile tests demonstrate clear differences in specimen failure mode (Fig. 1a-b), with typical dimple ductile fracture observed at early-life doses transitioning to brittle, transgranular cleavage fracture at end-of-life doses. Tensile tests performed at 320°C after irradiation demonstrated ductile failure mechanisms in all dose conditions studied (Fig. 1c-d).

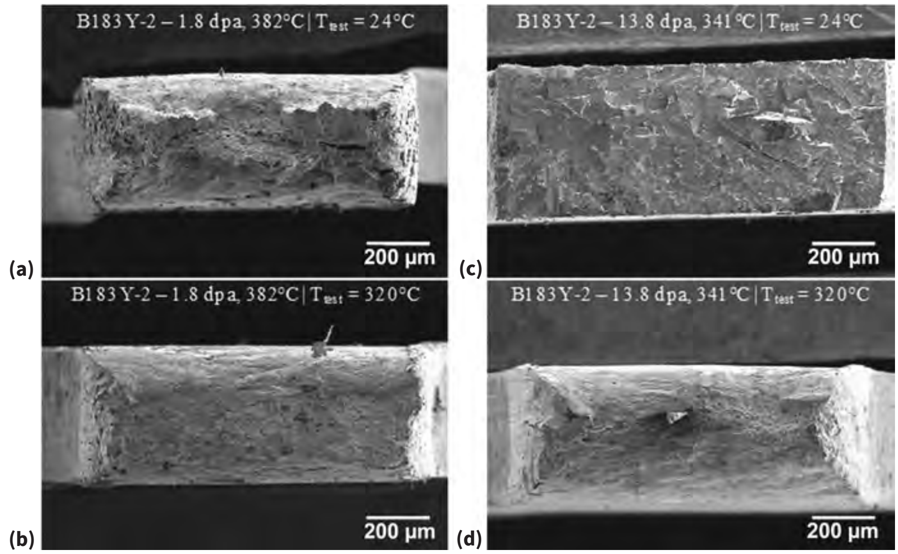


Fig. 1 — Comparison of fracture surfaces for Fe-18Cr-2.9Al alloy irradiated to 1.8 dpa (early life) and 13.8 dpa (near end-of-life) conditions.

Broken half-tensile heads from each material condition were then prepared, packaged for on-road shipping, and shipped to the general purpose small-angle neutron scattering (SANS) beamline at ORNL for diffraction-based analysis of nanoscale α′ precipitates in the microstructure. SANS is a nondestructive analysis technique in which an incident beam of neutrons is elastically scattered by interactions with nuclei or with the magnetic moment of unpaired electrons. Bulk samples used here (volume ~8 mm^3) pose a significant radiological threat, so special care is taken during SANS investigations to minimize user interaction. These larger specimens are necessary in order to fit the 4-mm-diameter aperture sizes and allow for sufficient scattering to maintain an adequate signal-to-noise ratio in the SANS data. Two dimensional diffractograms were collected at room temperature at three different detector configurations. An example of

TABLE 1 — Fe-Cr-Al MODEL ALLOY COMPOSITIONS INVESTIGATED IN THIS RESEARCH

Alloy		Fe	Cr	Al	Y	C	Si
Fe-10Cr-4.8Al	wt%	bal.	10.01	4.78	0.038	0.005	<0.01
Fe-12Cr-4.2Al	wt%	bal.	11.96	4.22	0.027	0.005	0.01
Fe-15Cr-3.9Al	wt%	bal.	15.03	3.92	0.035	0.005	0.01
Fe-18Cr-2.9Al	wt%	bal.	17.51	2.93	0.017	0.005	<0.01

*S, O, N, and P contents at or below 10 ppm.

TABLE 2 — SUMMARY OF Fe-Cr-Al ALLOY CAPSULE IRRADIATION CONDITIONS

Capsule ID	Exposure time (hours)	Neutron flux (n/cm^2s) E > 0.1 MeV	Neutron fluence (n/cm^2) E > 0.1 MeV	Dose rate (dps/s)	Dose (dpa)	Irradiation temperature (°C)
FCAY-01	120	8.54×10^{14}	3.69×10^{20}	7.7×10^{-7}	0.3	334.5 ± 0.6
FCAY-02	301	8.54×10^{14}	9.25×10^{20}	7.7×10^{-7}	0.8	355.1 ± 3.4
FCAY-03	614	8.84×10^{14}	1.95×10^{21}	8.1×10^{-7}	1.8	381.9 ± 5.4
FCAY-04	2456	8.74×10^{14}	7.73×10^{21}	7.9×10^{-7}	7.0	319.9 ± 12.7
FCAY-05	4914	8.74×10^{14}	1.55×10^{22}	7.8×10^{-7}	13.8	340.5 ± 25.7

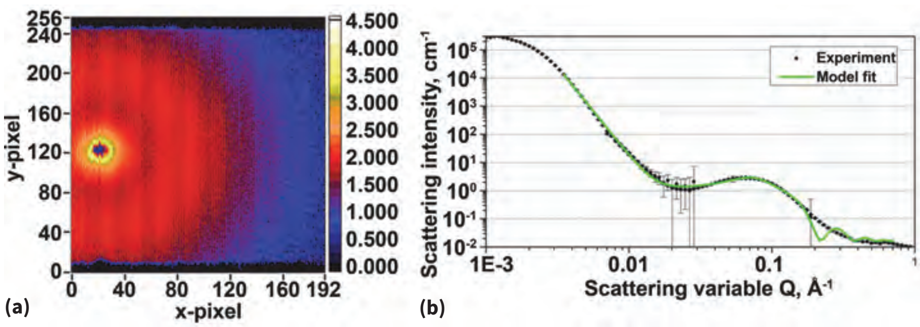


Fig. 2 — (a) Raw SANS diffractogram for Fe-18Cr-2.9Al alloy, irradiated to 13.8 dpa at 341°C. (b) Final SANS scattering intensity curve, combining data from three distinct detector configurations, and example fit of analytical model to the data.

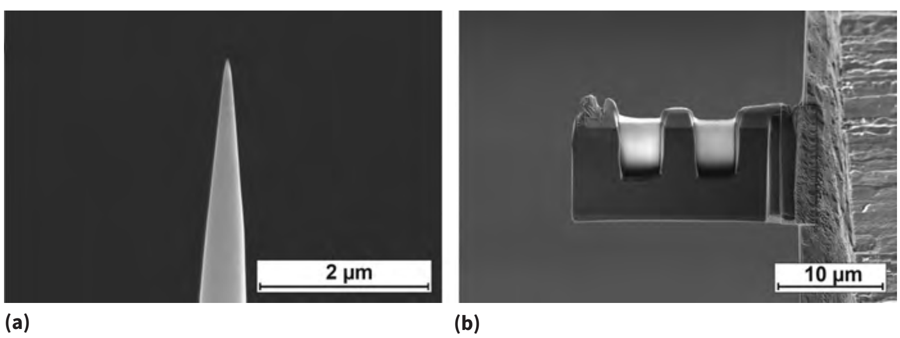


Fig. 3 — SEM micrographs of representative Fe-Cr-Al specimens prepared for (a) APT analysis, and (b) TEM and STEM analysis.

a diffractogram for the Fe-18Cr-2.9Al specimen irradiated to 13.8 dpa at 340°C is shown in Fig. 2a, in which the precipitate signal manifests as a red-orange contrast ring around the central zero-beam (black contrast). Radial reduction of these diffractograms to one-dimensional curves allows analytical models to be fit to the data, from which bulk-averaged precipitate morphology information can be extracted (Fig. 2b).

Sections from the remaining half-tensile specimens opposite the strained neck were cut using a low-speed saw in the hot cells and shipped to the Low Activation Materials Development & Analysis (LAMDA) facility at ORNL for FIB sample preparation. LAMDA is a specialized facility designed for state-of-the-art characterization of low-radiological threat fuel and metallic specimens[6]. Much less material is required for FIB sample preparation and subsequent analyses compared to SANS, and the volume reduction in the hot cell was sufficient to allow for out-of-cell hand polishing of specimens in LAMDA using standard metallographic techniques with appropriate personal protective equipment (PPE) and dosimetry. Polished specimens were then installed in a remotely-operated FEI Quanta 3D Dual-Beam FIB that is housed in a lead-lined room in order to shield personnel from radiation exposure. Operators of FIB equipment are specially trained to handle the radiological samples and a focus is placed on efficient loading practices to minimize exposure to the radioactive specimens. Standard lift-out techniques[7] were then used to prepare microtip needles for APT analyses and lamellae for scanning transmission electron microscopy (STEM) investigations, examples of which are shown in Fig. 3.

APT investigations allowed for an atomic-scale study of individual precipitate composition and morphology within a very small analysis volume. Data collection was performed using the Cameca Instruments Local Electrode Atom Probe (LEAP) 4000X HR at either the Center for Nanophase Materials Sciences (CNMS) at ORNL or at the Center for Advanced Energy Studies (CAES) at INL. Both facilities are capable of handling radiological samples,

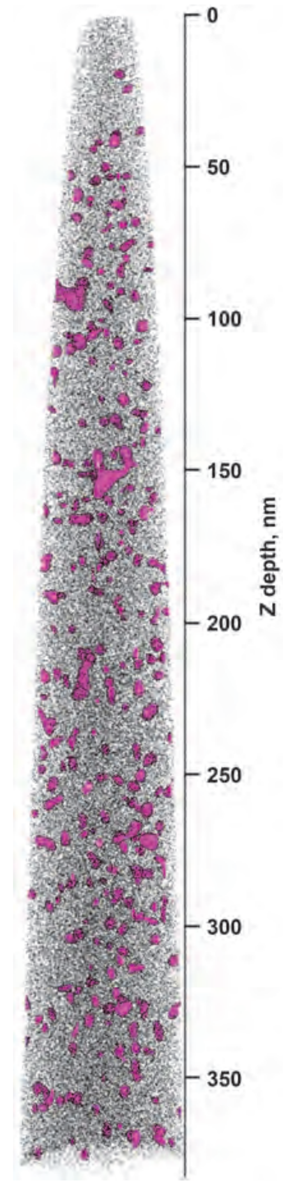


Fig. 4 — Atom probe reconstruction showing precipitate microstructure in Fe-15Cr-3.9Al alloy, irradiated to 7 dpa at 320°C. Precipitates are displayed using 34 at.% Cr concentration isosurfaces (purple) with 2% of total matrix Fe atoms shown (black).

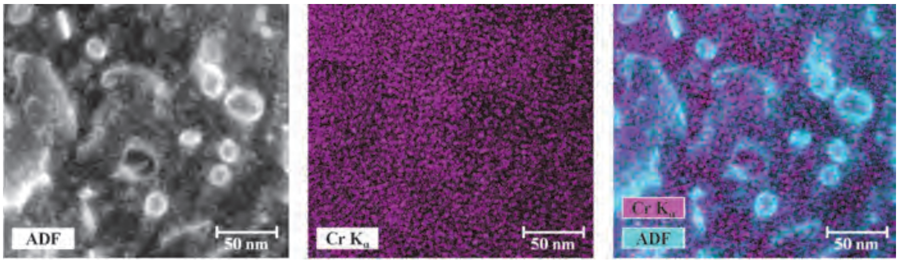
but the significant volume reduction (< 100 μm^3 on a single APT microtip coupon) promotes ease of handling when working at these laboratories. A representative reconstruction of a Fe-15Cr-3.9Al specimen irradiated to 7 dpa at 320°C is shown in Fig. 4. APT results reveal that Al additions appear to reduce the Cr content of precipitates when compared to binary Fe-Cr alloys[8], and the observed precipitate morphology trends are in agreement with those seen in the SANS study, validating the models used in the scattering analysis. Artifacts of nuclear transmutation resulting from neutron absorption are observed in the generated time-of-flight spectrum as manifested by peak ratios that are inconsistent with expected natural isotopic abundances of constituent elements. In addition, peaks corresponding to transmutation products, such as V and Mn, are also identified.

Due to the semi-coherency of α′ particles in an α-ferrite matrix, conventional diffraction contrast-based TEM techniques are not useful for studying the Cr-rich precipitates in this system, necessitating the use of chemically sensitive electron microscopy techniques[9]. As such, energy dispersive x-ray spectroscopy (EDS) was coupled with STEM to acquire spectral images of the precipitate microstructure. STEM/EDS data collection was performed on the FEI Talos F200X S/TEM located in the LAMDA facility. Due to the high efficiency of the FEI Talos system, care must be taken when performing data analysis as the energetic emissions of radioactive decay from irradiated samples can be easily detected along with the characteristic x-rays, potentially muddling the observed x-ray spectrum; this effect is significantly reduced with FIB prepared specimens over more traditional 3 mm disc specimens. The x-ray maps were collected concurrently with annular dark field (ADF) images on the [111] zone axis, allowing for simultaneous imaging of dislocation loops[10] and collection of EDS spectra in these materials. Figure 5 demonstrates that α′ precipitates appear to nucleate homogenously in the bulk material, with no bias for dislocation loop or other defect sites.

THE FUTURE OF NUCLEAR MATERIALS RESEARCH

Modern nuclear materials characterization capabilities allow for an in-depth, multifaceted investigation of nanoscale precipitation events in alloys, as demonstrated for α′ phase precipitation in neutron-irradiated Fe-Cr-Al alloys for accident-tolerant nuclear fuels applications. The resulting detailed analysis of the mechanisms and dependencies of precipitation in this system has informed design decisions within the Fe-Cr-Al alloy development program of the DOE's Advanced Fuels Campaign. Generation II Fe-Cr-Al engineering alloys with down-selected compositions based on this work are currently undergoing irradiation in HFIR to study the effect of minor alloying element additions on alloy radiation tolerance with the goal of recommending a nuclear-grade Fe-Cr-Al LWR cladding material in the near future.

Fe-Cr-Al alloys are just one of many material systems currently being developed for nuclear applications. For example, SiC is being extensively investigated both as an alternative LWR cladding material and for advanced TRISO particle fuel for gas-cooled reactor technologies. Late-blooming phases (LBP) that may embrittle reactor pressure vessel (RPV) materials are also under investigation in order to extend commercial LWR operating licenses from 60 to 80 years. Additionally, improved radiation tolerance of oxide-dispersion strengthened (ODS) and nanostructured materials is being explored for other in-core components.



(a) (b) (c)

Fig. 5 — (a) Colored STEM-ADF image, acquired on the [111] zone axis. (b) STEM/EDS map for Cr-K_α x-rays. (c) Color overlay of ADF image and EDS map. All images from the Fe-18Cr-2.9Al alloy irradiated to 7.0 dpa at 320°C.

Advancing nuclear materials technology and developing robust radiation-tolerant alloys, ceramics, and nuclear fuels is critical for both maintaining and improving the existing commercial LWR technologies in addition to enabling the construction of next-generation reactor designs with even more demanding environments for materials performance. The modern characterization capabilities discussed here have greatly facilitated high-quality characterization research on nanoscale radiation effects in materials. The continued adoption of advanced characterization techniques and equipment in addition to ample access to this equipment by nuclear materials researchers is anticipated to play a central role in enabling informed materials development for nuclear applications.

ACKNOWLEDGMENTS

Primary research funding was sponsored by DOE's Office of Nuclear Energy, Advanced Fuels Campaign of the Fuel Cycle R&D Program. Neutron irradiation of Fe-Cr-Al alloys at ORNL's HFIR user facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, DOE. APT was conducted at the CNMS, which is a DOE Office of Science User Facility and the MaCS Laboratory at the CAES at INL. A portion of this work was supported by the DOE Office of Nuclear Energy under DOE Idaho Operations Office Contract DE-AC07-051D14517 as part of a NSUF experiment. The FEI Talos F200X TEM is provided by the DOE, Office of Nuclear Energy, Fuel Cycle R&D Program and the NSUF. A portion of funding for SAB was provided by the DOE Office of Nuclear Energy's Nuclear Energy University Programs.

The authors would like to thank P.D. Edmondson and K.C. Littrell for assistance in developing analysis techniques used in this work. In addition, they thank Y. Yamamoto for providing the materials for irradiation and analysis, and R.H. Howard for leading irradiation capsule design efforts. Finally, the authors acknowledge the staff of the CAES, Irradiated Materials Examination and Testing (IMET) hot cell facility, LAMDA, and GP-SANS facilities in addition to the Thermal Hydraulics and Irradiation Engineering Group (THIEG) at ORNL for their support. **~AM&P**

For more information: Samuel A. Briggs, graduate research assistant, University of Wisconsin, is on permanent assignment at ORNL and may be reached at sabriggs2@wisc.edu or 503.302.4439.

References

1. G.S. Was and T.R. Allen, Intercomparison of Microchemical Evolution Under Various Types of Particle Irradiation, *J. Nucl. Mater.*, Vol 205, p 332-338, 1993.

2. S.J. Zinkle, et al., Accident Tolerant Fuels for LWRs: A Perspective, *J. Nucl. Mater.*, Vol 448, No. 1-3, p 374-379, 2014.

3. E.A. Little, Development of Radiation Resistant Materials for Advanced Nuclear Power Plant, *Mater. Sci. Technol.*, Vol 22, No. 5, p 491-518, 2006.

4. M.H. Mathon, et al., A SANS Investigation of the Irradiation-Enhanced α-α' Phases Separation in 7–12 Cr Martensitic Steels, *J. Nucl. Mater.*, Vol 312, No. 2-3, p 236-248, 2003.

5. K.G. Field, et al., Radiation Tolerance of Neutron-Irradiated Model Fe-Cr-Al Alloys, *J. Nucl. Mater.*, Vol 465, p 746-755, 2015.

6. C.M. Parish, et al., LAMDA: Irradiated-Materials Microscopy at Oak Ridge National Laboratory, *Microsc. Microanal.*, Vol 21, p 1003-1004, 2015.

7. M.K. Miller and K.F. Russell, Atom Probe Specimen Preparation with a Dual Beam SEM/FIB Miller, *Ultramicroscopy*, Vol 107, No. 2007, p 761-766, 2007.

8. P.D. Edmondson, et al., Irradiation-Enhanced α' Precipitation in Model FeCrAl Alloys, *Scr. Mater.*, Vol 116, p 112-116, 2016.

9. J. Ribis and S. Lozano-Perez, Orientation Relationships and Interface Structure of α-Cr Nanoclusters Embedded in α-Fe Matrix After α-α' Demixing in Neutron Irradiated Oxide Dispersion Strengthened Material, *Mater. Lett.*, Vol 74, p 143-146, 2012.

10. C.M. Parish, et al., Application of STEM Characterization for Investigating Radiation effects in BCC Fe-based Alloys, *J. Mater. Res.*, Vol 30, No. 9, p 1246-1274, 2015.

ADVANTAGES OF 3D LASER SCANNING CONFOCAL MICROSCOPY

3D laser microscopy is opening new areas of study for metallic alloys and coatings in power generation applications.

John Shingledecker, John Siefert, and Daniel Purdy,* Electric Power Research Institute, Charlotte, N.C.*
Jonathan Tedesco and Andrew Szafarczyk, Keyence Corp., Itasca, Ill.

Laser scanning confocal microscopy (Fig. 1) provides non-contact, nanometer level profile (depth) resolution to measure surface roughness and film thickness data on a variety of materials and components. This, along with the possibility of high resolution and large area mapping, are leading to new opportunities for materials characterization.

To measure surface profiles, traditional contact methods risk surface damage, particularly for soft materials. Additionally, surface changes smaller than the stylus tip cannot be measured or quantified. A laser microscope can be used on soft and hard materials alike because it is non-contact and the laser beam diameter is much smaller than a stylus, which provides more accurate data. The laser also makes it easier to measure surfaces on an entire area without the need to properly place a measuring device (Fig. 2).

Scanning electron microscopy (SEM) is typically used when resolution beyond optical microscopy is required. A key advantage of laser microscopy compared to SEM is that high resolution images (with magnifications up to 28,000×) are possible in air without additional surface preparation. This makes laser microscopy a faster option for imaging materials and components. With an automated stage and image stitching capabilities, large-area high-resolution images can be obtained while retaining spatial distances and associated 3D depth information, which is not possible with a SEM.

Fig. 1 — Keyence VK-X laser microscope.

Laser microscopy is used in a variety of industries. The ability to measure depth and soft/optical materials in a non-contact manner is particularly well suited for the electronics industry and chemical industries including a wide range of applications in CCD microlens characterization, MEMs measurements and failure investigations, silicon wafer roughness, and film thickness characterization of transparent materials such as glasses and lenses. Laser microscopy is also widely used for metals characterization, specifically surface roughness. In addition, the unique attributes of the laser microscope have led to expanded characterization applications for materials in power generation equipment in recent years.

CASE STUDIES

At the Electric Power Research Institute's (EPRI) laboratories in Char-

*Member of ASM International

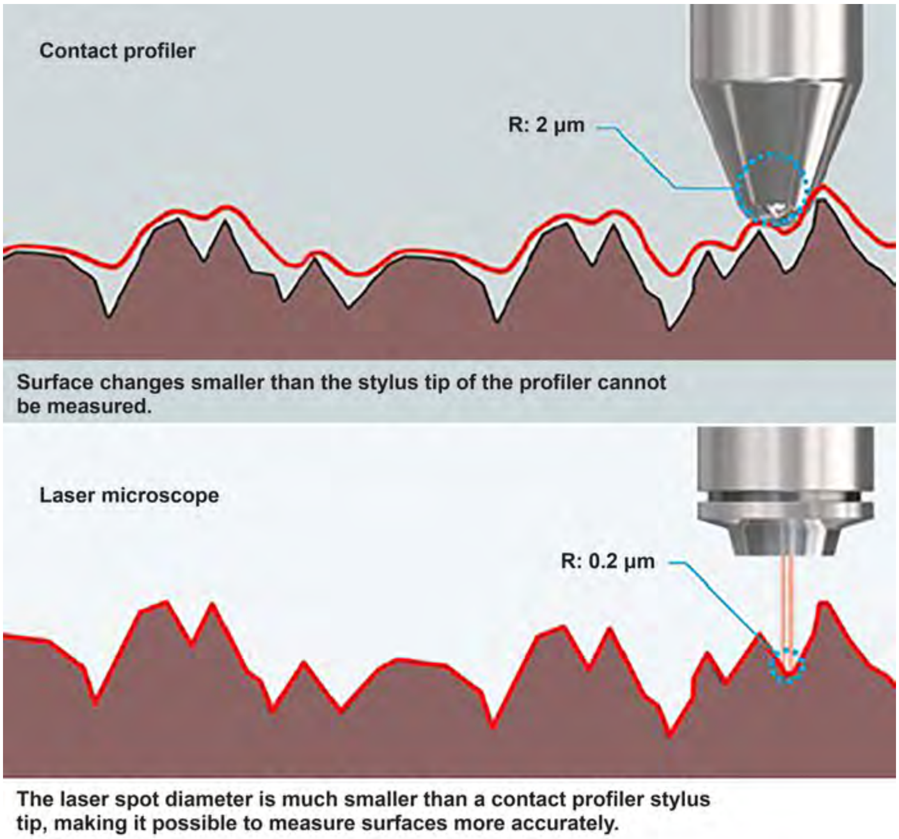


Fig. 2 — Comparison between contact profiler and laser microscope.

lotte, N.C., a Keyence VK-X160K has augmented, and in some cases replaced, metallic alloy characterization traditionally done using either optical microscopy or scanning electron microscopy. The VK-X160K uses a red semiconductor laser (658 nm wavelength) with maximum z-axis (depth) display resolution of 5 nm, maximum magnification of 19,200×, and motorized x-y stage with a range of 100 × 100 mm. The microscope's ability to show subtle differences in surface relief, develop high-resolution large area metallurgical maps, and quantify surfaces through non-contact profilometry has opened up new avenues in research.

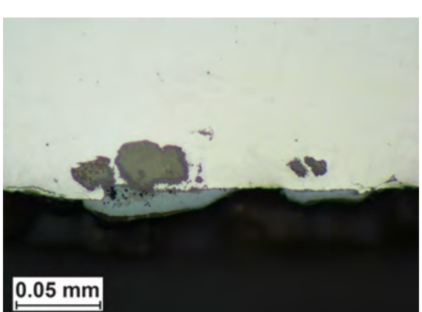


Fig. 3a — Optical image of steam-side scale.

Inner diameter (ID) shot-peening (SP) of austenitic stainless steel boiler superheater (SH) tubing has been used to manage short-term (<10,000 hour) steam-side oxidation and exfoliation in utility boilers[1]. To improve understanding of long-term behavior of this technique, EPRI received SP-304H SH tubing after exposure in a utility boiler for ~24,500 hours. Using careful sample preparation including a final ~20 hour vibratory polishing step, the optical image of the ID oxide scale was produced in Fig. 3a.

Internal and external oxidation in the form of nodules is clearly evident. However, little information about grain structure and oxide morphology is revealed, which is critical to understanding material behavior. Chemical etching could be used to evaluate such features, but experience shows this leads to artifacts in the oxide scales. Using the same as-polished sample and the laser microscope, large area and small area high-resolution images were obtained including detailed characterization of nodule regions. Figure 3b shows results at approximately 2000×. On the as-polished material, the grain structure of steel is observed.

Furthermore, slip bands are evident from the original SP process as well as clear evidence of multiple oxide scale morphologies and voids forming at the inner and outer oxide surface. Comparing Figs. 3a and 3b clearly shows that the laser microscope offers enhanced possibilities for detailed understanding of materials without the need for surface etching, which can damage scales and potentially lead to erroneous results.

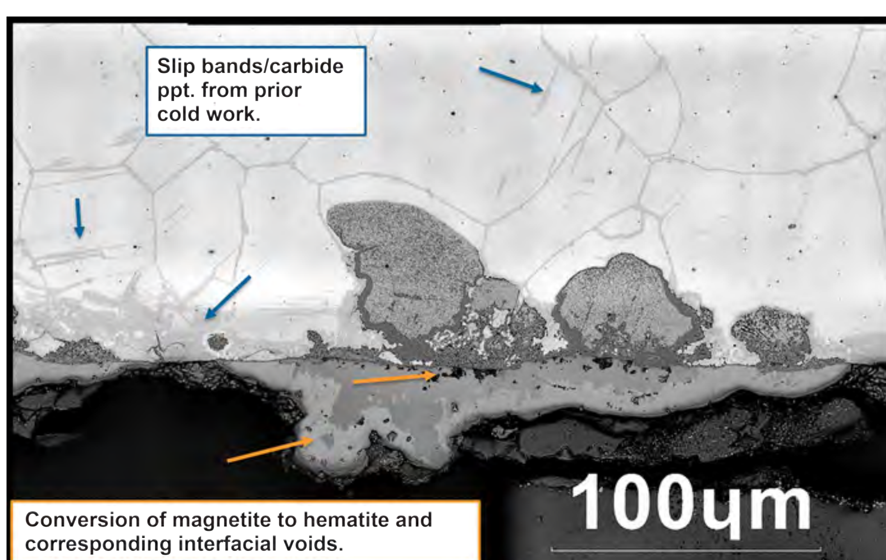


Fig. 3b — Unetched (as-polished) laser image of the inner diameter surface of a shot-peened 304 H stainless steel superheater tube after ~24,500 hours of service.

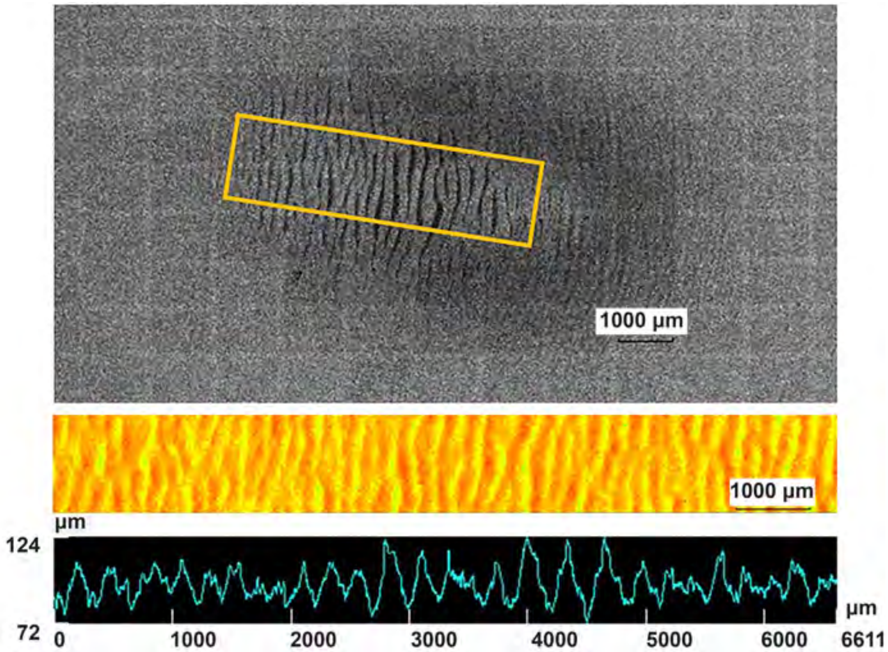


Fig. 4 — High resolution 3D color height map used to characterize a high-temperature erosion test coupon.

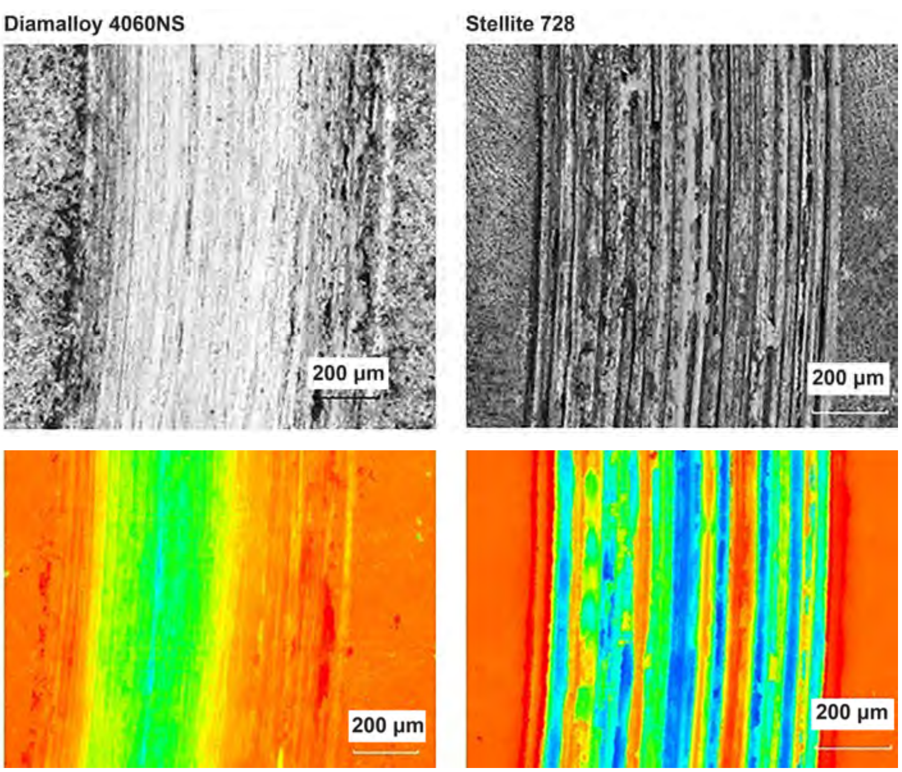


Fig. 5 — Quantifiable differences in high temperature wear behavior of potential high-temperature wear resistant coatings/claddings for valves after high-temperature laboratory wear testing.

LASER MICROSCOPY FOR HARDFACING ALLOYS

Tribology is the science of interactions between two bodies. In general, these interactions damage one or both components. Characterizing the rate of how this damage accumulates is critical for providing materials comparisons under expected service conditions. EPRI has been studying advanced hardfacing alloys for high-temperature valves and alternative materials and coatings for erosion resistance.

The value of laser microscopy in tribological examinations is that the microscope can quickly provide information above and beyond traditional profilometry techniques that generally provide a single 2D trace, which provides a linear profile or maximum depth of damage. Alternatively, a laser microscope can rapidly provide a detailed 3D topography map of the entire specimen and enable valuable and quantifiable parameters to be determined. Examples of these parameters include maximum and average depth of damage and total volume lost, as well as various surface textures such as adhesion.

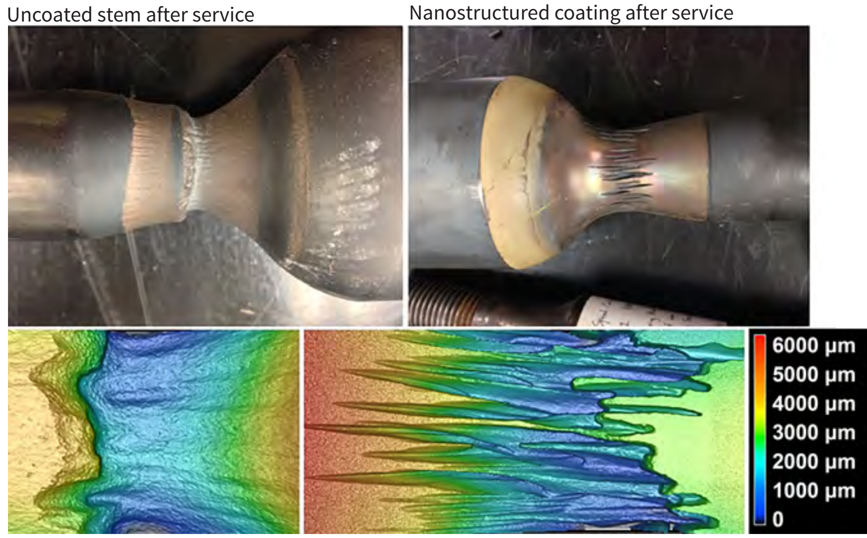
In solid particle erosion, 3D scanning via laser microscopy was used to quantify the above parameters in support of development of the ASTM G211 high-temperature erosion test standard[2]. In addition, the level of detail from laser microscopy can illuminate topographical features that may be relevant to the success or failure of a material or related to the test equipment's inconsistencies. Analysis of the sample in Fig. 4 shows surface perturbations forming a series of ripples. In this case, comparisons between the 3D laser images and 2D contact methods found that traditional 2D methods can under or overestimate actual metal erosion rates by more than 20%.

In elevated temperature sliding wear for advanced cycle power generation, various hardfacing materials behave differently as illustrated in Fig. 5. Here, the Diamalloy 4060NS HVOF spray coating shows a consistent low wear rate while the Stellite 728 experimental GTAW cobalt alloy shows significant gouging and adhesion under the same test conditions. The 3D height maps produced from laser microscopy can be used to reliably quantify these differences. In fact, research using these techniques has shown that current ASTM standards that only rely on optical comparisons can be explained in quantifiable terms[3].

EVALUATING EROSION DAMAGE

Erosion damage in steam turbine valve stems presents a reliability challenge to utilities. Steam-grown oxides appearing on the inside surfaces of high-temperature boiler tubes and pipes occasionally exfoliate and are entrapped in the steam system. In the valve stem, the combination of small hard oxide particles and high velocities can cause severe erosion damage in certain cases. Utilities and EPRI have been investigating material options for reducing the severity of erosion damage to extend maintenance intervals.

Figure 6 compares two valve stems from the same unit after nominally equivalent operating history (hours, starts, and cycles). In this case, the large depth of field of the laser microscope was used to quantify both the amount and depth of metal loss after service due to localized erosion. The data shows 1.6 to 2.4 times improvement in performance for a valve stem that included a ~30 μm thick, nanostructured TiSiCN coating. Without a tool like the laser microscope, quantification of materials performance would not be possible due to the tortuous nature and large area of the erosion damage.



	Area measured (mm^2)	Eroded volume (mm^3)	Eroded volume per 100 mm^2 (mm^3)	Average depth of erosion
Old stem (no coating)	49	303	618	6.2
Stem with 30 micron thick nanocoating	75	194	259	2.6
Improvement in erosion resistance due to coating		1.6	2.4	2.4

Fig. 6 — Laser microscope measurements on large steam turbine valve stems after service comparing performance of uncoated and coated stems after nominally equivalent operating history.

FUTURE POSSIBILITIES

3D laser microscopy is opening new areas of study for metallic alloys and coatings in power generation applications. Further studies are currently being conducted to quantify creep void distributions in large creep samples and service-exposed components on unetched samples, examine deformation characteristics of small sample tests including identification of size and location of cracking to build more accurate models of material behavior, and examine hardness indents to accurately measure depth and changes in surface profiles. The rapid, accurate, and high resolution quantification of materials is a unique combination that is hoped to be exploited in other areas to replace or augment optical and scanning electron microscopy and eliminate the need for 2D surface contact surface profilometry. ~AM&P

For more information: John Shingledecker is senior program manager, cross sector technologies, Electric Power Research Institute, 420 Hillview Ave., Palo Alto, CA 94304, 704.595.2619, jshingledecker@epri.com, www.epri.com.

References

1. A.S. Sabau, et al., Managing Oxide Scale Exfoliation in Bilers with TP347H Superheater Tubes, *Advances in Materials Technology for Fossil Power Plants*, Proceedings from the Seventh International Conference, Oct. 22-25, 2013, Waikoloa, Hawaii. Published by ASM International, p 821-831, 2014.

2. V.P. Swaminathan, J.S. Smith, and D.W. Gandy, High-Temperature Solid Particle Erosion Testing Standard for Advanced Power Plant Materials and Coatings, *Advances in Materials Technology for Fossil Power Plants*, Proceedings from the Seventh International Conference, Oct. 22-25, 2013, Waikoloa, Hawaii. Published by ASM International, 2014.

3. J. Siefert and S. Babu, Experimental Observations of Wear in Specimens Tested to ASTM G98, *Wear,* Vol 320, p 111-119, 2014.

TECHNICAL SPOTLIGHT

BIOMATERIALS TESTING AND CHARACTERIZATION

As scientists and engineers continue to develop and investigate replacement tissues for patient disease, injury, and aging, proper mechanical characterization of biological materials is critical.

Biological materials, both natural and engineered, are inherently multifaceted. As such, the biomaterials that make up one individual will not have the exact same mechanical properties as those that make up another. Given the natural variability in biological tissues—such as bone, tendons, ligaments, and hair—achieving consistent and repeatable metrics to effectively characterize the mechanical properties of a biomaterial may be challenging. In the physical materials testing industry, biomaterials can generally be broken down into two categories: soft and hard tissues. Mechanical characterization of both natural and engineered biomaterials is usually achieved using a combination of both static and fatigue testing.

Biological materials are *viscoelastic* because they feature both viscous and elastic properties. Viscosity is the measure of a fluid's resistance to flow, while elasticity is the tendency of a material to return to its original state after undergoing deformation. In mechanical terms, elasticity is modeled using a spring and viscosity is modeled using a dashpot, which resists motion via friction. Viscoelastic materials exhibit time-dependent properties and thus exhibit both creep and stress-relaxation.

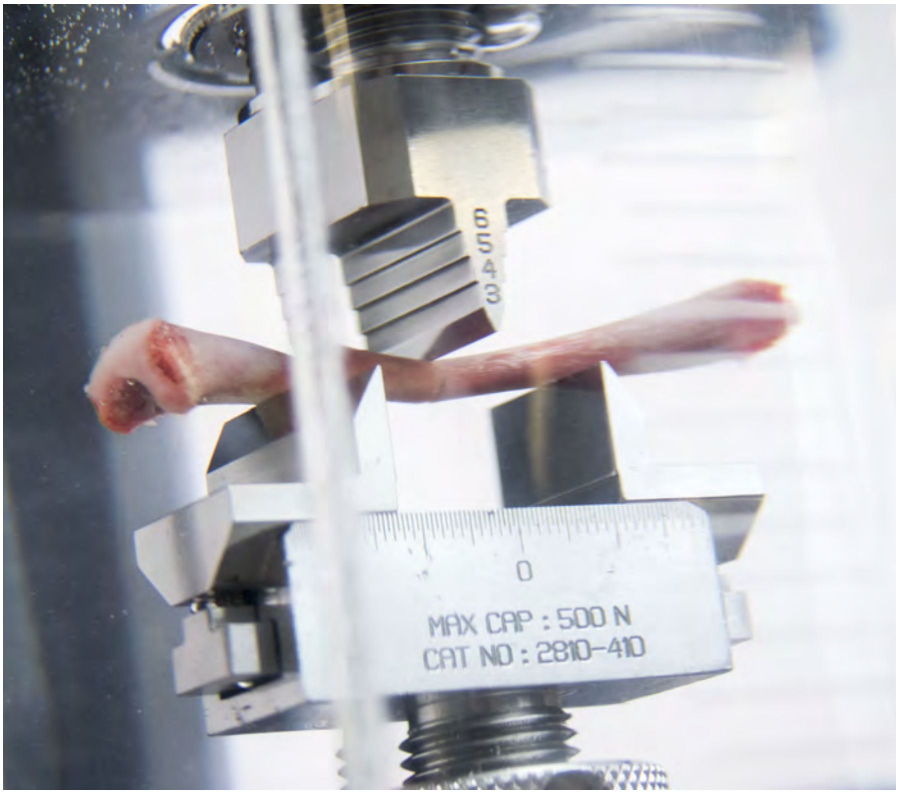
Tensile testing of biological tissues.

MECHANICAL TESTING TECHNIQUES

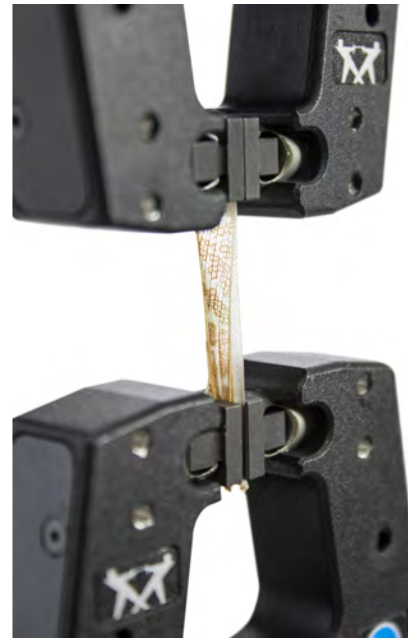
Static mechanical testing generally refers to monotonic compression, tensile, and flexural testing. However, it also encompasses simple cyclic testing, creep, and stress-relaxation testing, which can help properly characterize the viscoelastic properties of biological materials. When testing biomaterial strength, simple cyclic testing is often conducted at the beginning of the test and is referred to as pre-cycling. Pre-cycling soft tissues before failure helps align fibers to condition the material.

Creep testing is a type of static test that involves holding a specimen in tension under a constant load. In a purely elastic material, an applied load in tension or compression will create some displacement in a material that will not change over time. For example, if a weight were applied to a spring and held constant over time, the initial extension of the spring would not increase or decrease no matter how long the weight is kept on the spring. This behavior is expected in purely elastic materials. However, in viscoelastic materials such as a tendon containing mostly collagen fibers, under constant load, a tendon's strain or material extension will increase over time. Materials that exhibit creep will undergo plastic deformation under constant load.

Stress-relaxation is a static test that involves holding a specimen in tension at a constant strain or displacement. For example, if a purely elastic spring is pulled to a displacement, the resulting force or stress on the spring would remain constant over time. In materials that exhibit stress-relaxation, stress will decrease or relax in response to the same amount of strain over time. Biological materials primarily



Flexural testing of rat bone.



Tensile testing of an insect wing.

composed of smooth muscle, such as the bladder, will exhibit a high level of stress relaxation.

Fatigue testing, also known as dynamic testing, helps to characterize the lifetime properties of materials that experience constant mechanical loading. For example, a human heart beats approximately 35 million times per year. In order to ensure that an engineered tissue material used in the heart will last a minimum of 10 years, the tissue needs to be subjected to at least 350 million cycles of mechanical loading. In order to complete this testing in a reasonable amount of time, the testing machine needs to run at a high frequency. Using a dynamic testing machine at 100 Hz, a researcher could simulate 10 years of wear on a material in just a few weeks. Fatigue testing of biomaterials helps ensure that the material will not yield, become damaged, or fail within the required lifetime.

TESTING PARAMETERS

While mechanical testing of tissues can range from a quick pull or failure test to a months-long fatigue test, all testing methods are critical to understanding the bulk mechanical properties of biomaterials and predicting how those materials will behave in the human body. In addition to the variability inherent to biological materials, experimental setup for biomaterials testing also presents a challenge. Testing biological materials can be challenging due to the need for highly sensitive force measurement, delicate specimens that are difficult to grip, and strain measurement that is often impossible with traditional extensometers.

Highly sensitive force measurement is needed for mechanical testing of soft biomaterials. Soft tissue specimens are usually small and the loads required to pull these materials apart are generally in the gram-force range. With biological materials that require pre-cycling before pull to failure, it is important to ensure that the force transducer is not only verified for accuracy in the load range for pre-cycling, but also has the capacity to measure force at specimen failure.

Determining the proper gripping solution for testing biomaterials is crucial to achieving consistent results. The delicate nature of soft biomaterials in combination with testing in physiological conditions makes gripping a challenge. Testing tissues in physiological conditions to properly mimic the environment inside the human body is relatively easy to achieve using a heated bath. However, consistently inserting a specimen in a set of grips while submerged in a bath can be difficult. To save time and make sure specimen alignment is repeatable, using a bath with a lifting mechanism is recommended so that the specimen can be inserted into the grips and then submersed as the bath raises into place. When choosing a gripping mechanism for soft tissues, side-acting grips that are manual, spring actuated, or pneumatically closed generally work well. In addition, the gripping face should feature a material with high friction to avoid specimen slippage while minimizing the necessary clamping force.

When tensile testing soft tissues to failure to determine maximum force or maximum strain at break, calculating strain from the machine's extension is widely accepted as suitable. This method of measuring strain is calculated by taking the machine's crosshead extension data and dividing it by the initial grip separation. However, for hard tissues such as bone or dental enamel, using an extensometer to measure strain is required. Further, for both hard and soft tissues, it is considered best practice to use an extensometer to measure properties such as modulus. During a tensile or compression test, a force is applied to a material causing deformation. For hard tissues with high stiffness, the material will deflect under load and some test system deflection (compliance) will also occur. To avoid having system compliance factored into the specimen's strain measurement, using an extensometer is recommended. Because soft biomaterials are delicate and fragile, a noncontacting extensometer such as a laser or video model is required.

SUMMARY

Mechanical characterization of both natural and engineered biomaterials is done through a combination of static and fatigue testing to understand both bulk and lifetime material properties. Given the natural variability of biomaterials, it is critical that mechanical testing design reduces test variables such as low force measurement, specimen gripping, and strain measurement. While data sheets with mechanical test specifications are available for materials such as polymers, elastomers, and metals, biomaterial mechanical properties are still largely unknown or only published academically. As scientists and engineers continue to develop and investigate replacement tissues for patient disease, injury, and aging, proper mechanical characterization of biological materials is critical. **~AM&P**

For more information: Elayne Gordonov is biomedical assistant market manager, Instron, 825 University Ave., Norwood, MA 02062-2643, 800.877.6674, www.instron.us.

Tensile testing of hydrogels using a video extensometer.

METALLURGY LANE

Metallurgy Lane, authored by ASM life member Charles R. Simcoe, is a yearlong series dedicated to the early history of the U.S. metals and materials industries along with key milestones and developments.

THE DECLINE OF THE INTEGRATED STEEL INDUSTRY–PART I

FROM POOR LABOR RELATIONS AND DELAYED MODERNIZATION TO AN UNBALANCED PRODUCT MIX AND INCONVENIENT MILL LOCATIONS, THE U.S. STEEL INDUSTRY BEGAN ITS STEADY DECLINE IN THE LATE 1950s.

The strike of 1959 was a watershed moment in the history of the U.S. integrated steel industry. The strike itself was not the main event, but it exposed industrywide problems and failures that had existed for many years. Among them were poor labor relations, delayed modernization, an unbalanced product mix, and inconvenient mill locations in relation to the market. Other factors included steady price increases to cover union contracts rather than productivity improvements, pollution that had to be controlled by EPA rulings, and management staff who were paid the highest incomes of any industry.

In the early 1950s, David J. McDonald replaced Philip Murray, longtime president of the steelworkers union. McDonald was expected to be a moderate negotiator due to his limited experience in the steel union. However, he had a bitter rivalry with Walter Reuther of the auto union who had been elected head of the Congress of Industrial Organizations (CIO) after Murray. In subsequent negotiations, McDonald tried to keep the steelworkers ahead of the autoworkers: By 1959, steelworkers were earning roughly 38 cents per hour more than autoworkers (15% more).

Even so, McDonald never had the same support of union members as Murray did, so he always overreached in his attempt to keep them loyal. A serious strike took place in 1956 with workers out for 35 days. The final settlement included Clause 2b—for Bethlehem only—that gave the union a guarantee that new technology would not reduce the number of employees. This would be the source of labor problems at Bethlehem for years to come.

STRIKE OF 1959 AND LAGGING TECHNOLOGY ADOPTION

The 1959 strike was the longest in steel history at 116 days. Grievances included wages, pensions, and paid holidays. Bethlehem fought unsuccessfully to remove Clause 2b, and the strike became a pivotal point in the history of the U.S. steel industry. As the strike continued, customers became seriously concerned about supply sources and their only recourse was imported steel. The U.S. had been a net exporter of steel during the 20th century, but in 1959 imports exceeded exports for the first time with total imports reaching 4.7 million tons.

From 1957-1962, steel use declined 8% per capita although the total market was still growing slowly. At the same time, the steel industry was trying to upgrade and expand capacity. The lack of modern technology adoption was evident when Bethlehem built a new open hearth shop in 1958 at Sparrows Point for $200 million. Four years earlier, a small steel company in Michigan had installed the first basic oxygen furnace (BOF) and Jones & Laughlin Steel Corp. (J&L) installed these furnaces in 1959. The BOF process was invented in Austria after World War II. BOF vessels were similar to the earlier Bessemer converters, except they used oxygen instead of air to remove carbon from cast iron. A heat of steel could be processed in less than an hour compared to six or eight hours for open hearth furnaces. Importantly, BOFs accomplished this with fewer workers and produced a superior product. New plants built in Europe and Japan to replace those destroyed in the war implemented new BOFs.

Philip Murray, first president of United Steelworkers of America. Courtesy of usw.org.

There were 21 BOFs in operation before United States Steel Corp. (USS) built its first two at the Duquesne plant in Pittsburgh in 1963, and 29 units when Bethlehem Steel installed its first two at Sparrows Point in 1964, replacing 40-year-old open hearths. That same year, the company built two BOFs at Lackawanna, which produced 2.5 million tons per year. The U.S. produced only 17% of its steel using BOFs in 1965, while Japan produced 55%. As late as 1970, this ratio was 48% for the U.S. and 79% for Japan.

Reluctance to accept new technology extended to the continuous casting process—another European development made after WWII. Molten metal delivered from the blast furnace was

David J. McDonald, president of the steelworkers union during the strike of 1959. Courtesy of usw.org.

teemed into a machine that channeled the metal through a container where it solidified and was prepared for the rolling mills. This process replaced the casting of metal into large molds and reducing them by forging or rolling in preparation for the finishing mills.

LOFTY PROJECTS AMID RISING IMPORTS

In 1961, Bethlehem Steel built a central research laboratory to compete with USS who had built its own lab in 1957. Bethlehem built a huge campus on 1000 acres bulldozed from a mountain. The $35 million project—eight buildings and a reflecting pool—was not complete until 1969. It accommodated over 1000 research engineers and support personnel and was named Homer Research Laboratory after Bethlehem's chairman, Arthur B. Homer.

U.S. Steel corporate headquarters in downtown Pittsburgh.

Bethlehem Steel's Homer Research Laboratory. Courtesy of lehigh.edu.

From 1960 to 1970, imports increased from 8% to 17% of total U.S. steel consumption. Most of the imported steel came from Japan, which had increased production from 25 million to 100 million tons annually from nine new plants located at tidewater ports for ease of importing raw materials and exporting finished product.

In 1964, Bethlehem decided to build a new mill at Burns Harbor, Ind., featuring a new production layout and state-of-the-art equipment. It became the most efficient in the country. The first phase of construction was limited to finishing operations and ingots were shipped in from Johnstown and Lackawanna. Blast furnaces and BOFs were added later to make it a fully integrated plant.

CONSOLIDATIONS AND LAYOFFS

The first integrated steel company to be affected by the new market with increasing imports was J&L. They sold an interest in the company in 1968 to a Texas conglomerate, Ling-Temco-Vought (LTV)—who had zero experience in owning a steel mill. A few years later, the company completed total ownership of J&L and called it the LTV Steel Division. Thus, J&L became the first casualty in the decline of the integrated steel industry. The next company to disappear from the industry was Youngstown Iron Sheet and Tube Co. (YS&T). It closed the Campbell plant in Youngstown in the late 1970s and terminated 5000 employees. In 1979, it sold all remaining plants to LTV and joined J&L in the LTV Steel Division.

Though the winds of change had turned, both USS and Bethlehem built

Martin Tower, headquarters of Bethlehem Steel in Bethlehem, Pa.

new corporate headquarters in the early 1970s. USS was first, constructing a $50 million, 64-story office in downtown Pittsburgh. In 1972, Bethlehem built Martin Tower in Bethlehem, Pa., named for its chairman, Edmund Martin. The new 21-story tower cost $35 million. The same year, Bethlehem had peak production of 16 million tons of finished product and earnings of $200 million. Employment reached 120,000 in the mid 1970s, but 20 years later only 20,000 workers remained.

USS closed its Youngstown plant in 1979, a plant with open hearth furnaces and finishing mills as old as 1900 and a sheet mill installed in 1935. It had become obvious that the mills were too outdated to compete in the new market conditions.

Bethlehem finally built a continuous caster at Burns Harbor in 1975 after abandoning its first attempt to continuous cast slabs in 1969 at the Johnstown plant. At this time, the Japanese were continuous casting 46% of their steel capacity compared with 11% for the U.S. integrated steel industry. Foreign imports held steady at roughly 15% during the 1970s, but increased to 22% during the next decade.

In 1977, Bethlehem reported a loss of nearly $500 million and began closing parts of the Johnstown and Lackawanna plants. The company terminated 2500 white-collar workers that year, 800 from the Homer Research Laboratory and Martin Tower facilities built just seven years earlier.

For more information: Charles R. Simcoe can be reached at crsimcoe1@gmail.com.

TSS
Thermal Spray Society
ASM INTERNATIONAL

NOVEMBER/DECEMBER 2016 | VOL. 11 | ISSUE 4

iTSSe

INTERNATIONAL THERMAL SPRAY & SURFACE ENGINEERING

THE OFFICIAL NEWSLETTER OF THE ASM THERMAL SPRAY SOCIETY

THERMAL SPRAY COATINGS IN EMERGING TECHNOLOGIES


WE
DO
IT
ALL

TSS
Thermal Spray Society
ASM INTERNATIONAL

NOVEMBER/DECEMBER 2016 | VOL. 11 | ISSUE 4

iTSSe

INTERNATIONAL THERMAL SPRAY & SURFACE ENGINEERING

THE OFFICIAL NEWSLETTER OF THE ASM THERMAL SPRAY SOCIETY

International Thermal Spray & Surface Engineering™ is published quarterly by ASM International®, 9639 Kinsman Road, Materials Park, OH 44073; 440.338.5151; asminternational.org. Vol. 11, No. 4.

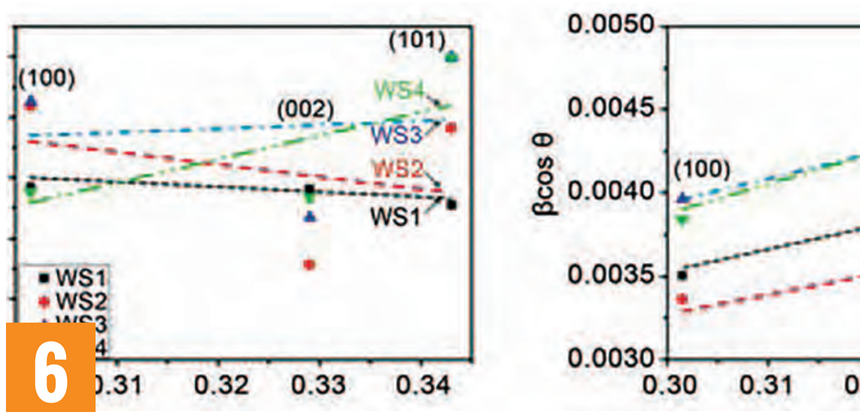
EDITORIAL OPPORTUNITIES FOR iTSSe IN 2017

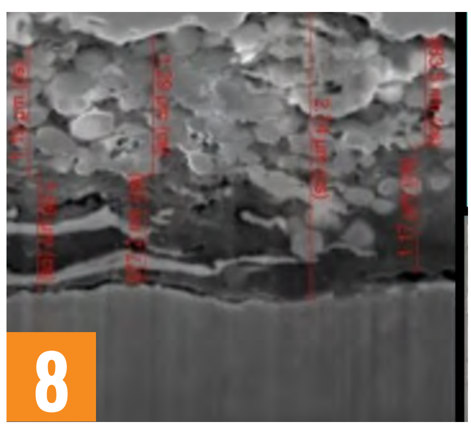
The editorial focus for iTSSe in 2017 reflects established applications of thermal spray technology such as power generation and transportation, as well as new applications representing the latest opportunities for coatings and surface engineering.

February Issue:
Aerospace Industry/Military Applications

To contribute an article, contact Julie Lucko at julie.lucko@asminternational.org.

To advertise, contact Erik Klingerman, at erik.klingerman@asminternational.org.





DEPARTMENTS

ABOUT THE COVER

The electric arc wire spray process uses two metallic wires, usually of the same composition, as the coating feedstock. The two wires are electrically charged with opposing polarity and fed into the arc gun at controlled speeds. When wires are brought together at the contact point, opposing charges create enough heat to continuously melt the wire tips. Compressed air is used to atomize molten material and accelerate it onto the workpiece surface to form the coating.

A HISTORY LESSON

Kay

Years ago, trying to be an encouraging father, I bought my children a book that outlined history in chart form showing different time periods with notable events including influential people, politics, wars, science, medicine, notable discoveries, art, music, and much more. My baseline to give some meaning was the white oak tree in my backyard in Ohio that is approximately 350 years old. I should also mention that at the time I was doing this, the internet was just beginning!

In that same vein, one TSS founding father and influence most of us know, Dr. Christopher Berndt, has undertaken similar initiatives to outline the history of thermal spray as well as influential people and companies. For many of us in the field, the history is rather fascinating when you see the impact and growth through decades of thermal spray use showcasing the advancements in science, technology, and everyday applications.

With this in mind, during the remainder of this year and into 2017, leading technologies focused on state-of-the-art advancements will be presented first at the Cold Spray Conference in Edmonton, Canada, and next year at the annual International Thermal Spray Conference (ITSC 2017) in Dusseldorf. Throughout this issue, you will find advancements that may influence thermal spray and cold spray technologies and how we think about these topics for decades to come. And the progress never stops—thanks to the high energy group of TSS volunteers and associates around the globe.

Just think, perhaps one day general history books will have a section devoted to thermal spray that children will bring home and share with their parents.

Happy spraying!

Charles M. Kay
iTSSe co-editor
ASB Industries

REVIEW ARTICLE AVAILABLE ON COLD SPRAY DEPOSITION

The September issue of *International Materials Reviews* features the article "The Unique Abilities of Cold Spray Deposition" by Victor Champagne and Dennis Helfritch. The review is useful to those new to cold spray, members of the cold spray community, and product developers in need of cold spray capabilities. Descriptions of equipment and techniques used to create cold-spray deposits are helpful to those seeking to produce products requiring similar characteristics. The journal is co-owned by ASM International and IOM3. Visit http://tinyurl.com/j5lp5lr to purchase and download the article.

NOMINATIONS SOUGHT FOR ASM THERMAL SPRAY SOCIETY BOARD

The ASM TSS Nominating Committee is currently seeking nominations to fill three board member positions. Candidates may be from any segment of the thermal spray community. Nominees must be a member of the ASM Thermal Spray Society and must be endorsed by five TSS members. Board members whose terms are expiring may be eligible for nomination and possible re-election on an equal basis with any other nominee. Nominations must be received no later than **March 1, 2017.** Forms can be found at tss.asminternational.org. For more information, contact Christian Moreau, nominating committee chair, at christian.moreau@concordia.ca.

SEEKING STUDENT MEMBERS FOR TSS BOARD

The ASM Thermal Spray Society is seeking applicants for the two student board member positions. Nominations are due by **March 1, 2017.** Students must be a registered undergraduate or graduate during the 2016-2017 academic year and must be studying or involved in research in an area closely related to the field of thermal spray technology. For more information, visit tss.asminternational.org.

TSS ANNOUNCES NEW BOARD MEMBERS

The ASM Thermal Spray Society elected officers and board members for 2016. **Douglas G. Puerta,** director–aerospace product qualifications, Element Materials Technology, succeeds as president of TSS, while **Christian Moreau, FASM, TS-HoF,** professor, Concordia University, remains on the board as immediate past president. **André McDonald,** associate professor, University of Alberta, is elected vice president. Officers serve a two-year term. In addition, **Dan C. Hayden,** president, Hayden Corp., continues on the board as secretary/treasurer for a one-year term.

New board members elected for a three-year term include **Komal Laul,** senior process engineer, Chromalloy; **James A. Ruud,** materials scientist, General Electric; **Maurice E. VandenBergh,** president, VandenBergh & Associates Inc.

Two student members were also appointed to the board for a one-year term: **Ankur Gupta,** University of Central Florida, and **Gregory Smith,** Stony Brook University.


Puerta


Moreau


McDonald


Hayden


Laul


Ruud


VandenBergh


Gupta


Smith

TSS PRESIDENT'S AWARD FOR MERITORIOUS SERVICE

This award recognizes exceptional service to the Thermal Spray Society and community. **Jeganathan Karthikeyan, FASM,** (right) director of research and development, ASB Industries, received the 2016 TSS President's Award for Meritorious Service from TSS President **Christian Moreau** on June 20 at a company picnic held in his honor.

ASM DISTINGUISHED LIFE MEMBER AWARD

Albert Kay, FASM, president, ASB Industries, Barberton, Ohio, received this year's ASM Distinguished Life Member Award "in recognition of outstanding innovation and early commercialization of emerging thermal spray technologies including high-velocity oxyfuel and cold spray; exemplary development of a business model that gained worldwide recognition; and for dedication and unselfishness, unstinting personal effort, and leadership on behalf of the Thermal Spray Society and ASM International." The award was established in 1954 and is conferred on leaders who have devoted their time, knowledge, and abilities to the advancement of the materials industries. His award will be presented during a future Akron Chapter meeting.

Kay

MAKE PLANS TO ATTEND THE NORTH AMERICAN COLD SPRAY CONFERENCE

The ASM Thermal Spray Society and the Canadian Cold Spray Alliance will jointly present the 2016 North American Cold Spray Conference from November 30 through December 1 at Alberta Innovates – Technology Futures, Canada. The biennial event showcases the latest technical insights from international experts in industry, government, and academia on new technology, exciting applications, and cutting-edge research. The conference also features a tabletop exhibition and ample networking opportunities for face-to-face interaction.

The two-day multidisciplinary program includes invited presentations from prominent speakers in the areas of additive manufacturing; applications—case studies, cost analysis, and optimization; equipment and quality control; fundamentals: physics, science, modelling, and characterization; science and engineering of powders; and the brand new, young professionals' student presentations. Visit asminternational.org/coldspray for more information.

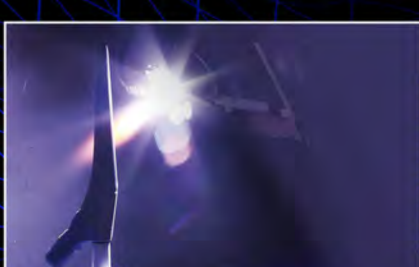
MEC®
SINCE 1967

TURNKEY SOLUTIONS FOR THERMAL SPRAY

7 AXES ROBOTIC FLAME SPRAY COATING

8 AXES HVOF & PLASMA SPRAY COATING

CNC WIRE FLAME SPRAY SYSTEM FOR OEM



ROBOTIC PLASMA SPRAY COATING FOR MEDICAL IMPLANTS

8 AXES ROBOTIC HVOF COATING SYSTEM

9 AXES ROBOTIC HVOF PLASMA & FLAME SPRAY

Q1 API Spec Q1 Registered
MECPL REG NO:- Q1-1731

ASM INTERNATIONAL
CRISIL RATINGS
CE
KUKA

METALLIZING EQUIPMENT CO. PVT. LTD.
E-101, M.I.A., BASNI II - PHASE, JODHPUR 342005 (INDIA)
Ph. : +91-291-2747601 Fax : +91-291-2746359
sales@mecpl.com; marketing@mecpl.com; trade@mecplcom
www.mecpl.com

COLD SPRAY: ADVANCED CHARACTERIZATION METHODS—X-RAY DIFFRACTION

This article series explores the indispensable role of characterization in the development of cold spray coatings and illustrates some of the common processes used during coatings development.

Dheepa Srinivasan, GE Power, GE India Technology Center, Bangalore

X-ray diffraction (XRD) is a versatile tool for preliminary characterization of phases in cold spray coatings. A collimated beam of x-rays with a wavelength of 0.5 to 2 Å is diffracted by the crystalline phases in the specimen according to Bragg's law. The diffraction pattern identifies the material's structural properties, such as structure, lattice parameter, strain (which is measured with great accuracy from the diffraction peaks), epitaxy, and size and orientation of the crystallites. The probing depth is a few micrometers to tens of micrometers, depending on the absorption coefficient of the material for the particular wavelength of the x-ray probe.

Cold spray coating analysis starts with powder characterization, followed by as-sprayed and heat treated coatings.

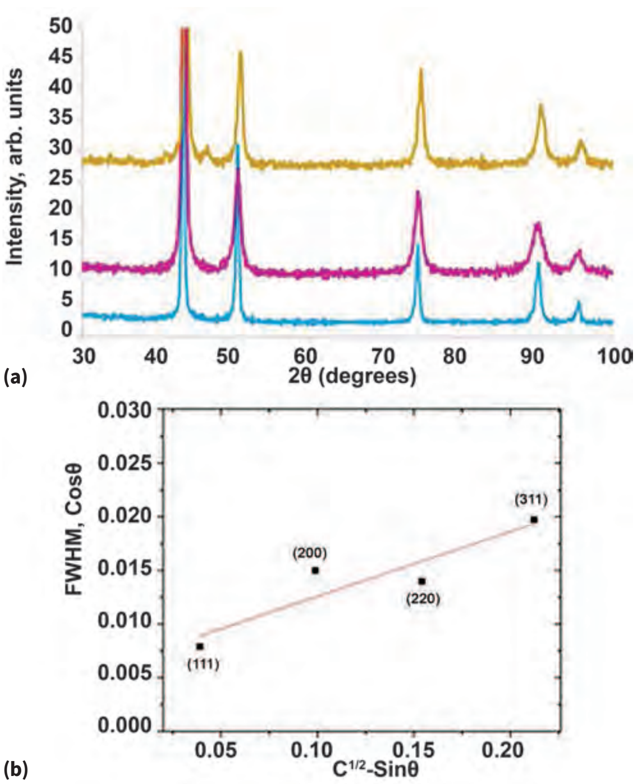


Fig. 1 — (a) X-ray diffractograms comparing IN625 (nickel-base superalloy) powders and cold spray IN625 coating show broadening of diffraction peaks in the coating, indicative of macroscopic strain. (b) Hall-Williamson plot taken from peak broadening data to calculate the extent of strain. FWHM, full width at half maximum.

Due to the severe plastic deformation that takes place during high-pressure cold spray, the characteristic diffraction pattern in the as-sprayed condition usually has broad peaks, as shown in Fig. 1(a) for an IN625 coating. Peak broadening in the as-sprayed coating compared to the powders can be due to two factors—particle size broadening and broadening due to strain (nonuniform). The full width at half maxima (FWHM) of the diffraction peak is estimated from the Scherrer formula for particle size broadening:

$$d = 0.9\lambda/\beta\cos\theta$$

where d is particle size, λ is the x-ray's wavelength, β is FWHM of the x-ray peak (in radians), and θ is peak angle. Typically, the Scherrer formula is based on the assumption that peak broadening is caused by crystallite size. The nonuniform strain-induced broadening effect of the FWHM is obtained by the Williamson-Hall (WH) plot, which is derived by the equation:

$$\varepsilon = \beta\cos\theta - A/2\sin\theta$$

where A is expressed as $C\lambda/d$, C is the correction factor (~1), and d is particle size. Figure 1(b) shows the WH plot for an IN625 coating, with a grain size of 35 nm. XRD can identify coating phases and distinguish any changes to the phase equilibria during cold spray. Figure 2(a) illustrates cold spray titanium

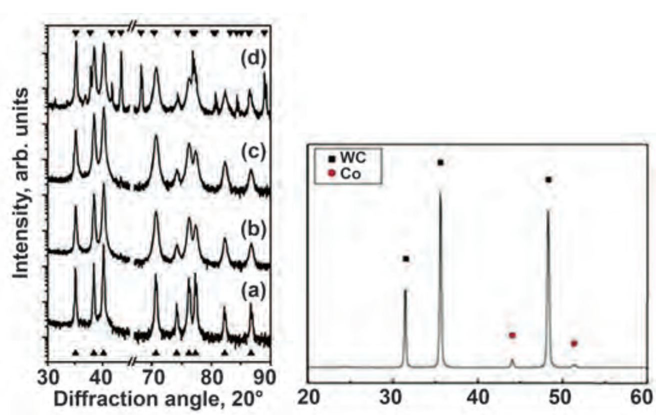


Fig. 2 — X-ray diffraction patterns from (a) cold spray titanium coating after removal of progressive layers from the coating compared with α titanium, indicating no phase transformation in the coating, and (b) cold spray WC-Co without decarburization after cold spray.

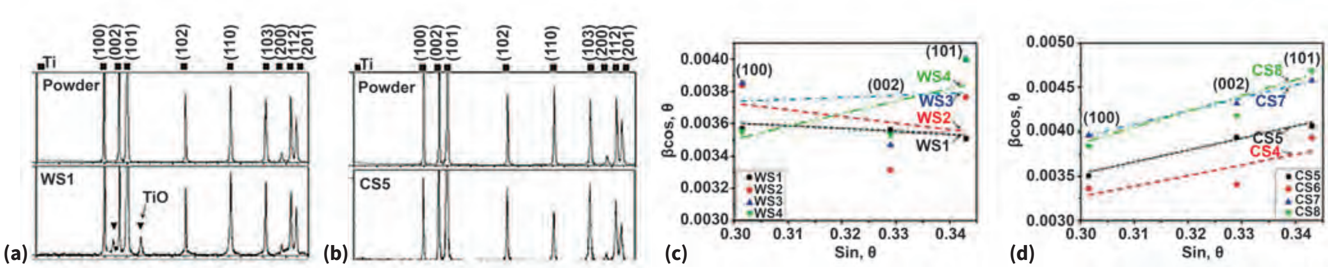


Fig. 3 — X-ray diffraction patterns comparing (a) warm spray and (b) cold spray titanium coatings, along with the respective starting powders indicate the presence of TiO oxides and metal in the warm spray coating. Williamson-Hall plots for the (c) warm spray and (d) cold spray coatings indicate a lower degree of strain in the warm spray coating.

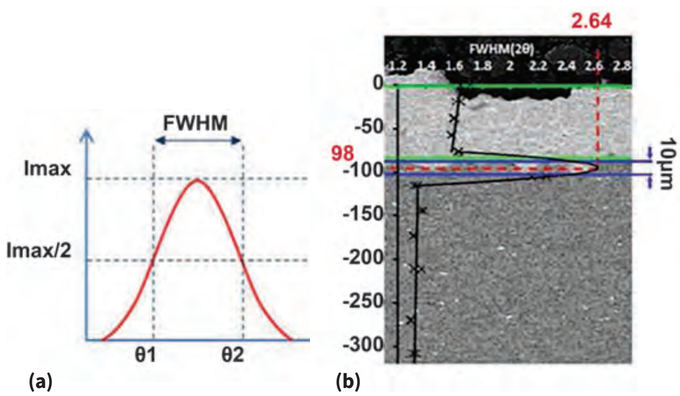


Fig. 4 — (a) Full width at half maximum (FWHM) from an x-ray diffractogram peak corresponding to a cold spray Al7075 coating. (b) Mapping the FWHM through coating thickness and substrate to estimate grain size.

coating characterization with progressive removal of layers. A comparison between the as-sprayed coating and α titanium indicates that no phase transformations take place during cold spray.

Another example includes characterization of the absence of decarburization in a WC-Co coating. In Fig. 2(b), a comparison between the powders and cold spray WC-Co coating indicates the absence of any decarburization. Moridi, et al., were able to distinguish between a cold spray coating and the substrate for a titanium alloy using XRD, and Rokni, et al., showed an extensive peak broadening that is typically characteristic of cold sprayed coatings in the as-sprayed condition. Upon heat treatment, the peaks become sharper, indicative of stress relaxation in the coatings. In all cases, coating residual stresses diminish with heat treatment to a more tensile value, as reflected by a sharpening of the peak, as shown in Fig. 1(a).

Cold spray coatings are nearly always crystalline with sharp diffraction peaks. X-ray diffraction can identify phase formation during variations in the process, such as in the case illustrated in Figs. 3(a) and (b). Kim, et al., have done extensive characterization of cold spray versus warm spray using XRD, wherein the presence of oxides of titanium is prevalent in the warm-spray coatings, and absent in cold spray. The authors conclude that solid-state spray can minimize or eliminate detrimental effects to the microstructure, such as oxide formation.

Figures 3(b) and (c) compare the WH plots between warm- and cold-spray titanium, indicating that the coatings contain more nonuniform strain as compared to the powder. However, between the two coatings, the cold spray coating shows a higher level of strain than those in the thermally softened process of warm spray coatings. Particle sizes indicated by the WH plots range between 38 and 40 nm.

Peak FWHM is used to interpret the surface state of the material. Figure 4(a) shows a representative FWHM taken from the work of Ghelichi and Bagherifard from their studies on an Al7075 cold spray coating onto an aluminum substrate. A big jump at the interface between the deposited material and the substrate is shown in Fig. 4(b), which represents the FWHM measured through coating thickness into the substrate. In this case, by applying the de Keijser method, material grain size ranges between 20 and 50 nm, from a convolution of the diffraction pattern.

Several reports have thus made use of XRD to characterize powders, as-sprayed coatings, extent of lattice strain, phase transformations in as-sprayed versus heat treated conditions, and grain size. Typically, flat samples are recommended in order to obtain precise values of lattice spacing from the x-ray diffractogram. Apart from this, no special care is needed during sample preparation. The top surface should be slightly ground off so that any oxides on the surface do not lead to artifacts or additional peaks in the XRD pattern. ~iTSSe

For more information: Dheepa Srinivasan is a principal engineer at GE Power, GE India Technology Center, Bangalore, dheepa.srinivasan@ge.com, www.ge.com. This article series is adapted from *Chapter 5, Cold Spray—Advanced Characterization,* in High Pressure Cold Spray—Principles and Applications, edited by Charles M. Kay and J. Karthikeyan (ASM, 2016).

AEROSOL METHOD FOR ROOM TEMPERATURE THICK-FILM DEPOSITION

Aerosol deposition offers an alternative to conventional thin film processes when mesoscale coatings are needed.

Pylin Sarobol, Andrew Vackel, Jesse Adamczyk,* Thomas Holmes, Mark Rodriguez, James Griego, Mia Blea, and Harlan Brown-Shaklee, Sandia National Laboratories, Albuquerque, N.M.*

Ceramic and metallic thick film (>5 µm) processing typically requires high temperatures or reactive environments that limit integration of film/substrate materials with drastically different melting points or thermal properties. In many spray processes, particle melting/solidification and splatting lead to highly defective microstructures, containing splat boundaries, porosity, oxide inclusions, and nonstoichiometric oxide formation. Moreover, melting/solidification associated with thermal spray can lead to loss of volatile elements, change in original crystal structure, and altered properties. The aerosol deposition (AD) process is being used to create readily integrated, high density ceramic and metallic thick films on a variety of substrates at room temperature.

AEROSOL DEPOSITION PROCESS

In the AD process, thick films are produced by accelerating submicron to micron sized particles onto a substrate in a low-vacuum environment chamber. AD is able to conformally deposit dense, thick films up to ~100 µm, which bridges the gap between coatings fabricated with thin film technologies and traditional thermal spray technologies. AD takes advantage of the low pressure within the vacuum by allowing spray particles to maintain velocity and consolidate in the solid state into a film on impact with the substrate and subsequent film. When the carrier gas for the aerosol powder hits the substrate, the gas compresses and creates a densified gas layer above the substrate, similar to the bow shock in cold spray. However, in AD the bow shock/particle interaction is reduced in the vacuum, allowing small particles with low momentum to penetrate the bow shock layer and impact the substrate with sufficient kinetic energy for deposition.

AD takes advantage of the small particle size and the ability of ceramic and metallic particles to plastically deform at a small length scale and bond as coatings. At Sandia National Laboratories, fundamental experiments were performed to understand how submicron ceramic particles deform and bond to the substrate in the AD process. Ultimately, the knowledge gained is being used in R&D work, yielding new potential applications.

*Member of ASM International

AD PROCESS RESULTS

Previous work has proven that submicron ceramic particles undergo dislocation nucleation/slip, plastic deformation, fracture, and consolidation in the AD process at room temperature. Submicron ceramic particles with diameters >100 nm capable of plastic deformation should be selected as feedstock for the aerosol deposition process. Submicron alumina particles can deform plastically via dislocation nucleation and slip, as well as fracture without fragmentation, under quasi-static compressive loading. Research also shows that impact at high velocity can cause submicron alumina particles to deform, change shape without fragmentation, and adhere to the substrate as an anchor layer.

Bonding between an individual splat and substrate is present near the middle of the splat whereas gaps remain around splat edges. Subsequent particles impacting on the splatted anchor layer produce a *tamping effect.* Gaps around the splatted particles and substrate are closed and bonding between the anchor layer and substrate is complete. Consequently, film buildup relies on the tamping effect to deform, fracture, and mechanically bond the arriving particles to the already deposited particles. An example of AD Al_2O_3 single splats and coatings is shown in Fig. 1. Splat boundaries within the coatings are undistinguishable from other grain boundaries. The consolidated coating is polycrystalline with 15-30 nm nanocrystals.

The fundamental knowledge on deformation and bonding gained from the previous work provides a strong foundation to mature the aerosol deposition process for fabricating ceramic, metallic, and composite films on metallic, glass, and plastic substrates at room temperature. Potential applications of aerosol deposition being investigated include direct applied multilayered ceramic capacitors, electrically conductive electrodes, thermally and chemically resistant barrier coatings, and electrically insulating films. Examples of AD material integration are shown in Fig. 2.

APPLICATION OF $BaTiO_3$ DIELECTRICS

The application of $BaTiO_3$ dielectrics for high temperature stable capacitors to enable high power electrical switching devices is explored. The high sintering temperature of $BaTiO_3$ (T>1000°C) often prevents successful integration with

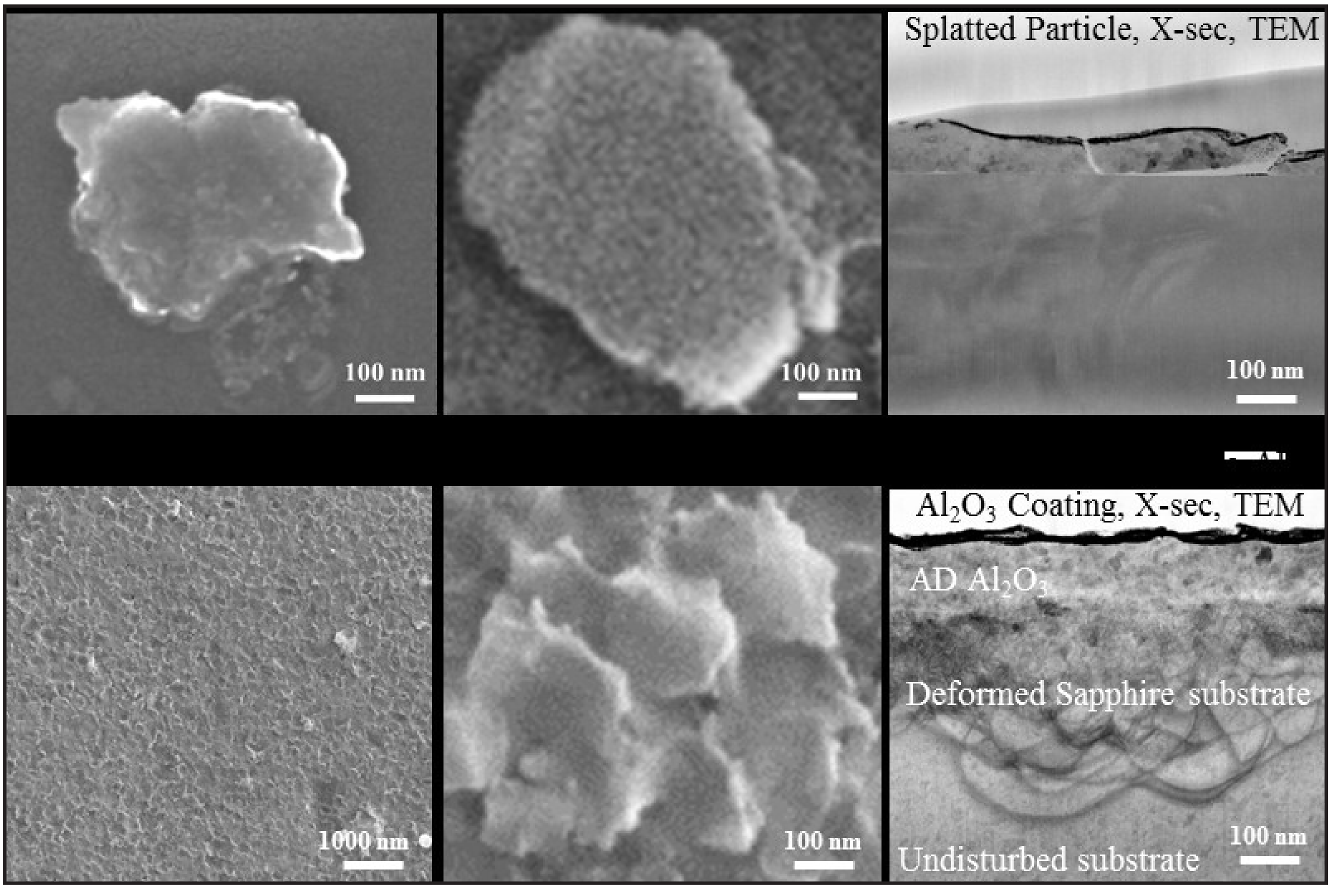


Fig. 1 — (Top row) SEM top view images of splatted Al_2O_3 particles from quasi-static loading (left) in the indentation experiment and impacting on the substrate in the AD process (middle). Cross-section TEM image of AD splatted particle showing nanocrystallinity (right). (Bottom row) SEM top view images of AD Al_2O_3 films at low (left) and high (middle) magnifications. Cross-section TEM image of AD Al_2O_3 coating on sapphire shows nanocrystallinity and deformed substrate (right).

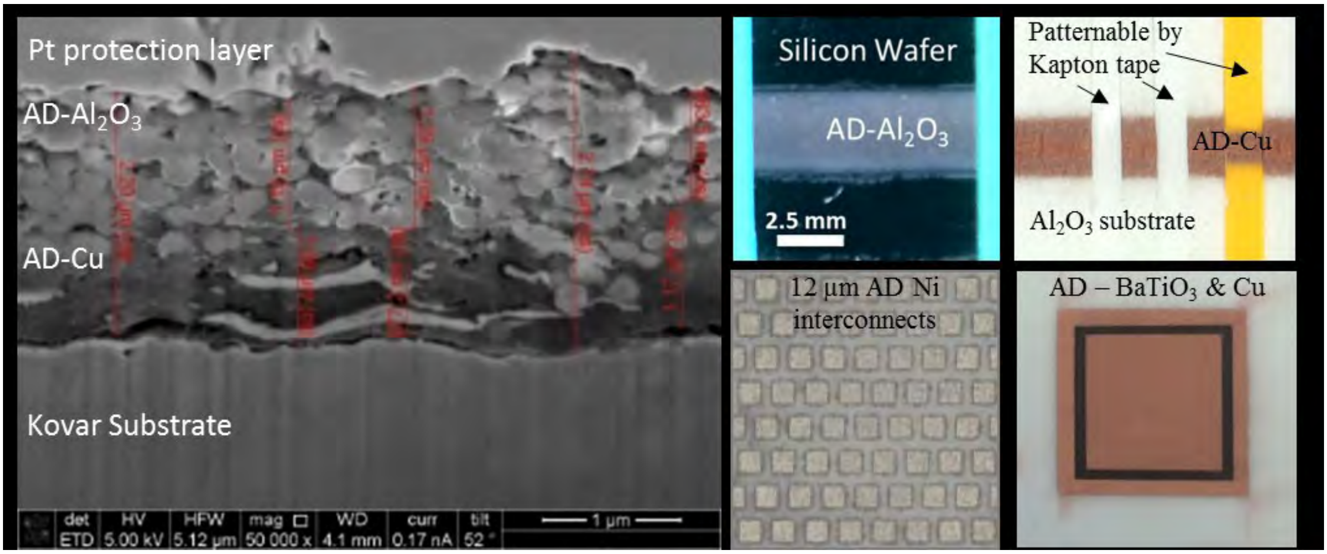


Fig. 2 — Examples of integrated AD coatings—Al_2O_3-Cu graded coating on Kovar, Al_2O_3 on Si, Cu on alumina, patterned Ni interconnect on alumina, and Cu-$BaTiO_3$-Cu on alumina.

low melting point substrates such as glass, metal, or plastic. Moreover, $BaTiO_3$ is susceptible to point defect formation by highly reducing thermal processes. Thermal spray lends itself to electron-rich point defect formation due to volatilization of typical A-site substitution cations, including bismuth or lead. In addition, oxygen vacancy formation that contributes to sub-band electrical transport is enhanced by the thermal processing and rapid quenching used in thermal spray processes. The associated dielectric properties, such as insulation resistance, dielectric constant, and dielectric loss are therefore significantly impacted by melting/solidification in traditional plasma spray.

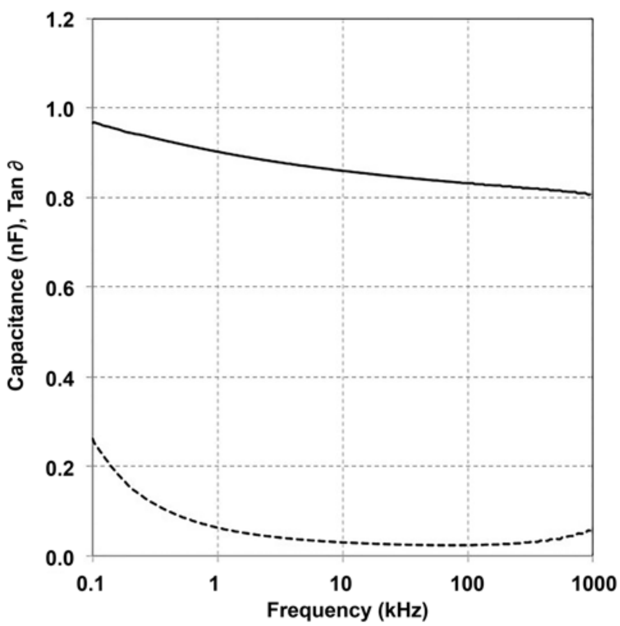


Fig. 3 — Frequency dependent capacitance and dielectric loss (tan ∂) of 4 mm² capacitors defined on as sprayed 5-7 µm thick films on Kovar. Low frequency dielectric loss is a common attribute of as-sprayed $BaTiO_3$ that exhibits significant space charge contribution to total polarization.

The AD process was used to successfully integrate high density thick $BaTiO_3$ films with copper (Tm = 1085°C) at room temperature. The aerosol deposited $BaTiO_3$ film crystal structure, grain size, residual strain, and dielectric properties are currently being investigated. Preliminary results show that 5-7 µm thick $BaTiO_3$ films produce 1 nF capacitors with a 2 x 2 mm (providing a 4 mm² area) gold electrode. A 3 x 3 matrix of electrodes was defined on the 1-cm deposition area. Each electrode spans a 4 mm² area. Seven of the nine 4 mm² electrodes demonstrate potential as capacitors with loss values indicative of continuous, non-cracked films. Figure 3 shows the frequency dependent capacitance and dielectric loss from 100 Hz to 1 MHz. As-aerosol-deposited films exhibit some space charge contribution to polarization. The permittivity of the films was calculated to be k~200, which is consistent with other published research.

Although polycrystalline $BaTiO_3$ capacitors generally exhibit k>1000 at room temperature, these films exhibit significant crystalline disorder and amorphous grain boundary volume. This combination of grain boundary volume and crystalline disorder reduces polarization by hindering Ti4+ mobility within the unit cell and reduces domain wall motion by defect pinning. In addition, these films exhibit significant in-plane crystallographic strain quantified by XRD analysis. The in-plane strain is approximately 1% as determined by XRD analysis (Fig. 4), which corresponds to an approximate compressive stress of 1 GPa.

The as-deposited coatings also have very small grain size (20-75 nm or less). Small grain size may be beneficial for structural ceramics, but larger grain size on the order of

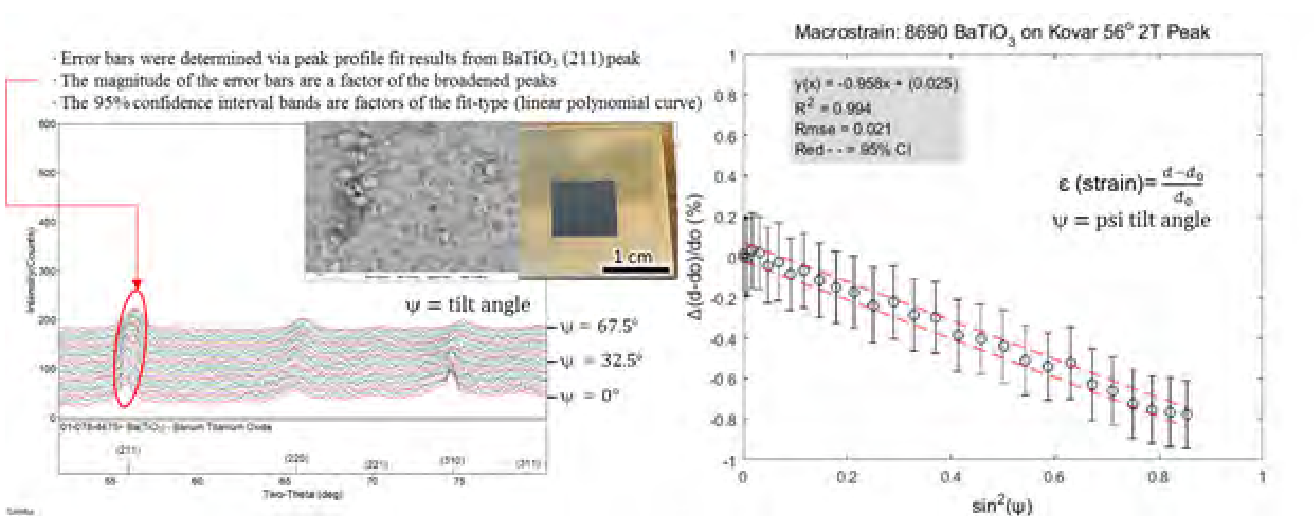


Fig. 4 — XRD measurement and strain calculation on the 4 µm thick $BaTiO_3$ film on Kovar substrate. The strain was on the order of ~1%, corresponding to ~1 GPa residual compressive in-plane stress.

100-1000 nm is generally required to maximize permittivity of ferroelectric ceramics. Post-deposition annealing treatment is required to achieve grain growth, improve crystal quality, and relieve residual stresses, which increases dielectric constant and promotes ferroelectricity.

CONCLUSION

Aerosol deposition is a solid state deposition process that is gaining interest as it can produce functional metallic, ceramic, and composite coatings at room temperature and can potentially further device design, fabrication, and integration. The fundamental knowledge behind particle plastic deformation and bonding for coating consolidation was investigated. The flexibility of the aerosol deposition process allows investigation of a variety of potential applications, including electrical interconnects, electrical insulating coatings, and dielectric coatings toward capacitor fabrication. The AD process offers an alternative to conventional thin film processes when mesoscale coatings are required. The AD process also offers an alternative to conventional thermal spray processes when near 100% coating density and compatibility with a thermally sensitive component is required. ~iTSSe

For more information: Pylin Sarobol is senior technical staff member, coatings and additive manufacturing R&D at Sandia National Laboratories, P.O. Box 5800, Albuquerque, NM 87185, 505.284.7699, psarobo@sandia.gov, www.sandia.gov.

Acknowledgment

The authors thank Donald Susan, Deidre Hirschfeld, Rick Kellogg, and Aaron Hall for their support. This work is supported by Office of Electricity and Laboratory Directed Research and Development Program at Sandia National Laboratories. Sandia National Laboratories is a multi-mission laboratory managed and operated by Sandia Corp., a wholly owned subsidiary of Lockheed Martin Corp., for the U.S. DOE's National Nuclear Security Administration under contract DE-AC04-94AL85000.

ONLINE DIAGNOSTIC TOOLS IMPROVE THERMAL SPRAY PROCESS

REASON TO CONSIDER RESURFACING

Keeping a particle's characteristics constant over time yields coatings with very similar properties. An experiment was conducted where 8%-YSZ is sprayed for 37 hours starting with a brand new set of electrons. The torch is stopped and restarted every hour. Visual inspection of the inside of the nozzle is carried out every hour and gun power is measured. Finally, particle characteristics are monitored using an online spray diagnostic tool from Tecnar, Canada. After 37 hours, the electrodes are worn, gun power has decreased, and the initial microstructure of the coating (TBC) has disappeared. However, after the 37 hours, spray parameters were modified to retrieve the initial particle properties as well as corresponding coating characteristics such as porosity, vertical crack network, and deposition efficiency (DE).

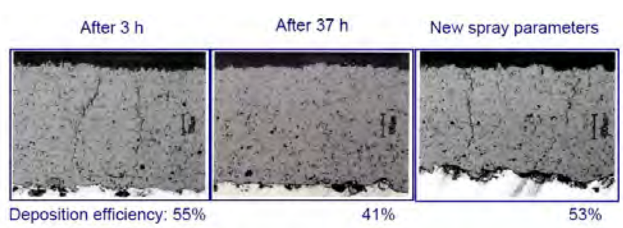


Fig. 1 — Effect of electrode wear on coating characteristics.

Fig. 2a — New electrode. **Fig. 2b —** Worn electrode.

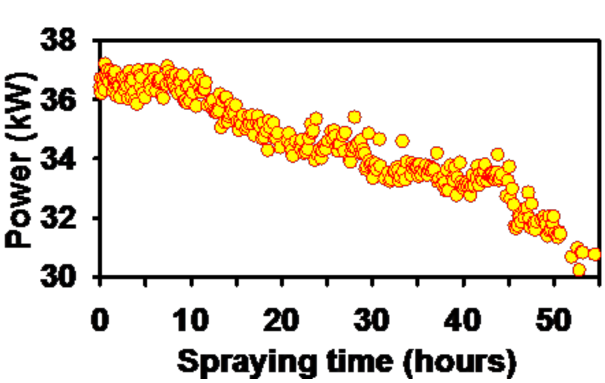


Fig. 3 — Evolution of plasma power over time.

VALUE OF INSPECTION

Nearly three decades ago, thermal spray researchers recognized the need for advanced diagnostic tools able to provide spray material characteristics just before impingement on the part, independently of upstream parameter controls. This need was focused mostly around fundamental understanding of the spray process, validation of thermo- and fluid-dynamic models, development of advanced materials, and development and optimization of spray equipment. Production managers, on the other hand, had different objectives in mind—namely improving coating reproducibility (P_{pks} and C_{pks}), extending electrode lifetime, reducing test coupons, improving DE, better forecasting of coating thickness, and spraying correctly the first time.

OPTION

It is well known that lot-to-lot variations exist for commercially available powders, and that powders supposed to be a direct replacement sometimes yield very different coatings. Below is an example where the same powder (reference) has been used for more than a decade. For various reasons, an alternate supplier is sought. Two other suppliers propose a direct replacement to the reference powder. The three materials are sprayed the same morning, with the same torch and hopper, in the same spray booth, and by the same operator. Coupons are produced and, for each spray run, particle characteristics are measured using an online spray diagnostic tool.

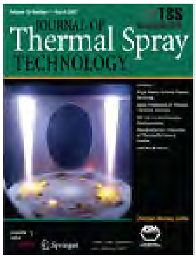
BENEFITS

Results clearly show that Alternate-1 succeeded in providing a direct replacement powder, which was confirmed by the coating microstructure. Alternate-2 provided a material significantly different from the sensor standpoint, which was also confirmed by coating microstructure. **~iTSSe**

Powder	Average velocity (m/s)	Average temperature (°C)	Microstructure
Original	613	1549	Reference
Alternate-1	616	1535	Same as reference
Alternate-2	652	1819	Different (denser coating)

For more information: Luc Pouliot is executive vice president, Tecnar, 1321 Hocquart St., Saint-Bruno-de-Montarville, QC, Canada, J3V 6B5, 450.461.1221 ext. 235, lpouliot@tecnar.com, www.tecnar.com.

Images courtesy of Christian Moreau/National Research Council of Canada.



The *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 the October and December issues, as selected by *JTST* Editor-in-Chief Armelle Vardelle, are highlighted here. The December issue also features the 7th Asian Thermal Spray Conference (ATSC-7). In addition to the print publication, *JTST* is available online through springerlink.com. For more information, visit asminternational.org/tss.

ESTABLISHED AND ADAPTED DIAGNOSTIC TOOLS FOR INVESTIGATION OF A SPECIAL TWIN-WIRE ARC SPRAYING PROCESS

Johannes König, Michael Lahres, Stephan Zimmermann, and Jochen Schein

In a twin-wire arc spray (TWAS) process developed by Daimler AG, known as LDS (Lichtbogendrahtspritzen), gas injection and arc feed play a crucial role in separating molten particles from the wire ends. This paper describes an investigation of the gas and particle behavior according to individual LDS process parameters. Coating problems are not considered. Measurements are separated into two different parts: Cold (without arc and particles) and hot (with arc and particles). Results provide the first detailed understanding of the effect of different LDS process parameters. A correlation between the gas parameter settings and the particle beam properties was found. Using established and adapted diagnostic tools, also used in conventional TWAS processes, this special LDS process was investigated and the results (gas and particle behavior) validated, thereby allowing explanation and comparison of the diagnostic methods, which is the main focus. Based on error analysis, individual instabilities, limits, and deviations during the gas determinations and particle measurements are explained in more detail. The paper concludes with presentation of the first particle-shadow diagnostic results and main statements regarding these investigations (Fig. 1).

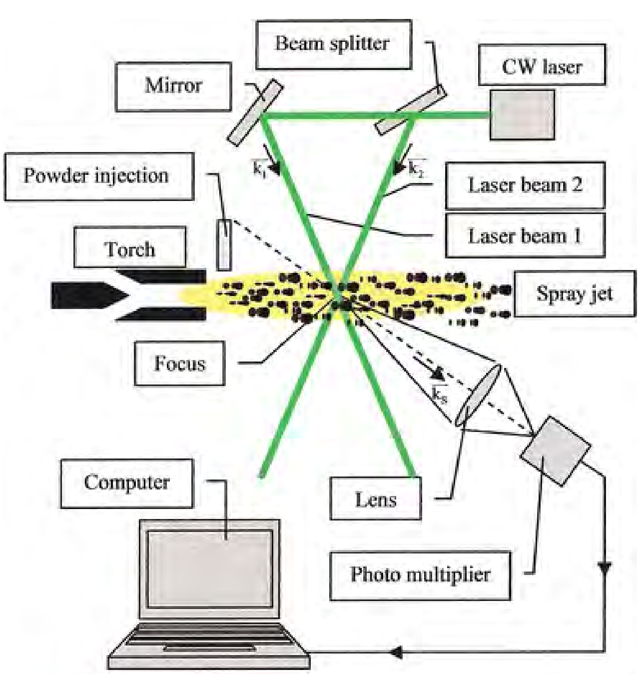


Fig. 1 — Laser doppler anemometry: Principle of setup.

MANUFACTURING AND PROPERTIES OF HIGH-VELOCITY OXYGEN FUEL HVOF)-SPRAYED FeVCrC COATINGS

Paolo Sassatelli, Giovanni Bolelli, Luca Lusvarghi, Tiziano Manfredini, and Rinaldo Rigon

This paper studies the microstructure, sliding wear behavior, and corrosion resistance of high-velocity oxygen fuel (HVOF)-sprayed FeVCrC-based coatings. Various process parameters were tested to evaluate their effects on coating properties, which were also compared to those of HVOF-sprayed NiCrBSi and Stellite-6 coatings. Fe-alloy coatings are composed of flattened splats, originating from molten droplets and consisting of a super-saturated solid solution, together with rounded particles, coming from partially unmolten material and containing V- and Fe-based carbide precipitates. All process parameters, apart from extreme settings with excess comburent in the flame, produce dense coatings, indicating that the feedstock powder is quite easily processable by HVOF. These coatings, with a microhardness of 650-750 HV0.3, exhibit wear rates of $\approx 2 \times 10^{-6}$ $mm^3/(Nm)$ in ball-on-disk tests against sintered Al_2O_3 spheres. They perform far better than the reference coatings, and better than other Fe- and Ni-base

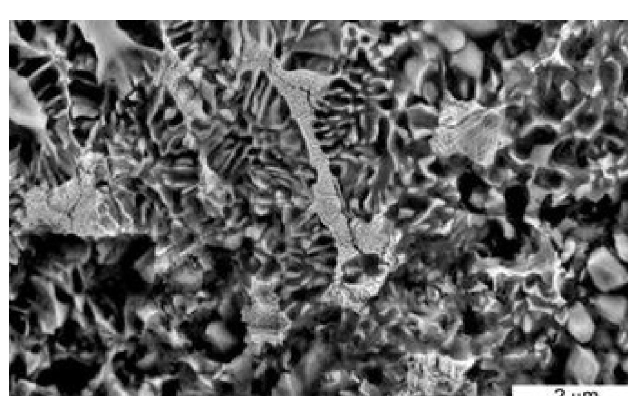


Fig. 2 — SEM micrograph of Sample 1 cross-section after electrochemical polarization test.

alloy coatings tested in previous research. On the other hand, corrosion resistance of the coating material (tested by electrochemical polarization in 0.1 M HCl solution) is quite low. Even in the absence of interconnected porosity, this results in extensive, selective damage to the Fe-base matrix. This coating material is therefore unadvisable for highly corrosive environments (Fig. 2).

EFFECT OF SOLID SHIELD ON COATING PROPERTIES IN ATMOSPHERIC PLASMA SPRAY PROCESS

Ting Liu, Lili Zheng, and Hui Zhang

This paper investigates the impact of shrouded shield structure on plasma spray processes and optimal shield structure selection. Plasma flame characteristics' response to solid shield structures is studied first, and experimental investigations are then performed for both atmospheric (APS) and shrouded (SPS) plasma spray processes. Use of a conical shield with a divergence angle of 5.5° and 90-mm length is effective to form a low-oxygen (<2%) and high-temperature (>3000 K) region in the plasma flame, which covers the main area where particles pass by. The average particle temperature is higher in SPS than APS with the given conditions, and such behavior is intensified as solid shield length increases. Using SPS, more disk-shaped splats are obtained and the oxygen concentration in the coating is significantly reduced. The degree of oxidation in the coatings is further reduced as the length of the solid shield increases from 50 to 90 mm. Applying the solid shield leads to high flame temperature and low oxidation; however, substrate overheating and velocity reduction may occur. For the cases studied, optimal shield length is around 90 mm (Fig. 3).

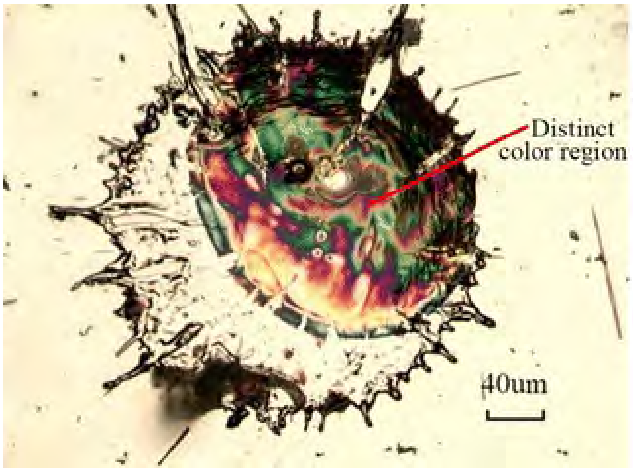


Fig. 3 — Optical micrograph of splat morphology: APS process.

COLONIZATION OF BACTERIA ON THE SURFACES OF COLD SPRAYED COPPER COATING ALTERS THEIR ELECTROCHEMICAL BEHAVIORS

Xinkun Suo, Peng Xia, Yi Liu, Leila Abdoli, Xiaotao Luo, Guanjun Yang, and Hua Li

Copper coatings were fabricated on stainless steel plates by cold spray. Attachment and colonization of *Bacillus sp.* on the surfaces of cold spray copper coatings in artificial seawater were characterized and their effects on anti-corrosion performance of coatings were examined. Attached bacteria were fixed and observed using field emission scanning electron microscopy (FESEM). Electrochemical behaviors including potentiodynamic polarization and electrochemical impedance spectroscopy with and without bacterial attachment were evaluated using the commercial electrochemical analysis station, Modulab. Results show that *Bacillus sp.* prefers to settle on low-lying spots of coating surfaces in an early stage, followed by recruitment and attachment of extracellular polymers (EPS) secreted through metabolism of *Bacillus sp.* The bacteria survives on coating surfaces with the protection of EPS. The attachment model is proposed to illustrate bacteria behaviors on the surface of copper coatings. Electrochemical data shows that current density within the *Bacillus sp.* environment decreases compared to that without. Charge-transfer resistance increases markedly in bacteria-containing ASW, suggesting that corrosion resistance increases and corrosion rate decreases. The influence mechanism of bacteria settlement on corrosion resistance of cold spray copper coatings is discussed (Fig. 4).

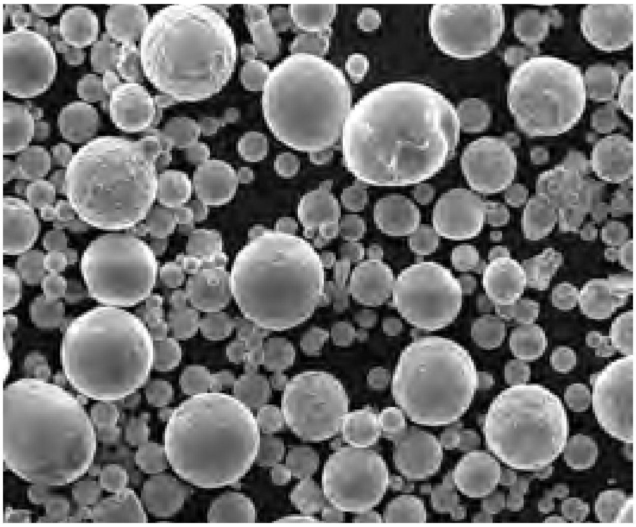


Fig. 4 — Surface morphology of copper powder.

CORRELATION OF IMPACT CONDITIONS, INTERFACE REACTIONS, MICROSTRUCTURAL EVOLUTION, AND MECHANICAL PROPERTIES IN KINETIC SPRAYING OF METALS: A REVIEW

Jaeick Kim and Changhee Lee

Most studies into kinetic spray technology focus on basic research, but a large portion of current research is devoted to industrial applications of the technology. However, to advance studies on industrial applications of kinetic spray requires a profound understanding of the scientific foundations of the process. Nevertheless, there is not yet a well-organized summary of the correlations among impact conditions, interface reactions, microstructural evolution, and mechanical properties across the whole field of kinetic spray technology. This paper provides an overview of these correlations for kinetic spray of metals. For each correlation, interactions between the given conditions and material properties of the metal feedstock powder are the most influential. These interactions are so complicated that it is difficult to systematically classify all cases into certain types. An attempt is made to explain and summarize the critical factors and their roles in each relationship (Fig. 5).

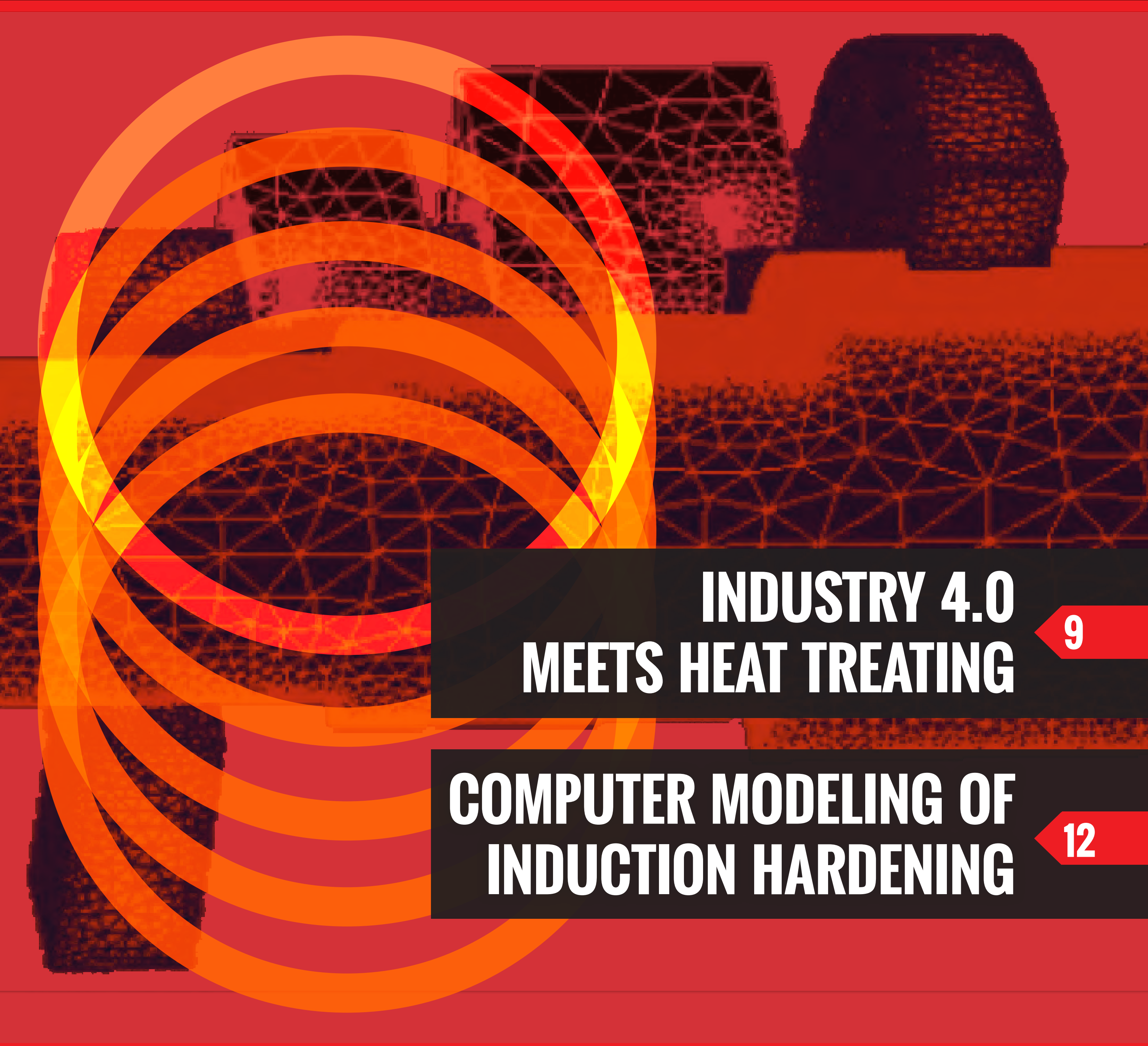
Fig. 5 — EBSD characterization of Ni coating after nanoindentation: Euler angle map.

HTS Heat Treating Society
ASM INTERNATIONAL

NOVEMBER/DECEMBER 2016 | VOLUME 4 | ISSUE 4

HTPRO

BUSINESS AND TECHNOLOGY FOR THE HEAT TREATING PROFESSIONAL



hts.asminternational.org

Heat Treating Society
ASM INTERNATIONAL

HTPRO

BUSINESS AND TECHNOLOGY FOR THE HEAT TREATING PROFESSIONAL

EDITOR
Frances Richards

TECHNICAL ADVISORS
Aymeric Goldsteinas
Stephen Feldbauer
Valery Rudnev
Olga Rowan
HTS R&D Committee

CONTRIBUTING EDITOR
Ed Kubel

PRODUCTION MANAGER
Kelly Sukol

NATIONAL SALES MANAGER
Erik Klingerman
440.338.5151, ext. 5574
erik.klingerman@asminternational.org

HEAT TREATING SOCIETY EXECUTIVE COMMITTEE
Stephen G. Kowalski, *President*
Roger Alan Jones, *Immediate Past President*
James Oakes, *Vice President*

DEPARTMENTS

ABOUT THE COVER

Complex components are routinely induction hardened. Courtesy of Inductoheat Inc., inductoheat.com

EDITORIAL OPPORTUNITIES FOR HTPro IN 2017

The editorial focus for *HTPro* in 2017 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.

June Testing and Process Control
September Thermal Processing in On/Off Highway Applications
November Atmosphere/Vacuum Heat Treating

To contribute an article to one of the upcoming issues, contact Frances Richards at frances.richards@asminternational.org.

To advertise, contact Erik Klingerman at erik.klingerman@asminternational.org.

HTPro is published quarterly by ASM International®, 9639 Kinsman Road, Materials Park, OH 44073, 440.338.5151, asminternational.org. Vol. 4, No. 4

HOW TO MOVE HEAT TREAT R&D FORWARD

Heat treating is an easily identifiable industry and ASM's Heat Treating Society (HTS) organizes successful exhibits and conferences. Likewise, the Metal Treating Institute organizes a successful Furnaces North America (FNA) event every two years. Both organizations and IHEA have course work available for heat treat training. There are clear issues around energy, safety, cost, and product performance. However, no one receives a degree in heat treating and there are few universities and national labs that boost their prestige and funding by investigating and solving issues around heat treatment.

Heat treating is an applied field relying on various aspects of mechanical engineering and materials science. It is an industry but not an academic field where advancements are self-propelled by motivated researchers at universities, institutes, and national labs competing against one another for dollars and prestige. More motivation for development comes from companies, but declining manufacturing infrastructure has certainly slowed this development within companies. It is with this backdrop that the HTS Research and Development Committee has laid out three key activities: 1) Identify, 2) Disseminate, 3) Encourage.

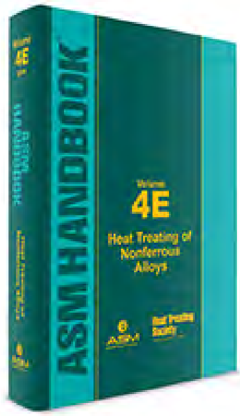
First, use the experience and knowledge of members to identify new work and the organizations that are performing development pertinent to the heat treating industry. The committee must track the work from Worcester Polytechnic Institute's Center for Heat Treating Excellence (CHTE), foreign universities, EWI, and from newly formed consortiums such as LIFT (Lightweight Innovations for Tomorrow), America Makes (National Additive Manufacturing Innovation Institute), and the Digital Manufacturing and Design Innovation Institute.

Second, the information must be disseminated if allowed. Bringing forward work for conference papers and publishing in *HTPro* are two ways to do that. Website updates and other member-friendly and timely ways must also be utilized.

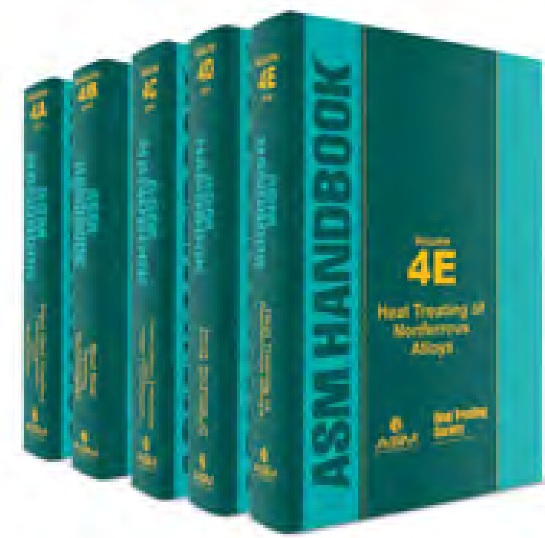
Lastly, research and development must be encouraged. The Thermal Manufacturing Industries Advanced Technology Consortium (TMI-ATC) is a great aid in this area. A roadmap was published showing the areas where research and development are needed to advance industries like heat treating. The committee must find ways to involve more universities, national labs, and companies in heat treat related research.

Michael Pershing
HTS R&D Committee Chair
Sr. Technical Steward at Caterpillar Inc.

NEW HEAT TREATING HANDBOOK JUST RELEASED



ASM Handbook, Volume 4E: *Heat Treating of Nonferrous Alloys,* edited by George E. Totten, is now available. This singular work gives engineers, analysts, and technicians a one-stop source on heat treating fundamentals and guidelines for a wide variety of nonferrous alloys. The new handbook completes the series of five volumes on heat treating, *ASM Handbook,* Volumes 4A, 4B, 4C, 4D, 4E *Heat Treating Set.* The full set is a comprehensive reference guide to all aspects of heat treating including steel heat treating fundamentals, processes, and technologies; selection of steels for heat treatment and the processing and properties of heat treated steels, components, and cast irons; induction heating and heat treatment; and heat treating of nonferrous alloys. For more information, visit asminternational.org/handbook4e, www.asminternational.org/heattreatset, or call the ASM International Service Center at 800.336.5152.



HEAT TREAT MEXICO DEBUTS TO SOLD-OUT CROWD

ASM's inaugural Heat Treat Mexico conference was held September 20-23, organized by the Heat Treating Society. The event was completely sold out and far exceeded expectations. The first day started with ASM's "non-metallurgical metallurgy" course taught in Spanish by Professor Rafael Colas at the Autonomous University of Nuevo Leon and eneded with a reception of more than 125 attendees welcoming the new Mexico Chapter to ASM. The 12-session technical program kicked off Wednesday with Spanish and English presentations simultaneously translated to over 274 attendees and ended with a networking reception with all attendees, including those from the 43 exhibiting companies. Due to the success of this new event, another one is planned for 2018.

ASM Mexico Chapter leadership, from left, Vic Zacharias, Francisco Granados, Erika Zarazúa, Carlos Carrasco, Adriana Michaca, and Fabian Mendez.

NOMINATIONS SOUGHT FOR GEORGE H. BODEEN HEAT TREATING ACHIEVEMENT AWARD

ASM's Heat Treating Society (HTS) is currently seeking nominations for the George H. Bodeen Heat Treating Achievement Award, which recognizes distinguished and significant contributions to the field of heat treating through leadership, management, or engineering development of substantial commercial impact. **Deadline for nominations is February 1, 2017.**

For nomination rules and forms, visit hts.asminternational.org and click on Membership & Networking and Society Awards. For more information or to submit a nomination, contact Joanne Miller at 440.338.5151 ext. 5513, joanne.miller@asminternational.org.

SOLICITING PAPERS FOR ASM HTS/BODYCOTE BEST PAPER IN HEAT TREATING CONTEST

The ASM HTS/Bodycote award was established in 1997 to recognize a paper that represents advancement in heat treating technology, promotes heat treating in a substantial way, or represents a clear advancement in managing the business of heat treating. The award is endowed by Bodycote Thermal Process-North America. The contest is open to all students, in full-time or part-time education, at universities (or their equivalent) or colleges. It is also open to students who have graduated within the past three years and whose paper describes work completed while an undergraduate or post-graduate student. The winner receives a plaque and check for $2500. To view rules for eligibility and paper submission, visit hts.asminternational.org, Membership & Networking, and Society Awards.

Paper submission deadline is March 1, 2017. Submissions should be sent to Joanne Miller, ASM Heat Treating Society, 9639 Kinsman Rd., Materials Park, OH 44073, 440.338.5151 ext. 5513, joanne.miller@asminternational.org.

NOMINATIONS SOUGHT FOR ASM HTS/SURFACE COMBUSTION EMERGING LEADER AWARD

The ASM HTS/Surface Combustion Emerging Leader Award was established in 2013 to recognize an outstanding early-to-midcareer heat treating professional whose accomplishments exhibit exceptional achievements in the heat treating industry. The award was created in recognition of Surface Combustion's 100-year anniversary in 2015. The award acknowledges an individual who sets the highest standards for HTS participation and inspires others to dedicate themselves to the advancement and promotion of vacuum and atmosphere heat treating technologies.

Deadline for nominations is April 1, 2017. For rules and nomination form, visit hts.asminternational.org and click on Membership & Networking, and Society Awards. For additional information, or to submit a nomination, contact Joanne Miller at 440.338.5151, ext. 5513, joanne.miller@asminternational.org.

HEAT TREATING SOCIETY SEEKS BOARD NOMINATIONS

The HTS Awards and Nominations Committee is seeking nominations for three directors and a vice president, student board member, and young professional board member. Candidates must be an HTS member in good standing. Nominations should be made on the formal nomination form and can be submitted by a chapter, council, committee, HTS member, or an affiliate society. The HTS Nominating Committee will consider any HTS member including those who have previously served on the HTS Board. **Nominations are due by February 1, 2017.**

For more information and the nomination form, visit hts.asminternational.org and click on Membership & Networking, and Board Nominations; or contact Joanne Miller at 440.338.5151 ext. 5513, joanne.miller@asminternational.org.

OBTAINING NADCAP ACCREDITATION: HELPFUL GUIDELINES FOR PASSING YOUR AUDIT, PART III

Learn how to simplify the process of obtaining Nadcap accreditation for your heat treating facility by paying heed to some of the challenges others have experienced.

Nathan Durham, Ipsen USA, Cherry Valley, Ill.

Aerospace Material Specification (AMS) standards and the Nadcap accreditation process play key roles in ensuring that manufacturers performing heat treating and other special processes adhere to consistent, high-quality standards for producing aerospace products. While preparing for a Nadcap audit can seem daunting, a series of articles has been developed that discusses some of the recommended resources for preparing for and executing a successful audit. The first part of this series (June 2016, *HTPro*) looked at preparing for and scheduling an audit. The second part (September 2016, *HTPro*) addressed the internal audit process, nonconformances, and completing the internal audit. This final article looks at the official audit process, auditor interactions, and completing the official audit.

During the Process segment of the Nadcap audit, the auditor will spend a significant portion of time reviewing specification checklists and both historical and live jobs. Images courtesy of Ipsen.

EXECUTING A SUCCESSFUL OFFICIAL AUDIT

After performing an internal audit and submitting the necessary documentation, the official audit is the next step in the Nadcap accreditation process. The official audit is an independent, objective activity designed to verify that you are operating under a specified state of control[1]. Having control over your processes and equipment—essentially every aspect involved in manufacturing aerospace components—places you in the best position to consistently meet and adhere to industry quality standards for all aerospace products.

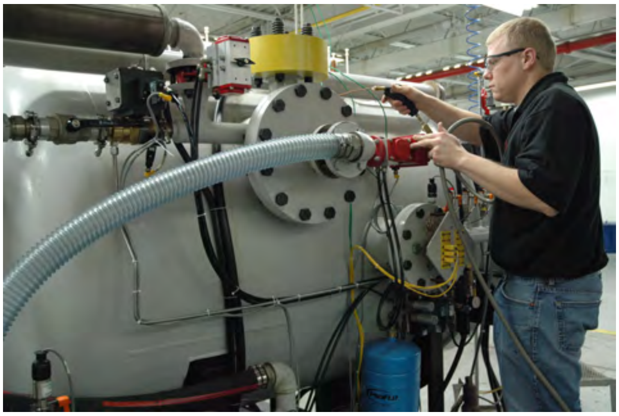
Close control of your processes is important as certain processes, such as heat treatment and brazing, are considered special processes (i.e., processes where it is not possible to completely verify that the final product meets the specified requirements). Therefore, these processes must be validated in advance to ensure products are free of defects and meet the aerospace industry's high quality standards. The only way to guarantee quality results is to guarantee the process and demonstrate control through a Nadcap audit.

In addition to preparing for the audit process and performing an internal audit, a few best practices are recommended for executing a successful audit; these include verifying the desired scope of accreditation, scheduling key personnel, and informing the auditor in advance of any schedule considerations.

Verifying the scope of accreditation: When starting the official audit, one of the first things you typically do with the auditor is verify that the scope of accreditation submitted when scheduling the audit has not changed. The standards and specifications you selected to be audited against are what the auditor will go over in detail during the audit process. It is also important to note that you cannot remove or add any accreditations once the auditor has finished verifying the scope.

Scheduling personnel: According to some companies that have undergone a Nadcap audit, it is important to have personnel who performed the internal audit available during the official audit process. Key personnel typically include those most knowledgeable about day-to-day processes, as well as those who regularly operate the equipment. It is also important to inform all personnel of the audit process and confirm that they understand their role in making the audit successful as the auditor may choose to talk with one of them during the official audit.

Informing the auditor of schedule considerations: Scheduling is crucial during the official audit. In most



It is important to inform all personnel of the audit process and confirm that they understand their role in making the audit successful as the auditor may choose to talk with one of them during the official audit.

cases, the auditor is required to be on-site for four to five full days. Therefore, you are required to inform the auditor at the start of the audit about the Primes for which you want to be approved, as well as what is scheduled with production over the next few days (e.g., if there are any upcoming furnace cycles). This enables the auditor to effectively evaluate your processes while maximizing productivity.

THE AUDIT PROCESS

The official audit is generally divided into two segments—the Quality System Audit and the Process Audit.

The Quality System Audit is approximately one day long. According to Performance Review Institute (PRI), the auditor will not perform this audit if you provided sufficient evidence of an acceptable quality system when scheduling the audit. If you do not have an acceptable quality system, the auditor will perform the Quality System Audit, which involves verifying that you meet all the requirements for a quality specification.

The Process Audit is approximately four days long. However, the Process segment could be longer, depending on the number of Primes for which you are being accredited, the scope of accreditation, and other factors. Typically, the auditor spends a significant portion of time reviewing specification checklists and both historical and live jobs, so a large number of both should be available for review (as defined on the eAuditNet website). Auditors typically request different historical jobs besides the ones prepared, as well as perform live job audits.

Interacting with the auditor occurs in various ways throughout the official audit process, from discussing your understanding of specifications to reviewing findings. A key part of the audit process is providing credible information and data on how the process is controlled, as well as explaining it in a clear, concise manner. Nadcap auditors are extensively trained to identify nonconformances and ensure adherence to all specifications within the set standards. Discussing nonconformances you received with the auditor enables you to better understand all aspects of the finding, as well as why you received it.

Recommended methods for interacting with the auditor include presenting your reasons for executing a particular process a certain way in a factual manner and discussing how you interpret a certain specification to determine where any misunderstanding might have occurred. We found it is vital to keep a few key best practices in mind to ensure interactions with the auditor are calm and professional, including:

- *Balancing the auditor's time.* The auditor's time is divided between the office and shop floor. Because there are only a few days for the auditor to review everything, and because each job audit can consume a lot of time, it is helpful to provide a daily reminder of when loads are being run and what times certain personnel are available. Being prepared, regardless of when the auditor wants to review a certain area or job audit, helps avoid any delays in the audit process.
- *Explaining your interpretation of a specification*. There may be times when you need to explain how you interpreted a specification. Others recommend having collected data, documented procedures, and additional information on hand for discussions. In addition, being able to provide an explanation for why you do things a certain way not only helps you better understand the nuances of various specifications, but also helps you decide if any refinements need to be made in your process.

FINISHING THE AUDIT PROCESS

Based on discussions with aerospace suppliers and information from PRI, it is recommended that you follow the proper steps after the official audit, regardless of whether you pass or fail. These steps are nearly identical to those followed after the internal audit, but with a few key differences.

Reviewing findings. The first and most important step is to review all findings with the auditor and make sure you understand each one. This step is vital because if you do not understand why you received a finding, you will not be able to determine the corrective action. After the audit is concluded, you must respond to all findings, making sure to include:

- Root cause of the nonconformance
- Product impact of all nonconformances
- Corrective action taken
- Date of corrective action implementation
- Action taken to prevent future reoccurrences
- Objective evidence

Performing root-cause analysis. The process of performing root-cause analysis for the official audit is identical to the steps applied during the internal audit. However, a recommended best practice is to take advantage of the root-cause training PRI offers; this training will help you better understand how to identify them as related findings can sometimes be grouped together, depending on the ultimate root cause. Having a similar root cause does not necessarily mean the number of findings will be reduced, but knowing how to perform root-cause analysis is an essential part of the official audit process.

Applying corrective actions. This is the next step after performing root-cause analysis. In making a corrective action, you should define the resolution made to address the issue and cite the timing of implementation. For example, if you determined that the root cause of a finding for improperly located load thermocouples is a lack of operator training, the corrective action would be to provide operator training at a specific frequency. You should also include the training schedule that has been followed since the corrective action was implemented.

When going through these steps after the audit, it is also important to note that you have 21 calendar days from the end of the official audit to submit your initial response concerning any findings. You then have seven calendar days to submit each subsequent response with four total responses allowed. Thirty extra days are allotted if more time is needed. However, using these extra days can affect your merit, and using more than the 30 extra days (or more than the allotted four responses) could lead to an audit failure[2].

Identifying successful practices from the accreditation process, as well as areas that could use refinement, ultimately helps contribute to your success on future audits. Continually refining your processes and methods enables you to enhance the quality and safety of the components you process.

CONCLUSIONS

Auditors are tasked with evaluating a specific set of standards and noting any findings that do not adhere to set specifications. They play a key role in ensuring suppliers successfully adhere to the high quality standards of the aerospace industry. It is important to remember that Primes have established global quality standards to ensure aircraft and traveler safety. The Nadcap accreditation process not only helps ensure the safety of all who use these products, but also helps companies continually refine and improve their processes. Whether it is your first or 10th Nadcap audit, continually refining and developing your audit process will contribute to a positive and successful experience each time. **~HTPro**

For more information: Nathan Durham is an electrical solutions manager at Ipsen. For technical information, contact technical@ipsenusa.com or 844.464.7736 (select 1), Ipsen USA, 984 Ipsen Rd., Cherry Valley, IL 61016, ipsenusa.com.

References

1. American Society for Quality, asq.org/learn-about-quality/auditing.

2. P. Evans and E. Jacklin, Nadcap Supplier Tutorial, Performance Review Institute, p 37, 2013.

INDUSTRY 4.0 MEETS HEAT TREATING

Industry 4.0 is in the early technology evolution stage with tremendous business expectations.

Satyam S. Sahay, FASM,* John Deere Technology Center India, Pune

Industry 4.0, or Internet of Things (IoT), is an emerging technology trend in the manufacturing arena, and is widely considered the fourth industrial revolution (Fig. 1). IoT, where convergence of the virtual and real world is envisioned, is characterized by:

- Physical and virtual connectivity across product development cycles
- Multifunctional synergies, including engineering, manufacturing, quality, and customer support
- Higher level of intelligence from connected databases, products, and process models, which could lead to more informed and autonomous decisions with self-learning intelligence developed within the organization

Key business drivers for this revolution include a higher level of efficiency and quality achieved through individualized solutions at the component or machine level, better leverage of networked production across the supply chain, and proactive equipment maintenance (Fig. 2). In addition, significant emphasis will be placed on organizational knowledge development over time.

The emergence of Industry 4.0 will significantly impact heat treating operations as well. The most significant change will be a change in the current mindset, i.e., changing from the current silo view of the heat treating process to considering it part of the connected production operation. A connected gear-making process is illustrated in Fig. 3. Data collected from heat treating operations, which is typically used for quality audit purposes, would be leveraged to provide part-level data sharing for improved pre- and post-operations. Transfer of specific data, for example, chemical composition data from the steel mill and the casting, could provide better control of reheating, annealing, and carburizing operations, which in turn would help mitigate and manage distortion in these precision components.

Further, identifying the location of specific components in batch type operations would help to better design recipes for machining and finishing operations. Such individualized solutions at the part level would bring a transformational change to quality control at the product level. One of the biggest changes in the heat treating operation would be with recipe management, which is now *heuristically driven*—mainly by trial and error. The significant amount of production and quality data together with their mathematical models would help to develop self-learning and self-evolving heat treating recipes, suitable for current production and quality needs with due consideration of the current furnace health and

FOUR INDUSTRIAL REVOLUTIONS
1. Invention of mechanical weaving loom (1784)
2. Assembly-line mass production (1870)
3. Programmable logic controller (PLC) automation (1969)
4. Virtual-real world convergence (today)

Fig. 1 — Four industrial revolutions in human civilization.

Fig. 2 — Key business drivers for Industry 4.0, or Internet of Things (IoT), the fourth industrial revolution.

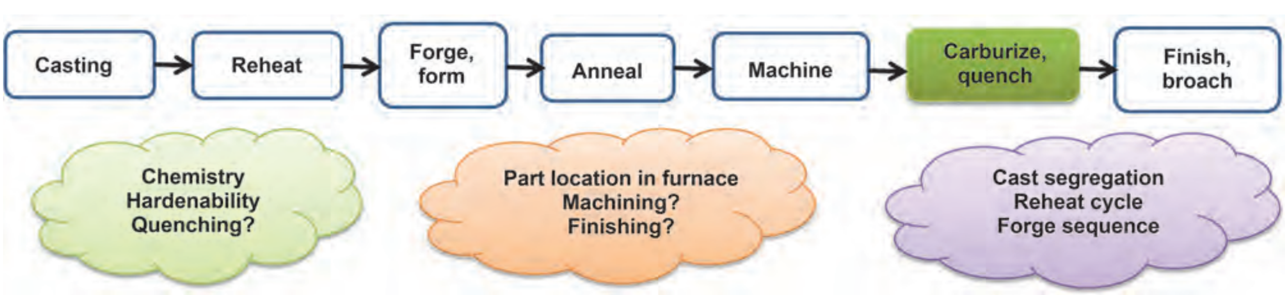


Fig. 3 — Integration of heat treating operations into the process chain for gear-making.

*Member of ASM International

operating condition. Aligned with the overall Industry 4.0 vision, these changes would also make heat treating more operationally efficient with robust, quality products.

INDUSTRY 4.0: HEAT TREATING STORY BOARDS

Three examples of Industry 4.0 will now be illustrated for heat treating operations, described in an IoT framework with key characteristics of sensing, connectivity, analytics and intelligence capabilities, and business value:

Recipe management: Although significant changes in furnace technology and automation have occurred, the heat treating recipe—which is the core of heat treating operations—continues to be heuristically developed. Furthermore, recipe selection for new parts is also heuristic, resulting in a significant amount of rework and reject levels at initial stages of production. It is expected that Industry 4.0 would have the most significant impact on how heat treating recipes are developed and managed. Recipe selection would primarily be accomplished through image recognition algorithms of a part, which would eliminate mistakes in selection. Further, self-learning algorithms would enable continuous evolution of recipes with feedback from historical quality data (Fig. 4). This would also enable recipe customization based on the current part chemistry and furnace health and operating condition. The system would also have self-evolving capabilities for heat treating recipes to become rationalized (Fig. 5) over time due to changes in product mix. Any recipe decision due to production disruption in pre- or post-steps could be better managed. Consequently, heat treating operations will be more efficient and product quality will be predictable. In the Industry 4.0 framework, heat treaters need: sensing capability for part shape, quantity, and weight in the furnace; connectivity and analytics capability to choose the correct reference recipe; intelligence capability to fine-tune the recipe based on instantaneous inputs and operating conditions; and long-term rationalization and recipe development to create value in terms of efficient operations and robust product quality.

Distortion control: Distortion control in precision transmission components, such as gears and shafts, is a significant heat treating challenge. Critical parameters including chemistry (Fig. 6), process parameters during reheating and forging, annealing and machining conditions, and carburizing and quenching conditions impact distortion. A key reason for distortion is inadequate information at different processing steps, which prevents efficient distortion-management strategies. For example, chemical composition is directly linked to hardenability and distortion. Significant control of distortion could be achieved if information on the composition is known at the heat treating step, and an instantaneous recipe created incorporating this information. Further, depending on quench-tank conditions and furnace

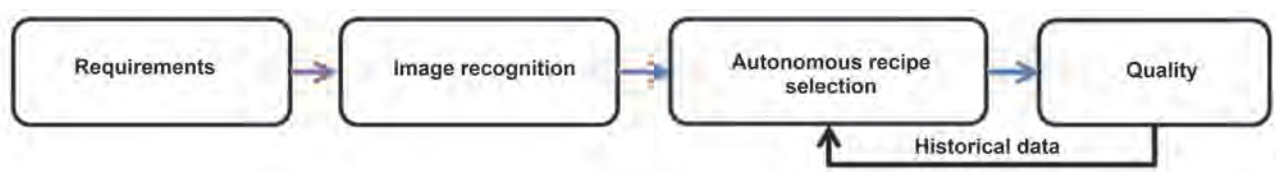


Fig. 4 — Self-learning algorithms and intelligence created for autonomous recipe selection, management, rationalization, and fine-tuning.

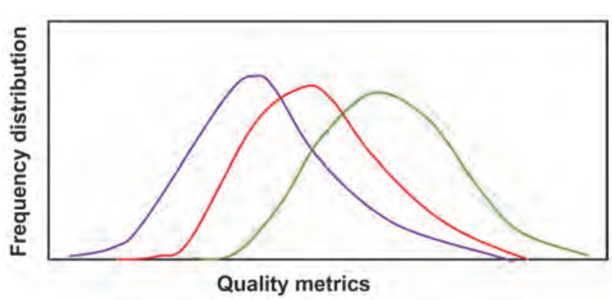


Fig. 5 — Overlapping frequency distributions of quality metrics obtained from different recipes indicate the possibility of recipe rationalization.

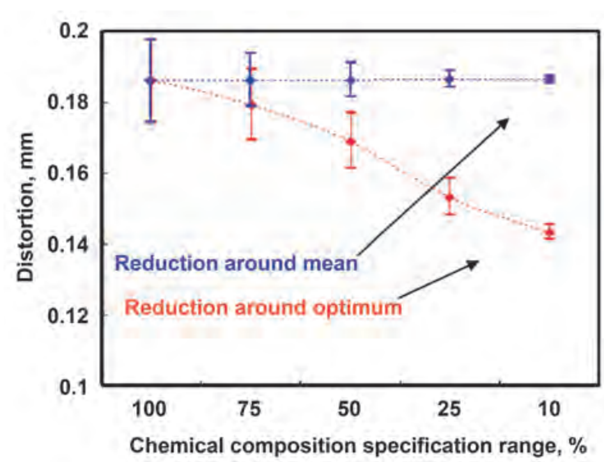


Fig. 6 — Influence of steel chemical composition (within specified chemistry range) on heat treating distortion, which presents an opportunity to control distortion by fine-tuning recipes for specific loads.

health, machining steps prior to carburizing could be fine-tuned to anticipate distortion and better manage distortion in the final product. Furthermore, depending on the location of the part in the carburizing step, finishing operations could be made part specific, thereby having highly predictable distortion in the product. In the Industry 4.0 framework, sensing capability is required for:

- Composition information
- The health of both furnace and oil-quench tank
- Part location
- Connectivity and analytics capability to predict hardenability and distortion
- Intelligence capability to fine-tune recipes based on instantaneous inputs
- Operating conditions
- Deriving location-specific finishing conditions to create value in terms of mitigating distortion in precision components

Furnace maintenance: In a heat treating shop with a battery of furnaces, the maintenance schedule is designed predominately on a time interval, with specific furnaces undergoing maintenance after a fixed time (Fig. 7). This often results in suboptimal operation, with furnaces undergoing maintenance too early or too late. In the Industry 4.0 framework, sensing capability for furnace temperature-time response is needed, such as the heat-up response cycle after a charge is loaded, together with skin temperature monitoring. Connectivity and analytics capability would help to identify influencing parameters, such as charge and basket weight, to provide intelligence capability for burner health. This would trigger furnace and burner maintenance requirements to create value in terms of proactive maintenance with efficient operation and energy reduction. The Industry 4.0 framework also provides architecture to connect the maintenance staff through appropriate alert systems, either through a computer system or mobility and wearable solutions.

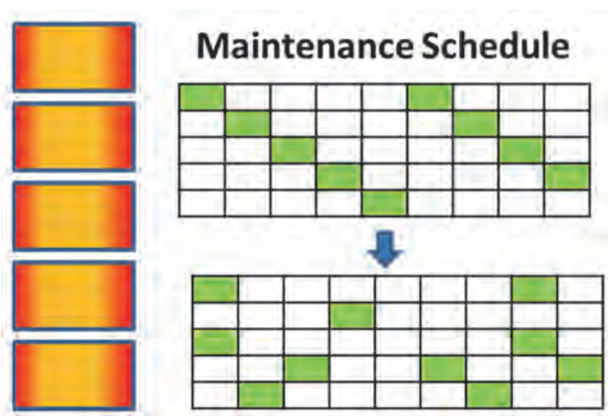


Fig. 7 — Time-based, staggered furnace maintenance schedule, which has a significant impact on efficiency if transformed into a furnace health-based schedule.

CONCLUSION

Industry 4.0 is in the early technology evolution stage with tremendous business expectations and significant research grants available to pursue the technology. Few organizations have taken the lead in identifying cases for using and applying this technology, while the IT infrastructure is nearly ready to be implemented. The challenge lies in identifying value-driven use cases, which could impact efficiency and product cost and quality. In the area of heat treating, an additional challenge will be finding talent with a deep understanding of physical metallurgy and the industrial heat treating domain, in addition to the ability to convert this understanding into analytics, algorithms, and self-learning intelligence that leverage IT systems. The immediate need for the future of this emerging technology is development of such interdisciplinary engineering skillsets (Fig. 8), which could be the biggest obstacle to successful scale-up of this technology. **~HTPro**

For more information: Satyam S. Sahay is a John Deere Fellow, materials engineering, John Deere Technology Center India, John Deere India Pvt. Ltd., Tower XIV, Cybercity, Magarpatta City, Pune 411 013, India, satyamsahay@yahoo.com.

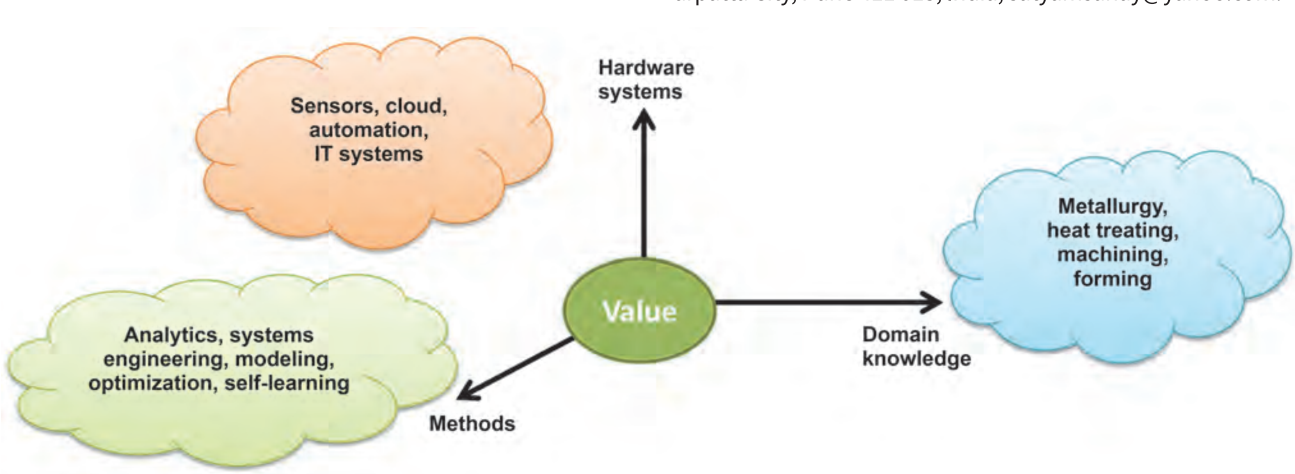


Fig. 8 — Interdisciplinary skills needed to successfully leverage Industry 4.0 in the context of heat treating.

COMPUTER MODELING SINGLE-SHOT INDUCTION HARDENING OF A POWER TRANSMISSION SHAFT

Computer modeling is used in induction hardening process design to improve component quality including hardness, beneficial stress distributions, and reduced distortion.

Zhichao (Charlie) Li* and **B. Lynn Ferguson, FASM,*** Dante Solutions Inc., Cleveland, and **Collin Russell*** and **Valery Rudnev, FASM,*** Inductoheat Inc., Madison Heights, Mich.

The automotive industry is implementing lightweighting initiatives in vehicle design to meet more stringent federal Corporate Average Fuel Economy regulations. New component designs involve material removal resulting in complex geometries containing longitudinal and/or transverse holes, grooves, shoulders, flanges, diameter changes, undercuts, teeth, splines, and more. Many of these components with complex shapes are surface hardened using induction hardening (Fig. 1). Four induction methods routinely used are scan, continuous or progressive, static, and single shot hardening[1].

Irregularities in component geometry distort the magnetic field generated by an inductor, which can cause temperature deviations, hot and cold spots, excessive shape distortion, undesirable microstructures, grain boundary liquation, and cracking. For example, scan hardening shafts with large shoulders, multiple-diameter changes of appreciable size, and other irregularities can produce severe nonuniform hardened patterns. Eddy current flow and temperature fields should be evaluated to determine appropriate process parameters and coil design to prevent cracking and minimize distortion.

Steel shafts and shaft-like components are traditionally induction surface hardened using scanning and single-shot methods. In the single-shot method, the part rotates rather than the shaft or coil moving relative to each other. The entire region to be hardened is heated at the same time. Single-shot inductors typically control hardness pattern and distortion better than scan and static hardening, particularly for stepped shafts. A single-shot inductor consists of two legs and two crossover segments. Crossover segments encircle only half of the workpiece circumference, and induced eddy currents primarily flow along the length of the part. An exception is crossover segments where eddy current flow is half circumferential. Longitudinal leg sections are profiled by relieving selected regions of the copper inductor to accommodate workpiece geometrical features, such as changes in diameter and undercuts.

Inductor configuration depends on factors such as workpiece geometry, temperature uniformity, required hardness pattern, and production rate. The design must take into account the tendency of certain geometrical features to produce heat surpluses and/or heat deficits upon induction heating. Required heat source control can be achieved by adjusting the current-carrying face of the appropriate inductor section, applying flux concentrators, and by varying the inductor-to-workpiece gap. Determining the appropriate inductor profile might be cumbersome and time-consuming.

Fig. 1 — Variety of complex geometry components that are routinely induction hardened.

For critical applications, single-shot inductors are CNC machined from solid copper to conform to the area of the part to be heated. This type of inductor requires the most care in fabrication because it usually operates at high power densities, and workpiece positioning is critical with respect to coil profile. Single shot hardening is also the preferred

*Member of ASM International

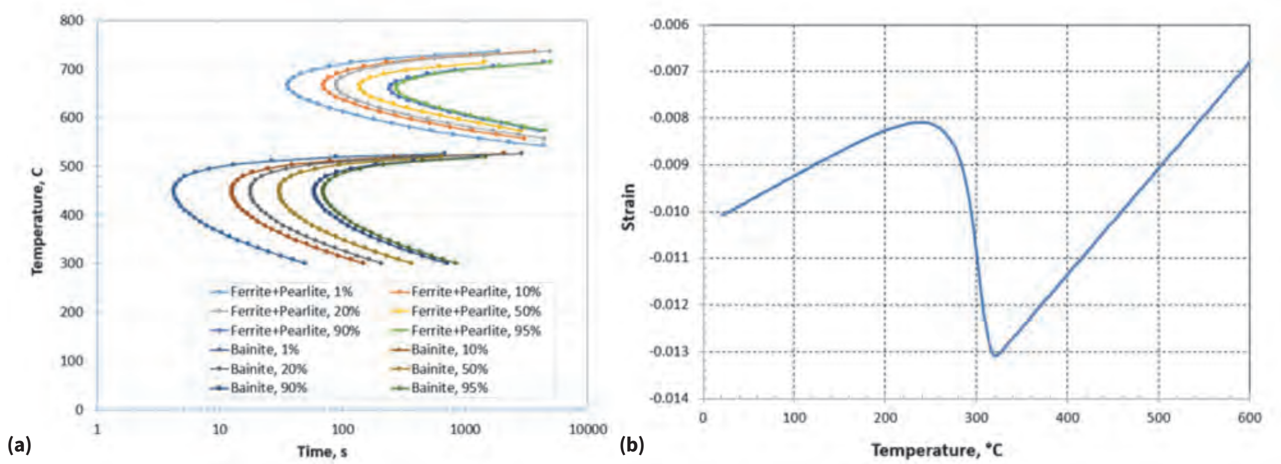


Fig. 2 — TTT diagram (a) and dilatometry strain curve (b) of martensite transformation for AISI 4140 generated from Dante database.

choice when shorter heat times/high production rates are desired. Heating time can be as short as two seconds, but is typically four to eight seconds. Sufficient rotation is critical with single-shot inductor design; at least 10 full rotations per heat cycle is desired. Spray quenching can begin immediately after austenitization is complete, or after a short delay. The duration of quench delay can range from a fraction of a second to a few seconds depending on the geometry of the component, material chemical composition, and hardness pattern specification.

COMPUTER MODELING SPECIFICS

The current production environment does not allow the luxury of process design via trial and error. Computer simulation enables induction heating specialists to quickly determine process details, which could be costly, time consuming, and difficult or impossible to resolve experimentally. Simulation enables prediction of how different interrelated and nonlinear factors could impact the transitional and final thermal conditions of the heated component. It also helps determine what must be accomplished to improve process effectiveness to establish the most appropriate process recipes. Computer modeling provides a comfort factor when designing new systems, avoids unpleasant surprises, shortens the learning curve, and reduces development time.

Today, most software programs are not capable of modeling single shot hardening. However, FEA software developed by Dante Solutions in combination with proprietary subroutines for Flux-3D developed by Inductoheat enable taking into consideration all critical features of the induction hardening process[2].

MODELING INDUCTION HARDENING OF A STEEL SHAFT

Consider a case study of induction hardening of an AISI 4140 alloy steel shaft with shoulders and numerous changes

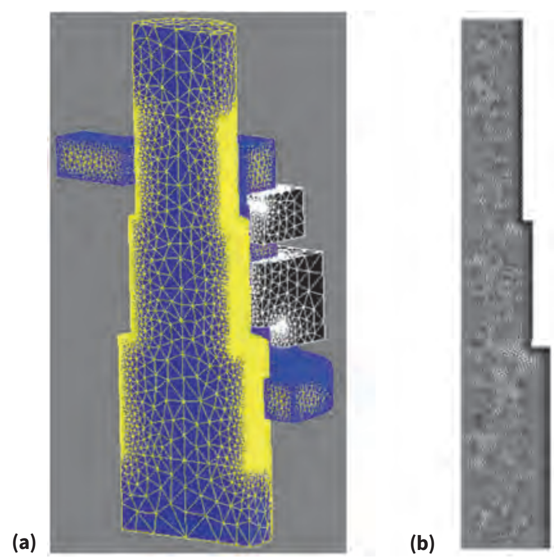


Fig. 3 — Finite element models: (a) electromagnetic model used by Flux-3D, and (b) 2D axisymmetric model used by Dante.

in diameter. Phase transformations occur during induction heating and spray quenching. The surface of the part transforms to austenite during heating, and during quenching, austenite can transform to ferrite, pearlite, bainite, or martensite depending on the cooling rate. Accurate descriptions of phase transformations and mechanical properties of individual phases are required for thermal stress analysis[3]. Figure 2(a) shows the TTT diagram of 4140 generated from the Dante database. The TTT diagram is not used in Dante models directly, but the analytical phase transformation models and database contain all TTT diagram information. Figure 2(b) shows a dilatometry strain curve during martensite formation under continuous cooling. The curve provides the martensite start temperature (M_s), martensite finish temperature (M_f), transformation strain, and coefficients of thermal expansion (CTE) for austenite and martensite. Because the cooling rate during spray quenching is severe, the main transformation in this study is austenite to martensite.

Using Flux-3D, the electromagnetic problem was solved and 3D heat source distribution was obtained at different heating stages. The finite element model is shown in Fig. 3(a). Calculated power distribution from the Flux-3D model was mapped into the Dante model for thermal, phase transformation, and stress analyses; the Dante mesh is shown in Fig. 3(b). After induction heating, the shaft was spray quenched using a polymer solution without delay. Different finite element meshes were used for the Flux and Dante models.

MAPPING FROM FLUX-3D TO DANTE

Copper inductor profiling was optimized and the most appropriate locations for magnetic flux concentrators were determined to address changes in shaft geometry. During the hardening process, the shaft was positioned inside the inductor with a rotation rate of five rotations/second, and a total heating time of five seconds, which translates into 25 full rotations during the heating cycle.

Because temperature distribution is relatively uniform circumferentially in the shaft, a 2D axisymmetric model was used in Dante to model temperature, phase transformation, and stress evolution during heating and quenching. Power distribution in terms of time predicted by Flux-3D was mapped into the Dante heat treatment model. A special mapping subroutine was developed because different finite element meshes were used in the Flux-3D and Dante models. A comparison of temperature profiles projected by Flux-3D and Dante reveals good correlation (Fig. 4).

STRESS AND PHASE TRANSFORMATION MODELING USING DANTE

Temperature, austenite pattern, axial displacement, radial displacement, and hoop stress distributions after two seconds of heating are shown in Fig. 5. At the early stage of heating before the surface transforms to austenite, the surface is under compression due to thermal expansion. After the surface transforms to austenite, the stress decreases to a magnitude close to neutral. Compression occurs under the austenite layer due to thermal expansion, with tensile stress in the core to balance the stress.

Figure 6 shows predicted results at the end of the five seconds of heating; the austenite layer is approximately 5 mm deep. Surface temperature is about 1100°C, and the radial displacement of the heated region is about 0.2 mm.

After heating is complete, the shaft is spray quenched using a 6% polymer solution; a convection coefficient of 15 KW/(m^{2}K) is applied on the shaft surface of the FEA model to represent the spray quench. After two seconds of quenching, surface temperature is about 204°C, which is below the M_s (320°C) of AISI 4140, and martensite formation started on the surface (Fig. 7). Surface stress shifts from tension to compression due to the volume expansion that accompanies martensite formation. Surface stress is tensile due to thermal shrinkage caused by quenching prior to martensite formation.

At about 10.4 seconds of quenching, almost all of the austenite layer transforms to martensite (Fig. 8). The surface temperature is about 70°C and core temperature is about 306°C. Hoop stress on the surface is about −310 MPa under compression, a tensile stress of about 390 MPa is observed under the austenitized layer, and the core is under slight compression of -80 MPa.

Cooling of the core after phase transformation is completed has a significant effect on the change in stresses in the shaft. Hoop stresses are −700 MPa at the surface, +450 MPa at the case-core location, and +150 MPa in the core after the shaft cools to room temperature (Fig. 9). Residual stress distribution affects shaft fatigue performance.

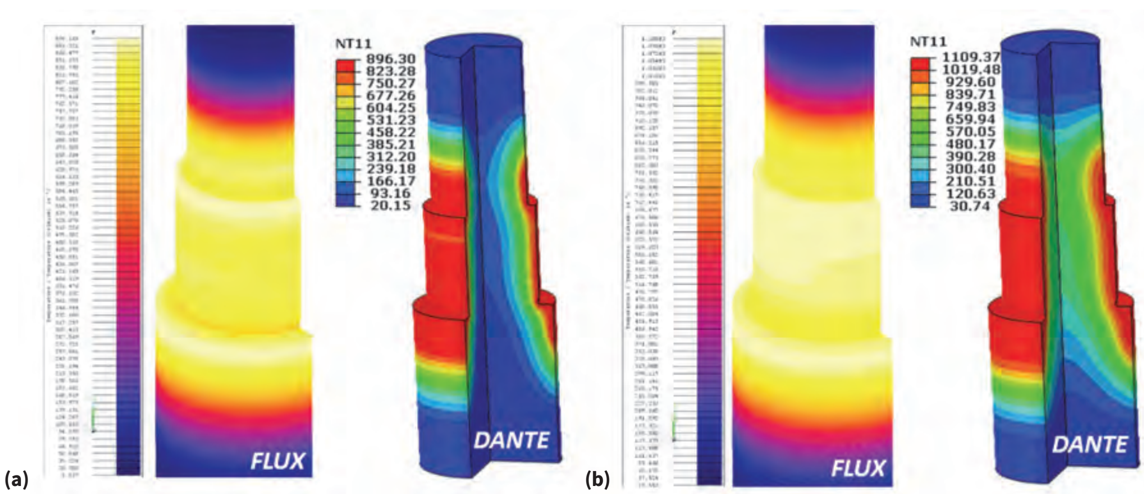


Fig. 4 — Predicted temperature distributions between Flux-3D and Dante during induction heating at 2 s (a) and 5 s (b).

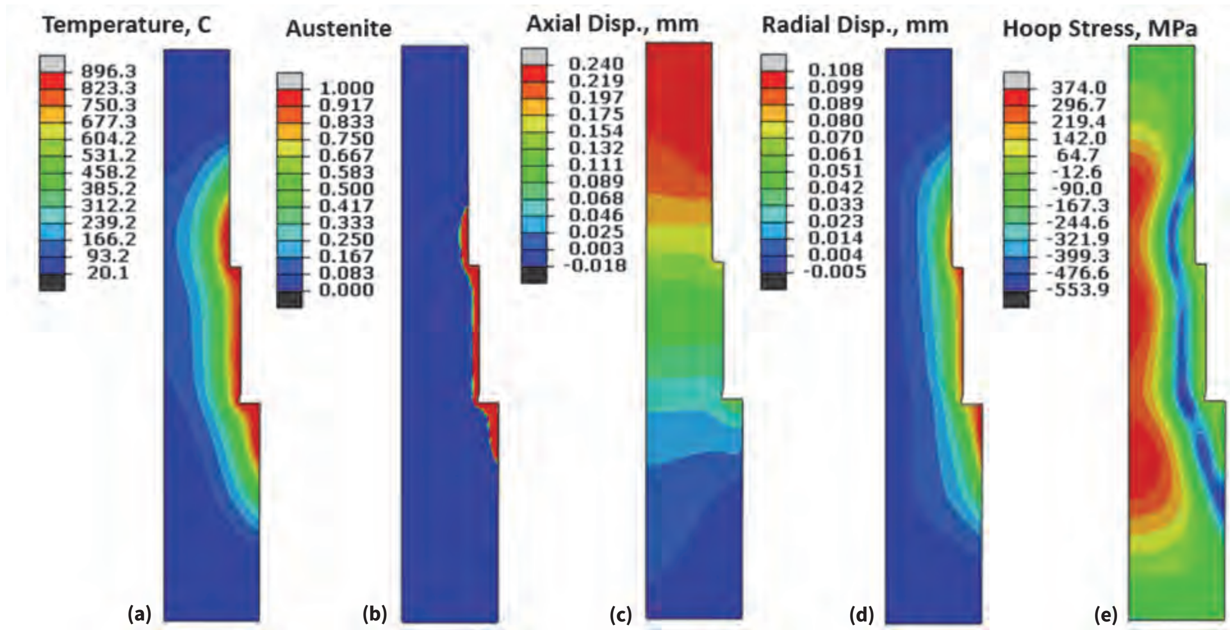


Fig. 5 — (a) Temperature, (b) austenite phase, (c) axial displacement, (d) radial displacement, and (e) hoop stress distributions at 2 s of heating.

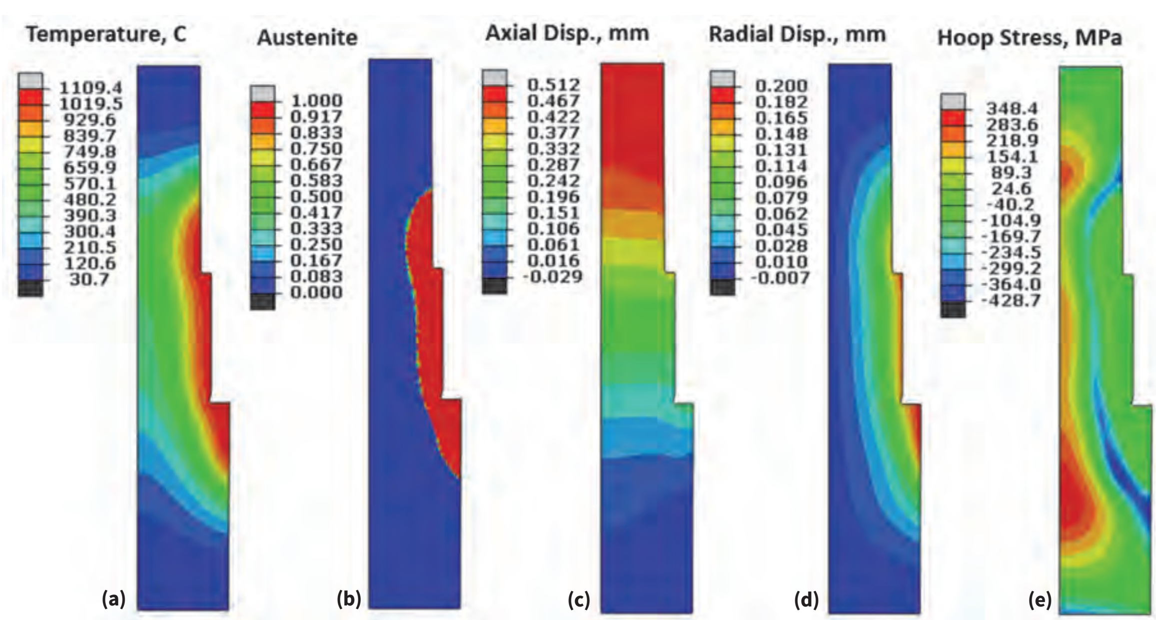


Fig. 6 — (a) Temperature, (b) austenite phase, (c) axial displacement, (d) radial displacement, and (e) hoop stress distributions at 5 s of heating.

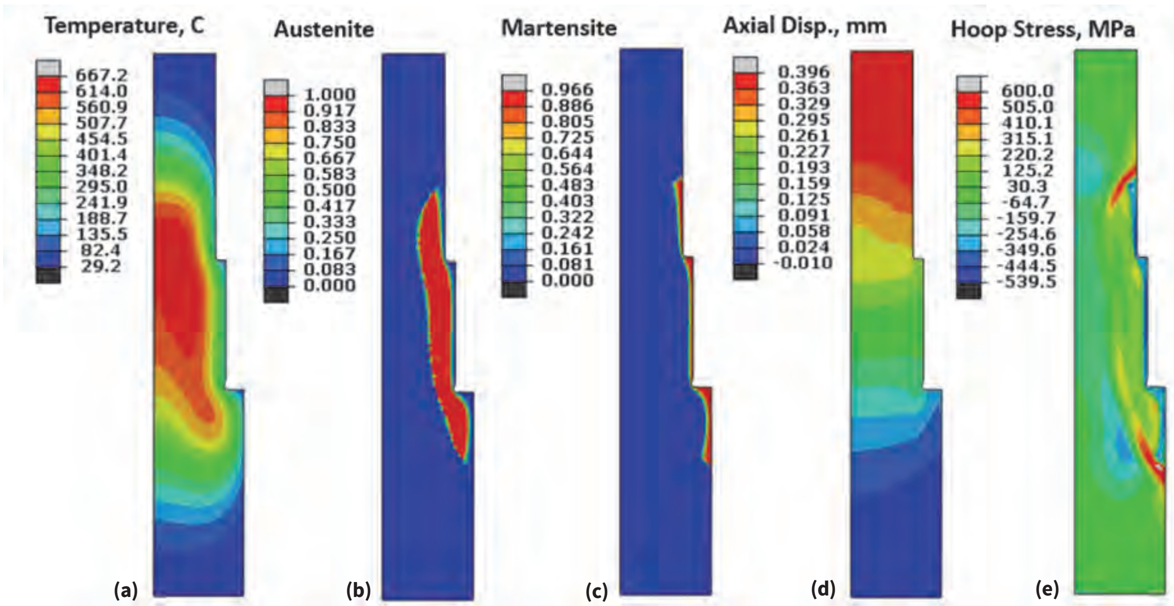


Fig. 7 — (a) Temperature, (b) austenite phase, (c) martensite phase, (d) axial displacement, and (e) hoop stress distributions at 2 s of quenching.

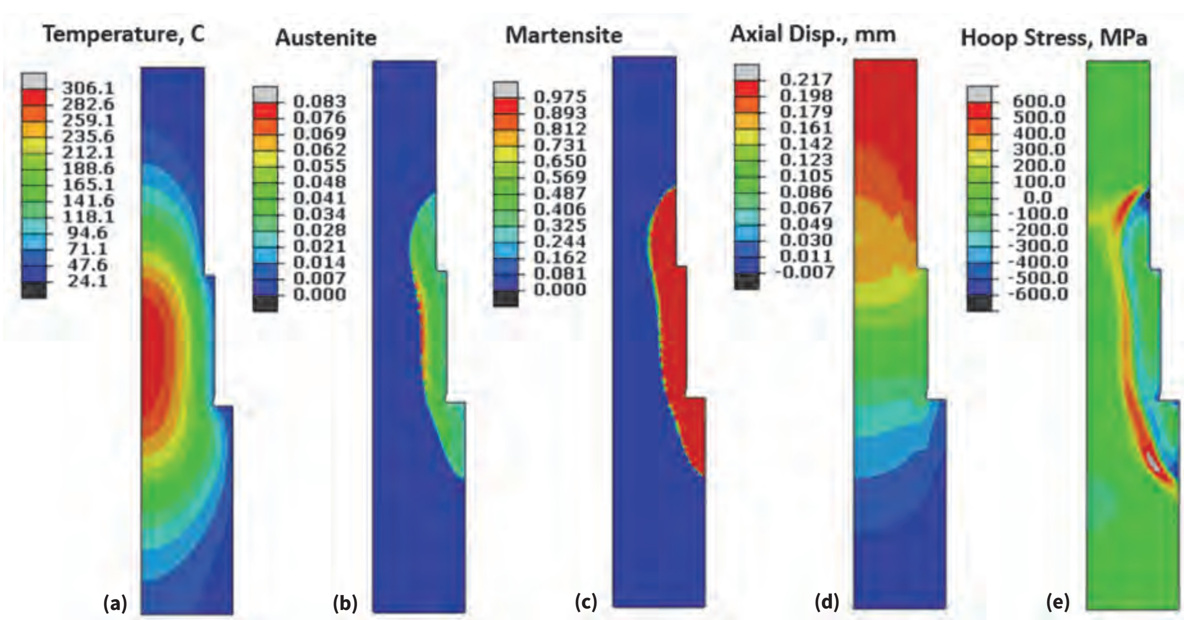


Fig. 8 — (a) Temperature, (b) austenite phase, (c) martensite phase, (d) axial displacement, and (e) hoop stress distributions at 10.4 s of quenching.

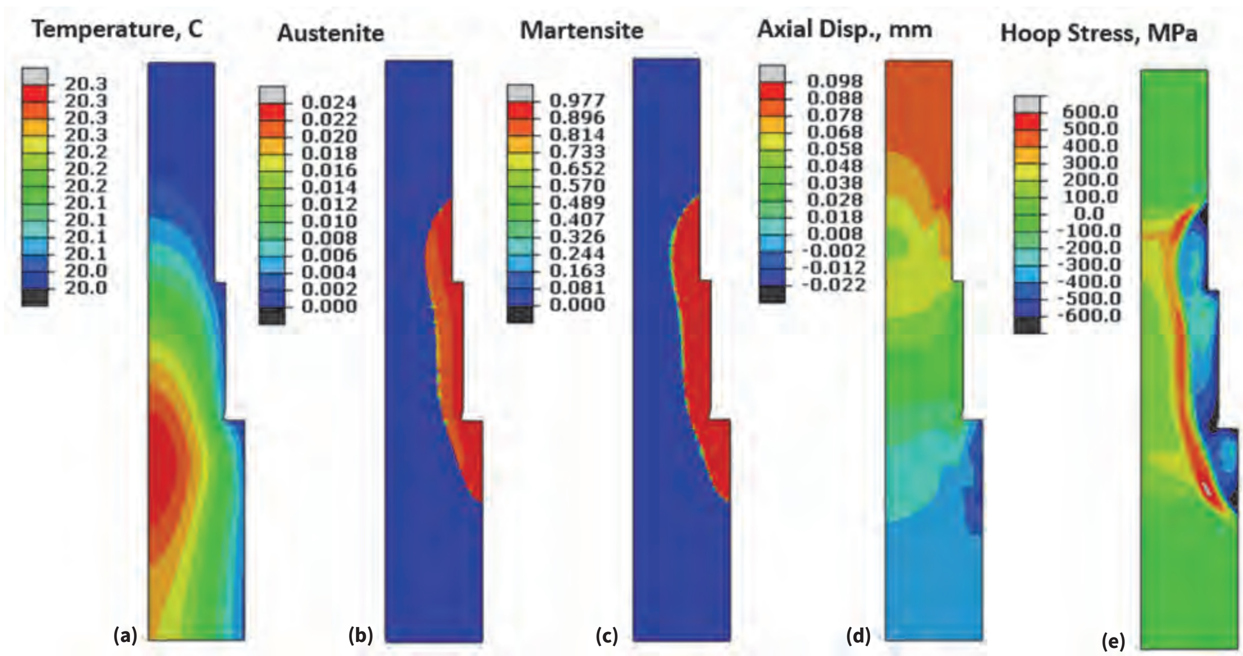


Fig. 9 — (a) Temperature, (b) austenite phase, (c) martensite phase, (d) axial displacement, and (e) hoop stress distributions at the end of quenching.

Compressive residual stresses are preferred on the surface. However, to balance surface compression, tensile stresses exist under the case or at the core, which may lead to failures if the material at those locations exhibits metallurgical or microstructural irregularities (e.g., material is not clean) or if the applied load is too high. Process optimization based on computer modeling is a critical factor to optimize the stress and hardness distribution in specific applications.

CONCLUSION

Years of experience, leveraged by advancements in high performance computers, have improved the cost effectiveness of applying computer simulation during the design and development stages for induction hardening processes. This shortens the learning curve, reducing development time and enabling accurate inductor design and process optimization.~**HTPro**

References

1. *Induction Heating and Heat Treating*, Vol 4C, *ASM Handbook*, V. Rudnev and G. Totten, eds., ASM Intl., 2014.

2. D. Bammann, et al., Development of a Carburizing and Quenching Simulation Tool: A Material Model for Carburizing Steels Undergoing Phase Transformations, *Proc. 2nd Intl. Conf. on Quenching and Control of Distortion*, p 367-375, 1996.

3. Z. Li, B. Ferguson, and A. Freborg, Data Needs for Modeling Heat Treatment of Steel Parts, *Proc. MS&T Conf.*, p 219-226, 2004.

For more information: Zhichao (Charlie) Li, Dante Solutions Inc., Cleveland, OH 44130, (440) 234-8477, charlie.li@dante-solutions.com, www.dante-solutions.com.

ECM USA inc
VACUUM FURNACES

ECM TREATED GEARS DRIVE THE WORLD

Low Pressure Vacuum Carburizing Systems

Innovation

ECM Technologies and ECM USA have installed over 1000 cells of heat treating capacity with almost every Automotive company in the world. These systems provide low pressure vacuum carburizing and gas quenching for millions of parts that bring motion and reliability into our daily lives. From automatic, manual transmissions, dual clutch, CVT, to axles and even airplane engine parts, ECM and our processes are part of your lives. Let us build a system for you.

Our Products

ICBP® Duo

ICBP® Flex

ICBP® Jumbo

ICBP® Nano In-line system

- Vacuum Carburizing
- Gas Quenching
- Oil Quenching
- Carbonitriding
- Neutral Hardening

ECM USA, INC.
9505 72nd Ave. Ste 400
Pleasant Prairie, WI 53158
T. 262.605.4810

GLOBAL MANUFACTURER OF INDUSTRIAL FURNACES

WWW.ECM-USA.COM

ASMNEWS

ASM AFFILIATE SOCIETIES ANNOUNCE NEW OFFICERS AND BOARD MEMBERS

In accordance with their Rules of Governance, five ASM Affiliate Societies have completed their elections for officers and board members for 2016. Please join us in welcoming the following appointments.

ASM Electronic Device Failure Analysis Society

Zhiyong Wang, executive director, Maxim Integrated, succeeds as president of EDFAS while **Cheryl Hartfield,** CDH Consulting, remains on the board as immediate past president. **Lee Knauss, FASM,** chief-technology transition, IARPA, is elected vice president, **James J. Demarest,** E.I.T., IBM, is elected finance officer, and **William Vanderlinde, FASM,** director–Office of Safe and Secure Operations, IARPA, is reappointed secretary. Officers serve a two-year term. **Sam Subramanian,** senior member of technical staff, NXP Semiconductors, was elected to the board for a two-year term as general chair of ISTFA.

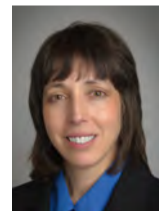
Rebecca Holdford, failure analyst, **Ted Lundquist,** consultant, and **Ryan Ross,** manager, Analysis and Test Laboratory, NASA Jet Propulsion Laboratory, were elected to the EDFAS board for a four-year term.

ASM Failure Analysis Society

Burak Akyuz, team lead–metallurgy and failure analysis, Applied Technical Services Inc., FAS president, and **Pierre Dupont,** account manager–industry, Schaeffler Belgium, FAS vice president, are pleased to welcome the inaugural FAS Board. **Roch Shipley,** principle engineer, Professional Analysis and Consulting Inc., was appointed treasurer and **James F. Lane,** senior engineer, Professional Analysis and Consulting Inc., was appointed secretary. Serving as immediate past president is **Erhan Ulvan,** manager–engineering and laboratories, Acuren Group Inc.

Appointed to board member positions are **Donato Firrao, FASM,** professor, Politecnico di Torino and **Tim Jur,** chief engineer emeritus, ED&T Corp. (one-year terms); **Erik Mueller,** materials research engineer, NTSB, and


Wang

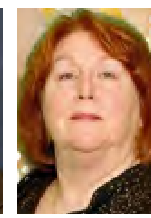

Hartfield


Knauss


Demarest


Vanderlinde


Subramanian


Holdford


Lundquist


Ross


Akyuz


Dupont


Shipley


Lane


Ulvan


Firrao


Jur


Mueller


Parrington


Dalley


Dennies

In This Issue

Submit news of ASM and its members, chapters, and affiliate societies to Frances Richards, editor, ASM News | ASM International
9639 Kinsman Road | Materials Park, OH 44073 | P 440.338.5151 ext. 5563 | F 440.338.4634 | E frances.richards@asminternational.org
Contact ASM International at 9639 Kinsman Road, Materials Park, OH 44073 | P 440.338.5151 ext. 0 or 800.336.5152 ext. 0 (toll free in U.S. and Canada) | F 440.338.4634 | E MemberServiceCenter@asminternational.org | W asminternational.org


Grice


Kelly


Kowalski


Oakes


Cryderman


Powell


Rowan


Wingens

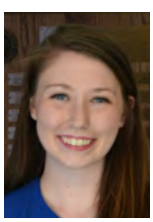

Jones


Noll


Whitley


Frafjord


Keeble


Lukas

Ron Parrington, FASM, director–industrial services, ESI Inc. (two-year terms), and **Amber Dalley,** director of business development–materials and environmental, RJ Lee Group, and **Daniel P. Dennies, FASM**, principal, DMS Inc. (three-year terms).

Daniel Grice, senior materials engineering, Materials Evaluation and Engineering Inc., was appointed emerging professional board member and **Sean M. Kelly,** Worcester Polytechnic Institute, was appointed student board member. Both are one-year appointments.

ASM Heat Treating Society

HTS president **Stephen G. Kowalski,** president, Kowalski Heat Treating Co., and HTS vice president, **James P. Oakes,** vice president business development, Super Systems Inc., welcome the following new members to the HTS board for a three-year term: **Robert Cryderman,** research associate professor, Colorado School of Mines; **Joseph Powell,** president, Akron Steel Treating Co.; **Olga Rowan,** senior engineer, Caterpillar Inc. **Thomas Wingens,** director, Wingens International Industry Consultancy, was appointed to a one-year term to complete an unexpired term.

Roger A. Jones, corporate president, Solar Atmospheres Inc., remains on the board as immediate past president.

Hannah M. Noll, superintendent, Bakers Powder Operations, ATI Specialty Materials, was reappointed young professional board member and **Blake M. Whitley,** Colorado School of Mines, was appointed student board member. Both are one-year appointments.

ASM International Metallographic Society

IMS president **Jaret J. Frafjord,** laboratory director, IMR Test Labs–Portland, announced the appointment of **Mike Keeble,** US labs and technology manager, Buehler, as an IMS director for one year, filling an unexpired term. **Rachel Lukas,** University of Pittsburgh, was appointed as a student board member for one year.

ASM Thermal Spray Society

The TSS electorate elected a vice president and three directors to the board; the TSS executive committee appointed two student board members; and the president-elect appointed a secretary/treasurer. See page 3 of *iTSSe* in this issue for the full story.

Seeking Applicants for SMST Fellowship

The International Organization on Shape Memory & Superelastic Technologies (SMST), an affiliate society of ASM International, is seeking applications for the 2017 SMST Fellowship. The intent of the fellowship is to provide a current use gift to a deserving graduate student(s) with the purpose of initiating interest in a unique path of research for shape memory materials such as Nitinol. The award, which is financially supported in 2017 by Edwards LifeSciences, includes a stipend up to $50,000. The recipient will present research results at the 2019 SMST Conference to be held in Constance, Germany. Application deadline is **January 9, 2017.** For more information visit http://bit.ly/29pjlkD or contact sarina.pastoric@asminternational.org.

SEEKING NOMINATIONS FOR NEW EDFAS AWARDS

EDFAS is pleased to announce two new awards to recognize the accomplishments of its members. The awards will be given annually, with the inaugural EDFAS Lifetime Achievement Award and the EDFAS Presidents Award being presented at ISTFA 2017. Deadline for both awards is **March 1, 2017.** For more information, visit asminternational.org/web/edfas/societyawards. Nominate a worthy colleague today!

EDFAS Lifetime Achievement Award

The EDFAS Lifetime Achievement Award was established by the EDFAS Board of Directors in 2015 to recognize leaders in the EDFAS community who have devoted their time, knowledge, and abilities to the advancement of the electronic device failure analysis industry.

EDFAS President's Award

The EDFAS President's Award shall recognize exceptional service to EDFAS and the electronic device failure analysis community. Examples of such service include committee service, service on the board of directors, organization of conferences or symposia, development of education courses, and student and general public outreach. While any member of EDFAS is expected to further the Society's goals through service, this award shall recognize those who provided an exceptional amount of effort in their service to the Society. For complete rules and nomination forms, visit edfas.org, click on Membership & Networking and then Society Awards or contact Joanne Miller at 440.338.5151 ext. 5513, joanne.miller@asminternational.org.

Deadline for both awards is **March 1, 2017.** For more information, visit asminternational.org/web/edfas/societyawards.

Nominations Due for 2017 ASM Nominating Committee

ASM International is seeking members to serve on the 2017 ASM Nominating Committee. The committee will select a nominee for 2017-2018 vice president (who will serve as president in 2018-2019) and three nominees for trustee. Committee candidates may only be proposed by a Chapter through its executive committee, an ASM committee or council, or an affiliate society board. Nominations are due **December 15.** For more information, contact Leslie Taylor at 440.338.5151, ext. 5500, leslie.taylor@asminternational.org, or visit asminternational.org/about/governance/nominating-committee.

Nomination Deadline for 2017 Class of Fellows is Fast Approaching

The honor of Fellow of the Society was established to provide recognition to members for distinguished contributions in the field of materials science and engineering, and to develop a broadly based forum for technical and professional leaders to serve as advisors to the Society. Criteria for the Fellow award include:

- Outstanding accomplishments in materials science or engineering
- Broad and productive achievement in production, manufacturing, management, design, development, research, or education
- Five years of current, continuous ASM membership

Deadline for nominations for the class of 2017 is **November 30, 2016**. View rules and past recipients at asminternational.org/membership/awards. To receive a unique nomination form link, contact Christine Hoover at christine.hoover@asminternational.org.

Annual ASM Award Nominations due Feb. 1, 2017

The deadline for the majority of ASM's awards is **February 1, 2017,** and we are actively seeking nominations for all of these awards, a few of which are listed below:

- Edward DeMille Campbell Memorial lectureship
- Distinguished Life Membership
- William Hunt Eisenman Award
- Gold Medal
- Silver Medal
- Bronze Medal
- Historical Landmarks
- Honorary Membership
- Medal for Advancement of Research
- Allan Ray Putnam Service Award
- Albert Sauveur Achievement Award
- Albert Easton White Distinguished Teacher Award
- J. Willard Gibbs Phase Equilibria Award

View rules and past recipients at asminternational.org/membership/awards. To receive a unique nomination form link, contact Christine Hoover at christine.hoover@asminternational.org.

WOMEN IN ENGINEERING

This new profile series introduces leading materials scientists from around the world who happen to be females. Here we speak with ***Ida Berglund,*** *materials design engineer for QuesTek Innovations LLC.*

Berglund

What does your typical workday look like?

It starts with a cup of coffee (or three) at my desk while going through emails and outlining my agenda for the day. There are often one or several internal project meetings during the day to discuss technical work and strategies, and the remaining time is spent by the computer on data analysis, reporting, communication, or doing basic research. There is also the daily lunch group effort on solving the Chicago Tribune crossword puzzle.

What's been your biggest technical challenge?

Being able to easily convey scientific concepts, ideas, and methods to a new audience. Also, despite almost six years in the U.S. I cannot appreciate magnitudes of inches, ksi, miles, and such, and find myself having to convert to SI units during conversations and presentations.

What part of your job do you like most?

The variety. There is a balanced combination of technical work, meetings, reporting, client or collaborator interactions, and proposal writing, as well as travel to conferences or client meetings. Further, QuesTek works with a variety of alloys, products, customers, and partners, thus the technical work varies significantly, requiring me to constantly expand my knowledge in not only alloy design, but in multiple fields, which I greatly enjoy.

What is your engineering background?

I started my undergraduate studies in materials design and engineering at the Royal Institute of Technology in Stockholm and continued through a master's program specializing in biomedical engineering while also focusing on metallurgy. This rather unusual combination of design, biology, and metallurgy prepared me for an ideal Ph.D. project on biomedical alloy design at University of Florida.

What are you working on now?

I'm currently involved in seven projects focusing on various classes of alloys or processes for different applications. While there is a large variety between the projects, the methodologies and tools I use are similar between them and utilize integrated computational materials engineering, with work spanning from alloy design concept generation to process assessment and property modeling. As an example, I'm looking at chemistry modifications of a commercial alloy and its effect on properties, by modeling the phase evolution with time using thermodynamic and kinetic models, and correlating the nano- and microstructure to macroscopic behaviors. The principles are the same whether you work with turbine blades or gum wrappers.

How many people do you work with?

QuesTek has 23 fulltime employees. The majority have Ph.D.s, in materials science, mechanical engineering, or physics, and while male dominated, there is a variety of cultural backgrounds, ages, and personalities among us. The small size of the company makes personal day-to-day interactions inevitable, and I'm happy to be around people who are not only intelligent, but also fun and kind.

If a young person approached you for career advice about pursuing engineering, what would you tell them?

Engineering is probably not what you think it is, or at least only part of it. Engineering skills can be applied to almost any field, regardless of discipline. As long as you enjoy what you do and are willing to learn new things, you can make a career out of it. Therefore I would encourage any engineering studies or training. I would further suggest getting involved in professional organizations within fields of interest by attending meetings and networking to get a better understanding of the society you're considering going into. This could provide a deeper insight than, for example, textbook reading or listening to people's opinions and experiences. It could also give you an idea of the structure of the community, which could identify paths needed to reach a certain position or environment you are interested in.

Hobbies?

Traveling, skiing, dining, baking, and hiking. Basically anything involving new experiences or food and drinks.

Last book read?

"Porten mot evigheten" by Ulf Westblom and John Ågren.

For more information about ASM's Women in Materials Engineering Committee, visit asminternational.org/wime.

VOLUNTEERISM COMMITTEE

Profile of a Volunteer

Beth Matlock, Senior Materials Engineer, Technology for Energy Corp. (TEC)

Matlock

When you're blazing a new trail, it's good to have help. In the 1970s, Beth Matlock was the only woman among 15 classmates in materials engineering at North Carolina State University. She started out as a pre-med student, but a chance encounter discussing super-sharp scissors while working at J.C. Penney led to a job in an x-ray diffraction lab and the decision to change her major. After winning an ASM academic paper contest, she was off and running. "It was maybe only $25, but to me that award was huge," recalls Matlock. "I got support and mentoring from people in the industry."

Matlock graduated in 1977 and moved to Lynchburg, Va., to work in failure analysis for Babcock and Wilcox. Her boss made it clear to all employees: "You will be active in ASM." Matlock served on the executive committee and laughs recalling her "greatest contribution:" changing invitations from "bring your wife" to "bring your spouse." She and her husband moved to Knoxville, Tenn., in 1982 for his job and welcomed their first child. Working as a consultant, Matlock joined the Oak Ridge Chapter and helped with membership outreach.

Employed by TEC for 32 years, Matlock works in the materials testing division. She's served in numerous roles with the Oak Ridge Chapter including chair in 2010 and marvels at tremendous programs in this active chapter. Her two children attended meetings and both became engineers. "My son went to more meetings than most members!" she says with a smile, remembering topics like Cummins Engine Co., the Titanic, and the legend of the Damascus sword.

Matlock is still an active ASM volunteer for membership outreach. She also runs her own beef cattle farm and plays piano for her church. "We were put on this earth to give back," she says. "ASM has given much to me. I need to pass that on to young students and engineers."

CHAPTERS IN THE NEWS

Oak Ridge Hosts Materials Camp

Participants and volunteers of the Oak Ridge Chapter's 2016 Materials Camp held this summer at the University of Tennessee in Knoxville.

ASM Announces Mexico Chapter

ASM welcomed its newest Chapter—Mexico—during September 20-23 at the inaugural Heat Treat Mexico conference. Bienvenido! See page 3 in *HTPro* for the full story.

Adriana Michaca and Carlos Carrasco.

MEMBERS IN THE NEWS

Lampman Named Alpha Sigma Mu Fellow

Lampman

ASM's very own **Steve Lampman** was recognized as a Fellow of Alpha Sigma Mu at a ceremony held during October at MS&T16 in Salt Lake City. Lampman has served as an editor of the *ASM Handbook* series for nearly three decades. Alpha Sigma Mu is the international professional honor society dedicated to encouraging and recognizing excellence in the materials engineering field. Members consist of students, alumni, and other professionals who have demonstrated exceptional academic and professional accomplishments. Founded in 1932, Alpha Sigma Mu has since expanded to include all materials disciplines.

Doll Awarded Visiting Fellowship

Doll

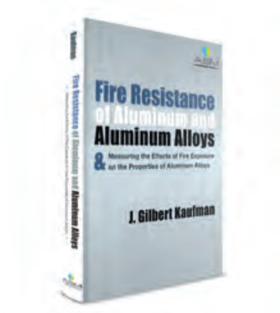
University of Akron (UA) professor **Gary Doll** has been awarded a Royal Academy of Engineering Distinguished Visiting Fellowship allowing him to improve his interactions with the UK's research base in tribology and surface engineering and to facilitate collaborative research at The University of Manchester. "I hope to continue my research on the surface engineering of materials to address friction, wear, and corrosion, and lubrication strategies for challenging environments," says Doll, Timken Professor of surface engineering and director of the Timken Engineered Surfaces Laboratory.

Seifi named Director of ASTM's Additive Manufacturing Programs

Seifi

ASTM International recently hired **Mohsen Seifi** as a technical consultant in additive manufacturing (AM) to support standards development and related needs. Seifi, a post-doctoral research associate at Case Western Reserve University, will serve as director of AM programs. Seifi is a globally recognized expert in process-structure-property relationships for metal AM. As director, he will track key trends, drivers, and innovations while also building relationships with ASTM members, external networks, and professional organizations. Seifi will play a key role in collaborating with ASTM's Committee on Additive Manufacturing Technologies (F42) as well as other committees interested in AM.

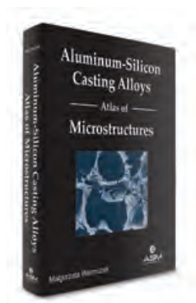
ASM INTERNATIONAL



Fire Resistance of Aluminum and Aluminum Alloys and Measuring the Effects of Fire Exposure on the Properties of Aluminum Alloys

Author: J. Gilbert Kaufman
Hardcover | **Product code:** 05917G | **ISBN:** 978-1-62708-106-1

PRICE: $149.00 | **MEMBER PRICE:** $109.00

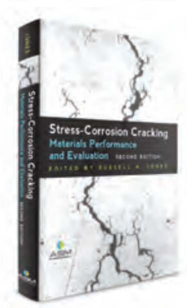


Aluminum-Silicon Casting Alloys: Atlas Of Microstructures

Author: Małgorzata Warmuzek
Hardcover | **Product code:** 05919G | **ISBN:** 978-1-62708-108-5

PRICE: ~~$199.00~~ **$179.00** | **MEMBER PRICE:** ~~$149.00~~ **$129.00**

Prepublication prices good through November 30, 2016



Stress-Corrosion Cracking: Materials Performance and Evaluation, Second Edition

Editor: Russell H. Jones
Hardcover | **Product code:** 05509G | **ISBN:** 978-1-62708-118-4

PRICE: ~~$199.00~~ **$179.00** | **MEMBER PRICE:** ~~$149.00~~ **$129.00**

Prepublication prices good through December 31, 2016

Life Lessons of a Failure Analyst

Author: McIntyre R. Louthan, Jr.
Softcover | **Product code:** 05921G | **ISBN:** 978-1-62708-110-8

PRICE: $29.00 | **MEMBER PRICE:** $22.00

WWW.ASMINTERNATIONAL.ORG/STORE

ADVANCED MATERIALS & PROCESSES

EDITORIAL PREVIEW

JANUARY 2017

Advanced Manufacturing Processes

Highlighting:
- Next-Generation Steels
- High-Pressure Die Casting
- Bulk-Nanostructured Metal

Advertising Bonus:
- Corporate Spotlights

Advertising closes December 5

FEBRUARY 2017

Microscopy/Metallography/Materialography

Highlighting:
- Emerging Topics in Microscopy
- Jacquet-Lucas Award Winner
- Advanced Imaging Techniques

Special Supplement:
International Thermal Spray and Surface Engineering newsletter covering coatings in the aerospace and defense industries.

Bonus Distribution:
M&M 2017 + IMS Annual Meeting
August 6-10, St. Louis, Mo.

Advertising closes January 5, 2017

Subscriptions/Customer Service:
800.336.5152 ext. 0
MemberServiceCenter@asminternational.org

Sales Staff:
***AM&P/iTSSe*/ASM Web Media**
Erik Klingerman, *National Sales Manager*
440.338.5151 ext. 5574
erik.klingerman@asminternational.org

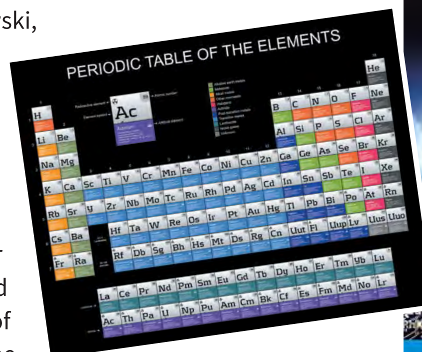
Affiliate Sponsorships
Kelly Thomas, CEM.CMP, *National Account Manager*
440.338.1733
kelly.thomas@asminternational.org

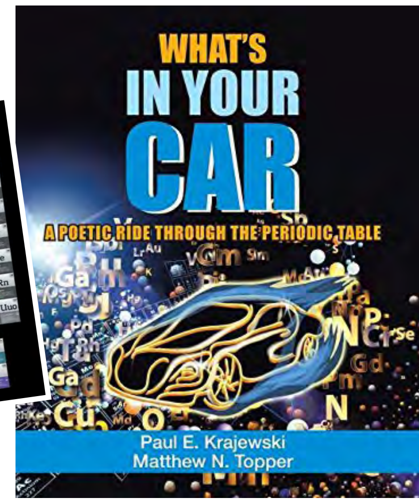
AD INDEX

STRESS RELIEF

POETRY BOOK PUTS THE PEDAL TO THE METAL

A new book of poetry coauthored by an ASM Fellow provides a playful glimpse into the complex field of automotive materials. “What’s in Your Car: A Poetic Ride Through the Periodic Table,” by Paul E. Krajewski, FASM, and Matthew N. Topper uses a combination of catchy poems and interesting pictures to explore how elements of the periodic table are used to make a car. Thirty-one elements are shown with images of the raw materials and their application within a vehicle. Materials are described with simple poems that can serve as a starting point for deeper exploration into the world of materials and automobiles. The book aims to appeal to children of all ages as they learn how different materials come together to create today’s automobiles. Included here are a few excerpts.





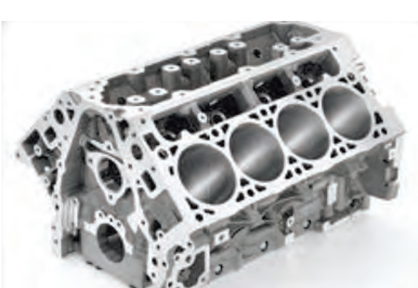
“What’s in Your Car,” by Paul E. Krajewski and Matthew N. Topper.

Introduction

The elements are bricks that
construct all things,
From magnificent buildings to
diamond rings.
They give objects properties, and
each one has a role
That can make it unique, or part
of the whole.
Shiny or light, heavy or strong,
Conductive or bright, you can’t go wrong.
This book tells the story of what’s in a car.
From aluminum to zinc, they help you
drive far.
So buckle your belt and adjust your view.
Here’s a book about elements in a car
just for you.

Aluminum

We use aluminum almost every day,
But I’ll bet you didn’t know it
could be used this way.
Aluminum is applied all over the car;
You could say it’s a lightweight superstar.
Wheels are forged and engines cast,
Making cars that are both cool and fast.
Aluminum’s surface refuses to rust,
It’s a metal for hoods that
engineers trust.



Aluminum was once viewed as more precious than gold and silver.

Molybdenum

Molybdenum’s a tongue twister,
an out-and-out folly,
So most people just use its
nickname, moly.
Adding it to steel is an absolute must,
To make that material more
resistant to rust.
Moly and sulfur create a slick actor,
Making lubes and greases
for your car or tractor.

Molybdenum is used as a fertilizer for cauliflower and broccoli.

Vanadium

Vanadium is found in many
a mineral and deposit.
The Model T could not have
been made without it.
Vanadium makes steel
wear resistant and strong,
Creating axles and crankshafts
that can last long.
It also helps make battery acid and more,
like the performance valve
material Titanium 64.

Vanadium is found in ocean algae.

3D PRINTSHOP

DIGGING UP A NEW ADDITITVE APPLICATION

Scientists at Oak Ridge National Laboratory (ORNL), Tenn., are designing and producing the world's first 3D-printed excavator, a prototype that will leverage large-scale additive manufacturing (AM) technologies and explore the feasibility of printing with metal alloys. The scientists are collaborating with industry partners and student engineers from the University of Illinois at Urbana-Champaign who won a design competition for the project. Three excavator components will be printed at the DOE's Manufacturing Demonstration Facility at ORNL: the cab, the stick (a large hydraulically articulated arm), and a heat exchanger. The stick will be fabricated with the Wolf System using a freeform technique for printing large-scale metal components, and the heat exchanger will be printed on a Concept Laser machine that produces metal parts through a powder-bed-based laser melting process. During the nine-month fabrication and assembly period, the team aims to develop processes to improve material performance and printability and validate models to reduce residual stress and distortion. *ornl.gov.*

3D-printed excavator made of carbon fiber-reinforced ABS plastic. Courtesy of ORNL.

3D PRINTERS VULNERABLE TO SMARTPHONE SPIES

Researchers at the University of Buffalo, N.Y., discovered that 3D printers can be hacked with a common smartphone, threatening intellectual property. While many 3D printers have security features such as encryption and watermarks designed to foil cyberattacks, the Buffalo team sidestepped those safeguards and programmed a smartphone's built-in sensors to measure the machine's electromagnetic energy and acoustic waves, which allowed them to map the print nozzle's movements as it printed. The phone, held 20 cm from the printer, gathered enough data to replicate a simple object with 94% accuracy. For complex objects such as an automotive part, the accuracy rate was still above 90%. To counter this vulnerability, researchers suggest several ways to make 3D printers more secure: Put a moat around the printer because the ability to obtain accurate data for simple objects diminished to 87% at 30 cm and 66% at 40 cm; or increase the print speed—emerging materials may enable faster printing, reducing the ability to track the print nozzle. Other options include using software and hardware-based solutions to program the printer to operate at different speeds or installing acoustic and electromagnetic shields. *buffalo.edu.*

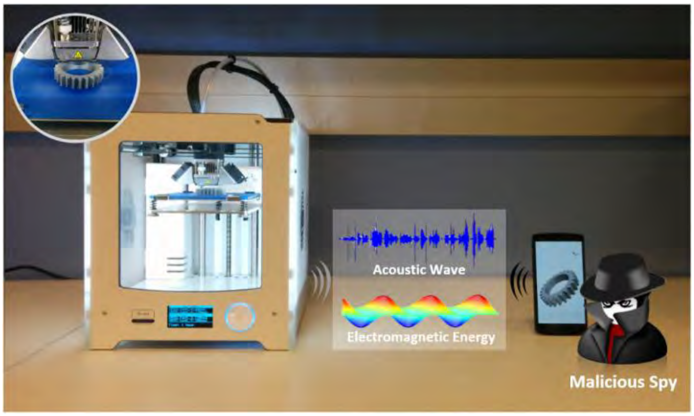


Illustration of a smartphone hacking a 3D printer.

COMING TOGETHER IN COLORADO

A public-private partnership has been established to advance AM methods for Nitinol, a nickel-titanium alloy increasingly used in the medical industry. The Colorado School of Mines, Confluent Medical Technologies, and the Development of Additive Processing Technologies (ADAPT) program will join forces to optimize the superelastic properties of AM-synthesized Nitinol, which exhibits unique shape memory properties in addition to being superelastic.

"Over the next few years, we expect additive manufacturing technologies to have a profound effect on the medical device industry," says Tom Duerig, founder of Confluent, a contract manufacturer of Nitinol-based medical devices. As part of the collaboration, the company established the Confluent Medical Postdoctoral Fellowship at the School of Mines with an initial $100,000 gift. In addition, a full-time postdoctoral researcher was hired for the project by ADAPT, a new R&D consortium dedicated to developing next-generation data informatics and advanced characterization technologies for AM. *mines.edu, confluentmedical.com, inside.mines.edu/ADAPT-Home.*