

APRIL 2021 | VOL 179 | NO 3

ADVANCED MATERIALS & PROCESSES

AEROSPACE MATERIALS AND TESTING TITANIUM MICROTTEXTURE PART I

P. 14

19

Near Net Shape Extrusion
for Aerospace

23

South American
Historic Metals

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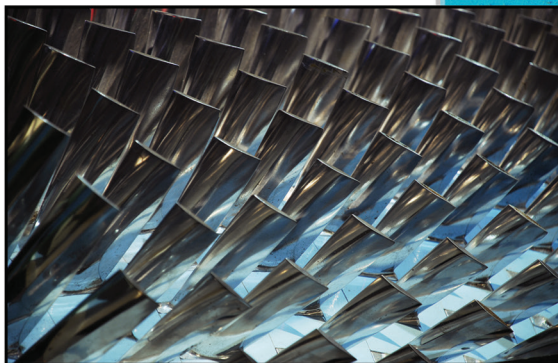
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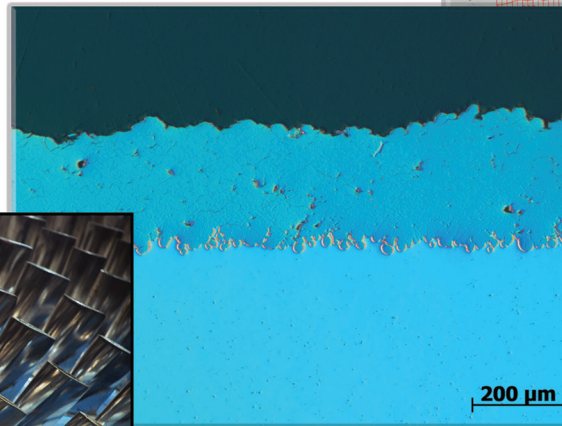
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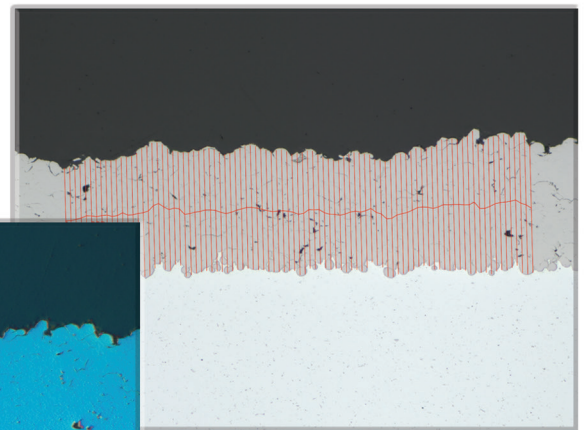
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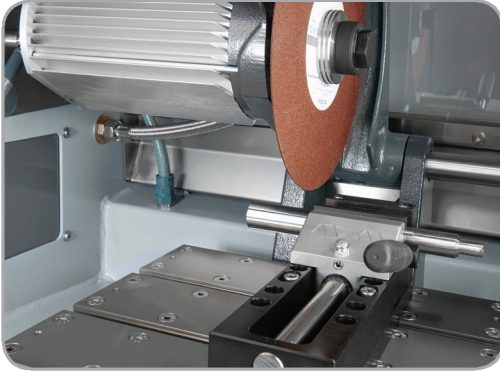
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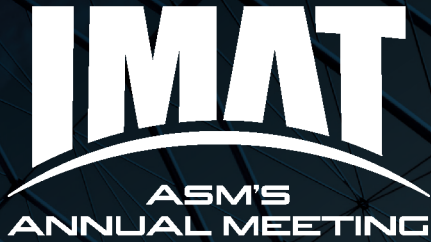
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Planning is well underway for IMAT 2021, ASM's reimagined Annual Meeting. A diverse group of materials experts, including the ASM Programming Committees, AeroMat Committee, and all six of ASM's Affiliate Societies, are heavily involved in building the technical symposiums, which will have a strong focus on application-oriented, real-world technologies that can be put to use today.

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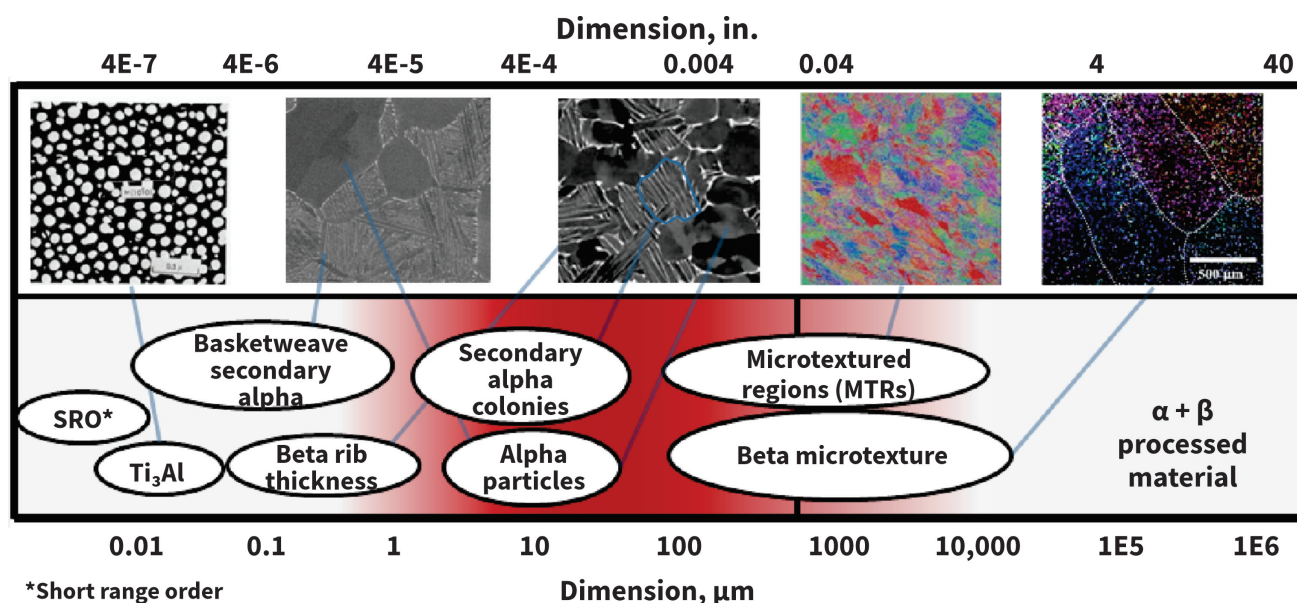
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TITANIUM MICROTEXTURE 101 – PART I

Michelle Harr, Adam Pilchak, and Lee Semiatin

Despite decades of research, fundamental questions still exist regarding the role of microtexture on the deformation, fatigue, and fracture behavior of titanium alloys.

On the Cover:

The Collins Aerospace integrated landing system for the A350-1000 features more than two dozen titanium components as part of the landing gear, wheels, and brakes.

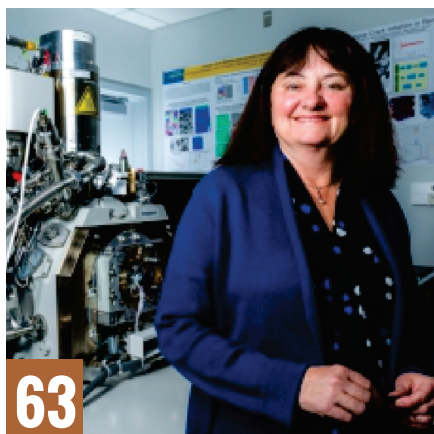


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EPA SAYS COPPER SURFACES HELP FIGHT COVID-19

Harold T. Michels

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19 ADVANCEMENTS IN NEAR NET SHAPE EXTRUSION FOR AEROSPACE APPLICATIONS

Phani P. Gudipati and Michael B. Campbell

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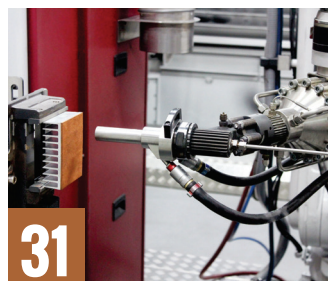


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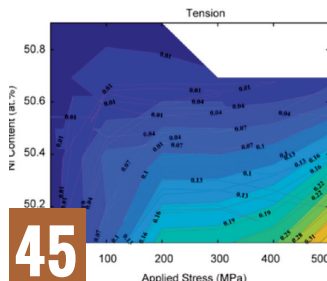
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The official newsletter of the International Organization on Shape Memory and Superelastic Technologies (SMST). This biannual supplement covers shape memory and superelastic technologies for biomedical, actuator applications, and emerging markets, along with SMST news and initiatives.

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GIVING LIFT TO AEROSPACE RECOVERY



It's been a yearlong, bumpy ride for the aerospace industry. But despite experiencing a pandemic-induced sharp descent, analysts forecast a recovery beginning this year (albeit quicker for the defense sector than commercial). And engineering will be key to that recovery. According to Deloitte, technological advancements and innovation could transform the industry and lead to long-term growth. Out on the horizon, areas of emerging technologies include advanced aerial mobility (AAM is being advanced by NASA and

the Federal Aviation Administration), hypersonics (such as defense glide vehicles and cruise missiles), electric propulsion (e.g., Rolls-Royce's hybrid version of the M250 gas turbine), and hydrogen-powered aircraft (e.g., a planned zero-emission aircraft from Airbus).

While those innovations may sound too high in the clouds, in this issue we share some titanium advancements related to current aviation technology. First, "Titanium Microtexture 101"—Part I of a two-part series—explains this long-standing fatigue-related phenomenon in aerospace components. Second, we share how recent improvements to near net shape titanium extrusion make it a viable manufacturing option, not just for long structural components as it has been traditionally used, but for additional types of aircraft parts.

Back on the ground, Patricia Silvana Carrizo—who prompted the formation of ASM's Archaeometallurgy Community and has been leading its efforts—dug around in South America to bring us a review of copper artifacts found in her homeland of Argentina. Her study of these historic metals is fascinating as it traces their usage from before the 1500s through post-colonial times. Look for more archaeometallurgy articles later this year as outgrowths of this new ASM Connect community.

In other copper news, we celebrate a recent announcement by the U.S. Environmental Protection Agency (EPA). Certain copper alloys have now been recognized by the EPA as having long-term efficacy against the virus that leads to COVID-19. In our Materials Science and Coronavirus series, Harold Michels shares his insights into what the announcement means for existing and future copper product registrations. In addition, the EPA statement provides further rationale for hospitals, extended care homes, and public transportation facilities to coat high-touch surfaces with copper alloys. We applaud the announcement!

Likewise, the U.S. Food and Drug Administration (FDA) recently released new guidance on the use of Nitinol in medical devices. We are honored to have authors from the FDA add context to the government document in their article in our *SMST NewsWire* supplement in this issue.

And finally, circling back to aerospace, Mitchell Dorfman, FASM, TSS-HoF of Oerlikon Metco, will be viewed on computer screens around the globe as the joint keynote speaker at our co-located AeroMat and ITSC Virtual Events this May. Check out the show previews for both events included in this issue. Dorfman will describe how current challenges faced by the airlines and engine manufacturers—compounded by COVID-19—necessitate innovations in engine efficiency along with environmentally friendly advancements. He believes reliable and cost-effective solutions will be required related to higher engine operating temperatures, new lighter weight components, and unique surface solutions for increased longevity of parts. As the aerospace industry makes its gradual ascent, technological innovations based on materials science will be more critical than ever.

Joanne Miller

joanne.miller@asminternational.org

RESEARCH TRACKS



Machine learning will be used to make 3D printing of metallic alloys cheaper and faster for the aerospace industry.

MACHINE LEARNING FOR AEROSPACE ALLOYS

A new research initiative called Project MEDAL (Machine Learning for Additive Manufacturing Experimental Design) aims to accelerate the product development lifecycle of aerospace components by using a machine learning model to optimize additive manufacturing (AM) processing parameters for new metal alloys—and at a lower cost than current approaches. The project is being led by two U.K. entities, Intellegens and the University of Sheffield, along with Boeing, headquartered in Chicago. Intellegens is a University of Cambridge spin-off specializing in artificial intelligence. Project MEDAL's research will concentrate on metal laser powder bed fusion—the most widely used industrial AM process—and will focus on the key parameter variables required to manufacture high density, high strength parts.

The project is part of the U.K.'s National Aerospace Technology Exploitation Program. Intellegens will produce a software platform with an underlying machine learning algorithm based on one of its previous innovations. The project was conducted for a leading OEM and a new alloy was designed,

developed, and verified in 18 months instead of the typical 20-year timeline, saving about \$10 million.

While the new method is being developed with aerospace in mind, the team believes it will have applications in other sectors as well. “The opportunity for this project is to provide end users with a validated, economically viable method of developing their own powder and parameter combinations. Research findings from this project will have applications for other sectors including automotive, space, construction, oil and gas, offshore renewables, and agriculture,” says Ian Brooks, AM technical fellow at University of Sheffield North West. *intellegens.ai*.

MORE EFFICIENT MATERIALS FOR ELECTROLUMINESCENCE

New research from an international team of scientists offers insight into how electroluminescent materials could be designed to work more efficiently. Two years ago, theoretical chemist Andrew Rappe of the University of Pennsylvania (Penn) visited the lab of Tae-Woo Lee at Seoul National University (SNU) to see if they could develop a theory to explain some of their experimental results. The material being studied was formamidinium lead

bromide, a type of metal-halide perovskite nanocrystal (PNC). Results collected by the Lee group indicated that green LEDs made of this material were working more efficiently than expected.

PNCs like formamidinium lead bromide are used in photovoltaic devices, where they can store energy as electricity or convert electric current into light in LEDs. To make sense of the SNU results, the team developed a computational model of the material's unexpected efficiency and designed follow-up experiments. Using their new model, researchers found that the PNCs were more efficient if the size of the quantum dots were smaller, although reducing the size also meant increasing the surface-to-volume ratio, creating more surface area prone to defects. The team found that replacing formamidinium with a larger organic cation called guanidinium made the particles smaller while also preserving structural integrity. Building on this approach, the team found other strategies to improve efficiency, including the addition of long-chain acids and amines to stabilize surface ions and adding defect-healing groups to repair any vacancies that might form.

Besides Penn and SNU, other researchers came from the Korea Advanced Institute of Science and Technology, Ecole Polytechnique Fédérale de Lausanne, University of Tennessee, University of Cambridge, Universitat de Valencia, Harbin Institute of Technology, and University of Oxford. *upenn.edu*.



A new study explores how a class of electroluminescent materials can be made to work more efficiently. Courtesy of Penn.

MACHINE LEARNING | AI



AI enables autonomous design of nanoporous materials. Courtesy of University of Toronto.

AI IMPROVES CHEMICAL SEPARATION

Led by researchers at the University of Toronto (U of T) and Northwestern University, Evanston, Ill., a new study is using machine learning and artificial intelligence (AI) to create the best building blocks in the pool of materials used for a specific application, such as improving chemical separation in industrial processes. “We built an automated materials discovery platform that generates the design of various molecular frameworks, significantly reducing the time required to identify the optimal materials for use in this particular process,” says U of T researcher Zhenpeng Yao.

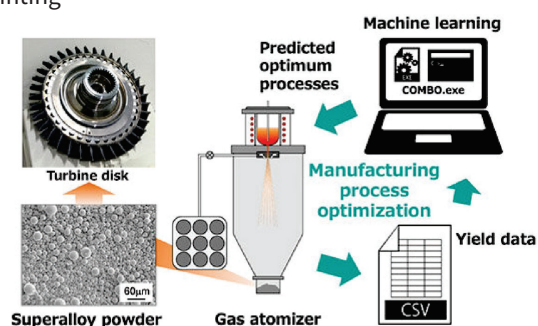
The team focused on development of metal-organic frameworks (MOFs)

that are considered the ideal absorbing material for removing CO₂ from flue gas and other combustion processes. “The new approach uses machine learning algorithms to learn from the data as it explores the space of materials and actually suggests new materials that were not originally imagined,” explains professor Randall Snurr of Northwestern. www.utoronto.ca.

MACHINE LEARNING SUPPORTS AEROSPACE

Using machine learning, researchers from the National Institute for Materials Science (NIMS), Japan, determined the optimum parameters for manufacturing high quality, Ni-Co-based superalloy powders at high yields—a promising development for aircraft engine materials. The team then demonstrated that these parameters led to low-cost manufacturing of powders suitable for high-pressure turbine disk production. Because metal 3D printing has been rapidly adopted in aerospace engine production, there is growing demand for the alloy powders these printing techniques require. When the materials are used to produce high-pressure turbine disks, they must be heat resistant, highly plastic, and homogeneous superalloy powders that can be processed into spheres. They also must be produced at high yields to reduce costs. Superalloy powders are commonly produced for this purpose using large gas atomizers.

The team used machine learning in an attempt to optimize gas atomization processes for the manufacturing of Ni-Co-based superalloy powders without relying on the knowledge of experts. As a result, the team succeeded in manufacturing fine-grained powders that can be processed into spheres. In addition, use of the parameters dramatically increased production yields from the conventional 10-30% to approximately 78% after performing experiments only six times without using previously collected data. The powder manufactured in this research was approximately 72% cheaper than commercially available powders when the prices of the raw materials were compared. The prediction accuracy of machine learning models increases as they receive more training data, and superalloy powder manufacturers possess largely unexploited manufacturing process data. Integrating this data may further improve the ability to predict optimum parameters, leading to even higher quality powders at lower cost. www.nims.go.jp/eng.

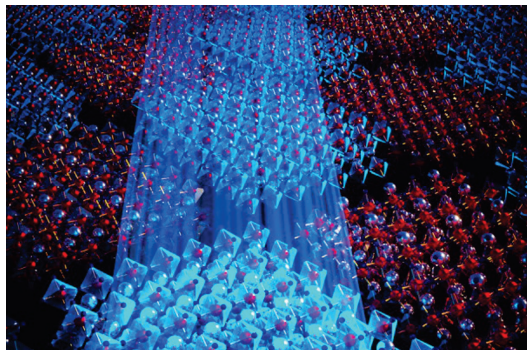


Optimization of superalloy powder manufacturing processes using machine learning. Courtesy of NIMS.

BRIEF

Researchers at **UCLA** developed a new image autofocusing technique to digitally bring a given microscopy image into focus without the need for special equipment during the image acquisition phase. The approach is based on deep learning, where an artificial neural network is trained to take a single defocused image as its input to rapidly create an in-focus image of the same sample. ucla.edu.

PROCESS TECHNOLOGY



Checkerboard patterns surprisingly formed at the nano scale of thin films, similar to structures fabricated in costly, multistep processes. Courtesy of Jalan Group/University of Minnesota.

ONE-STEP METAMATERIALS PROCESS

While studying a thin-film material called strontium stannate (SrSnO_3), researchers at the University of Minnesota Twin Cities, Minneapolis, discovered a groundbreaking one-step process for creating metamaterials. They demonstrated the realistic possibility of designing similar self-assembled structures with the potential of creating built-to-order nanostructures with wide applications in electronics and optical devices. In the new work, researchers studied a thin-film material called strontium stannate. During their research, they noticed the surprising formation of checkerboard patterns at the nanoscale similar to the metamaterial structures fabricated in the costly, multistep process. “At first we thought this must be a mistake, but soon realized that the periodic pattern is a mixture of two phases of the same material with

different crystal structures,” says researcher Bharat Jalan. The material had spontaneously organized into an ordered structure as it changed from one phase to another. During a first-order structural phase transition process, the material moved into a mixed-phase in which some parts of the system completed the transition and others did not. The team then demonstrated a process for the first-ever self-assembled, tunable nanostructure to create metamaterials in

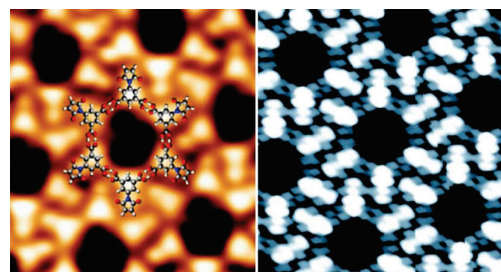
just one step. They were able to tune the ability to store electrical charge property within a single film using temperature and laser wavelength, effectively creating a variable photonic crystal material with 99% efficiency. Using high-resolution electron microscopes, the researchers confirmed the unique structure of the material. They are now looking to future applications for their discovery in optical and electronic devices. twin-cities.umn.edu.

GRAPHENE FUNCTIONALIZATION

An international research team has demonstrated a new process to modify the structure and properties of graphene. This chemical reaction, photocycloaddition, modifies the bonds between atoms using ultraviolet light. Although graphene has outstanding physical, optical, and mechanical properties, it currently has limited

use in electronics. “No other material has properties similar to graphene, yet unlike semiconductors used in electronics, it lacks a band gap. In electronics, this gap is a space in which there are no energy levels that can be occupied by electrons. Yet it is essential for interacting with light,” says Professor Federico Rosei of the Institut National de la Recherche Scientifique’s (INRS) Énergie Matériaux Télécommunications Research Centre in Quebec City, Canada.

The multidisciplinary group of researchers from Canada, China, Denmark, France, and the U.K. succeeded in modifying graphene so as to create a band gap. According to Rosei, current research is rather fundamental but could have repercussions over the next few years in optoelectronics, such as in the fabrication of photodetectors or in the field of solar energy. “These include the manufacture of high-performance photovoltaic cells for converting solar energy into electricity, or the field of nanoelectronics, for the extreme miniaturization of devices,” he added. www.inrs.ca/en.

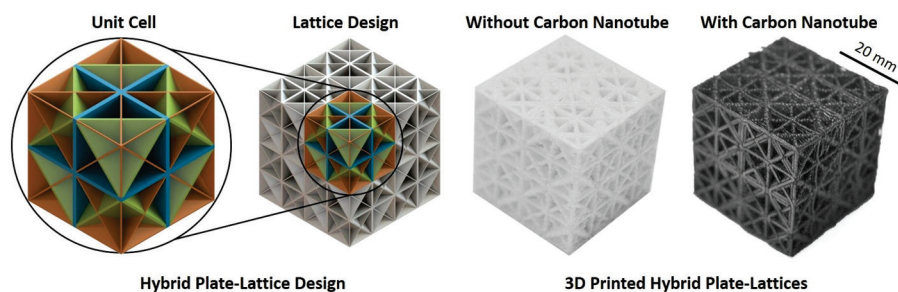


Magnified experimental and simulated scanning tunneling microscopy images of the molecule network on graphene. Courtesy of INRS.

BRIEF

Researchers from **Kanazawa University**, Japan, report a major improvement in recovering silver and palladium ions from aqueous acidic waste. Recovery of the metals in elemental, metallic form is straightforward—simply burn the extraction material and collect the remaining metal after further heating. www.kanazawa-u.ac.jp.

METALS | POLYMERS | CERAMICS



Various types of plate-lattices were designed and built by engineers in Glasgow, in the pursuit of lightweight engineering. Courtesy of University of Glasgow.

TOUGHER THAN ALUMINUM METAMATERIAL

A team led by University of Glasgow, U.K., engineers developed a new plate-lattice cellular metamaterial capable of impressive resistance to impacts. Scientists say their new 3D-printed material, made by combining commonly used plastics with carbon nanotubes, is tougher and lighter than similar forms of aluminum. The material could lead to the development of safer structures for use in the aerospace, automotive, renewables, and marine industries. The composite uses mixtures of polypropylene, polyethylene, and multiwall carbon nanotubes.

The researchers used their nano-engineered filament composite as the feedstock in a 3D printer that fused the filaments together to build a series of plate-lattice designs. Those designs were then subjected to a series of impact tests. The hybrid design, which amalgamated elements of all three typical plate-lattice designs, proved to

be the most effective in absorbing impacts, with the polypropylene version showing the greatest impact resistance. Using specific energy absorption as a measure, the team found that the polypropylene hybrid plate-lattice could withstand 19.9 joules per gram—a superior performance over similarly designed microarchitected metamaterials based on aluminum. www.gla.ac.uk.

MAKING SUPERHARD METALS

Researchers from Brown University, Providence, R.I., created a new method to customize metallic grain structures by smashing individual metal nanoclusters together to form solid macroscale hunks of solid metal. Mechanical testing of the metals manufactured using the technique showed that they were up to four times harder than naturally occurring metal structures. The researchers made centimeter-scale coins using nanoparticles of gold, silver, palladium, and other metals. Items of this size could be useful for making high-performance coating materials, electrodes, or thermoelectric

generators. But the researchers think the process could easily be scaled up to make superhard metal coatings or larger industrial components.

The key to the process, scientists say, is the chemical treatment given to the nanoparticle building blocks. Metal nanoparticles are typically covered with ligands, which generally prevent the formation of metal-metal bonds between particles. The team found a way to strip those ligands away chemically, allowing the clusters to fuse together with just a bit of pressure. In theory, the technique could be used to make any kind of metal and even metallic glass. The team has patented the technique and foresees widespread potential in both industry and the scientific research community. brown.edu.



This gold “coin” was made from nanoparticle building blocks. Courtesy of Chen Lab/Brown University.

IMPROVING CERAMIC FUEL CELL PERFORMANCE

Researchers at the Korea Advanced Institute of Science and Technology (KAIST) developed a new technology

BRIEF

Researchers from the **University of California's** San Diego and Berkeley campuses, **Carnegie Mellon University**, and the **University of Oxford** produced islands of amorphous, noncrystalline material inside high-entropy alloys. Potential applications include landing gear, pipelines, and automobiles, with the new material able to make these structures lighter, safer, and more energy efficient, according to the team. ucsd.edu.

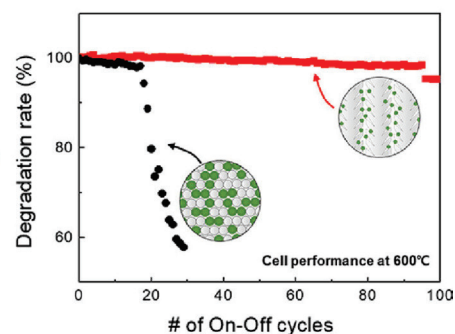
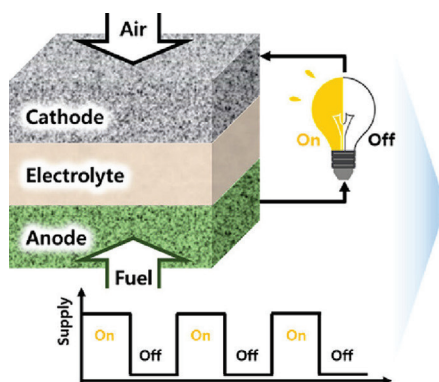
that suppresses the deterioration brought on by the reduction-oxidation cycle, a major cause of ceramic fuel cell degradation, by significantly reducing the quantity and size of the nickel catalyst in the anode using a thin-film technology.

Ceramic fuel cells generally operate at high temperatures—800°C or higher. Therefore, inexpensive catalysts, such as nickel, can be used in these cells, as opposed to low-temperature polymer electrolyte fuel cells, which use expensive platinum catalysts. Nickel usually comprises approximately 40% of the anode volume of a ceramic fuel cell. However, since nickel agglomerates at high temperatures, when the ceramic fuel cell is exposed to the oxidation and reduction processes that accompany stop-restart cycles, uncontrollable expansion occurs. This results in the destruction of the entire ceramic fuel cell structure. This fatal drawback has prevented the generation of power by ceramic fuel cells from applications that require frequent start-ups.

To overcome this challenge, the team developed a new concept for an

anode that contains significantly less nickel, just 1/20 of a conventional ceramic fuel cell. This enables the nickel particles in the anode to remain isolated from one another. To compensate for the reduced amount of the nickel catalyst, the nickel's surface area is drastically increased through the realization of an anode structure where nickel nanoparticles are evenly distributed throughout the ceramic matrix using a thin-film deposition process. In ceramic fuel cells using this novel anode, no deterioration

or performance degradation of the ceramic fuel cells was observed, even after more than 100 reduction-oxidation cycles, in comparison with conventional ceramic fuel cells, which failed after fewer than 20 cycles. Moreover, the power output of the novel anode ceramic fuel cells was improved by 1.5 times compared to conventional cells, despite the substantial reduction of the nickel content. www.kaist.ac.kr/en/.



Conceptual diagram of the oxidation-reduction cycle of ceramic fuel cells and new concept vs. deterioration rate of conventional fuel plates. Courtesy of Korea Institute of Science and Technology.

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Researchers Gerard Verbiest, Ruben Guis, and Martin Robin.
Courtesy of Delft University of Technology.

NANOMAPPING WITH ULTRASOUND

Researchers at Delft University of Technology and ASML, both in the Netherlands, recently developed a new imaging technique based on ultrasound that can explore materials at the nanoscale. “Existing nondestructive imaging techniques for nanoelectronics, such as optical and electron microscopy, are not accurate enough or applicable to deeper structures,” explains Delft researcher Gerard Verbiest. “A well-known 3D technique on a macroscale is ultrasound. But resolution is typically determined by the wavelength of the sound used.

“To improve this, ultrasound has already been integrated into an atomic force microscope (AFM),” Verbiest continues. “The advantage here is that it isn’t the wavelength, but the size of the tip of the AFM, that determines

the resolution. But at the initial frequencies, the AFM response was unclear. So the team increased the frequency of the sound used even further.

Increasing the frequency is something that has only become possible recently, Verbiest explains. They achieved this through photoacoustics and integrated the technique into an AFM.

The new method is particularly interesting for nanoelectronics, but the researchers say there are potential applications outside of electronics as well. It could be used to make detailed images of single living cells and also aid heat transport research in materials science. www.tudelft.nl/en, www.asml.com/en.

IMPROVING OPTICAL FIBERS

For the first time, researchers from the University of Southampton, U.K., and Université Laval, Canada, successfully measured back reflection in cutting-edge hollow-core fibers that is around 10,000 times lower than conventional optical fibers.

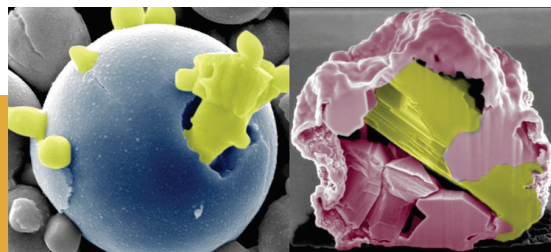
This discovery highlights yet another optical property in which hollow-core fibers are capable of outperforming standard optical fibers. Improving optical

fibers is key to enabling progress in numerous photonic applications. Most notably, these would improve Internet performance, which heavily relies on optical fibers for data transmission and where current technology is starting to reach its limits.

Backscattering in optical fibers is often highly undesirable as it causes attenuation of signals propagating down the optical fiber and limits the performance of many fiber-based devices, such as fiber optic gyroscopes that navigate airliners, submarines, and spacecrafts.

However, the ability to measure backscattering reliably and accurately can be beneficial in other instances, such as the characterization of installed fiber cables where the backscatter is used to monitor the condition of a cable and identify the location of any breaks along its length.

The latest generation of hollow-core nested antiresonant nodeless fibers (NANFs) exhibit backscattering that is so low that up until this point it remained unmeasurable. The researchers developed an instrument that enables them to reliably measure the extremely weak signals back-scattered in their latest hollow-core fibers—confirming that scattering is over four orders of magnitude lower than in standard fibers, in line with theoretical expectations. www.southampton.ac.uk, www.ulaval.ca/en.



A new dealloying method brings the most reactive components to the surface, forming stalagmite-like spikes (left), while leaving the least reactive components in the core surrounded by metal-oxide spikes (right).

BRIEF

Engineers at **Iowa State University**, Ames, developed technology capable of recovering precious metals from electronic waste. Using controlled applications of oxygen and relatively low temperatures, the team says they can dealloy a metal by slowly moving the most reactive components to the surface where they form spikes of metal oxides. iastate.edu.

DETECTING ULTRA-TRACE ELEMENTS

Chemists at the DOE's Pacific Northwest National Laboratory (PNNL), Richland, Wash., developed a simple and reliable method that holds promise for transforming how ultra-trace elements are separated and detected. Low levels of naturally occurring radioactive elements like uranium and thorium atoms are often embedded in valuable metals like gold and copper. It has been extraordinarily difficult, impractical, or sometimes even impossible to tease out how much is found in samples of ore mined across the globe.

Yet sourcing materials with very low levels of natural radiation is essential for certain types of sensitive instruments and detectors, like those searching for evidence of currently undetected particles that many

physicists believe comprise most of the universe. Scientists locate extraordinarily rare atoms from the huge field of ordinary atoms by sending their samples through a series of isolation chambers. These chambers first filter and then collide the rare atoms with simple oxygen, creating a "tagged" molecule of a unique weight that can then be separated by its size and charge. In this case, the sophisticated counter is a mass spectrometer.

The central innovation is the collision cell chamber, where charged atoms of thorium and uranium react with oxygen, increasing their molecular weight and allowing them to separate from other overlapping signals that can disguise their presence. Among other uses, the innovation may allow chemists to further hone the chemistry that produces the world's purest electroformed copper. The copper forms

a key component of sensitive physics detectors, including those used for international nuclear treaty verification. pnnl.gov.



PNNL scientists Khadouja Harouaka (seated), and Isaac Arnquist, prepare samples in an ultra-clean laboratory, which is necessary to ensure accurate mass spectrometry measurements. Courtesy of Andrea Starr/PNNL.



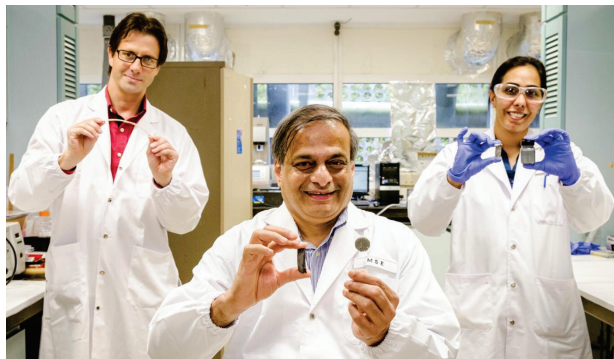
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EMERGING TECHNOLOGY



From left: NTU faculty, Terry Steele, Raju V. Ramanujan, and Richa Chaudhary holding up various soft and hard materials bonded by their new magnetocuring glue. Courtesy of NTU Singapore.

MAGNETOCURING ADHESIVE

Scientists at Nanyang Technological University, Singapore, invented glue that is activated by a magnetic field. Their “magnetocuring” glue has applications in environmental conditions where current adhesives do not work well. When the adhesive is sandwiched between insulating material like rubber or wood, traditional activators like heat, light, and air cannot easily reach it.

The new adhesive is made by combining a commercially available epoxy adhesive with specially tailored magnetic nanoparticles made by the scientists. It does not need to be mixed with any hardener or accelerator, unlike two-component adhesives, making it easy to manufacture and apply.

The adhesive bonds the materials when it is activated by passing through a magnetic field, which is easily generated by a small electromagnetic device. This eliminates the need for large

industrial ovens to cure the glue, resulting in a smaller manufacturing footprint and less energy consumption.

Potential applications include high-end sports equipment, automotive products, electronics, energy, aerospace, and medical manufacturing processes. Laboratory tests have shown that the new adhesive has a strength up to seven megapascals,

on par with many of the epoxy adhesives on the market. Moving forward, the team hopes to engage adhesive manufacturers to collaborate on commercializing their technology. They have filed a patent through NTUitive, the university’s innovation and enterprise company. www.ntu.edu.sg.

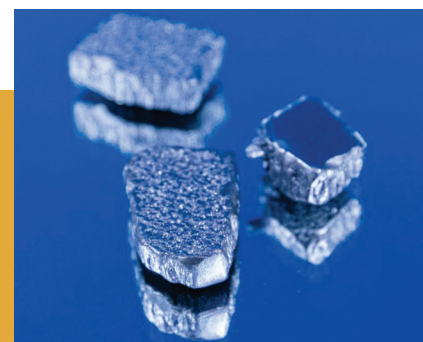
MORE DURABLE TOUCHSCREENS

Using a new theoretical model, scientists at the University of Tsukuba, Japan, gained a better understanding of vibrational spread through disordered materials such as glass. They found that as the degree of disorder increased, sound waves traveled less and less like ballistic particles, and instead began diffusing incoherently. This work may lead to new heat- and shatter-resistant glass for smartphones and tablets. Understanding the possible vibrational modes in a material is important for

controlling its optical, thermal, and mechanical properties. The propagation of vibrations in the form of sound of a single frequency through amorphous materials can occur in a unified way, as if it were a particle. However, this approximation can break down if the material is too disordered, which limits the ability to predict the strength of glass under a wide range of circumstances.

Now, researchers have developed a new theoretical framework that explains the observed vibrations in glass with better agreement with experimental data. They demonstrate that thinking about vibrations as individual phonons is only justified in the limit of long wavelengths. On shorter length scales, disorder leads to increased scattering and the sound waves lose coherence. The equations for low frequencies start looking like those for hydrodynamics, which describe the behavior of fluids. The researchers compared the predictions of the model with data obtained from soda lime glass and showed that they proved a better fit compared with previously accepted equations.

“Our research supports the view that this phenomenon is not unique to acoustic phonons, but rather represents a general phenomenon that can occur with other kinds of excitations within disordered materials,” the researchers say. Future work may involve utilizing the effects of disorder to improve the durability of glass for smart devices. www.tsukuba.ac.jp/en.

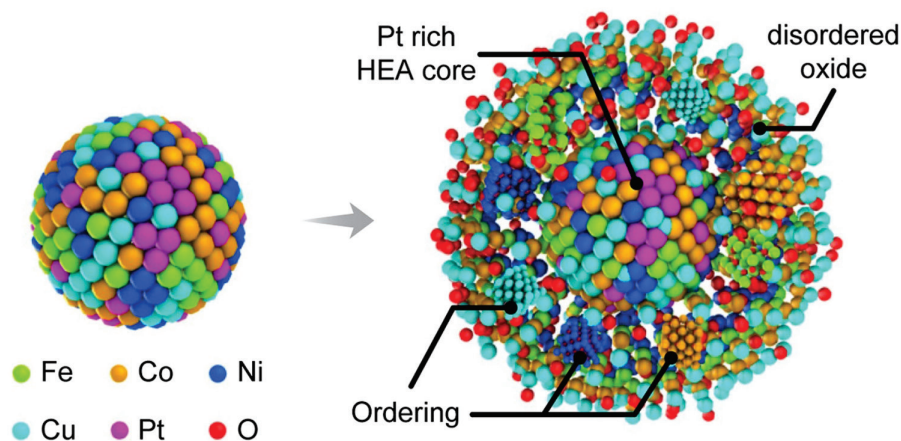


Iron-nickel permanent magnets. Courtesy of DOE Ames Laboratory.

BRIEF

The DOE’s **Critical Materials Institute**, Ames, Iowa, developed a low-cost, high-performance permanent magnet by drawing inspiration from iron-nickel alloys found in meteorites. The new magnet rivals widely used alnico magnets in magnetic strength and has the potential to fill a strong demand for magnets that are rare-earth free and cobalt free. ameslab.gov/cmi.

NANOTECHNOLOGY



HEA NP

Oxidation in air

Depiction of the movement of different molecules during the oxidation of high-entropy alloy nanoparticles. Courtesy of University of Illinois Chicago.

are able to resist oxidation much better than general metal particles.

To perform the TEM, scientists embedded the nanoparticles into a silicon nitride membrane and flowed different types of gas through a channel over the particles. A beam of electrons probed the reactions between the particles and the gas, revealing the low rate of oxidation and the migration of certain metals—iron, cobalt, nickel, and copper—to the particles' surfaces during the process. The researchers say their discoveries could benefit energy storage and conversion technologies, such as fuel cells, lithium-air batteries, supercapacitors, and catalyst materials. *anl.gov*.

DIVERSE NANOPARTICLES

Scientists from University of Illinois Chicago (UIC) and the DOE's Argonne National Laboratory, Lemont, Ill., recently discovered that certain types of alloy nanoparticles display exceptionally high stability and durability during a chemical reaction that often quickly degrades catalytic materials. The nanoparticles could have many applications, including water-splitting to generate hydrogen in fuel cells, reduction of carbon dioxide by capturing and converting it into useful materials like

methanol, more efficient reactions in biosensors to detect substances in the body, and solar cells that produce heat, electricity, and fuel more effectively.

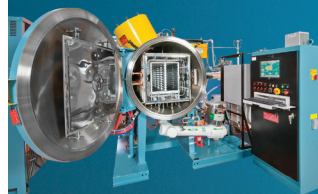
The team of researchers focused on high-entropy alloy nanoparticles and used Argonne's Center for Nanoscale Materials (CNM) to characterize the particles' compositions during oxidation. Using flow transmission electron microscopy (TEM) allowed them to capture the entire oxidation process in real time and at high resolution. They found that the high-entropy alloy nano-particles



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BRIEF

Scientists from the **University of Manchester** in the U.K. achieved a Guinness World Record for weaving threads of individual molecules together to create the world's finest fabric. The team produced a 2D-molecularly woven fabric with a thread count of 40-60 million, overtaking the finest Egyptian linen with a thread count of roughly 1500. *manchester.ac.uk*.

TITANIUM MICROTEXTURE 101— PART I

Michelle Harr,* Adam Pilchak,* and Lee Semiatin, FASM*

Air Force Research Laboratory, Wright-Patterson Air Force Base, Ohio
University of Dayton Research Institute, Ohio

Despite decades of research, fundamental questions still exist regarding the role of microtexture on the deformation, fatigue, and fracture behavior of titanium alloys.

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Microtexture is a feature of titanium alloy microstructures that has received considerable renewed attention in the past several years as it impacts materials processing, properties, and inspectability of titanium alloys. This article describes the microstructural evolution processes that result in microtexture formation, while Part II of this series will further elaborate on the effects of microtexture on deformation behavior.

Titanium alloys feature an excellent blend of high strength, low density, and good corrosion resistance. It is the fatigue performance, however, that helped titanium find its way into fracture-critical rotating hardware in gas turbine engines. Following the 1989 crash of a DC-10 in Sioux City, Iowa, due to the presence of a melt-related anomaly known as “hard α ,” the titanium industry set out to refine melt practices and improve nondestructive inspection methods in order to prevent such an event from occurring again.

Fast forward nearly three decades and we can say with confidence that those involved significantly advanced the state of the art in titanium melt practices. Following the event, the Federal Aviation Administration (FAA) convened the Jet Engine Titanium Quality Committee (JETQC), which still exists today. This group is responsible for ensuring that the premium quality titanium that finds its way into gas turbine

engines is safe and free from melt-related anomalies. The entire industry shares its experience with such anomalies in the interest of aviation safety.

In fact, the industry has cleaned up titanium so much that it is rare to find hard α during the now-routine ultrasonic inspections performed on billets and forgings. With no other hard second phases, inclusions, or pores (when properly processed), fatigue cracks tend to initiate at microstructural weak links. In the case of titanium alloys, these are known as microtextured regions (MTRs) or macrozones. These terms are used interchangeably to describe a feature of the microstructure that is actually an aggregation of the lower length scale microstructural features, specifically when those features share a common crystallographic orientation. These features have long been known to cause issues related to dwell fatigue of near- α alloys^[1]. However, it has only recently come to light that the industry workhorse Ti-6Al-4V alloy may also be susceptible under certain combinations of stress, temperature, microstructure, and composition^[2].

The salient microstructural features, which span about seven orders of magnitude, are depicted in Fig. 1. Titanium alloys used for rotating components are most often used in the so-called bimodal condition, which consists of a mixture of primary α particles that may

take equiaxed or elongated morphology, and secondary α or “transformed β ,” which can take on either basketweave or colony morphology. As discussed in this article, the mechanism by which MTRs develop results in crystallographically aligned primary α particles, so the solution heat treatment temperature is often selected to be high enough in the α/β phase field that the primary α particles are isolated from one another and separated by the transformed β . While this strategy is generally effective, under certain conditions the secondary α can adopt a similar orientation to its adjacent primary α ^[3-5]. The causes and implications of such an event are also discussed.

The goal of this article series is to bring awareness to this microstructural feature and highlight what the community currently knows about microtexture, draw attention to some gaps in understanding, and highlight opportunities for future research.

MICROTEXTURE EVOLUTION

The evolution of microtextured regions in near- α and α/β titanium alloys such as Ti-6242 and Ti-6Al-4V is related to industrial thermomechanical processes used to convert large cast ingots (~800-1000 mm in diameter) into semi-finished mill products such as billets, slabs, and plates. Typically, ingots are synthesized via techniques such

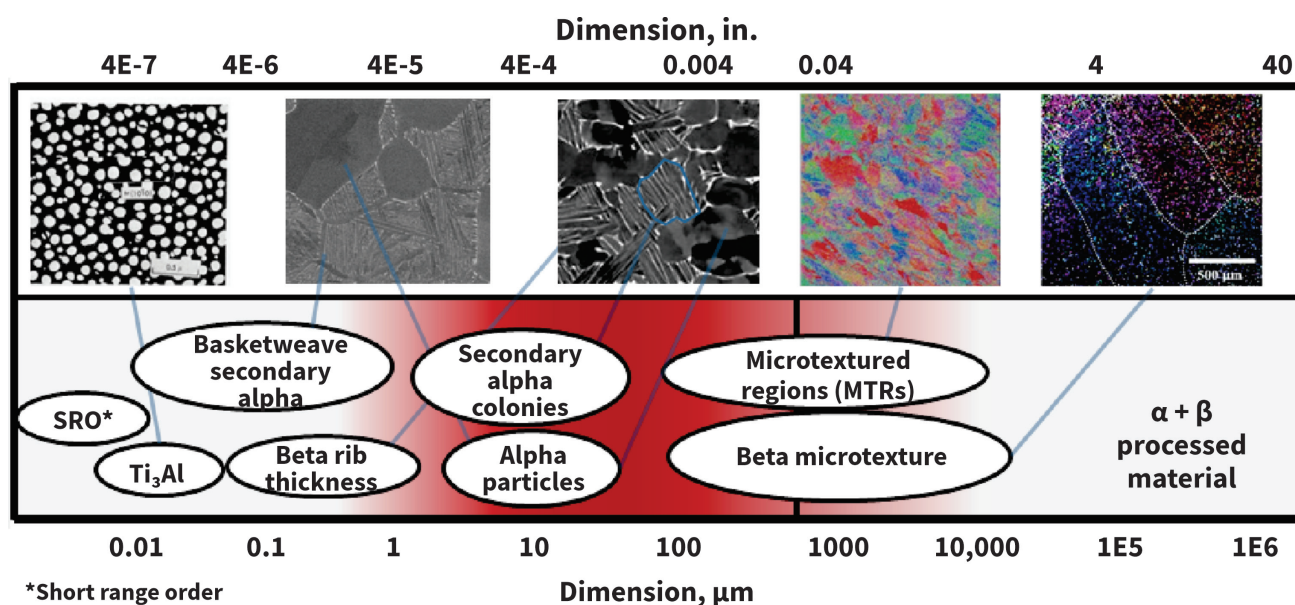


Fig. 1 — Length scale of salient microstructural features in α/β titanium alloys.

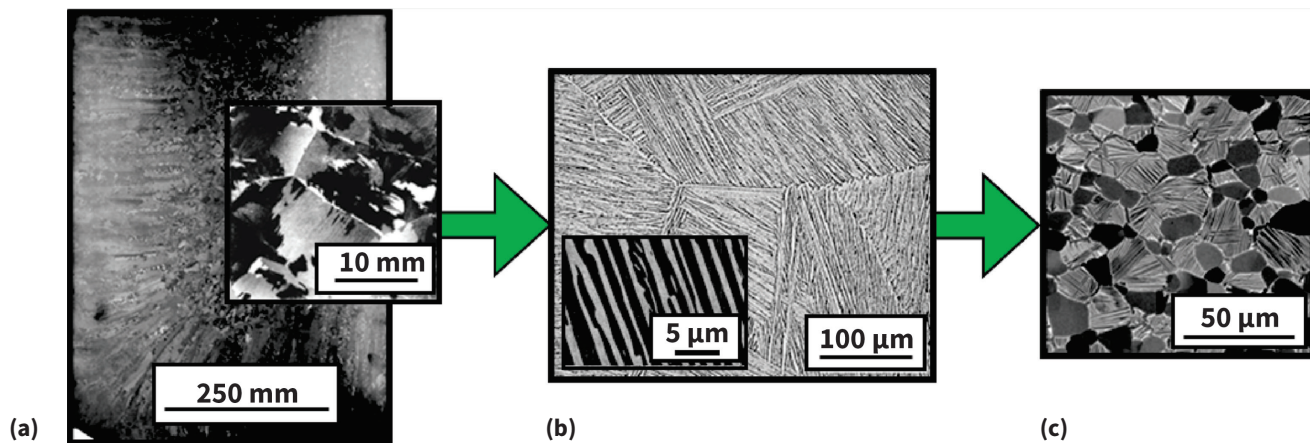


Fig. 2 — Microstructure evolution during thermomechanical processing of α/β titanium alloys: (a) as-cast ingot; (b) after recrystallization; and (c) after spheroidization of the lamellar α microstructure via α/β hot working^[6].

as vacuum arc or electroslag melting/remelting, electron beam melting, or plasma cold hearth melting to control interstitial content and remove high-density/low-density inclusions. However, slow cooling following solidification leads to coarse, columnar grains that are multiple millimeters in length and diameter (Fig. 2a)^[6].

A number of hot working and heat treatment steps are subsequently performed (principally in the high temperature, single phase bcc β field) to obtain billet or slab with recrystallized, equiaxed β grains, typically with a size of ~2-3 mm in diameter (Fig. 2b). Following β recrystallization, the relatively slow cooling associated with the thermal inertia of large-section workpieces results in the decomposition of each β grain into a microstructure comprising a number of ~0.5-to-1.5-mm-diameter colonies of hcp α lamellae (Fig. 2b), each of which has its own crystallographic orientation relative to a specified set of reference coordinates such as the radial and axial directions of the cylindrical billet. Due to a Burgers orientation relationship (BOR) between the high temperature β phase and low temperature α phase, the number of possible orientations of the α colonies (also called α variants) within a given β grain cannot exceed 12, and typically lies in the range of 3-10.

Each colony of α lamellae formed during cooling from the β field can be thought of as a nascent MTR, or a region in which all of the α phase has the same

(or nearly the same within a specified tolerance limit) crystallographic orientation. The objective of subsequent hot working steps performed in the α/β phase field is therefore twofold: (1) break down each colony to develop a uniform, fine, equiaxed structure of globular α particles within the β matrix (Fig. 2c), and (2) randomize the orientation of each α particle relative to its neighbors to minimize (or eliminate) the extent of MTRs. Accomplishing these objectives can be quite difficult due to the plastic anisotropy of the hcp α phase. The anisotropy translates to a sizeable difference (of the order of three times) between the material flow stress when deformation is imposed along the c-axis (i.e., the normal to the close-packed planes of the hcp Ti crystal) versus perpendicular to it, and this results in very inhomogeneous deformation. “Soft” colonies (having c-axes oblique to the forging direction) undergo large deformation, and “hard” colonies (with c-axes parallel or nearly parallel to the forging direction) suffer relatively small strains (Fig. 3)^[7].

Depending on the hot working temperature, the difference in local stresses and strains may lead to generation of deleterious cavities between the harder and softer colonies (Fig. 4)^[8-12]. Equally important, the hard oriented, less deformed colonies may retain their nature as relatively equiaxed microtextured features. By contrast, deformation within the softer colonies can be relatively large overall, but it tends to

be inhomogeneous due to the presence of neighboring hard colonies, the shape of the lamellae per se, and other such factors. Thus, it is quite common to see microstructural features such as kinked lamellae, lamellae that have rotated to

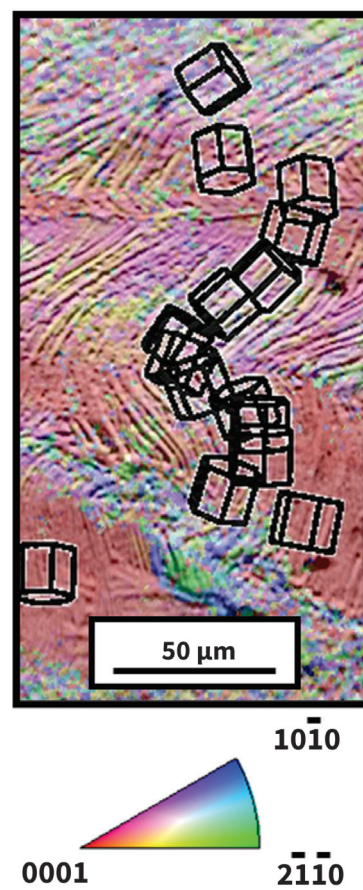


Fig. 3 — EBSD compression axis, inverse pole figure map for a region in a Ti-6Al-4V pancake forging illustrating the variation in deformation among colonies with hard orientations (red) and soft orientations (other colors). Courtesy of T.R. Bieler.

lie along the direction of primarily metal flow, and regions of very high strain near β grain boundaries.

High local strains within the soft colonies lead to the development of dislocation walls within individual α lamellae, which promote spheroidization during deformation (i.e., dynamically), especially if a multiplicity of slip systems has been activated. Further, the combination of high local strains,

evolution of high angle boundaries (associated with dislocation walls), and strain gradients may serve to rotate individual portions of long lamellae to different degrees and thus result in both spheroidization and randomization of the orientation of the resulting α particles, thereby eliminating MTR-like features, at least locally. On the other hand, lamellae within a given prior colony (or several adjacent colonies)

that have rotated to lie along the primary metal flow direction with little or no spheroidization or orientation randomization may form especially long and deleterious MTRs (Fig. 5)^[13].

HEAT TREATMENT CONSIDERATIONS

During static heat treatment following hot working, remnant stored work (dislocation substructure) may promote static spheroidization without additional changes in the misorientation of the α particles in a prior colony and thus result in little mitigation of MTR characteristics. The severity of MTRs associated with spheroidized (but not randomized) α particles can be exacerbated by the tendency of surrounding secondary α plates with a similar orientation to form in the β matrix during cooling following hot working or final heat treatment^[3-5]. Such a tendency can be mitigated somewhat by imposing a high cooling rate following hot working or heat treatment to develop multiple α variants within the β grains surrounding each α particle^[14]. A second heat treatment step at a lower temperature may then be applied to coarsen the secondary α produced in the first step^[15].

It is worth noting that such a condition can only occur if the β phase does not recrystallize dynamically during deformation or subsequent static heat treatment and hence retains a long range common orientation (denoted as “ β microtexture” in Fig. 1). Begley et al.^[16] hypothesized that this could occur under two conditions: (1) α and β phases co-rotate during deformation such that the orientation relationship is preserved, or (2) where very little deformation occurs such that there is insufficient strain to cause recrystallization. Moreover, extended recovery processes that occur during near but sub-transus annealing can result in substantial changes to the β phase texture^[17]. A great need exists for additional research in this area because β microtexture is believed to be a key contributor to dwell fatigue, and it has also been implicated in abnormal grain growth in β annealed structural titanium forgings.

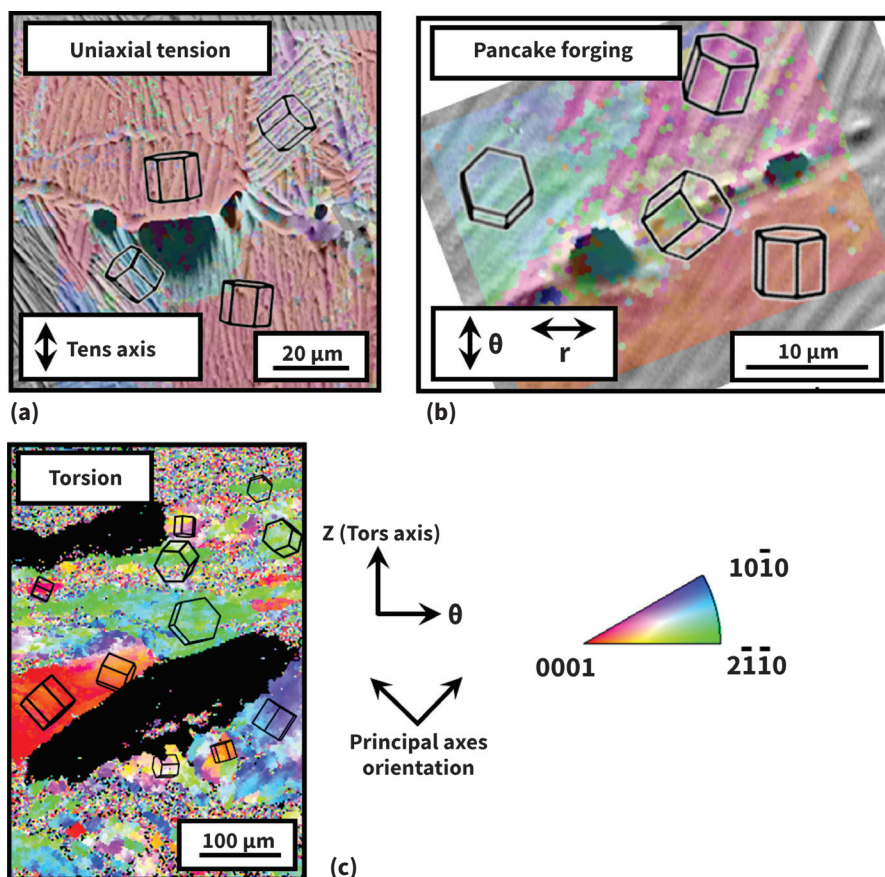


Fig. 4 — EBSD inverse pole figure maps for Ti-6Al-4V samples with an initial colony—a microstructure in which cavities developed during hot deformation at 1089 K (815°C) via (a) uniaxial tension^[9], (b) pancake forging^[10], or (c) torsion testing^[11]. Hexagons indicate the orientations of hard and soft colonies adjacent to some of the cavities.

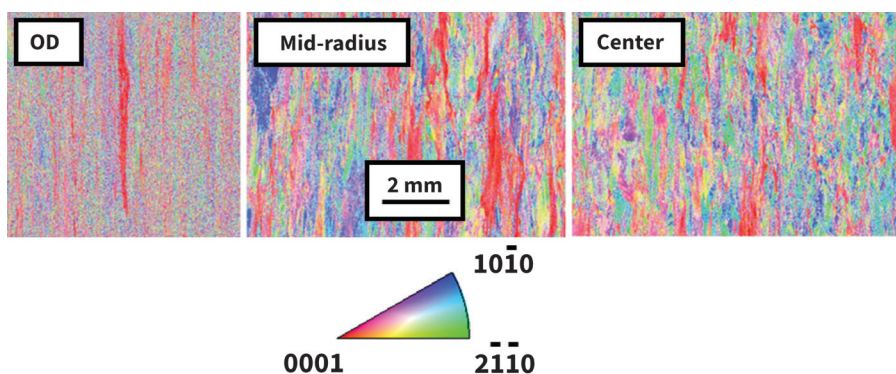


Fig. 5 — EBSD radial direction, inverse pole figure maps illustrating (red) microtextured regions in a 209-mm-diameter Ti-6242 billet at various radial locations^[13].

Due to the debit in mechanical properties and service performance associated with MTRs, a number of attempts have been made to lessen their severity. Various techniques include breakdown of the colony microstructure via low-temperature superplastic forging, multistep processing at successively lower temperatures, and multi-axial forging^[18-20]. For example, a change in strain path involving redundant work can be effective in eliminating MTRs in forgings^[14]. The usefulness of a strain-path change such as elongation followed by compression was also recently suggested using crystal plasticity finite element method simulations for Ti-6242^[21]. Finally, an approach consisting of α/β hot working of workpieces that have been β annealed and water quenched to produce a basketweave α microstructure (i.e., multiple variants at any given location) has been suggested^[22]. However, this specific method is likely only suitable for section sizes in which the basketweave microstructure can be developed during water quenching.

Part II of this series will further elaborate on the effects of microtexture on deformation behavior and will be available in a future issue of *AM&P*.

Acknowledgments

The authors wish to extend their gratitude to the following collaborators for many useful discussions related to microtexture: T.R. Bieler, T.F. Broderick, S. Daly, M.G. Glavicic, P.D. Nicolaou, V. Venkatesh, J.C. Williams, and A. Woodfield. Michelle Harr wishes to acknowledge the support of the National Science Foundation Graduate Research Fellowship under Grant No. 1256260 DGE and the Air Force Research Labs under Contract #FA8650-16-C-5235 during the completion of this work. ~*AM&P*

For more information: Adam Pilchak, Air Force Research Laboratory, AFRL/RXCM, Wright-Patterson Air Force Base, Ohio, 45433, adam.pilchak.1@us.af.mil.

References

1. A.P. Woodfield, et al., Effect of Microstructure on Dwell Fatigue Behavior of Ti-6242, *Titanium '95: Science and Technology*, p 1116-1123, 1995.
2. France-BEA, Accident to the AIRBUS A380-861 Equipped with Engine Alliance GP7270 Engines Registered F-HPJE Operated by Air France on 30 September 2017 in Cruise over Greenland (Denmark), 2020.
3. L. Germain, et al., Analysis of Sharp Microtexture Heterogeneities in a Bimodal IMI 834 Billet, *Acta Mater.*, Vol 53, p 3535-3543, 2005.
4. I. Bantounas, D. Dye, and T.C. Lindley, The Role of Microtexture on the Faceted Fracture Morphology in Ti-6Al-4V Subjected to High-Cycle Fatigue, *Acta Mater.*, Vol 58, p 3908-3918, 2010.
5. J.L.W. Warwick, et al., In Situ Observation of Texture and Microstructure Evolution During Rolling and Globularization of Ti-6Al-4V, *Acta Mater.*, Vol 61, p 1603-1615, 2013.
6. S.L. Semiatin, An Overview of the Thermomechanical Processing of α/β Titanium Alloys: Current Status and Future Research Opportunities, *Metall. Mater. Trans. A*, Vol 51, p 2593-2625, 2020.
7. T.R. Bieler and S.L. Semiatin, The Origins of Heterogeneous Deformation During Primary Hot Working of Ti-6Al-4V, *Int. J. Plast.*, Vol 18, p 1165-1189, 2002.
8. S.L. Semiatin, et al., Cavitation and Failure During Hot Forging of Ti-6Al-4V, *Metall. Mater. Trans. A*, Vol 30, p 1411-1424, 1999.
9. T.R. Bieler, P.D. Nicolaou, and S.L. Semiatin, An Experimental and Theoretical Investigation of the Effect of Local Colony Orientations and Misorientation on Cavitation During Hot Working of Ti-6Al-4V, *Metall. Mater. Trans. A*, Vol 36, p 129-140, 2005.
10. T.R. Bieler, M.G. Glavicic, and S.L. Semiatin, Using OIM to Investigate the Microstructural Evolution of Ti-6Al-4V, *JOM*, Vol 54, p 31-36, 2002.
11. P.D. Nicolaou, J.D. Miller, and S.L. Semiatin, Cavitation During Hot-Torsion Testing of Ti-6Al-4V, *Metall. Mater. Trans. A*, Vol 36, p 3461-3470, 2005.
12. P.D. Nicolaou and S.L. Semiatin, An Analysis of Cavity Growth During Open-Die Hot Forging of Ti-6Al-4V, *Metall. Mater. Trans. A*, Vol 36, p 1567-1574, 2005.
13. A.L. Pilchak, et al., Characterization of Microstructure, Texture, and Microtexture in Near-Alpha Titanium Mill Products, *Metall. Mater. Trans. A*, Vol 44, p 4881-4890, 2013.
14. N. Gey, et al., Texture and Microtexture Variations in a Near- α Titanium Forged Disk of Bimodal Microstructure, *Acta Mater.*, Vol 60, p 2647-2655, 2012.
15. M.D. Gorman, A.P. Woodfield, and B.A. Link, US Patent 6,284,070, 2001.
16. B.A. Begley, et al., Prediction of Relative Globularization Rates in $\alpha + \beta$ Titanium Alloys as a Function of Initial Crystal Orientation, *J. Mater. Res.*, Vol 35, p 1113-1120, 2020.
17. A.L. Pilchak, G.A. Sargent, and S.L. Semiatin, Early Stages of Microstructure and Texture Evolution During Beta Annealing of Ti-6Al-4V, *Metall. Mater. Trans. A*, Vol 49, p 908-919, 2017.
18. G.A. Salishchev, O.R. Valiakhmetov, and R.M. Galeev, Formation of Submicrocrystalline Structure in the Titanium Alloy VT8 and its Influence on Mechanical Properties, *J. Mater. Sci.*, Vol 28, p 2898-2902, 1993.
19. G.A. Salishchev, S.Y. Mironov, and S.V. Zharebtsov, Mechanisms of Submicrocrystalline Structure Formation in Titanium and Two-Phase Titanium Alloy During Warm Severe Processing, *Rev. Adv. Mater.*, Vol 11, p 152-158, 2006.
20. G.A. Salishchev, et al., Development of Ti-6Al-4V Sheet with Low Temperature Superplastic Properties, *J. Mater. Process. Technol.*, Vol 116, p 265-268, 2001.
21. R. Ma, et al., Modeling the Evolution of Microtextured Regions During α/β Processing Using the Crystal Plasticity Finite Element Method, *Int. J. Plast.*, Vol 107, p 189-206, 2018.
22. B.P. Bewlay, et al., US Patent 6,387,197, 2002.

ADVANCEMENTS IN NEAR NET SHAPE EXTRUSION FOR AEROSPACE APPLICATIONS

The aerospace industry could benefit from recent progress in making near net shape titanium extrusions for applications beyond the long structural components of an aircraft.

Phani P. Gudipati* and Michael B. Campbell

Plymouth Engineered Shapes, Hopkinsville, Kentucky

Extruded shapes are often ideal for long aircraft components that require consistent cross sections throughout the length of the part^[1]. The most common aerospace extrusions include seat tracks produced using the workhorse titanium alloy, Ti-6Al-4V (Ti-64). However, major advancements have occurred in the use of extruded titanium shapes deployed in a variety of other applications. Uses range from specialized parts in subsonic systems to components in unmanned submarines that take advantage of titanium's favorable strength-to-density relationship and superior corrosion resistance. Billet temperatures used in the extrusion of titanium alloy shapes are typically above the beta transus temperature of the material, and the reduction ratios are higher than those used in other product types. Gross titanium extrusion, while producing radical savings in materials due to closer shape approximation, also requires less machining to achieve the finished product.

In an effort to further reduce overall manufacturing costs and improve the buy-to-fly ratio, the R&D division of Plymouth Engineered Shapes (PES) has successfully developed an innovative process and manufactured near net shape Ti-64 extrusions, roughly 30 ft long, on a production scale that includes various geometries. This article

presents different near net shapes extruded at PES and provides the mechanical properties, microstructures, and dimensional tolerances consistent throughout the full length of the extrusion.

METAL EXTRUSION

The metal extrusion process can be broadly classified into two main categories—direct and indirect^[2]. In the process described here, the direct or forward extrusion process was employed, where the die and ram are on opposite ends and the billet travels in the same direction as the ram (Fig. 1). Typically, the cross section of the work billet is much larger than the cross section of the extruded part. To relate the cross section of the workpiece to that of the extruded product, a value commonly called the extrusion ratio was established, which is defined as the ratio of the area of the original billet cross section (A_o) to that of the extruded product (A_f). The extrusion ratio, or reduction ratio, can be expressed as (A_o/A_f). Depending on final part geometry, a wide range of extrusion ratios for extruding different titanium products is available. Typically, most titanium extrusion presses are water hydraulic systems with remarkably high strain rates in the range of 10 s^{-1} or higher^[3]. A beta extruded titanium billet will yield products

with an elongated grain structure that is often recrystallized by hot stretch straightening and annealing. The resultant structure consists of recrystallized prior beta grains with colony alpha, which offers an excellent combination of strength, fracture toughness, and fatigue life^[4].

MATERIALS AND PROCESSING

Ti-64 billets, with nominal composition per AMS 4935^[5] ranging from 6.0 to 9.25 in. in diameter, were induction heated to a temperature above the beta transus and extruded into two distinct geometric profiles. These two shapes were selected for their uniqueness in terms of geometry and application (Fig. 2). While the "T profile" is often used for aerospace structural components, the shape with more complex



Fig. 1 — Forward extrusion process where the ram pushes the hot metal through the die.

*Member of ASM International

geometric features (NNS-3) is utilized in aeroengine applications. Dies used for the extrusion process are designed and manufactured in-house with modified tool steel. Parameters for the extrusion process are selected based on billet size, extrusion ratio, and the profile to be extruded. These profiles are designed for a thickness of 0.150 in. for a planned 30 ft of extruded length, although typical extrusions can be up to 40 ft long, depending on initial billet size.

Extruded products are then subjected to hot straightening and annealing, in accordance with AMS 4935, to achieve the mechanical properties and key characteristics such as flatness across the width of the part, straightness (bow/camber), and twist along the full length of the extrusion. To enhance machinability, finished extrusions are chemically treated to remove the thin layer of surface alpha case. The sequence of operations is schematically illustrated in Fig. 3.

RESULTS AND DISCUSSION

Dimensions. The two selected profiles were successfully extruded to

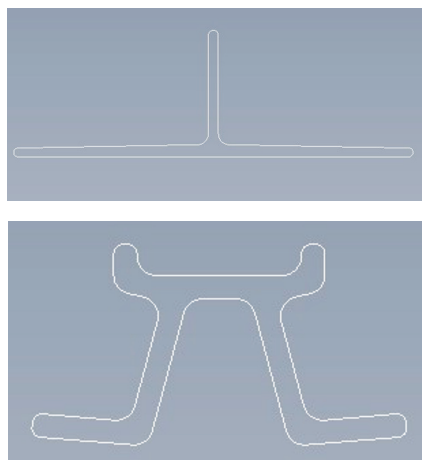


Fig. 2 — Geometric profiles of two selected near net titanium shapes.

over 30 ft long (Fig. 4). Dimensional stability (uniform dimensions with minimal variation) and straightness of the extrusions play a major role in machining the finished product. Figure 5 shows the different dimensions measured for each profile and the location of samples for room temperature mechanical testing and microstructural evaluation. Results of dimensional measurements and variation to nominal planned dimensions for both profiles are shown in Figs. 6 and 7. While AMS 2245^[6] allows for a deviation of ± 0.060 in. on a given feature, Plymouth's near net extrusions have outperformed the specification by presenting a variation less than 50% of the total permissible limit from the nominal.

Straightness. Machining of titanium extrusions requires extreme control



Fig. 4 — Near net extrusions produced in Ti-64. A sample cut from the T-profile (top) and complex geometrical profile (bottom) are beta extruded to over 30 ft long.

of tolerances on key characteristics such as transverse flatness, straightness, twist, and angularity along the full length of the extruded part. With reduction in the envelope due to near net shape profiles, holding these tolerances along the length of the extrusion is of paramount importance to successfully machine the final part. Due to space constraints, only the transverse flatness tolerances on the north and south ends of the near net extrusions are presented in Table 1. The maximum level of bow and twist observed over the full length of the extrusion are also shown in the table. It can be observed that the average values of critical characteristics are not only well within allowable limits, but the near net extrusions described here also offer much tighter tolerance limits than specified in AMS 2245, thus making them exceptionally favorable for machining.

Mechanical Properties & Microstructure. Specimens for mechanical testing and microstructure were obtained from the location indicated in Fig. 5. Room temperature mechanical

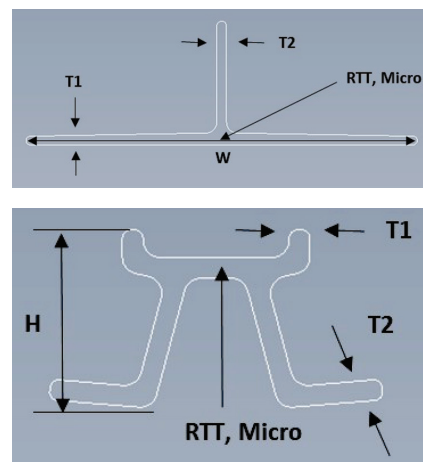


Fig. 5 — Location on the two extruded profiles where dimensions were measured and samples for mechanical testing and microstructure are excised.

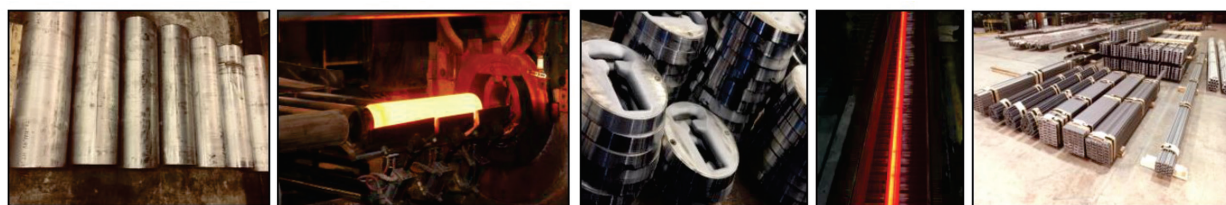


Fig. 3 — Titanium billets are extruded in the beta field using required dies, thermally straightened, surface alpha case chemically removed, and packed for shipment.

testing on samples in the longitudinal direction was performed according to ASTM E8^[7], and optical microscopy was performed using a Keyence digital

microscope. As observed in Table 2, mechanical testing results are in full compliance with the strength requirements of AMS 4935 for both

geometric profiles. In addition, optical micrographs present a microstructure resulting from beta processed Ti-64, with colony alpha along the recrystallized beta grain boundaries (Fig. 8). Average grain size, measured according to ASTM E112^[8], was found to be 100 μm . Longitudinal and transverse surface roughness was also measured using a standard profilometer that resulted in a range of 80-130 Ra for both profiles.

Buy-To-Fly. Each preform process such as forging, casting, and plate stock incurs a considerable loss of material during machining due to the excessive material envelope built around the finished product. Extrusions considerably reduce the buy-to-fly ratio and provide advantages not offered by alternative preform processes. With the development of near net shape extrusions, the buy-to-fly ratio is further reduced, thus decreasing the costs associated with machining.

It must be stated that the original standard extrusion for a T-profile was already an improvement over its previous preform (plate). Table 3 presents further reduction in the buy-to-fly ratio.

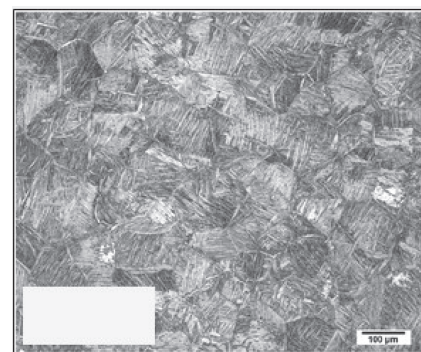
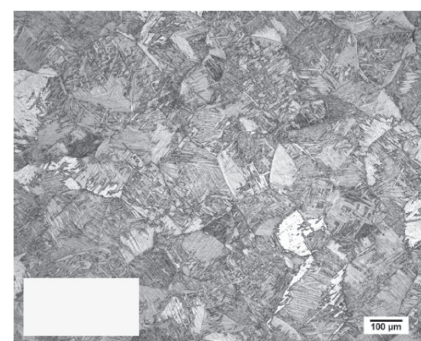


Fig. 8 — Optical micrographs from the near net shape extrusions: T-profile (top) and NNS-3 (bottom) indicate colony alpha along the recrystallized beta grains. Samples were etched with Kroll's reagent after polishing.

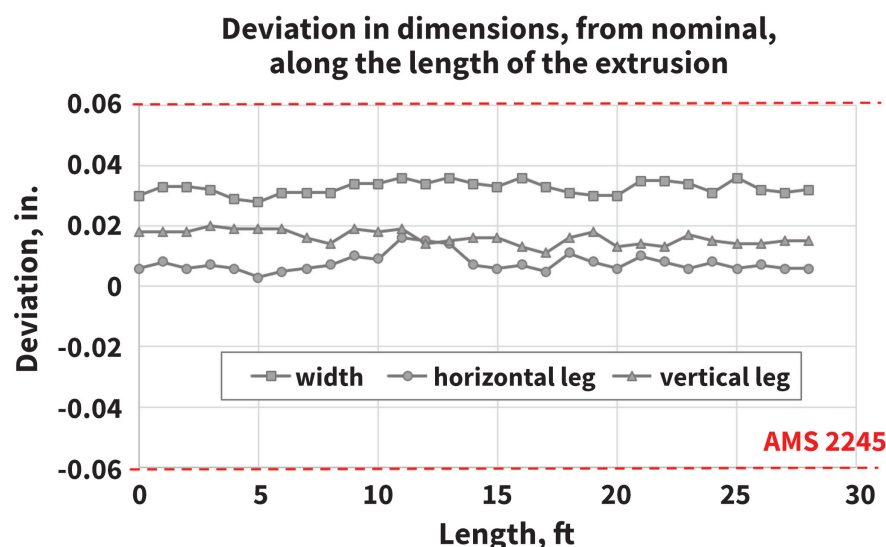


Fig. 6 — Deviation from the nominal planned dimensions on the T-profile. Note that the near net extruded profile exhibits consistent dimensions with minimal variation along the full length of the extrusion, much less than the permissible limits per AMS 2245.

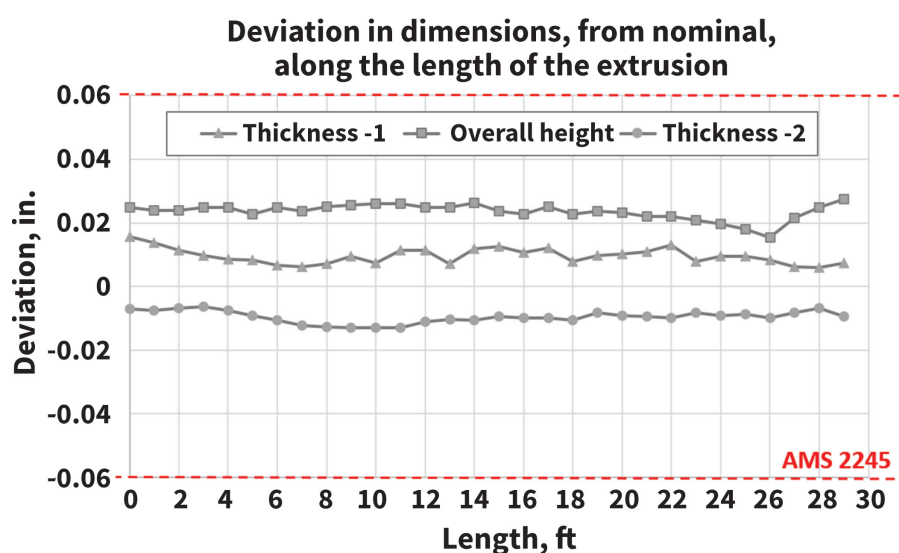


Fig. 7 — Deviation from the nominal planned dimensions on NNS-3. Note that the near net extruded profile exhibits consistent dimensions with minimal variation along the full length of the extrusion, much less than the permissible limits per AMS 2245.

TABLE 1 – AVERAGE TOLERANCES ON KEY CHARACTERISTICS OF NEAR NET EXTRUSIONS

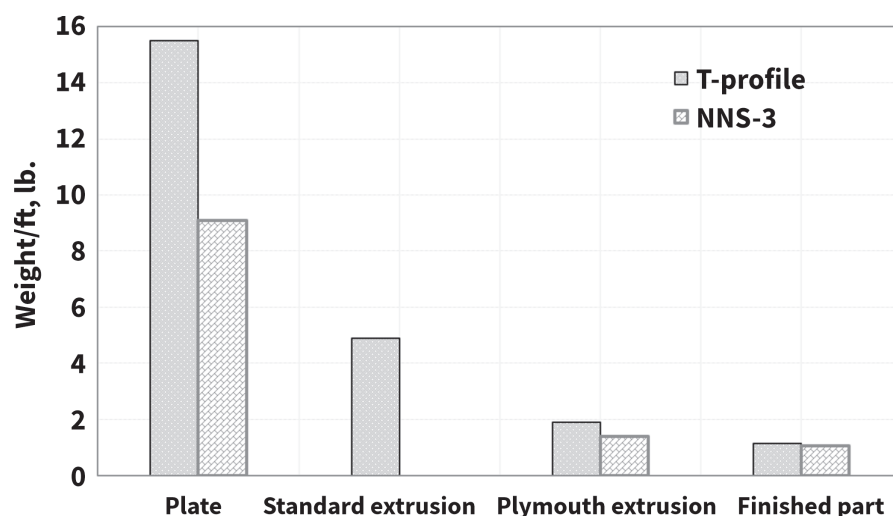
Product type	Location	Flatness, in.	Max. bow, in.	Max. twist, deg.
T-profile	North	0.013	0.055	0.3
	South	0.008		
NNS-3	North	0.000	0.010	0.7
	South	0.000		

TABLE 2 – ROOM TEMPERATURE MECHANICAL PROPERTIES OF NEAR NET EXTRUDED PRODUCTS

Product type	YS, ksi	UTS, ksi	EL, %	RA, %
AMS 4935	120.0	130.0	10.0	20.0
T-profile	130.0	146.0	19.0	38.0
NNS-3	132.0	147.0	16.0	31.0

TABLE 3 – BUY-TO-FLY RATIOS FOR TWO GEOMETRIC PROFILES

Product type	T-Profile	NNS-3
Plate	13.47	8.66
Standard extrusion	4.26	-
Plymouth extrusion	1.64	1.32
Finished parts	1.00	1.00

**Fig. 9** — Comparison of weight/ft to produce the finished part for both profiles from various product forms.

Although most finished product for NNS-3 is currently being machined from plate, the near net extrusion method developed by PES can provide significant savings over the current manufacturing method. Figure 9 shows the weight/ft for each product type, clearly exhibiting the advantages of using near net extrusion technology to create these shapes.

SUMMARY

Economic advantages of reducing the buy-to-fly ratio include reduction in tooling costs and material usage, lower parts count resulting from the ability to extrude complex shapes over length in a single operation, and less downstream machining and finishing^[9]. PES

has successfully developed techniques to reduce the buy-to-fly ratio by manufacturing near net shape extrusions on a production scale. Microstructure, room temperature mechanical properties, and extrusion-critical characteristics such as transverse flatness and straightness are in accordance with AMS 4935 and AMS 2245, respectively, for the near net shape extrusions. Considering the merits of titanium alloys, the authors believe that the aerospace industry could significantly benefit from these technological advancements in the production of near net shape extrusions by using this novel concept in applications beyond the long structural members of an aircraft. ~AM&P

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References

1. R.R. Boyer, E.R. Barta, and J.W. Henderson, *Journal of Materials*, p 36-39, March 1989.
2. G.E. Dieter, *Classification of Extrusion Processes*, Mechanical Metallurgy.
3. G. Legate, *Titanium 2013*, Las Vegas, 2013.
4. R. Wanhill and S. Barter, Springer Briefs in Applied Sciences and Technology.
5. *Titanium Alloy Extrusions and Flash Welded Rings Ti-6Al-4V Annealed Beta Processed*, AMS 4935 Rev. L, issued 1959-06, Revised 2017-09.
6. *Tolerances, Titanium and Titanium Alloy Extruded Bars, Rods and Shapes*, AMS 2245 Rev. B, issued Dec. 1973, Revised June 2003.
7. *Standard Test Method for Tension Testing of Metallic Materials*, ASTM E8/E8M – 16a.
8. *Standard Test Method for Determining Average Grain Size*, ASTM E112 – 13.
9. D. Sanders, et al., *ITA*, Atlanta, 2012.

AN EXPLORATION OF HISTORIC METALS IN SOUTH AMERICA

A study of metal artifacts from the Mendoza region of Argentina reveals the influences of native migration and European conquest.

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The Mendoza province in Argentina is endowed with a great variety and richness in metal mineral deposits. Archaeometallurgical study of metal artifacts provides examples of precapitalist metalworking technologies, including the Mapuches in South America who through their jewelry sought a link with the universe. Although Mapuche jewelry is best known for silver work (Mapuche Silverware), this was not the only material on which they would have worked, and various studies indicate that they began to work with copper (Vergel Culture). Although they went through various stages of development and improvement of their silver work, they never abandoned copper.

Copper, which they called “anta,” was used instead of iron to make weapons of war, knives, carpenter’s tools, pins to hold women’s cloaks, or hoes to dig the earth. The native Andean miner was intimately linked with the land, “Pachamama,” and the mineral was treated as one more living element of nature, which was reproduced, manipulated, and “harvested” as a crop. The native metallurgists, more than mere artisans, were intermediaries between humans and deities, owners of both technical and esoteric knowledge.

CHANGING POPULATIONS

The south of the province of Mendoza was part of a southern border between territories controlled by the original peoples, and the colonial government, which later became the Argentine Republic. This made the region a place of convergence for different types of societies with very different

economic, productive, and social systems. The conquest of this territory by the Spanish began with the first exploration by Francisco de Villagra around 1551 and the subsequent foundation of the city of Mendoza around 1561^[1].

Roughly around the same time, the migration of the Mapuche-speaking peoples from Chile, known as “araucanización” began, bringing instability to the southern border of the Viceroyalty of the Río de la Plata, which allowed the border to move south with the native peoples. With this exchange and circulation of people, networks, and goods, it is likely that knowledge about the manufacture of metals was also dispersed, including information about its significance and use.

STUDYING ARCHAEOMETALLURGY IN MENDOZA

Archaeometallurgy uses metallographic and chemical techniques, and historical and bibliographic review to answer various archaeological questions. In this case, archaeometallurgy was used to study the relationship with metallurgy and metals by native peoples.

The effective use of metals in Argentine territory dates back well before the 1500s, although territorially it is restricted to the northwestern region of Argentina^[2]. In southern Mendoza, known use of metals corresponds to post-colonial times. However, recent evidence shows that use of metals may have been practiced in the centuries prior to the arrival of Europeans to the region^[3]. Metallic elements corres-

ponding to both time periods were found at sites such as the Mapuche Cemetery of Cerro Mesa, Malargüe, Mendoza.

As the Mapuches were divided by the Los Andes range, those who occupied the Argentine pampas were called Puwelches, particularly those found south of the Rio Salado. Those who lived in Chile were called Guluches, and they occupied the region from the Bío-bío River to the south in the Araucanía area, maintaining a great exchange on both sides of the Andes mountain range, an ideal mechanism to expand families and strengthen the transfer of goods through the region.

Through the study of historical pieces of Mapuche origin, it is possible to find connections of great importance from before the expansion of the Inca Empire. It is already known that a tradition of metalworking (Vergel Culture) would have existed in the southern regions of Chile and Argentina. In the same way, the arrival of the Spanish to these latitudes left a mark regarding the use of other techniques for metalworking and the mode of supply of raw materials.

In the different areas of the vast Andean territory, the path that metallurgy followed acquired its own peculiarities. The production and technical innovations of metals in the Andes was driven primarily by their use for social status and in the religious sphere where metals served as elements of connection with supernatural powers^[4]. As Heather Lechtman^[5] pointed out, Andean metallurgy was, above all, a way to communicate with the gods.

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HOOP RING IN THE EL VERGEL TRADITION

The El Vergel metalworking tradition (1000 to 1500 A.D.), includes objects such as square notched hoops shown in Fig. 1, flat circular hoops, simple circular hoops, bracelets, and rings. These pieces are characterized by being small in size, not very thick, made up of a single piece, without moving or articulated parts, and without engraved or cut-out and/or relief decorations. All these pieces are made of copper.

The type of notched quadrangular ring belonging to the El Vergel metallurgical tradition surely ceased to be manufactured at some point before the 18th century, but may have continued in use as it passed from generation to generation.

The quadrangular notched ring (also called “Chawai Chapel”) presents,

macroscopically, a good state of conservation and only a gentle cleaning was necessary to begin studies. The chemical composition shows it is composed of copper (94.83%) and arsenic (4.32%), and the rest are trace elements. The data on the copper-arsenic alloy is very interesting and allows this piece to be located on the timeline, because this alloy tends to be the dominant alloy before the 1500s.

According to metallographic studies, the simultaneous concurrence of an approximately constant composition and an adequate proportion of arsenic (<8%) results in the typical structure: a solid copper-arsenic solution made up of a single phase^[6]. Then, arsenic would have been added with intention or considered a fortuitous event according to the characteristics of the minerals in the region: enargite (CuAsS_4) and tennantite (Cu_3AsS_3), and it would also be

considered valid. In other words, although they could not know that they “technically” manufactured a copper-arsenic alloy, they could take into account that when processing the enargite they had a metal with better qualities and a more red color than that resulting from the processing of malachite [$\text{Cu}_2\text{CO}_3(\text{OH})_2$].

The microhardness test shows that the manufacturing process was by successive reduction of thickness by forging and reheating of the original stem to stretch it both longitudinally and transversely. The average value of microhardness in the longitudinal direction is 154 HV and in the transverse direction it is 150 HV. The microhardness value of a current Cu-As alloy is: 151 HV. The Cu-As alloy improves properties such as resistance, hardness, malleability, and ease of fusion, which indicates the degree of technological development of the Mapuche to achieve a resistant material.

BRASS ALLOY CONES

A study of brass alloy cones illustrates the change of raw materials used in the region around 1800 A.D., and is attributable to the processes of social and political transformation strongly fueled by the arrival of Europeans and the creation of the border. Both indigenous and Spanish-Creole metal artisans (criollos) would participate in this process, adapting to the aesthetics of both worlds, leading to changes in the universe of the manufactured pieces^[7]. The cones (Fig. 2) indicate a process of recycling of materials, their chemical compositions are very uniform and the interesting fact is that brass was introduced in the Southern hemisphere by the conquerors. It is possible that these cones were integrated into pectoral pendants called ‘Sikil’ but made of copper. The ‘Sikil’ made of silver seem to arise after the 19th century, approximately in 1860 A.D.^[8].

From the microhardness measurement it is possible to suggest the manufacturing process of the receptacle. It could be that the original ingot had a greater plastic deformation at one of its ends to form a thin sheet, which was laminated in a certain way using, for

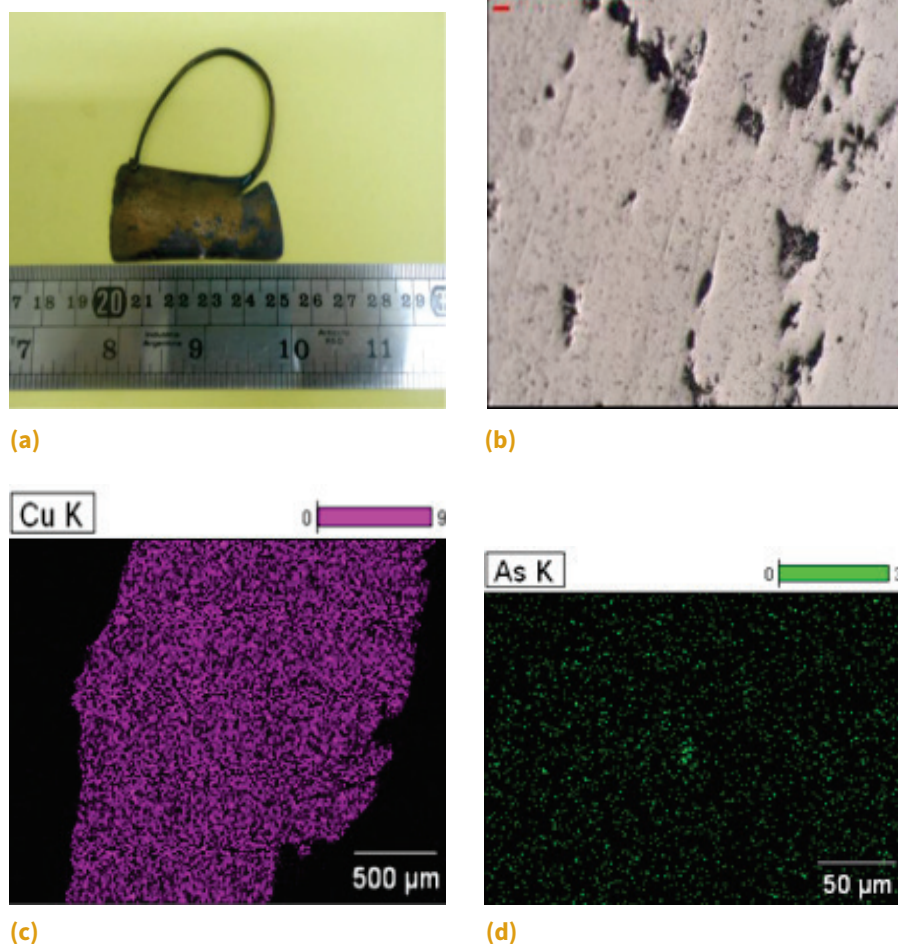


Fig. 1 — (a) A quadrangular hoop with notch as found in Mendoza, (b) the microstructure, and (c) and (d) chemical composition.

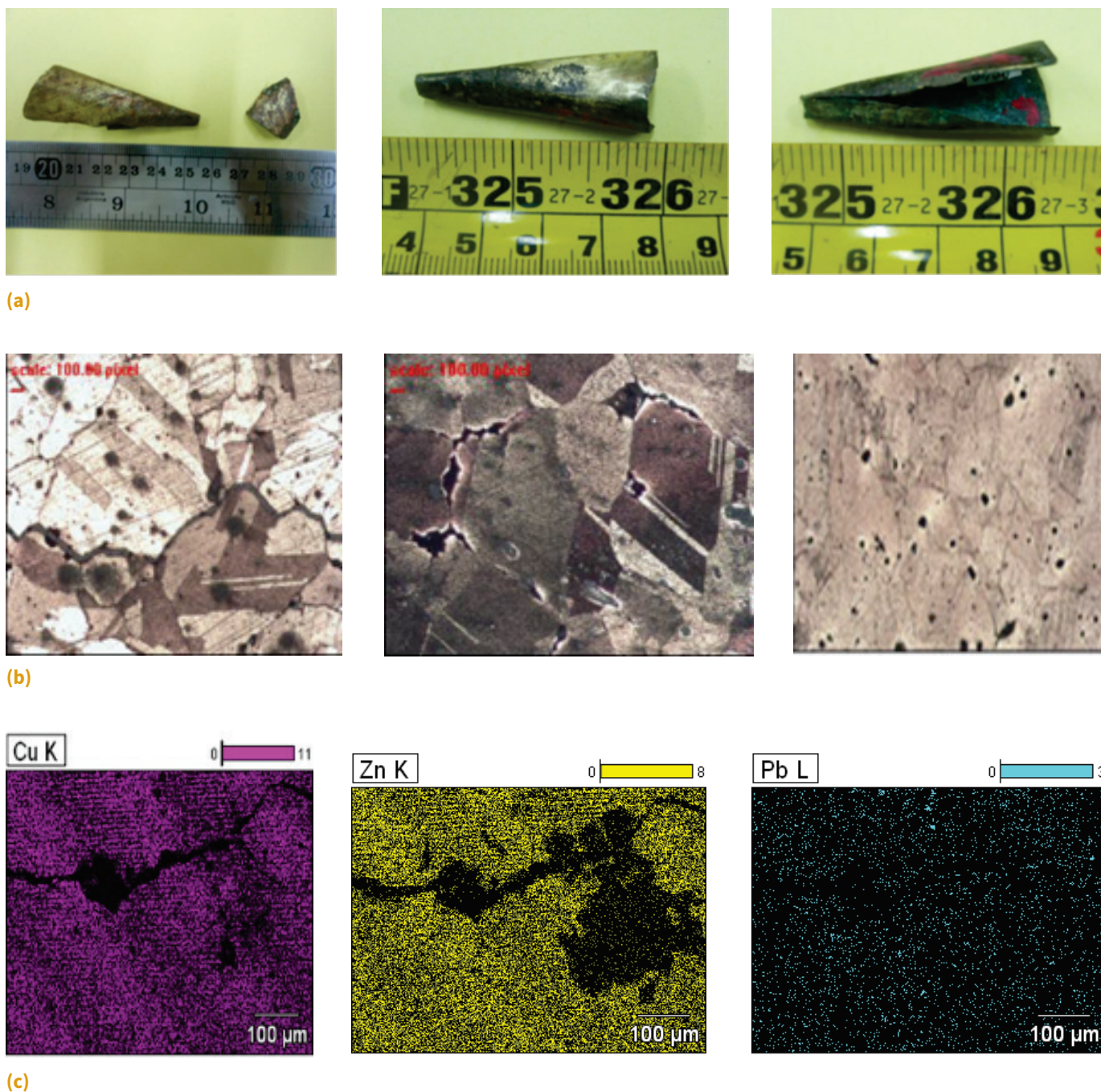


Fig. 2 — (a) Brass cones from the post-1600s era, (b) their corresponding microstructures, and (c) chemical compositions.

example, a piece of wood as a guide in its interior. The average microhardness data for Cone 1 is 121 HV, Cone 2 is 109 HV, and Cone 3 is 144 HV.

Observing the three cones macroscopically, their morphology is very similar (Fig. 2a). From the metallographic study (Fig. 2b), the images show structures consistent with α -brass and leaded brass (Cu-Zn-Pb). The metallographic structure is very similar to brass alloy (Cu-Zn) and lead appears as small

dark particles at the grain boundaries. The images show equiaxed grains, polygonal grains, annealed twins, slip lines, and void segregation.

The chemical composition values as shown in Fig. 2 are: for Cone 1: Cu is 71.1% and Zn is 24.4%; for Cone 2: Cu is 66.7% and Zn is 27.9%; for Cone 3: Cu is 66.6% and Zn is 26.2%. The chemical analysis of the three cones corresponds to the current alloys for forged brass α , that is, brass type α with lead

(Cu-Zn-Pb), precisely it is cartridge brass 70% C36000 for Cone 2 and Cone 3. In the case of Cone 1, the alloy is equivalent to that of cartridge brass 70% C26000^[10].

CONCLUSIONS

The work done to study these artifacts from the era before the arrival of the Spanish reiterates the role metallurgists played in their communities. They didn't consider metalworking an

industrial process, more as a way of interpreting the universe and creating offerings to their gods. Archaeometallurgical study shows the influence of migration and European settlement in the area. ~AM&P

Acknowledgments

The author wants to especially thank the staff of the Natural Museum of San Rafael, Mendoza, Argentina for allowing the study of these historical-heritage assets. This research study is part of the Border Archaeometallurgy Project (MATUNME code 004308) developed at the Metallurgy Laboratory, National Technological University, Faculty Mendoza Regional.

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References

1. N.E. Sosa Morales, Historia de un Pueblo La Villa Vieja [History of a Town Village The Old], Serie Libros N°1, Museo de Historia Natural, Dirección Municipal de Cultura, San Rafael, Provincia de Mendoza, Argentina, 1979.
2. H. Lagiglia, Hacha insignia ceremonial de bronce Santamariana hallada en Mendoza [Santamariana bronze ceremonial insignia axe found in Mendoza], *Actas Jornadas de Arqueología del Noroeste*, Buenos Aires, Universidad del Salvador, p 74-84, 1979.
3. R. Campbell, Entre el Vergel y la Platería Mapuche: El Trabajo de los Metales en la Araucanía Post Contacto (1550-1850 D.C.) [Between the Vergel and the Mapuche Silverware: Metalworking in Post-contact Araucanía (1550-1850 A.D.)], 2004.
4. J. Antinao, Sociedad y Cosmovisión en la Platería Mapuche [Society and Cosmvision in Mapuche Silverware]. Universidad Católica de Temuco, Temuco, Chile, 2010.
5. H. Lechtman, Reflexiones Sobre la Metalurgia de América. En Metalurgia de la América Precolombina [Reflections on the Metallurgy of America. In Metallurgy of Pre-Columbian America], p. 301-306. Memorias 45° Congreso Internacional de Americanistas. Banco Popular, Bogotá, Colombia, 1988.
6. M. Hansen, Constitution of Binary Alloys, Metallurgy and Metallurgical Series. Mac Graw-Hill Book. Segunda Edición, 1958.
7. L. González, Bronce Bajo el sol. Metalurgia Prehispánica en el Noroeste Argentino [Bronze under the Sun. Pre-Hispanic Metallurgy in the Argentine Northwest], pp. 97-131. En: Masked Histories. Editores. Stenberg, P. y Muñoz, A., Etnologiska Studier 43, Gotemburgo, 1999.
8. Antinao, J.: op. cit., 2010 *ASM Handbook*, Volume 9: *Metallography and Microstructures*, ASM International, 2004.
9. *ASM Handbook*, Volume 2: *Properties and Selection: Nonferrous Alloys and Special-purpose Materials*, ASM International, 1990.

GET ENGAGED, GET INVOLVED, GET CONNECTED

Join the conversation about historic metals. The archaeometallurgy community on ASM Connect is very active and welcomes new participants. Recent discussions include metallurgy during the Iron Age, pigment analysis, misconceptions in archaeometallurgy, and a call for papers to present at IMAT 2021. Visit the ASM Connect home page at connect.asminternational.org, search for the Archaeometallurgy Committee, and get into the conversation!



Patricia Silvana Carrizo prompted the formation of the Archaeometallurgy Community and has been leading its efforts.

MATERIALS SCIENCE AND CORONAVIRUS SERIES

EPA OFFICIALLY SAYS COPPER SURFACES HELP FIGHT COVID-19

The story behind the Environmental Protection Agency's registration of copper surfaces against the virus that causes COVID-19.

Harold T. Michels,* consultant and retired senior vice president, Copper Development Association, Manhasset, New York

In May 2020, a short article^[1] describing why copper has the potential to help in the fight against COVID-19 was published in *Advanced Materials & Processes* magazine. The article cites a short pioneering study^[2] illustrating that SARS-CoV-2, the virus that causes COVID-19, was inactivated in 4 hours on 99.9% copper surfaces, but remained infective for a prolonged period on plastic and 304 stainless steel.

The *AM&P* article also discussed a 2015 publication^[3] on another human coronavirus (Hu-CoV-229E) that also causes lung disorders. Six copper alloys, ranging from 100% to 60% Cu, balance Zn, as well as a series of copper nickel alloys were challenged by Hu-CoV-229E. Rapid inactivation of Hu-CoV-229E was observed on different copper alloy surfaces within 10 minutes.

Although these two articles^[2,3] used different strains of human coronavirus, both viruses are essentially structurally identical. They display the now familiar spherical shapes, but with slight differences in their spike proteins. It was surmised that both of these coronaviruses would be inactivated by copper by the same mechanism of attack because of their similar structures.

A more recent paper^[4], published on January 2, 2021, showed that the COVID-19 causing virus, SARS-CoV-2, was inactivated by copper in as little as 1 minute. This paper confirms the speculation made in the *AM&P* article^[1] that

because copper was effective against Hu-CoV-229E, it would also be effective against SARS-CoV-2. Copper is effective against both of these human coronaviruses because they have very similar structures. All of this information was made available to the U.S. Environmental Protection Agency (EPA).

EPA ANNOUNCEMENT

On February 10, 2021, the EPA announced that it has registered certain copper alloys that have demonstrated effectiveness against viruses, including SARS-CoV-2, the virus that causes COVID-19^[5]. (See "EPA Registers Copper Surfaces for Residual Use Against Coronavirus.") Note that this is the first antimicrobial product that is registered for "residual" use against viruses. Traditional disinfectants only kill viruses and bacteria on the surface at the time they are used, while in contrast, "residual" antimicrobial disinfectants kill pathogens that come in contact with the surface days, weeks, or years after the product is applied. This residual antimicrobial property of copper alloy surfaces provides a unique advantage in the fight against COVID-19 infections.

FUTURE PANDEMICS

The EPA acted very quickly because of the seriousness of the COVID-19 crisis. Thus copper alloy surfaces can now be used in the fight against SARS-CoV-2 as well as other pandemic viruses

AM&P'S MATERIALS SCIENCE AND THE CORONAVIRUS SERIES

This article is the eighth installment in an *AM&P* series on materials science and coronavirus. Below is a list of the first seven articles:

- Can Copper Help Fight COVID-19? May/June 2020.
- Copper's Conductivity and Antimicrobial Properties Inspire Renewed Interest, July/August 2020.
- Antimicrobial Copper-Containing Stainless Steels Show Promise, September 2020.
- Using Digitally Distributed Manufacturing to Address Critical Needs, October 2020.
- Development and Validation of High-Performance SARS-CoV-2 Antiviral Coatings for High-Touch Surfaces, November/December 2020.
- Supersonically Deposited Antiviral Copper Coatings, January 2021.
- Optimizing 3D-Printed, Reusable Metal N95 Filters by 3D Characterization and Modeling, February/March 2021.

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that may emerge in the future. A variety of new viral infections seems to rapidly spread around the world, perhaps because of the ease of air travel. Thus it seems logical to deploy copper touch surfaces as a first line of defense in transportation facilities, including airports, train and bus stations, as well as planes, rail cars, and buses. Of course, nursing homes, extended care facilities, and hospitals should also be outfitted with copper alloys because they house a very susceptible demographic.

~AM&P

Lead image: 2019-nCoV spike protein, courtesy of Jason McLellan/University of Texas at Austin.

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References

1. H.T. Michels and C.A. Michels, Can Copper Help Fight COVID-19?, *Advanced Materials & Processes*, Vol 178, No. 4, p 21-24, 2020.
2. N. van Doremalen, et al., Aerosol and Surface Stability of SARSCoV-2 as Compared with SARS-CoV-1, *N. Engl. Jour. Med*, 2020, DOI: 10.1056/NEJMc2004973.
3. S.L. Warnes, Z.R. Little, and C.W.

Keevil, Human Coronavirus 229E Remains Infectious on Common Touch Surface Materials, *mBio*, American Soc. Microbiology, Vol 6, e01697-15, 2015.

4. C. Bryant, S.A. Wilks, and C.W. Keevil, Rapid Inactivation of SARS-CoV-2 on Copper Touch Surfaces Determined Using a Cell Culture Infectivity Assay, *bioRxiv*, DOI: 10.1101/2021.01.02.424974.

5. EPA Registers Copper Surfaces for Residual Use Against Coronavirus, February 10, 2021, [epa.gov/newsreleases/epa-registers-copper-surfaces-residual-use-against-coronavirus](https://www.epa.gov/newsreleases/epa-registers-copper-surfaces-residual-use-against-coronavirus).

EPA REGISTERS COPPER SURFACES FOR RESIDUAL USE AGAINST CORONAVIRUS

The U.S. Environmental Protection Agency (EPA) announced that certain copper alloys provide long-term effectiveness against viruses, including SARS-CoV-2, the virus that causes COVID-19. As a result of EPA's approval, products containing these copper alloys can now be sold and distributed with claims that they kill certain viruses that come into contact with them. This is the first product with residual claims against viruses to be registered for use nationwide. Testing to demonstrate this effectiveness was conducted on harder-to-kill viruses.

In this action, EPA is granting an amended registration to the Copper Development Association for an emerging viral pathogen claim to be added to the label of Antimicrobial Copper Alloys- Group 1 (EPA Reg. No. 82012-1), which is made of at least 95.6% copper. Amended registrations allow previously registered products to make label changes (e.g., changes to product claims, precautions and/or use directions) and/or formulation changes. In this case, the amended registration is adding virus claims to the product registration.

New efficacy testing supported by the Copper Development Association and conducted according to EPA's protocols demonstrated certain high-percentage copper alloy products can continuously kill viruses that come into contact with them. Based on testing against harder-to-kill viruses, EPA expects these products to eliminate 99.9% of SARS-CoV-2, the virus that causes COVID-19, within two hours.

Antimicrobial copper alloys can be manufactured into a wide range of surfaces, including doorknobs and handrails. These high-percentage copper alloy products will be added to the List N Appendix, the Agency's list of residual antiviral products that can be used to supplement routine cleaning and disinfection to combat SARS-CoV-2.



AEROMAT²¹

32nd CONFERENCE AND EXPOSITION

CO-LOCATED EVENT:



MAY 24-26, 2021 | VIRTUAL EVENT

VIRTUAL SHOW PREVIEW

AeroMat 2021 is pivoting to a 100% virtual conference and expo platform. The good news is that AeroMat will still co-locate with the ITSC 2021 Conference and Exposition. The virtual AeroMat 2021 will offer the same vibrant technical programming, and impactful networking and engagement opportunities that you have come to expect from an in-person conference.

Here is a snapshot of what you can expect:

- **TWO** conferences for the price of **one**! More than 300 researchers and experts will present their work during AeroMat and ITSC.
- On-demand access to technical presentations for up to 30 days following the conferences.
- Keynote Sessions and Industry Panel Sessions
- Virtual Exhibit Hall Experience—Visit and chat with exhibitors through this highly interactive and engaging platform and learn about the latest advancements in technology.
- Engage and Interact—Connect with your colleagues from around the world in the dedicated AeroMat and ITSC attendee lounges, as well as via live chats during sessions and networking events.

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KEYNOTE SESSIONS

MONDAY, MAY 24 | 10:00-11:00 a.m. EDT



Joint Keynote Session with ITSC
Mitchell R. Dorfman, FASM, TSS-HoF
Product Portfolio Manager
High Temperature Materials
Oerlikon Metco (US) Inc.

Present and Future Thermal Spray Challenges and Opportunities in the Aerospace Industry

The market need for increased engine efficiency and environmentally friendly solutions remain the key drivers for the aerospace industry. These efficiency gains will be achieved by meeting the challenges of higher engine operating temperatures, new lighter weight components, and the surface solutions required for increased component longevity. This presentation will look at the thermal spray market related to aerospace, its suppliers, and review the challenges and opportunities for future growth.

MONDAY, MAY 24 | 3:15-4:15 p.m. EDT



Dr. James Ruud
Senior Principal Scientist
GE Global Research Center

Suspension Thermal Spray Coatings

Thermal spray processing using suspension feedstocks has produced new technological, manufacturing, and product opportunities. Suspension spray has produced novel coatings from ceramics, metals, and cermets. It has unique advantages for rapid research development yet also scales well into manufacturing. This talk will describe opportunities and challenges in developing and applying suspension thermal spray coatings for industrial aerospace applications.

TUESDAY, May 25 | 10:30-11:15 a.m. EDT



Dr. Ron Aman
Principal Engineer
EWI

Putting Materials and Process Research to Work—Transitioning from the R&D lab to Application

The speed of innovation is increasing at a growing rate with the application of ICME and data science methods. EWI is using these tools and others in a wide variety of applications and materials to bring lab-scale breakthroughs to the aerospace industry. A review of past successes along with current internal and Additive Manufacturing Consortia research will be presented.

PANEL

Emerging Additive Manufacturing of Materials

Wednesday, May 26 | 10:00 - 11:30 a.m. EDT

EDUCATION COURSES

Fatigue and Fracture

Instructor: Kumar Jata, Ph.D.
Thursday, May 27 | 12:00 - 4:00 p.m. EDT

Additive Manufacturing/Failure Analysis

Daniel P. Dennies, Ph.D., PE, FASM
Thursday, May 27 | 12:00 - 4:00 p.m. EDT

EXHIBITOR LIST

Ardleigh Minerals, Inc.
CenterLine SST
Fujimi Incorporated
GTP
Impact Innovations GmbH
IMR Test Labs
Leica Microsystems
Metallizing Equipment Co. Pvt. Ltd.
Nel Hydrogen

Oerlikon Metco
Oseir Oy
Quaker Houghton
Saint-Gobain
Salloytech
TECNAR Automation Ltd.
Thermo Fisher Scientific

Exhibitor list current as of March 15.

EXHIBIT HOURS

May 24-26
Monday - Wednesday
9:30 a.m. – 1:30 p.m. Daily

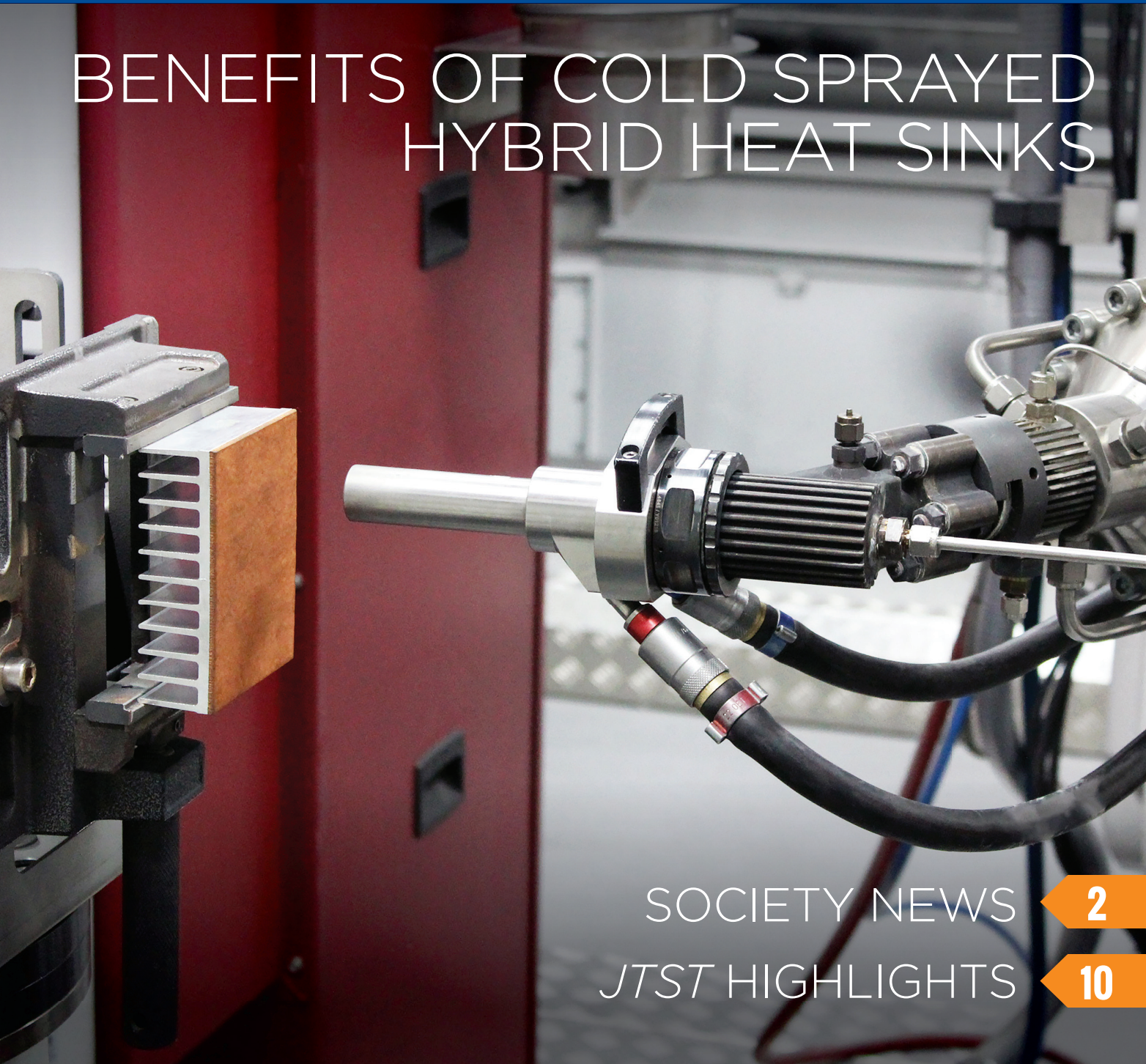
Exhibition hours are subject to change.

iTsSe

INTERNATIONAL THERMAL SPRAY & SURFACE ENGINEERING

THE OFFICIAL NEWSLETTER OF THE ASM THERMAL SPRAY SOCIETY

BENEFITS OF COLD SPRAYED HYBRID HEAT SINKS

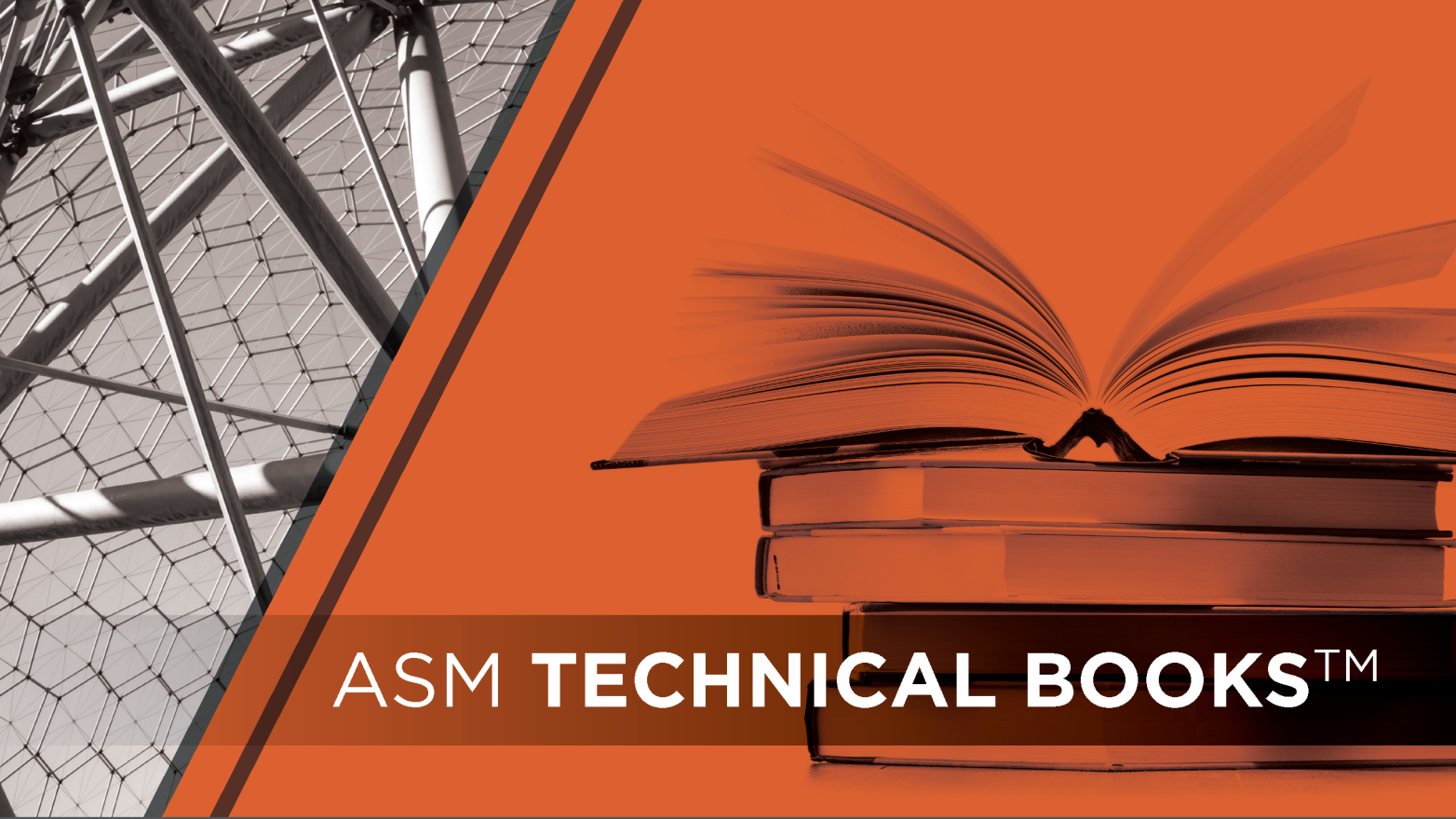


SOCIETY NEWS

2

JTST HIGHLIGHTS

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ASM TECHNICAL BOOKS™

ASM Technical Books Added to ASM Digital Library!

More than 50 of ASM International's best-selling materials engineering books are now available in the ASM Digital Library platform.

ASM International is proud to announce the launch of the new ASM Technical Books site in the ASM Digital Library. The new site provides convenient access to more than 50 book titles on topics related to materials selection, processing, properties, performance, and characterization. For the first time ever, subscribers will be able to gain access to a broad collection of ASM Technical Books and receive the highest value of all Access Options that the brand has to offer. With additional legacy titles from the ASM collection and new titles being added throughout 2021 and beyond, subscribers will experience enhanced value and benefits well into the future.

The launch of the ASM Technical Books site marks the first time that these valuable content resources are available in a unified content delivery platform offering advanced search, browse, filtering, linking, and content access.

"ASM has been a leading publisher of books on materials engineering for more than one hundred years. Our goal with the ASM Digital Library is to make all of ASM's content available in a single location to help ASM Members and other users quickly find the answers they need. We are very pleased that this collection of books is now on the site."

- Ron Aderhold, Acting Managing Director of ASM International

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EDITORIAL OPPORTUNITIES FOR iTSSe IN 2021

The editorial focus for iTSSe in 2021 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.

July/August:

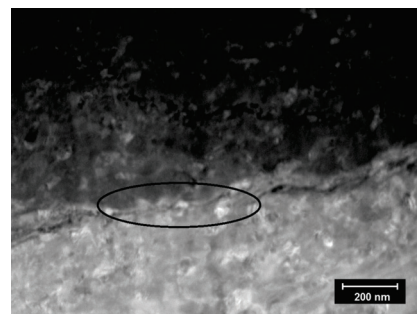
Energy and Power Generation

To contribute an article, contact Joanne Miller at joanne.miller@asminternational.org.

To advertise, contact Kelly Johanns at kelly.johanns@asminternational.org.

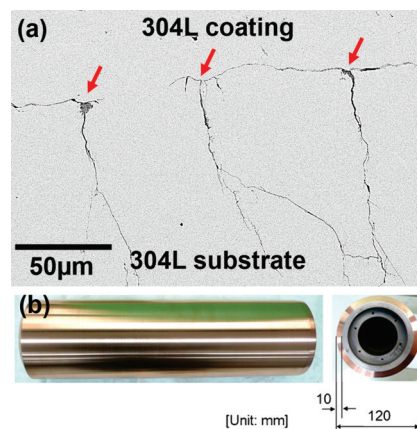
5

HYBRID HEAT SINK MANUFACTURING BY COLD SPRAY



8

NEXT GENERATION PLASMA SPRAY TORCH



DEPARTMENTS

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3 | ITSC VIRTUAL SHOW PREVIEW

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ABOUT THE COVER

A copper layer is deposited via cold spray technology onto a base plate of an extruded aluminum heat sink. Courtesy of Impact Innovations.

CAN YOU SEE OUR TSS SCREEN?

“Can you see my screen? Do you see my presentation?” These words have become part of our daily lives over the past year as many of us, especially in the academic world, have been working remotely and sharing research online. We have all been challenged in various ways and forced to develop and use new communication tools to minimize physical contact while maintaining safe in-person activities when there is no virtual option.

Graduate students all around the world have been adapting to the situation and have tried to make the best of it. While research lab access has been limited—with some periods where labs were completely closed—students have been models of resilience and found creative ways to make up for the lack of lab time. Cancellation of the vast majority of international conferences in 2020 was potentially one of the most detrimental aspects of the pandemic for these students, career-wise, preventing them from direct contact and feedback from world experts. Conferences are always a highlight in a student’s life and offer opportunities to get a better feel for the field, meet potential employers, and receive guidance on career options. As one solution, I started a new model of interactive, virtual sessions at my university to expose students to the real world.

Likewise, Charles Kay, FASM (ASB Industries/HRC), challenged TSS leaders to provide a short-term solution to this dilemma for graduate students in the thermal spray community, with a longer-term goal of continuing to offer them the best training environment possible to reach their career objectives. With the overwhelming support of ASM and ASM/TSS members André McDonald, FASM (University of Alberta, Canada), William Lenling, FASM, TSS-HoF (Thermal Spray Technologies Inc.), Rogerio Lima (National Research Council of Canada), and Jeanelle Harden (ASM events manager), the TSS Open Mic Series was created. The main objective is to ensure that students have direct access to thermal spray world leaders not only to receive science and engineering updates, but more

importantly to learn about their views of our industry and how to better prepare to enter the workforce.

Once a month, the TSS Open Mic Series welcomes a world expert who shares their field experience and vision and provides tips and guidance to attendees. The 10-15 minute presentation is followed by a Q&A session that aims to mimic exchanges that would normally occur between talks at an in-person conference session. The goal is to promote a relaxed discussion and exchange of information at both the technical and non-technical level.

Dr. Christopher Berndt, FASM, TSS-HoF (Swinburne University of Technology, Australia), introduced the series with a talk in which he shared guidelines that have served him well, along with some tough lessons and advice on living a balanced life. (Surf’s up!) At the second Open Mic event, Dr. Sanjay Sam-path, FASM, TSS-HoF (Stony Brook University), provided a thorough historical review on thermal spray with key advice to students about the importance of knowing what has already been done in order to build on it and move technology forward. In the third talk, Dr. Y.C. Lau, TSS-HoF (General Electric), delivered a comprehensive review on thermal barrier coatings along with his vision of the future for thermal spray.

While primarily designed for graduate students, the first three Open Mics were attended by thermal sprayers in both academia and industry, from more than 34 countries, confirming that the series is filling a need. For this first year, talks are scheduled once a month through June. We invite everyone to register for free at tss.asminternational.org and join us to learn and exchange with our students. We hope to “see you” at the next one.

Prof. Bertrand Jodoin
University of Ottawa, Canada



Jodoin

SOLICITING STUDENT MEMBERS FOR TSS BOARD

The ASM Thermal Spray Society is seeking applicants for the two student board member positions. Students must be a registered undergraduate or graduate during the 2021-2022 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 on the eligibility and benefits of this position, visit tss.asminternational.org.

Nominations are due by April 30.

SEEKING NOMINATIONS FOR THERMAL SPRAY HALL OF FAME

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

Nominations are due September 30.



ITSC2021

INTERNATIONAL THERMAL SPRAY
CONFERENCE AND EXPOSITION

CO-LOCATED EVENT:

AEROMAT²¹
32nd CONFERENCE AND EXPOSITION

MAY 24-28, 2021 | VIRTUAL EVENT

itscevent.org

VERSATILE SURFACE ENGINEERING FOR ENVIRONMENTAL SOLUTIONS

VIRTUAL SHOW PREVIEW

Plan now to attend the 2021 International Thermal Spray Conference & Exposition, the premier annual event for the global thermal spray community to meet, exchange information, and conduct business. While we were looking forward to seeing everyone in-person at ITSC 2021 in Quebec City this May, the safety and well-being of our attendees and exhibitors is our top priority.

Based on concerns related to the coronavirus pandemic and its continued impact on our international community's ability to travel to the conference, **ITSC 2021 is pivoting to a 100% virtual conference and expo platform.**

Moving our face-to-face conference to a fully online format presents us with the unique opportunity to showcase the best of what the online modality affords—flexibility, meaningful collaboration, and the ability to network and connect with colleagues around the world. We hope that you will join us for this reimagined event and celebrate our collective resilience and dedication to forging connections without boundaries.

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CONFERENCE HIGHLIGHTS

Oerlikon Metco Young Professionals Competition – Virtual Edition!

Wednesday, May 26 | 11:00 a.m. EDT

Sit in on this lively session that showcases oral presentations from young innovative scientists/professionals, all vying for a chance to win a prize package, courtesy of Oerlikon Metco.

Physical barriers are practically eliminated! The virtual platform offers a more convenient option for individuals who might not normally attend ITSC due to financial or travel restrictions. ITSC 2021 is a fraction of the cost of an in-person conference, offering reduced registration fees and no travel-related expenses for flight, hotel, and other ancillary expenses.

Networking opportunities galore! Attendees will have multiple ways to connect with fellow attendees, speakers, and exhibitors, via the networking lounge, exhibit hall, and during sessions. Attendees will be able to see who is attending ITSC and can also connect with them privately, via message or chat.

Convenient Schedule

May 24-28 | 8:00 a.m. – 1:00 p.m. EDT

The half-day virtual format makes it more convenient for international attendees to participate in live sessions and programming. The condensed daily schedule also offers the flexibility to be able to take care of business and family commitments.

Access to the same robust technical programming you have come to expect from an in-person ITSC. We will have more than 200 presentations within the three symposia areas: Thermal Spray Applications, Fundamentals/R&D, and a joint session with AeroMat on Advanced Coatings for the Aerospace Industry. If you miss a live session, attendees will have on-demand access to all sessions for up to 30 days after the conference. Attendees will also have full access to AeroMat 2021 programming.



Dorfman

Joint Keynote Session with AeroMat

Monday, May 24 | 10:00-11:00 a.m. EDT

“Present and Future Thermal Spray Challenges and Opportunities in the Aerospace Industry,” featuring Mitchell R. Dorfman, FASM, TSS-HoF, Oerlikon Metco (US) Inc.

EXHIBITOR LIST

Ardleigh Minerals, Inc.
CenterLine SST
Fujimi Incorporated
GTP
Impact Innovations GmbH
IMR Test Labs
Leica Microsystems
Metallizing Equipment Co. Pvt. Ltd.

Nel Hydrogen
Oerlikon Metco
Oseir Oy
Quaker Houghton
Saint-Gobain
Sallyotech
TECNAR Automation Ltd.
Thermo Fisher Scientific

Exhibitor list current as of March 15.

EXHIBIT HOURS

May 24-26
Monday - Wednesday
9:30 a.m. – 1:30 p.m. Daily

Exhibition hours are subject to change.

HYBRID HEAT SINK MANUFACTURING BY COLD SPRAY

Hybrid heat sinks produced via cold spray technology can be a cost effective and lightweight alternative for cooling electronics.

Reeti Singh

Impact Innovations GmbH, Rattenkirchen, Germany

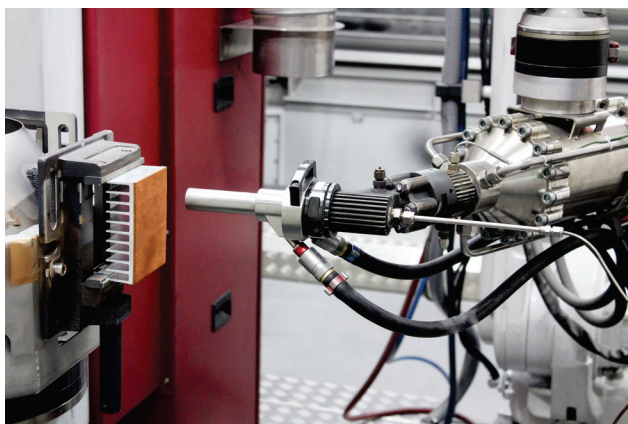
Electronic devices, such as those used in telecommunications and high-power systems, generate heat during normal operation. Yet this heat must be dissipated to avoid junction temperatures exceeding tolerable limits, as it can lead to performance inhibition and deterioration of reliability. It has been shown that every 10 K reduction in the junction temperature will increase the device's life and performance. Thus, maintaining the junction temperature below the maximum allowable limit is a primary issue.

The most common way to cool devices has been air/liquid cooling using a heat sink. Conventionally, copper and aluminum heat sinks are used in combination with such cooling systems. Copper is a preferred choice for heat sinks due to its cooling capacity superiority over aluminum; however, copper's weight and cost limit the size, especially for large electronics systems. Whereas, due to lower thermal conductivity, aluminum heat sinks do not spread the heat quickly enough; thus, a large surface area or taller fins are required, which is not a plausible option in many cases. Moreover, a problem arises if a heat sink is substantially larger than the integrated circuit devices it resides on. If the electronic device generates heat faster than the heat sink spreads, portions of the heat sink far away from the device do not contribute much to heat dissipation. In other words, if the base is a poor heat spreader, much of its surface area is wasted. Furthermore, to connect the aluminum heat sink with electronic devices, a thermal interface material is generally used because soldering of aluminum with direct bond copper of the electronic device is difficult. Typically, this material has a very low thermal conductivity, affecting the overall aluminum heat sink's performance.

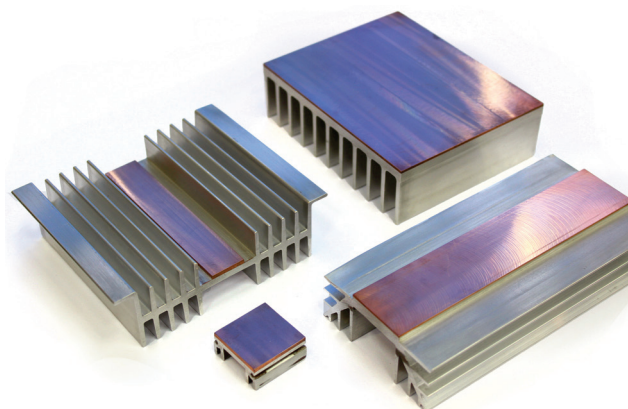
HYBRID HEAT SINKS

A hybrid heat sink—combining the thermal benefits of copper with lightweight aluminum—presents an exciting alternative to overcome the issues associated with conventionally available copper and aluminum heat sinks. In such a concept, the portion of the heat sink that comes in contact with the electronic device is made of copper, while the other portion is made of cheaper and lighter aluminum. However, joining aluminum and copper is a difficult challenge. Soldering and brazing is commonly used to join aluminum with copper in industrial refrigeration, air conditioning, and heat exchangers.

However, there are many issues associated with soldering and brazing, such as corrosion at the interfaces and solder materials with different electrical resistance, as well as thermal expansion mismatch.



(a)



(b)

Fig. 1 — Cold sprayed hybrid heat sinks: (a) application of copper powder coating and (b) resulting sprayed plates.

The cold spray (CS) technique is an innovative solution to join copper and aluminum and overcome the issues associated with soldering and brazing. The CS process is known to deposit the powder particles in solid-state far below the material's melting point; thus, it can avoid common temperature-induced problems such as high-temperature oxidation, thermal stresses, and phase-transformation. Cold spray is a powder-based technology in which micron-size powder particles are accelerated in the supersonic flow of a compressed working gas through a de Laval nozzle. These powder particles impact the substrate, plastically deform, and create bonding with the substrates. CS offers short production times, virtually unlimited component size capability, and flexibility for localized deposition.

THERMAL RESISTANCE EXPERIMENTS

To demonstrate the process, the Impact Innovations ISS 5/11 cold spray system and Impact's cold spray grade copper powder (iMatP_Cu01) were used to produce hybrid-heat sinks. A copper layer was deposited on a base plate of a commercially available extruded aluminum heat sink, as shown in Fig. 1. The thickness of such a copper layer can be adjusted to the electronic device's design and operational temperature.

When discussing a heat sink's performance, its cooling capability is typically quantified in terms of the thermal resistance, a measure of the temperature rise above ambient on the top of the device per dissipated unit of power. The lower the value of thermal resistance, the higher the cooling ability of the heat sink. To demonstrate the performance of hybrid heat sinks, Impact Innovations conducted experiments to compare the performance of identically structured copper, aluminum, and hybrid heat sinks. The experiment was performed three times, each time with a different heat sink design. Thermal impedance and thermal resistance were measured. The thermal impedance of heat sinks was evaluated by running power cycles at specific load currents heating the device until reaching the thermal equilibrium. Then the load current was switched off, and the voltage drop was recorded. When an aluminum heat sink was tested, a maximum temperature of 438 K was registered. This value corresponds to a thermal resistance of 0.7 K/W. For the copper heat sink, the maximum temperature was just 348 K, and the corresponding thermal resistance was

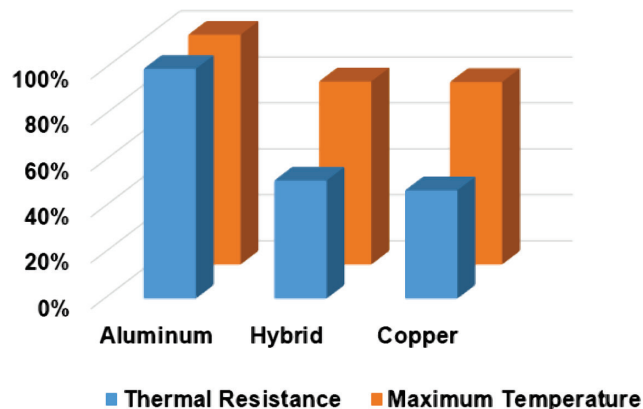


Fig. 2 — Thermal resistance and maximum temperature obtained at the device using aluminum, hybrid, and copper heat sinks.

0.33 K/W. Testing the hybrid-heat sink, the maximum temperature was just slightly higher at 349 K, and the thermal resistance was 0.36 K/W.

These results show that the copper and hybrid-heat sinks have almost identical thermal results and outperformed the aluminum heat sink in a substantial fashion, thus showing the importance of quick heat spreading along the base. At the same time, the hybrid heat sink weighed and cost less than the copper heat sink.

Indeed, hybrid heat sinks manufactured by cold spraying have slightly higher production cost than commercially available aluminum heat sinks. However, adding a layer of copper on an aluminum heat sink decreases its thermal resistance by 48%. This has a direct affect on the production costs because the semiconductor area can be decreased by 94%. In addition, the deposition efficiency and deposition rates of copper powder by the cold spray process are 95% (including overspray) and 10 kg/h, respectively, indicating the potential of the CS process to realize a cost-effective large-scale industrial production. ~iTSSe

For more information: Dr. Reeti Singh, principal scientist, Impact Innovations GmbH, Bürgermeister-Steinberger-Ring 1, 84431 Rattenkirchen, Germany, info@impact-innovations.com, www.impact-innovations.com.

NEXT GENERATION CERAMIC SUSPENSIONS

Suspension sprayed coatings create a performance advantage.

Treibacher Industrie AG is headquartered in Althofen, Austria and is more traditionally known for its ferro alloy, hard metal, and rare earth products. However, over the last decade, the company has been building its position as a leading developer and producer of next generation thermal spray materials.

Treibacher was a first mover in the development and commercialization of ceramic suspensions for suspension spraying. AuerCoat suspensions are available in a variety of different solvents with solid contents from 20 to more than 70 wt.%. They are manufactured in Austria with seamless scale-up capabilities from lab to full production. Some products can be manufactured according to AS9100 if required.

Suspension spraying has the capability to generate a range of coating microstructures that create value for a wide range of applications. For example, the technology is used to obtain feather-like thermal barrier coatings that show excellent thermal strain tolerance. Compared to electron-beam physical vapor deposition, it is a cost effective alternative



State-of-the-art Thermal Spray Center
in Althofen, Austria.

and opens up these coating structures to much larger components, which brings great advantages to aero and industrial gas turbines. Suspension spraying can also create highly dense coatings that offer improved corrosion resistance during the plasma etch process for the semiconductor industry and superior wear resistance for print and paper applications. Furthermore, these high solid content suspensions combined with mod-

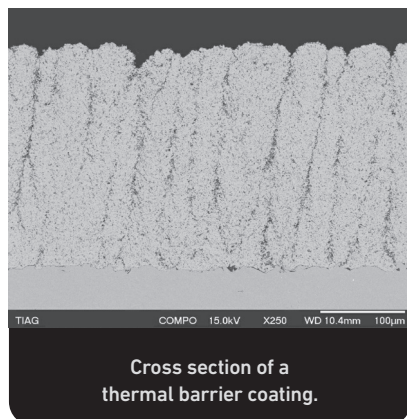
ern spray equipment make suspension spraying even more economically viable.

To ensure a complete market offering, Treibacher also has a Thermal Spray Center, equipped to spray both suspensions and spray powders and a comprehensive application-testing lab. This gives customers a significant competitive edge. The Thermal Spray Center is available for joint development projects with customers and hosts workshops on suspension spraying.

Contact Treibacher to see how suspension sprayed coatings can create a performance advantage for you.



Treibacher offers ceramic suspensions
for thermal spraying.



Cross section of a
thermal barrier coating.



Innovation is our tradition.

For more information, contact:
Treibacher Industrie AG, Austria
P: +43 4262 505-0 / F: +43 4262 2005
thermalspray@treibacher.com
www.treibacher.com

NEXT GENERATION PLASMA SPRAY TORCH

Innovations built into a new plasma spray torch address shortcomings of previous axial injection equipment.

Ash Kamble

Northwest Mettech Corp., Vancouver, BC, Canada

The Axial III, launched in 1994, is a patented plasma spray torch with axial injection of feedstock. In comparison to the traditional radial injection, axial injection technology has proven to offer greater deposition efficiency, throughput, and cost savings on numerous applications^[1]. Despite these benefits, the original Axial III M600 torch had several limitations due to its size, mass, and number of components. To address these shortcomings, a smaller, lighter, and more ergonomic version of the torch was developed, called the Axial III Plus. This article highlights the innovations in this new torch and the potential applications it opens for the plasma spraying industry.

RADIAL VS. AXIAL INJECTION

Conventional plasma spraying equipment uses a radial injection design whereby feedstock is injected radially into the plasma plume. Due to the size distribution of the feedstock, particles have different momentums as they enter the plasma stream, in turn leading to different trajectories^[2]. The larger and heavier particles tend to pass through the plume with little deflection toward the substrate whereas the smaller and lighter particles may be unable to penetrate the plume and simply bounce off. The result is that only a portion of the injected particles, being of an optimum size, are successfully carried by the plasma plume toward the substrate.

The axial injection technology enables the injection of feedstock along the axis of the plasma plume. This overcomes many of the issues posed by radial injection because more particles are completely entrained by the plume, leading to a more uniform particle heating and a much less dispersed trajectory. The major benefits that stem from this are ^[1]:

- Higher deposition efficiencies
- Higher deposition rates
- Better reproducibility of coatings
- Denser, lower porosity coatings
- More homogeneous coatings
- Less overspray
- Lower defect rate
- More effective spraying of nanoparticles in suspension

LIMITATIONS OF AXIAL INJECTION

To enable axial injection, the current Axial III design uses three electrodes symmetrically positioned around a central feed passage. This configuration inadvertently increases the

size of the torch, thereby limiting the size and geometry of substrates that can be coated. A larger torch also means it is heavier and thus takes more effort to handle. Furthermore, having three electrodes instead of one increases the number of components in the torch, making assembly and disassembly more difficult and time-consuming.

INNOVATIONS IN THE NEW TORCH

In the 15 years of service of the original Axial III M600, stakeholders in industrial and research sectors have assessed its performance and identified a list of potential upgrades for the torch. The Axial III Plus was thus developed, incorporating those upgrades. Fig. 1 shows the two torches side-by-side.

Improvements to the torch include:

- 90° cable connections to reduce the overall length of the torch. This not only enables the spraying of smaller internal bores, but also allows easy access to the fasteners for assembly and disassembly of the torch.
- Optimized part features and dimensions to reduce the overall mass of the torch.
- Larger gas jet lines to reduce back pressure.
- Integrated features to reduce the total number of parts.
- Multi-start threaded nozzle cap and threaded cathodes for quick and easy fastening.
- Pre-aligned suspension injector assembly to eliminate the need for manual alignment.
- Modified anode retainer design and tolerances for better electrode alignment.
- Refined electrical cable contacts for manual assembly and disassembly, greater longevity, and more secure electrical connection.

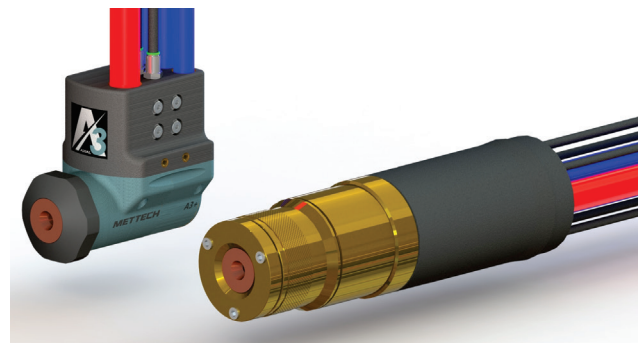


Fig. 1 — Axial III Plus (left) and Axial III M600 (right).

FEATURE

TABLE 1 – SUMMARY OF IMPROVEMENTS

Feature	A3M600	A3+	Improvement
Length (inches)	23.6	6.1	74% reduction
Mass (pounds)	15.75	6.70	57% reduction
Average Assembly/ Disassembly Time (min:sec)	5:21	2:28	54% reduction
Number of Parts	81	64	21% reduction
Jet Back Pressure (psi) @ 250 slm	27	20	26% reduction

Table 1 summarizes and quantifies changes to the design, parameters, and ergonomics between the two torches.

COATING QUALITY

The scope of this project was to repackage the torch in a smaller, lighter, and more ergonomic unit to address the aforementioned limitations. Because the parameter-related internal anatomy of the torch was virtually unchanged, the coating quality between the two torches is almost identical. The next stage of development is to optimize the design of parameter-related components such as nozzles, cathodes, anodes, and convergences to produce better quality coatings as per industry demand.

POTENTIAL APPLICATIONS

Plasma spraying is the most versatile thermal spray process with very few limitations on sprayed materials as well as substrate material, size, and shape^[3]. The coating quality, characterized by density, uniformity, and reproducibility, is in general higher than that obtained with flame spraying^[4]. These benefits, along with those brought about by axial injection such as high deposition efficiency and throughput, make axial plasma spraying a valuable investment for producing industrial coatings.

Some applications of plasma sprayed coatings include^[4]:

1. Combustion chambers and turbine blades of airplane engines
2. Cylinder bores, piston rings, valves, conrods, and exhaust parts of automotive engines
3. Shifter forks, gearbox, and clutch discs of transmissions
4. Brake discs of road vehicles
5. Impeller blades of mixers or pumps
6. Rollers in the paper and printing industry

7. Spools and threaded guides in the textile industry
8. Hip, knee, and dental implants in the medical industry
9. Sputtering targets and other components in the semiconductor industry

The Axial III Plus supplements these applications with its compact, light, and user-friendly design. Its compactness enables the spraying of internal bores as small as 10-in. in diameter (assuming a 3-in. stand-off distance). The torch is also easier and safer to use with complex part geometries and coating around tight radii. This may unlock more opportunities and further expand the list of applications. Moreover, its low mass, minimized number of parts, and optimized features simplify assembly, disassembly, handling, and maintenance. The time and effort saved here can add up and be of significant value especially in a production process when frequent replacing and maintenance of components such as nozzles, cathodes, anodes, and convergences is required.

CONCLUSION

Axial injection has several proven advantages over radial injection and thus appears to be the future of feedstock injection technology. The plasma spraying process in general has enabled technology in a wide range of industries, but to tap into the fullest potential of applications, continual innovation in the torch is necessary. The Axial III Plus is one step forward in this process. ~iTSSe

For more information: Ash Kamble, product development engineer, EIT, Northwest Mettech Corp., 19335 96th Ave., Vancouver, BC, Canada, +1 778-891-7407, ash.kamble@mettech.com, www.mettech.com.

References

1. Northwest Mettech Corp. Axial III Core Technology. (n.d.). <https://mettech.com/coating-technology/axial-III-core-technology.php>.
2. A. Ganvir, "Microstructure and Thermal Conductivity of Liquid Feedstock Plasma Sprayed Thermal Barrier Coatings," *Licentiate Thesis Production Technology*, University West, No. 9, p 19-20., 2016.
3. F. Miranda, et al., *Atmospheric Plasma Spray Processes: From Micro to Nanostructures*, 2018, doi: 10.5772/intechopen.80315.
4. P.L. Fauchais, et al., *Thermal Spray Fundamentals: From Powder to Part*, New York, 2014, doi: 10.1007/978-0-387-68991-3.



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 December 2020 and January 2021 issues are highlighted here. The January issue is a special issue on Selected and Expanded Papers Based on Abstracts Submitted for the 2020 International Thermal Spray Conference (ITSC 2020) (Event canceled due to the COVID-19 pandemic). The last three articles highlighted below are from this special issue. In addition to the print publication, *JTST* is available online through springerlink.com. For more information, visit asminternational.org/tss.

PARAMETER DEVELOPMENT VIA IN SITU RESIDUAL STRESS MEASUREMENT AND POST-DEPOSITION ANALYSIS OF COLD SPRAY CuNi COATINGS

Robert F. Brown, Gregory M. Smith, John Potter, and Timothy J. Eden

Developing dense, well-adhered coatings with minimal heterogeneity via cold spray deposition has been a challenge because in-flight and layer-by-layer diagnostics has not yet been adequately adapted to complement existing post-deposition analyses and parameter process mapping. This study presents an empirical and analytical approach for development and optimization of requisite spray parameters via the cold spray solid-state consolidation process using the in situ beam curvature technique to assess particle dynamics during deposition, in conjunction with post-deposition analyses. Here, CuNi (62/38) powder was deposited onto CuNi (70/30) substrates and monitored with an in situ beam curvature sensor to provide real-time feedback of the coating's evolving stress states during the deposition process and subsequent residual stress determination immediately after deposition. (Fig. 1)

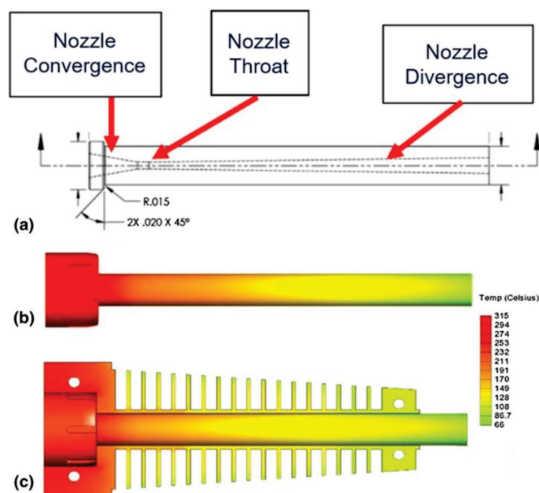


Fig. 1 — (a) Nozzle section view and temperature distribution profile (b) without and (c) with heat exchanger.

THERMAL SPRAYED PHOTOCATALYTIC COATINGS FOR BIOCIDAL APPLICATIONS: A REVIEW

Yi Liu, Jing Huang, Xiaohua Feng, and Hua Li

There have been ever-growing demands for disinfection of water and air in recent years. Efficient, eco-friendly, and cost-effective methods of disinfection for pathogens are vital to the health of human beings. The photocatalysis route has attracted worldwide attention due to its highly efficient oxidative capabilities and sustainable recycling, which can be used to realize the disinfection purposes without secondary pollution. In this review, various photocatalytic materials and corresponding inactivation mechanisms for virus, bacteria, and fungus are briefly introduced. The thermal-sprayed photocatalysts and their antimicrobial performances are summarized. Finally, the future perspectives of the photocatalytic disinfection coatings for potential applications are discussed. (Fig. 2)

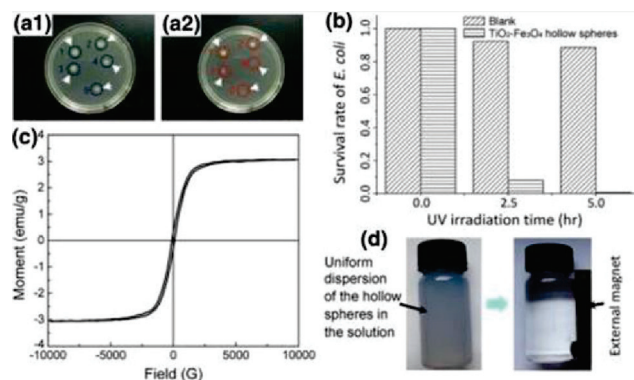


Fig. 2 — Recyclable iron oxides-containing photocatalysts for bacterial inactivation.

PERSPECTIVES ON ENVIRONMENTAL BARRIER COATINGS MANUFACTURED VIA AIR PLASMA SPRAY ON CERAMIC MATRIX COMPOSITES: A TUTORIAL PAPER

Kang N. Lee, Dongming Zhu, and Rogerio S. Lima

There are many sets of information in the literature (e.g., papers, books, and websites) about the great achievements

that are expected for aerospace gas turbine engines by the employment of ceramic matrix composites (CMCs) and thermally sprayed environmental barrier coatings (EBCs) in their hot zones (e.g., combustion chambers, vanes, shrouds, blades, and afterburners). Among these achievements, those typically highlighted are: (i) turbine weight reduction, (ii) reduced fuel consumption, (iii) higher operation temperatures, (iv) superior thrust-to-weight ratio, and (v) lower emission of toxic gases to the atmosphere. The objective of this tutorial paper is to present to the reader how these feats are achieved by the concomitant combination of imaginative engineering. It will explain the non-stop driving force for increasing combustion temperatures; show the basic concepts of CMCs, the paramount need of EBCs, and the complexity of creating EBC architectures via air plasma spray (APS). Finally, highlights on how EBCs/CMCs are tested at high temperature will be provided. (Fig. 3)

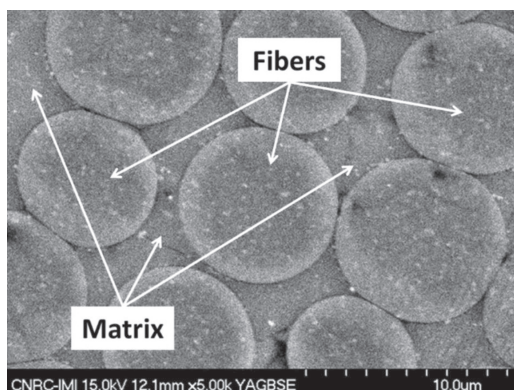


Fig. 3 — SEM picture of a polished cross-section of a CMC.

HIGH RATE DEPOSITION IN COLD SPRAY

Ozan C. Ozdemir, Patricia Schwartz, Sinan Muftu, Forest C. Thompson, Grant A. Crawford, Aaron T. Nardi, Victor K. Champagne, Jr., and Christian A. Widener

Industrialization of cold spray introduces concerns regarding the cost and time efficiency of cold spray procedures.

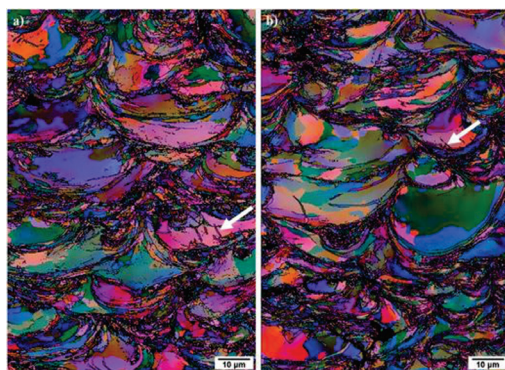


Fig. 4 — EBSD Euler orientation maps for coatings deposited at (a) 5.0%, (b) 8.4% particle loading rates. Black pixels indicate unindexed locations or high angle grain boundaries (>10°). Arrows identify representative lenticular grains.

In this work, high rate deposition of tantalum was studied computationally and experimentally. Quasi-1D multiphase fluid simulations predicted minimal effects on the bonding conditions of particles with 5% to 14% increase in powder-to-gas mass flow ratio. Experimental specimens were produced to observe the mechanical and microstructural effects of increased powder stream loading. Adhesion and hardness tests as well as thermal conductivity, optical microscopy, and electron backscatter diffraction examinations only exhibited minor differences in the mechanical and microstructural properties of the specimens. (Fig. 4)

BONDING MECHANISMS IN COLD SPRAY: INFLUENCE OF SURFACE OXIDATION DURING POWDER STORAGE

Maryam Razavipour, Saeed Rahmati, Alejandro Zúñiga, Denise Criado, and Bertrand Jodoin

Cold spray is a solid-state process in which solid particles are subjected to severe plastic deformation to form a coating. The effect of naturally occurring oxides on bonding in the cold spray was investigated in this work. Deposition characteristics of copper powder with different surface oxide thicknesses on steel substrate were examined using a local pull-off test. This enables the investigation of individual particle/substrate interfaces. X-ray photoelectron spectroscopy was used to study thoroughly the powder surface chemistry and the oxide thickness as a function of exposure time. (Fig. 5)

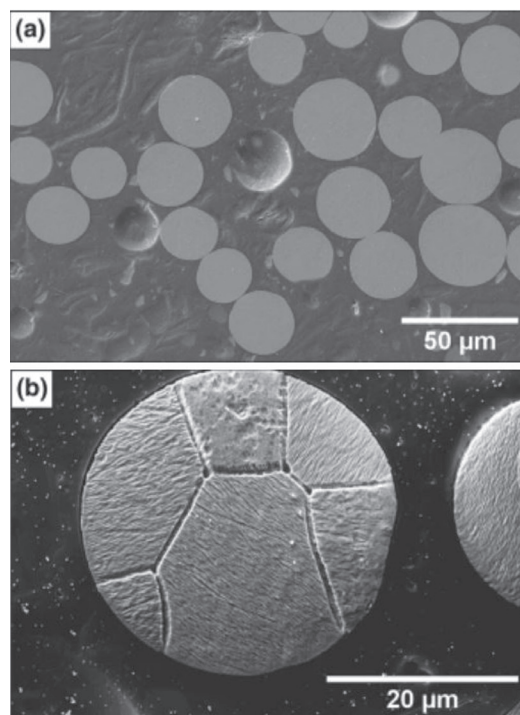


Fig. 5 — Cross-section SEM images of as-received copper powder (a) before etching and (b) after etching.

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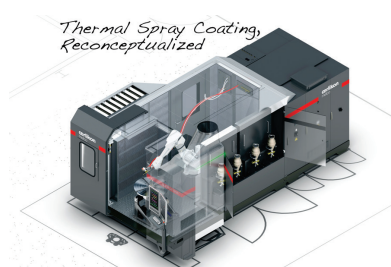
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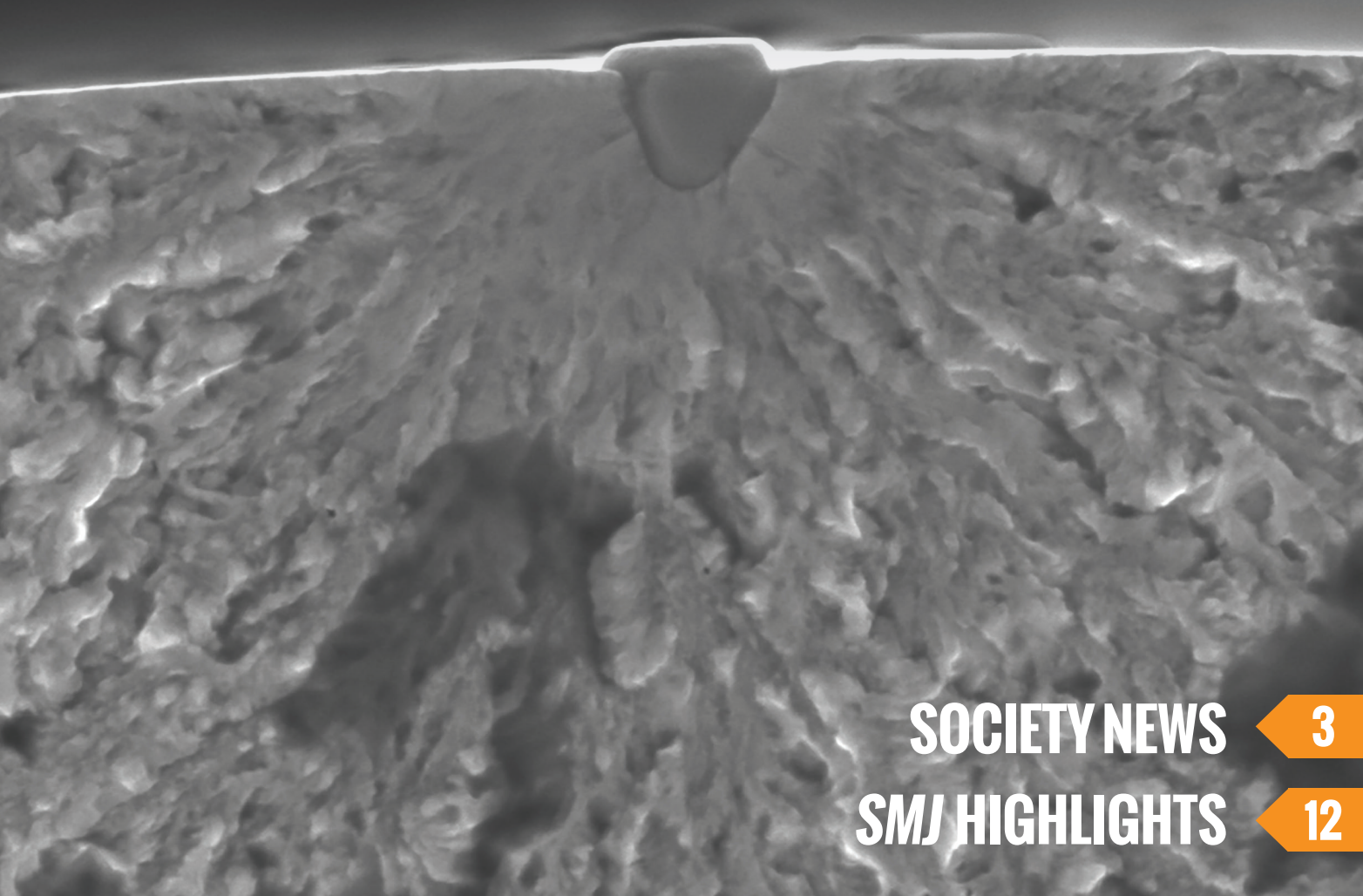
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SMST NEWSWIRE

THE OFFICIAL NEWSLETTER OF THE INTERNATIONAL ORGANIZATION ON
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FDA GUIDANCE ON NITINOL DEVICES



SOCIETY NEWS

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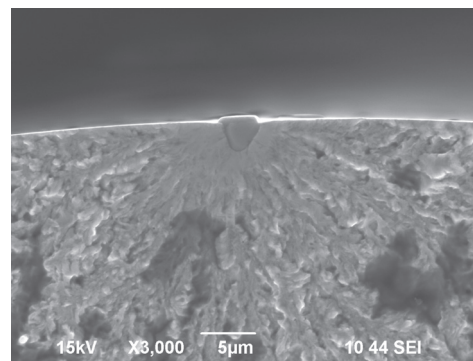
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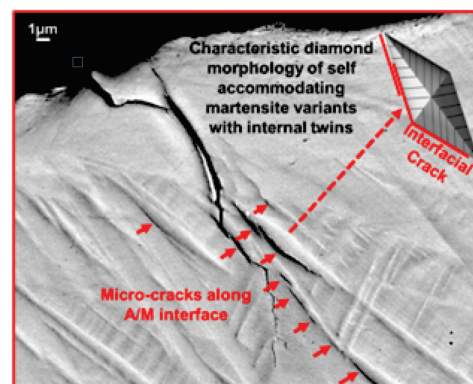
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FDA GUIDANCE ON MEDICAL DEVICES CONTAINING NITINOL



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DIRECT ELECTRICAL ACTIVATION OF THE SHAPE MEMORY EFFECT



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TECHNOLOGIES NEWS

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ABOUT THE COVER

A triangular-shaped inclusion is present at the fatigue crack initiation site for a Nitinol specimen subjected to cyclic fatigue loading. Courtesy of the FDA.

EDITORIAL OPPORTUNITIES FOR SMST NEWSWIRE IN 2021

The editorial focus for the SMST Newswire in 2021 reflects shape memory and superelastic technologies for biomedical, actuator applications, and emerging markets.

April | October

To contribute an article, contact Joanne Miller at joanne.miller@asminternational.org.

To advertise, contact Kelly Johanns at kelly.johanns@asminternational.org.

COVID'S IMPACT ON SMST AND THE MEDICAL DEVICE COMMUNITY

As some readers may be aware, there's a virus going around. It's doubtful that there are any industries, professional organizations, or scientific meetings unaffected by COVID-19. Nitinol production and research remains dominated by medical device procedures such as heart valve repair and replacement, stenting, aneurism repair, and the like. These procedures are generally critical to life, so our industry has been impacted less than most. Still, Nitinol is also extensively used in diagnostic and elective procedures, which are being delayed both by patients and caregivers. A very informal poll of Nitinol medical device CEOs indicates a 10-20% decline in 2020 revenue versus pre-pandemic levels, both in Europe and the United States. At the same time, there is confidence that things will return to normal, and unfortunately, perhaps even see a "bounce" because many patients will undoubtedly return with more advanced disease states.

While the impact to revenues is small compared to many other industries, the effect on professional and scientific activities has been more pronounced. In-person lab activities have been severely cut back at most industrial and academic institutions, as have many live teaching events. The ICOMAT conference, which in many ways is the precursor to the SMST conferences, has been postponed to mid-March of 2022 (<https://www.icomat2020.org/>). The joint SMST-AeroMat event originally scheduled for May 2020 was



Duerig

cancelled entirely, and the SMST meeting originally scheduled for May 2021 has now been postponed to May of 2022, still to be held in San Diego (<https://www.asminternational.org/web/srst-2022>).

Despite that, the SMST board has decided to proceed with the \$50,000 Founders' Grant as originally planned. The Grant Selection Committee received fifteen submissions this year and after a great deal of debate decided to award the grant to William LePage of the University of Tulsa for his proposed work on elucidating the role of inclusions on fatigue crack nucleation, certainly a topic of great interest to the medical community. Perhaps indicative of where our industry focus lies, six of the submissions focus on high temperature Nitinol (Hf,Zr) alloys, and five of those pertain to additive manufacturing.

And speaking of fatigue, the CardioVascular Implant Durability (CVID) conference intends to press forward with its October 11-13, 2021 dates (<https://cvidconference.org/>), but will likely offer virtual as well as in-person registration options. The founders of the CVID conference wish to point out that its highly successful debut conference (CVID-2019) literally went viral (at least in name). We hope to duplicate the success of the 2019 event and convince search engines that it isn't simply a typo for "COVID."

Tom Duerig, FASM

Board Member and Co-founder, International Organization on Shape Memory and Superelastic Technologies
Organizing Committee Member, SMST 2022

2021 SMST FOUNDERS' GRANT

Congratulations to the 2021 SMST Founders' Grant Recipient: Dr. William LePage of the University of Tulsa Department of Mechanical Engineering. Dr. LePage's proposal, "Zooming in on Inclusions and their Role in the Fatigue of NiTi Devices," will receive the grant of \$50,000 over two years. The Grant Selection Committee looks forward to results of this study to increase the life-span and predictability of failure for NiTi devices through targeted, micromechanical experiments that study inclusions and voids.



LePage

While the SMST Founders' Grant is usually presented at the SMST conference, the recipient will be asked to provide a mid-grant report at the conference rescheduled for 2022. The next grant will then be given at the following SMST conference in 2024.

The SMST Founders' Grant provides funding for early, exploratory research related to shape memory and superelasticity. The funds are expected to be used as a "seed grant" to test a concept and lay a foundation for obtaining further funding from industry or government agencies.

The SMST Founders' Grant was generously endowed in 2019 by Dr. Tom Duerig, FASM, for which the community is extremely grateful, allowing the Grant to be offered in perpetuity.

CASMAST 4TH STUDENT DESIGN CHALLENGE

CASMAST STUDENT DESIGN CHALLENGES are held biennially as part of the SMST conference where students are tasked with designing "the material" or designing "with the material" using shape memory alloy (SMA) technology. Students have the opportunity to showcase their creativity by applying engineering theories and design principles, and leveraging CASMAST and SMST members' experience to address SMA design challenges in aeronautics, astronautics, medical industry, energy, and else. This year, given SMST 2021 was postponed, the 4th CASMAST Student Design Challenge will be hosted virtually and will take place May 17-20. To learn more and see the final presentations in May, visit <https://www.asminternational.org/web/sms-2022>.

Teams include:

1. Team "**Return of the MAC**" (Texas A&M University): John Broucek, James "Trey" Royalty (Advisors: William Trehern, Dr. Ibrahim Karaman)
2. Team "**SMAlloygator**" (Iowa State University): Madison Harrington, Elizabeth Krotz, Alana Pauls, Edward Wagner, and Jonathan Zaugg (Advisors: Dr. Jun Cui, Dr. Shraddha Vachhani)

3. Team "**Heavy Metal**" (University of North Texas): J. Eli McCool, Neha S. John, Jessica Rider, and Jordyn M. Ward (Advisor: Dr. Marcus Young)
4. Team "**SmartGrip**" (Universität des Saarlandes): Dominik Scholtes, Carmelo Pirritano, and Andre Schieler (Advisors: Dr. Paul Motzki, Dr. Stefan Seelecke)
5. Team "**SMAUG**" (Texas A&M University): Sharon Pearl-nath, Jacob Spurgers, Brandon Tong (Advisors: Nathan Hite, Dr. Ibrahim Karaman)

SEEKING APPLICATIONS FOR SMST STUDENT BOARD MEMBERS

The International Organization on Shape Memory and Superelastic Technologies (SMST) is seeking applicants for its student board member position. Students must be a registered undergraduate or graduate during the 2021-2022 academic year and must be studying or involved in research in an area closely related to shape memory and superelastic technologies. **Nominations are due by April 30.**

For information, visit smst.asminternational.org or contact Mary Anne Jerson at maryanne.jerson@asminternational.org.

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FDA GUIDANCE ON MEDICAL DEVICES CONTAINING NITINOL

New FDA guidance for Nitinol used in medical devices includes clarification on the use of computational modeling, as well as corrosion and biocompatibility updates.

*Matthew Di Prima, David Saylor, Shiril Sivan, and Jason D. Weaver
U.S. Food and Drug Administration, Center for Devices and Radiological Health,
Office of Science and Engineering Laboratories, Silver Spring, Maryland*

On October 15, 2020, the U.S. Food and Drug Administration (FDA) released the guidance “Technical Considerations for Non-Clinical Assessment of Medical Devices Containing Nitinol^[1].” While the draft of the guidance was released in April of 2019, the roots of the guidance can be traced back to the 2012 Workshop “Cardiovascular Metallic Implants: Corrosion, Surface Characterization, and Nickel Leaching.” Since that workshop, Nitinol specific considerations were addressed in the 2015 guidance “Select Updates for Non-Clinical Engineering Tests and Recommended Labeling for Intravascular Stents and Associated Delivery Systems” and some gaps in regulatory science were addressed through numerous research papers from the Center for Devices and Radiological Health (CDRH). With the increased adoption of Nitinol in non-cardiovascular applications, it became necessary to provide the agency’s thinking on the technical considerations for Nitinol in a broad, non-product specific document. This culminated in the guidance “Technical Considerations for Non-Clinical Assessment of Medical Devices Containing Nitinol,” which contains pre-market submission recommendations covering general Nitinol information, mechanical testing, corrosion testing, biocompatibility, and labeling considerations.

CHANGES FROM DRAFT GUIDANCE

While the public comment period for the draft guidance was open from April to June 2019, per CFR 10.115(g) (5), FDA guidance dockets remain open indefinitely for public feedback (<https://www.regulations.gov/document?D=FDA-2019-D-1261-0002>). During the open comment period, FDA received 93 comments from 11 commenters who spanned industry, test labs, and private individuals. While a majority of the comments were editorial in nature, multiple commenters asked about inclusion of the R-phase, clarification of what was intended by “established surface finishes” for Nitinol, and clarification on using computational modeling to predict Nitinol fatigue behavior. While these issues were addressed in the final version of the guidance, no major changes in the document were needed to address the comments, which implied that there was general agreement

with the content of the guidance. This was expected, as the content was either in line with or updated from the 2015 guidance; therefore, industry and test houses had several years to understand FDA’s perspective on Nitinol testing.

GENERAL INFORMATION

The performance and behavior of Nitinol depends on multiple factors spanning material chemistry to heat treatment parameters. To ensure the correct considerations are applied for performance testing, the guidance calls for the material composition, manufacturing processes, expected Nitinol behavior (shape memory vs. pseudoelastic), and transformation temperatures to be provided. While it is anticipated that most Nitinol will conform to a recognized standard (e.g., ASTM F2063), the guidance asks for the material composition and a description of specific properties in cases where it does not. For manufacturing, the guidance asks for a high-level flowchart of the Nitinol manufacturing process from raw material to final device sterilization (if relevant), with special attention given to thermal and surface processes. As transformation temperatures are critical to the behavior and mechanical performance of Nitinol, the guidance asks for key transformation temperatures to be provided based on whether the Nitinol is intended for a shape memory or pseudoelastic application.

MECHANICAL TESTING AND COMPUTATIONAL MODELING

While mechanical testing on devices made from conventional metals like stainless steel is relatively common and well described in other standards and FDA documents, the unique thermomechanical response of Nitinol necessitates additional considerations to ensure accurate and consistent results. Among these considerations is the test environment, which is typically liquid to represent a physiologically relevant environment and to dissipate any heating effects caused by phase transformation^[2]. Given how drastically the stress-strain behavior can vary with temperature^[3], in order to ensure clinical relevance as well as consistency in results, the environment should be maintained at a physiologic

temperature. Computational modeling of medical devices containing Nitinol—especially of fatigue loading, such as that shown in Fig. 1—can be complex and requires the use of a Nitinol-specific constitutive law. Furthermore, given the sensitivity of Nitinol to thermal processing and surface treatments, the guidance recommends that experiments to determine constitutive law parameters or constant life curves (i.e., for fatigue safety factor calculations) be conducted on test specimens representative of the final manufactured device. Given the unique sensitivity of Nitinol to pre-strain (e.g., as occurs when crimping a stent onto a delivery catheter), which can increase or decrease a Nitinol device's fatigue performance^[4,5], the guidance also highlights the need to take pre-strain into account when estimating fatigue safety factors. Finally, although also relevant for conventional materials, the guidance emphasizes the importance of the computational methodology used to calculate mean and alternating strain^[6] as well as validation of the computational model. As the use of computational modeling in medical devices increases, it will become increasingly important to document adequate credibility of computational models for their context of use^[7,8].

CORROSION

Passive device alloys, such as Nitinol, are potentially susceptible to corrosion through a number of different mechanisms: pitting, crevice, uniform, galvanic, and fretting corrosion. The corrosion properties of a Nitinol device are directly influenced by its manufacturing processes. For example, thermal treatments used to create desired thermo-mechanical properties tend to grow the native TiO₂ surface layer. The result can include the formation of nickel-rich phases and microstructural defects within the surface oxide that may significantly degrade the resistance of the material to one or more corrosion mechanisms. Removal of this

thermally grown oxide through surface processing steps such as chemical etching, mechanical polishing, or electropolishing, followed by passivation, tends to result in a thin (< 10 nm) and uniform oxide layer that provides imparts corrosion resistance to the alloy. Based on these observations, the guidance recommends different corrosion evaluations depending on the processing history of the Nitinol device components and device design. These evaluations are captured by the flow chart shown in Fig. 2, which illustrates that all prolonged and permanent contacting devices that contain Nitinol should be assessed using potentiodynamic polarization testing per ASTM F2129. If the results pass a predefined acceptance criterion and an established surface processing method is used to remove the thermal oxide that may develop during heat treatments, no further assessments are necessary. However, if either of these criteria are not met, the guidance recommends conducting an extended in vitro assessment to evaluate nickel leaching kinetics using ASTM F3306. Because device design considerations may also enhance corrosion and metal ion release, galvanic corrosion testing per ASTM F3044 is recommended in scenarios where the Nitinol component(s) is in contact with dissimilar metals. Finally, if fretting corrosion is a concern (e.g., overlapping devices), microscopic evaluations can be conducted following accelerated durability testing or as described in device-specific standards and/or guidance documents.

BIOCOMPATIBILITY AND LABELING

The potential for Nitinol to release nickel ions means there may be biocompatibility considerations in addition to those found in FDA guidance “Use of International Standard ISO 10993-1 Biological Evaluation of Medical Devices—Part 1: Evaluation and Testing within a Risk Management Process.” Specifically, there is a need for a risk assessment to be performed comparing in vitro nickel release to tolerable intake (TI) values when nickel ion release testing is performed. While the guidance recommends a systemic non-oral TI of nickel of

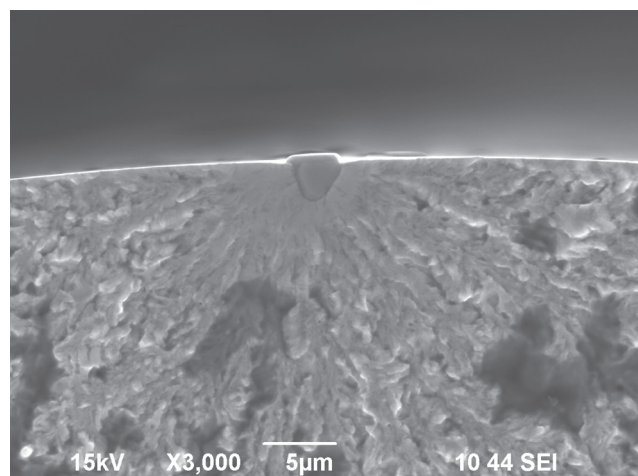


Fig. 1 — A triangular-shaped inclusion is present at the fatigue crack initiation site for a Nitinol specimen subjected to cyclic fatigue loading.

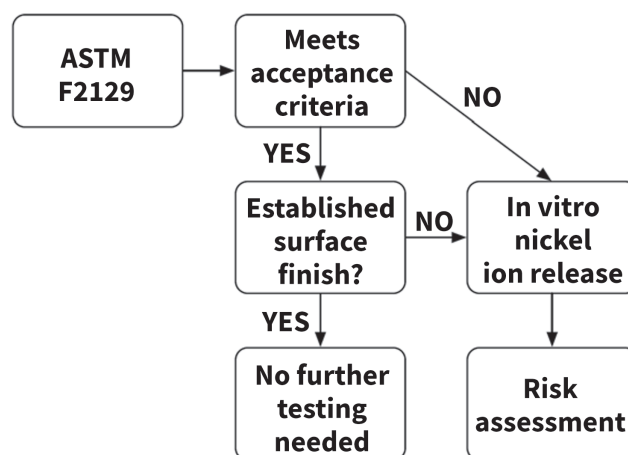


Fig. 2 — Corrosion testing paradigm flow chart.

0.5 µg/kg/day, it is important to stress that this value is not protective against local effects of nickel exposure nor does it protect against nickel hypersensitivity. As there is no known lower limit of nickel exposure for eliciting an adverse reaction, the guidance recommends a warning label be included to counsel patients on the materials contained in the device, as well as potential for allergy/hypersensitivity to these materials. This recommendation aligns with feedback on patient communication received from the Nov. 13-14, 2019 Immunology Devices Panel of the Medical Devices Advisory Committee meeting^[9].

RESEARCH SUMMARY

The regulatory science-based research at the FDA's Office of Science and Engineering Laboratories (OSEL) at the CDRH has focused on addressing key gaps in our understanding of the performance of medical devices containing Nitinol, especially with regards to durability and corrosion (Fig. 3). One of the key unknowns was whether the manufacturing process for Nitinol could influence its corrosion susceptibility and in vivo performance. Corrosion susceptibility assessments were combined with surface characterization to validate the appropriateness of acceptance criteria for corrosion resistance and their clinical relevance^[10]. The impact of pre-strain on fatigue life^[3] and whether the presence of fatigue^[11] or fretting damage could impact the corrosion susceptibility^[12] was also assessed. Least burdensome test methods to predict Ni ion release from Nitinol based medical devices using a shorter timeframe^[13] were also studied along with computational models to predict Ni ion diffusion into the peri-implant tissue to determine local and systemic nickel exposure^[14]. Novel digital image correlation techniques were also developed for characterizing strain at the micro level to better validate Nitinol computational models which may improve future fatigue performance predictions^[15]. The impact of applied potentials on fatigue life was studied and an electrochemical technique to monitor fatigue processes in real time was developed^[16]. The effect of test specimen surface area on the measured pitting corrosion resistance was also explored^[17]. Ongoing work at OSEL focuses on evaluating in vitro to in vivo correlation for Nitinol performance, the impact of reactive oxygen species on corrosion resistance, and how test environment conditions used for bench testing can impact device performance. CDRH is also collaborating with the standards community to evaluate the fatigue to fracture methodology for medical devices containing Nitinol.

To summarize, the research efforts at OSEL strive to develop least burdensome pre-clinical engineering test methods to enable safe and reliable medical devices containing Nitinol to reach the U.S. market quickly.

CLOSING THOUGHTS

Nitinol's unique shape memory and pseudoelastic behavior have allowed for a number of innovations in medical

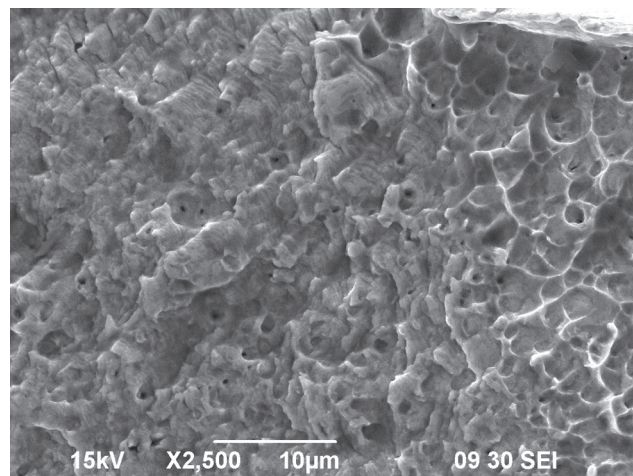


Fig. 3 — Nitinol fatigue fracture surface after 29 million loading cycles. Fatigue striations from crack growth can be seen on the left while the dimpled region on the right is indicative of the final tensile overload.

devices. However, these same properties and behaviors can make the assessment of devices containing Nitinol more involved than when other materials are used. Although the FDA's guidance "Technical Considerations for Non-Clinical Assessment of Medical Devices Containing Nitinol" elucidates critical considerations for Nitinol characterization, additional technical considerations may apply depending on the specifics of the medical device and/or patient population. ~SMST

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References

1. Available at <https://www.fda.gov/regulatory-information/search-fda-guidance-documents/technical-considerations-non-clinical-assessment-medical-devices-containing-nitinol>.
2. Wagner, et al., Structural Fatigue of Pseudoelastic NiTi shape Memory Wires, *Materials Science and Engineering A*, Vol 378, p 105-109, 2004.
3. A.R. Pelton, J. Dicello, and S. Miyazaki, Optimization of Processing and Properties of Medical Grade Nitinol Wire, *Minimally Invasive Therapy & Allied Technologies*, Vol 9, No. 2, p 107-118, 2000.
4. S. Gupta, et al., High Compressive Pre-strains Reduce the Bending Fatigue Life of Nitinol Wire, *Journal of the Mechanical Behavior of Biomedical Materials*, Vol 44, p 96-108, 2015.
5. K. Senthilnathan, et al., Effect of Prestrain on the Fatigue Life of Superelastic Nitinol, *J Materials Engineering and Performance*, Vol 28, Nov. 2, 2019.

FEATURE

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6. R. Marrey, et al., Validating Fatigue Safety Factor Calculation Methods for Cardiovascular Stents, *Journal of Biomechanical Engineering*, Vol 140, No. 6, 2018.

7. ASME V&V 40, Assessing Credibility of Computational Modeling through Verification and Validation: Application to Medical Devices.

8. FDA Guidance "Reporting of Computational Modeling Studies in Medical Device Submissions," available at <https://www.fda.gov/regulatory-information/search-fda-guidance-documents/reporting-computational-modeling-studies-medical-device-submissions>.

9. Available at <https://www.fda.gov/advisory-committees/advisory-committee-calendar/november-13-14-2019-immunology-devices-panel-medical-devices-advisory-committee-meeting-announcement>.

10. S.J. Sullivan, et al., The Effects of Surface Processing on in-vivo Corrosion of Nitinol Stents in a Porcine Model, *Acta Biomaterialia*, Vol 62, p 385-396, 2017.

11. M. Di Prima, E. Gutierrez, and J. Weaver, The Effect of Fatigue on the Corrosion Resistance of Common Medical Alloys, *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, Vol 105, No. 7, p 2019-2026, 2017.

12. D.A. Siddiqui, et al., Effect of Wire Fretting on the Corrosion Resistance of Common Medical Alloys, *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, Vol 105, No. 8, p 2487-2494, 2017.

13. D.M. Saylor, et al., Temperature Dependence of Nickel Ion Release from Nitinol Medical Devices, *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, 2020.

14. D.M. Saylor, et al., Predicting Patient Exposure to Nickel Released from Cardiovascular Devices using Multi-scale Modeling, *Acta Biomaterialia*, Vol 70, p 304-314, 2018.

15. K.I. Aycok, et al., Full-field Microscale Strain Measurements of a Nitinol Medical Device using Digital Image Correlation, *J Mech Behav Biomed Mater*, Feb. 2021; Vol 114, Article 104221, doi: 10.1016/j.jmbbm.2020.104221, Epub Nov. 23, 2020.

16. S. Sivan, M. Di Prima, and J.D. Weaver, Effect of Applied Potential on Fatigue Life of Electropolished Nitinol Wires, *Shape Memory and Superelasticity*, Vol 3, No. 3, p 238-249, 2017.

17. G.M. Sena, et al., Larger Surface Area Can Reduce Nitinol Corrosion Resistance. *npj Materials Degradation*, Vol 4, No. 1, p 1-8, 2020.



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DIRECT ELECTRICAL ACTIVATION OF THE SHAPE MEMORY EFFECT

New research explores a zirconia-based shape memory ceramic that exhibits a unique type of electrically triggered martensitic transformation.

Christopher A. Schuh, FASM, and Alan Lai, Massachusetts Institute of Technology*

Shape memory ceramics (SMCs) exhibit a number of significant advantages over the more common shape memory metals. Zirconia-based SMCs, for example, can achieve much higher transformation stresses (~GPa) and be used at much higher temperatures (> 500°C) than metal-based versions. Recent work exploring SMCs in small volumes^[1] and single crystal forms^[2] has discovered significant capacity for sustained cyclic transformation, avoiding the cracking that characterized earlier work on ceramics undergoing a martensitic transformation^[3]. One particularly unique advantage of SMCs over shape memory metals is that they do not conduct electricity; as dielectrics, SMCs can sustain electric fields. In the authors' recent work, they have shown that such applied electric fields couple to the martensitic transformation^[4], opening the door to a new class of what they term paraelectroactive shape memory ceramics.

MODES OF SHAPE MEMORY ACTUATION

As actuators, shape memory alloys are valued for their very high specific work output. For their size and weight, they can actuate over relatively long stroke distances with high forces. This is shown in the figure of merit map where contours of specific work increase toward the upper right-hand corner (Fig. 1). However, a perennial challenge for shape memory alloys as actuators is that they are activated by heating, either directly with heaters or indirectly via electrical resistance heat. In either case, the kinetics of heat flow limit the speed of two-way actuation.

It is highly desirable to trigger shape memory transformations directly with fields instead of temperature, avoiding the kinetics of heat transfer and permitting actuation on demand through control electronics. The field-driven shape memory effect, through martensite domain flipping, has been demonstrated in magnetic shape memory alloys and ferroelectrics. In these cases, the martensite domains carry an orientation of magnetization or electrical polarization, respectively, and an applied field can bias the formation of domains that are aligned with it^[5,6].

In order to achieve actuation through a martensitic transformation, the austenite and martensite phases must have a difference in some physical property, as highlighted in Table 1 for various different thermodynamic cases. In thermal transformations, this is simply the entropy difference between the phases. If there is a strain/shape difference between the phases, this results in shape memory and superelastic properties. In each case, the work input favors transformation to the phase more responsive to it. For magnetic shape memory and ferroelectric materials, the applied field couples with the inherent magnetic or electric axes, i.e., the magnetization or the polarization, respectively, and favors domains that are aligned.

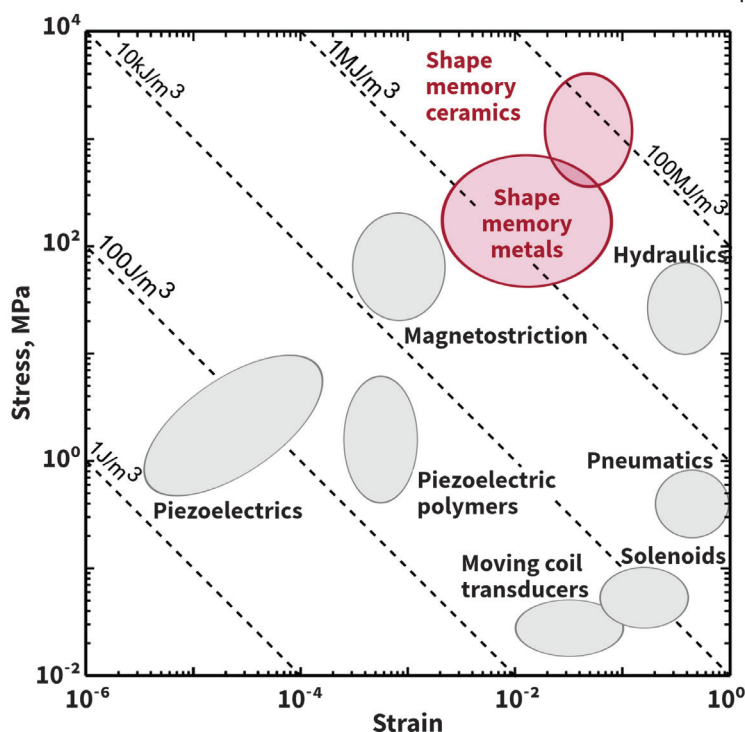


Fig. 1 — An Ashby map compares actuation technologies on the basis of stress and strain, with the product of those quantities leading to the figure of merit (specific work) captured by the dashed lines. Shape memory materials are solid state options with the highest combination of stress and strain^[1].

*Member of ASM International

ELECTRICALLY DRIVEN TRANSFORMATION IN ZIRCONIA

In this landscape, shape memory ceramics such as zirconia offer a unique set of properties.

TABLE 1 – CATEGORIZATION OF SHAPE MEMORY EFFECTS

Work input	Property difference between phases	Transformation type
Temperature change	Entropy, ΔS	All phase transformations
Mechanical stress	Strain, $\Delta \epsilon$	Shape memory and superelasticity
Magnetic field	Magnetization, ΔM	Magnetic shape memory
Electric field	Polarization, ΔP	Ferroelectric shape memory
	Electric susceptibility $\frac{dP}{dE} = \chi_e$	Paraelectric shape memory

Categorization of shape memory effects on the basis of the applied triggering effect (work input) and property mismatch it works upon. In addition to well-known intrinsic property mismatches ΔS , $\Delta \epsilon$, ΔM , and ΔP , it is possible to induce significant mismatches and cause phase transformations by virtue of a second-order (field-dependent) property such as electrical susceptibility.

Like shape memory metals, SMCs exhibit a reversible martensitic transformation that involves significant strains, matched with a very high intrinsic strength as shown in Fig. 1. What is more, SMCs are dielectric, so the two phases— austenite and martensite—can exhibit mismatches in electrical properties of the kind shown in Table 1.

In the authors' recent work^[4] they studied the martensitic transformation of single crystal zirconia doped with 2 mol% yttria, between its low-temperature monoclinic phase (martensite) and its high-temperature tetragonal phase (austenite). Figure 2 shows hysteresis curves for this transformation, measured using in situ single-crystal x-ray diffraction during a heating and cooling cycle. As expected, upon heating the monoclinic phase reflections decline and the tetragonal phase reflections emerge, with the transformation being complete at about 600°C. This is a thermal martensite-to-austenite transformation enabled by an entropy difference; it is also accompanied by the shape (strain) mismatch between the phases as noted in Table 1. It is a reversible transformation as well, and can be realized thermally upon cooling, albeit at lower temperatures as shown in Fig. 2.

Also shown in Fig. 2 is a new result for shape memory zirconia, namely, that the same martensite-to-austenite transformation can be driven directly with an electric field. The same single crystal specimen, in the form of a ~125 μm thick plate, was outfitted with electrodes and subjected to short ~1 s bursts of applied electric field. As shown by the yellow data series, voltage causes the transformation to occur at 550°C, below where temperature alone would

trigger it. This result was also shown to be reversible (cooling reverted to martensite) and reproducible, and unlike shape memory alloys, did not involve significant current flow or joule heating. Rather, this is a demonstration of direct electrically triggered martensitic transformation, and in this specific crystal orientation gives rise to a ~1% linear strain.

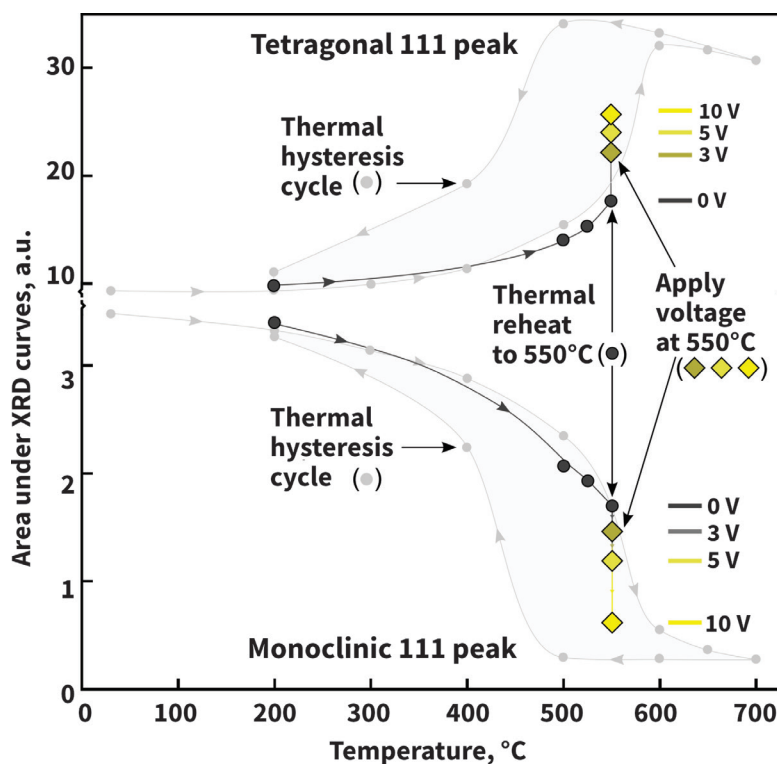


Fig. 2 — Experimental results on the tetragonal-monoclinic shape memory transformation in yttria-doped zirconia, measured by tracking single-crystal diffraction 111 peaks in situ during transformation. Gray hysteresis loops show the thermally triggered transformation during a full cycle. The data series in black and yellow show an experiment in which an applied electric field was used to directly trigger the monoclinic-to-tetragonal transformation. Reproduced from Lai^[4].

PARAELECTRIC SHAPE MEMORY

What is perhaps most unexpected about the electrical activation result in Fig. 2 is the fact that although zirconia is dielectric, it is not ferroelectric. In Table 1, the ferroelectrics occupy a special position due to their inherent electrical polarization; their crystal structures have built-in asymmetries that allow them to carry a permanent or spontaneous polarization and when a field is applied, it interacts directly with the polarization field inherent to the material. In contrast, zirconia is paraelectric with no spontaneous polarization to drive the transformation, because both its austenitic tetragonal and martensitic monoclinic phases are centrosymmetric and have no such intrinsic polarization^[7].

The authors suggest that in zirconia, the transformation seen in Fig. 2 is driven by a second-order property—electrical susceptibility—which is dependent on the field itself. When a field is applied, the structure of zirconia can distort slightly and develop a polarization; this induced polarization increases as the field does. Because the two phases involved have different susceptibilities, they can develop a significant polarization difference between them, which can drive the transformation to the more polarizable phase, in this case to tetragonal austenite. The contrast between this mode of electrical activation and that widely known in ferroelectrics is important, because it could foreshadow a new class of electroactive shape memory materials outside of the ferroelectrics family. These results encourage a search for other

materials that, like zirconia, exhibit a martensitic transformation between paraelectric phases, but also have enough of an electrical susceptibility mismatch to drive the transformation. Such paraelectroactive shape memory materials could present an interesting new frontier for actuation technology.

This susceptibility-mismatch shape memory transformation could foreshadow other couplings among combinations of thermal, mechanical, magnetic, or electrical properties. Table 2 lists a number of such second-order properties, many of which are named and tabulated, but not yet recognized as driving phase transformations by themselves. Some are other simple susceptibilities like elastic compliance or magnetic susceptibility that only involve a single work type (the diagonal terms), while others are properties that link multiple work inputs (off-diagonal terms) such as piezoelectric and magnetoelectric constants. An interesting direction for future research is to identify materials that have no spontaneous difference in a first-order property (entropy, strain, magnetization, polarization), but which can develop a large phase difference by virtue of these second-order properties, triggering the martensitic transformation and shape memory effects. As indicated by the number of material properties listed in Table 2, the potential material search space could be very large.

THERMODYNAMIC IMPLICATIONS

One intriguing aspect of this susceptibility-mismatch

TABLE 2 – PROPERTY MISMATCH AND SHAPE-MEMORY TRANSFORMATIONS

Property mismatch	Potential susceptibility mismatch			
	Temperature dependent property	Stress dependent property	Magnetic field dependent property	Electric field dependent property
Entropy ΔS	Heat capacity $\frac{dS}{dT} = c_p$	$\frac{dS}{d\sigma}$	$\frac{dS}{dH}$	$\frac{dS}{dE}$
Strain $\Delta \varepsilon$	Thermal expansion $\frac{d\varepsilon}{dT} = \alpha$	Elastic compliance $\frac{d\varepsilon}{d\sigma} = S_{el}$	Indirect piezomagnetic $\frac{d\varepsilon}{dH} = h_m$	Indirect piezoelectric $\frac{d\varepsilon}{dE} = h_e$
Magnetization ΔM	Pyromagnetic $\frac{dM}{dT} = p_m$	Direct piezomagnetic $\frac{dM}{d\sigma} = d_m$	Magnetic susceptibility $\frac{dM}{dH} = \chi_m$	Magnetoelectric susceptibility $\frac{dM}{dE}$
Electrical polarization ΔP	Pyroelectric $\frac{dP}{dT} = p_e$	Direct piezoelectric $\frac{dP}{d\sigma} = d_e$	Electromagneto susceptibility $\frac{dP}{dH}$	Electric susceptibility $\frac{dP}{dE} = \chi_e$

For each type of property mismatch that can trigger a shape-memory transformation, there are second-order properties, or susceptibilities, which can also trigger the transformation. These susceptibilities imply that upon application of a driving force (temperature, stress, magnetic field, or electric field), the phase properties change; if this change leads to a large mismatch between austenite and martensite phases, the transformation will be favored. The results in Fig. 2 speak to the electrical susceptibility mismatch transformation in the lower-right corner of this table.

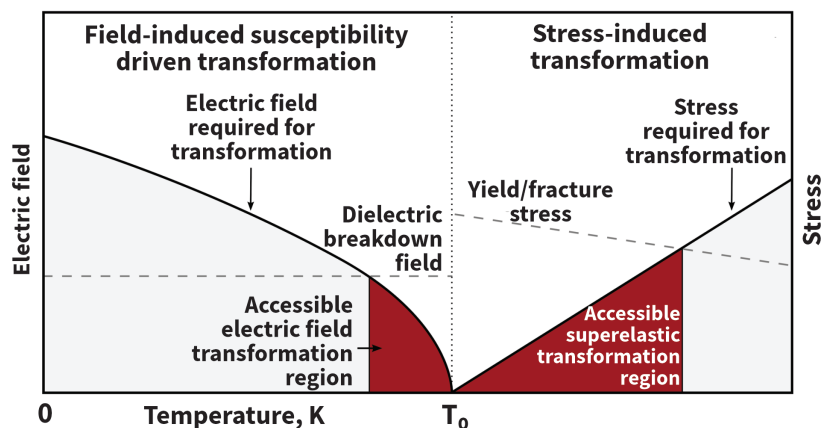


Fig. 3 — A schematic phase diagram for both thermally and electrically triggered martensitic transformation in zirconia. Under zero stress and field, transformation occurs at T_0 , the equilibrium temperature. As stress is applied (right side), the phase boundary rises linearly following the Clausius-Clapeyron equation, and the transformation can be triggered by stress in the maroon-shaded region, up until the yield or fracture occur instead. However, when an electric field is applied (left side), the phase boundary is not linear but parabolic, creating a curved region where a field-driven transformation can occur, provided the field is below the point of dielectric breakdown.

shape memory effect is the nature of the phase boundary. In the shape memory field, researchers are accustomed to a linear phase boundary given by the Clausius-Clapeyron equation. For example, in classical (thermomechanical) shape memory, temperature and stress trade off linearly for one another, as shown for zirconia on the right-hand side of Fig. 3. In the case of a susceptibility mismatch, however, the second-order nature of the property plays out literally as a higher order correction to the Clausius-Clapeyron equation: It provides a parabolic dependence. The left-hand side of Fig. 3 illustrates the effect in the case of shape memory zirconia, where there is no linear term (no spontaneous polarization), and only the parabolic term (from susceptibility mismatch) separates the two phases. From the equilibrium transformation temperature, the phase boundary increases with a square-root dependence on field.

There are many practical implications of this second-order thermodynamic effect for actuation. For example, as Fig. 3 shows for zirconia, electric field can be viewed as a third axis for control, with an opposite dependence from stress and a negative Clausius-Clapeyron slope; stresses promote martensite, but fields promote austenite. What is more, the slowly changing temperature dependence of the parabolic phase boundary suggests that there may be large design windows where the transformation field is less affected by changes in temperature, compared with a typically tight temperature window for stress-based activation. And whereas the mechanical yield or fracture stress provides an upper bound on the stresses achieved in shape memory alloys, the dielectric breakdown provides an analog for the upper limits on field, as shown in Fig. 3.

CONCLUSION

In paraelectroactive shape memory ceramics, the intersection and collaboration among stresses, field, and temperature require more detailed elaboration both theoretically and practically. At the same time, development of new materials in this category is likely to unveil new complexities and competitions. But the opportunity to directly trigger shape memory strains with applied electric fields remains a tantalizing prospect for faster, more addressable, and distributable actuation. The extension of field-activated shape memory to paraelectric ceramics may provide new paths toward that goal. ~SMST

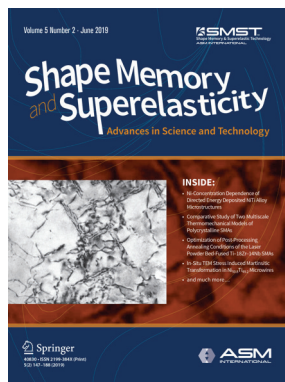
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References

1. A. Lai, et al., Shape Memory and Superelastic Ceramics at Small Scales, *Science*, Vol 341, p 1505-1508, 2013.
2. I. Crystal, A. Lai, and C. Schuh, Cyclic Martensitic Transformations and Damage Evolution in Shape Memory Zirconia: Single Crystals vs. Polycrystals, *J. Am. Ceram. Soc.*, Vol 103, p 4678-4690, 2020.
3. P. Reyes-Morel, J.-S. Cherng, and I.-W. Chen, Transformation Plasticity of CeO₂-Stabilized Tetragonal Zirconia Polycrystals: II, Pseudoelasticity and Shape Memory Effect, *J. Am. Ceram. Soc.*, Vol 71, p 648-657, 1988.
4. A. Lai and C. Schuh, Direct Electric-Field Induced Phase Transformation in Paraelectric Zirconia via Electrical Susceptibility Mismatch, *Phys. Rev. Lett.*, Vol 126, Issue 1, 2021.
5. K. Otsuka and C.M. Wayman, Shape Memory Materials, Cambridge University Press, 1998.
6. D. Damjanovic, Ferroelectric, Dielectric and Piezoelectric Properties of Ferroelectric Thin Films and Ceramics, *Reports Prog. Phys.*, Vol 61, p 1267, 1998.
7. R. Hannink, P. Kelly, and B. Muddle, Transformation Toughening in Zirconia-Containing Ceramics, *J. Am. Ceram. Soc.*, Vol 83, p 461-487, 2000.



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FATIGUE CRACK INITIATION IN THE IRON-BASED SHAPE MEMORY ALLOY FeMnAlNiTi

R. Sidharth, W. Abuzaid, M. Vollmer, T. Niendorf, and H. Sehitoglu

The newly developed FeMnAlNiTi shape memory alloy (SMA) holds significant promise due to its desirable properties including ease of processing, room temperature superelasticity, a wide superelastic window of operation, and high transformation stress levels. In this study, we report single crystals with tensile axis near $\langle 123 \rangle$ exhibiting transformation strains of 9% with a high transformation stress of 700 MPa. The functional performance revealed excellent recovery of 98% of the applied strain in an incremental strain test for each of the 40 applied cycles. Concomitantly, the total residual strain increased after each cycle. Accumulation of residual martensite is observed possibly due to pinning of austenite/martensite (A/M) interface. Subsequently, under structural fatigue loading with a constant strain amplitude of 1%, the recoverable strains saturate around 1.15% in local

residual martensite domains. Intermittent enhancement of recoverable strains is observed due to transformation triggered in previously untransformed domains. Eventually, fatigue failure occurred after 2046 cycles and the dominant mechanism for failure was microcrack initiation and coalescence along the A/M interface. Thus, it is concluded that interfacial dislocations, which play a crucial role in the superelastic (SE) functionality, invariably affect the structural fatigue performance by acting as the weakest link in the microstructure (Fig. 1).

PSEUDOELASTIC NITINOL IN ORTHOPAEDIC APPLICATIONS

David Safranski, Kenneth Dupont, and Ken Gall

Pseudoelastic Nitinol presents an attractive material option for devices used in clinical orthopaedic applications. The capacity of the material to exert sustained compression during shape recovery aligns well with the mechanobiological factors associated with bone healing, particularly in

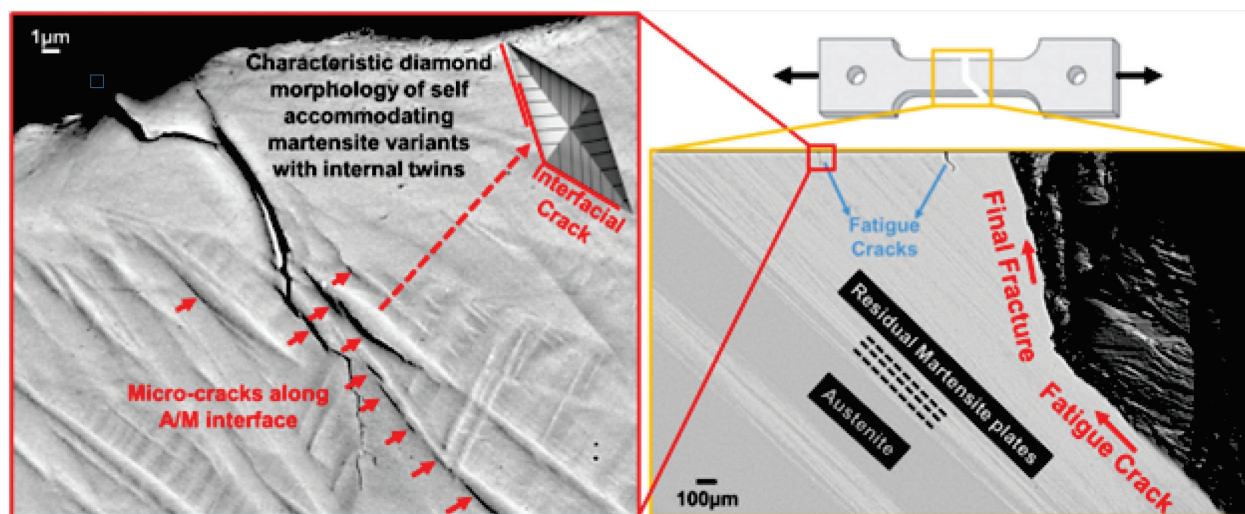


Fig. 1 — SEM-BSE image of the sample surface post-fatigue fracture. The low-magnification image on the right shows the residual martensite near the fatigue crack. The fatigue crack has propagated parallel to the activated martensite variant. A high-magnification image of the other fatigue cracks near the sample edge taken at 35,000x is shown on the left. Several microcracks have formed along the A/M interface.

applications such as fracture healing and joint fusion. Orthopaedic medical devices that have incorporated Nitinol are increasing in number, with two noted examples including staples and intramedullary nails. Early Nitinol devices utilized shape memory Nitinol, but the logistical difficulties with maintaining a cold state in the clinic or limited force-generation for materials warmed from room temperature to body temperature have led to pseudoelastic Nitinol devices dominating clinical usage. Both pre-clinical biomechanical and clinical studies have shown that these devices do exert sustained compression beyond the abilities of competing static devices, and largely have resulted in superior clinical outcomes, such as higher fusion rates and faster times to fusion. Given these results, continued adoption of existing Nitinol devices and future development of new orthopaedic devices utilizing the material should continue (Fig. 2).

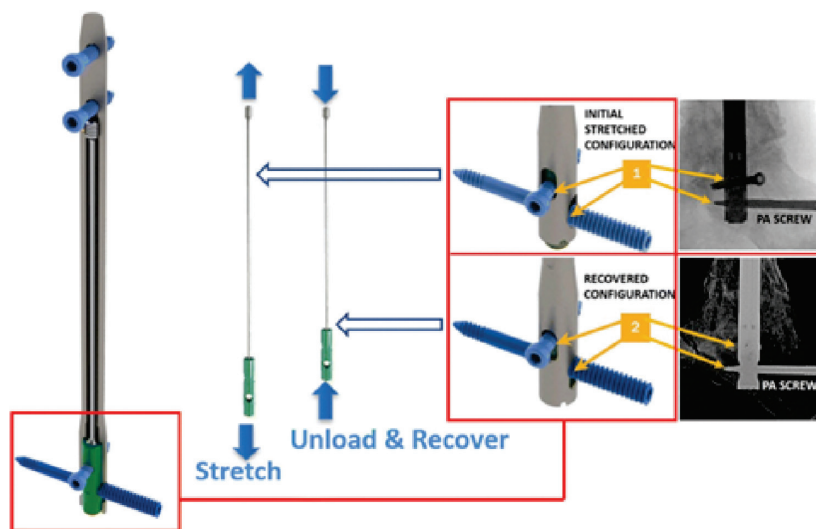


Fig. 2 — Intramedullary nail incorporating pseudoelastic Nitinol element. Recovery of the stretched element during the joint fusion process can be observed and quantified using longitudinal x-rays.

EFFECT OF C/O RATIO ON PHASE CHANGE AND STABILITY OF INCLUSIONS IN Ti-Ni ALLOYS FABRICATED BY A COMMERCIAL PRODUCTION PROCESS

Fumiyoshi Yamashita, Yasunori Ide, Hiroshi Akamine, Kouji Ishikawa, and Minoru Nishida

The effects of carbon/oxygen concentrations in Ti-51 at.% Ni on the morphology and phase changes of inclusions in a commercial wire manufacturing process were investigated. Whereas, cast material fabricated with a carbon to oxygen mass concentration ratio (C/O ratio) of 1.0–1.5 were found to contain single-phase Ti(C,O), when hot working was performed, some of the Ti(C,O) exhibited a phase

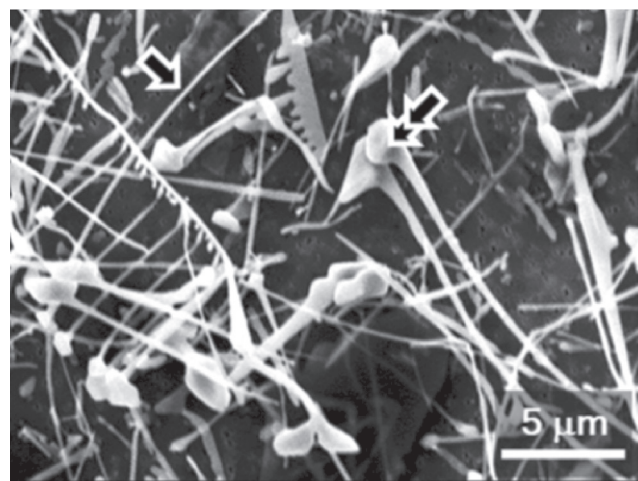


Fig. 3 — Scanning electron microscopy (SEM)-secondary electron (SE) images of inclusions extracted from cast specimen with C/O ratio of 1.3 by the selective potentiostatic etching by electrolytic dissolution (SPEED) technique.

change to $Ti_2Ni(O)$, and a mixed phase structure was found in specimens with a C/O ratio of less than 1.5. On the other hand, single-phase Ti(C,O) was found to remain in the wire produced from the specimens with a C/O ratio of 1.5 (Fig. 3).

PROCESSING AND SCALABILITY OF NiTiHf HIGH-TEMPERATURE SHAPE MEMORY ALLOYS

O. Benafan, G.S Bigelow, A. Garg, R.D. Noebe, D.J. Gaydosch, and R.B. Rogers

Development of melting and processing techniques for NiTiHf high-temperature shape memory alloys at the laboratory scale has resulted in pronounced success and repeatability for actuation purposes. Even the Ni-rich NiTiHf formulations, which are more challenging from a compositional control standpoint since small changes in chemistry can result in large transformation temperature variations, are reproducibly processed at the laboratory scale. Since properties of the slightly Ni-rich NiTiHf alloys have proved promising, large-scale production of such alloys now requires renewed attention. In this work, several melting techniques were used to process NiTi-20Hf (at.%), ranging from vacuum induction melting to plasma arc melting, with heats ranging in size from 0.4 to 250 kg with a target composition of $Ni_{50.3}Ti_{29.7}Hf_{20}$ (at.%). All cast ingots were subsequently hot extruded into bar. The resulting chemistries, microstructures, and inclusion types and sizes were evaluated as a function of melting technique. Finally, the thermophysical, mechanical and functional properties were measured for a number of material heats that varied in size and primary processing technique. The results indicated that various melting techniques could result in alloys with slightly different end compositions that can affect the mechanical and functional

correlated to the actual compositions of each heat, through corrections based on differential scanning calorimetry measurements, indicating that most scatter in properties can be explained by slight chemistry variations (Fig. 4).

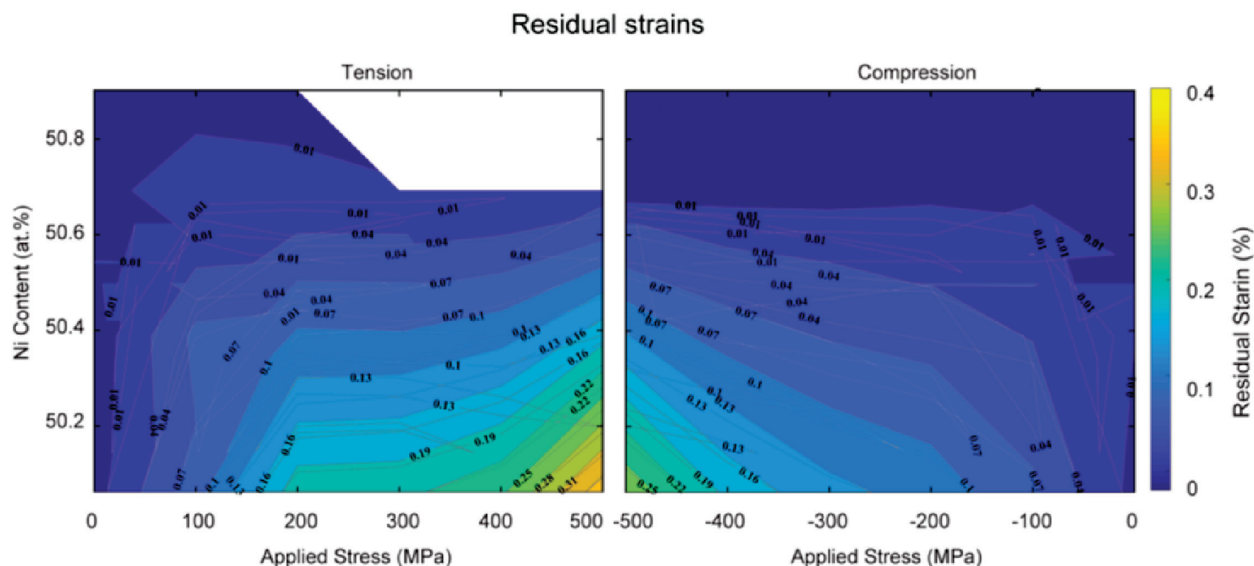


Fig. 4 — Effect of adjusted composition and applied stress on the residual strains for alloys aged at 550°C for 3 h and air cooled, tested in (a) tension and (b) compression.



Open Mic Series



DR. ROCCO LUPOI

WEDNESDAY, APRIL 21, 2021 – 9:00 A.M. ET

"Cold Spray: the journey began around 25 years ago, where are we now?"

Cold Spray is a method that is relatively new compared to other spray processes. It was discovered nearly by accident, and since then it has attracted considerable academic attention with some key industrial applications.

Join Dr. Lupoi for a brief overview of key Cold Spray innovations from past to present, with an overview around the current situation and the potential future for this process.

ASMINTERNATIONAL.ORG/CONFERENCES

FURRER TO CHAIR 2021 NOMINATING COMMITTEE

Members of the 2021 Nominating Committee have been selected and **Dr. David U. Furrer, FASM**, senior fellow discipline lead, Pratt & Whitney, was elected to serve as chair by the ASM Board of Trustees. Furrer has been a member of ASM International since 1985 and served as ASM trustee in 2010-2013 and as ASM President in 2019. He earned both his bachelor's and master's degrees in metallurgical engineering from the University of Wisconsin-Madison, and a doctorate of engineering from the Universität Ulm in Germany. Furrer joined Pratt & Whitney in 2010 and leads the Pratt & Whitney Materials Discipline Chiefs and Materials Fellows in development of technical strategies, new materials and processes, and engineering standards and procedures. He is also responsible for the materials engineering discipline health relative to technical talent growth, sustainability, and succession planning. Prior to Pratt & Whitney, Furrer was chief of strategic materials and process technology and fellow of materials process modeling at Rolls-Royce. Furrer is actively involved with ASM chapters, committees/councils and Affiliate Societies and most recently helped to establish the formation of technically focused committees, such as the Residual Stress Committee, for ASM. Furrer is a member of the Connecticut Academy of Science and Engineering and was installed as an ASM Fellow in 2003.



Furrer

The committee is responsible for selecting a nominee for vice president-trustee (one-year term) and for nominating three trustees (three-year terms). Members do not select a candidate for president of the Society, because Article IV, Section 3 of the Constitution states that the office of president shall be filled for a period of one year by succession of the vice president. The 2021 Nominating Committee's nominee for vice president will serve as ASM's president in 2023.

2021 Nominating Committee Members Include:

Dr. Andrew S.M. Ang, research engineer, Swinburne University of Technology, Melbourne, Victoria, Australia (nominated by the Thermal Spray Society); **Robert Goldstein, FASM**, executive director of product development and strategic planning, Fluxtrol Inc., Auburn Hills, Mich. (nominated by the Heat Treating Society); **Dr. M. Kamaraj, FASM**, professor, Indian Institute of Technology, Chennai, India (nominated by the Chennai Chapter); **Dr. Guiru Nash Liu, FASM**, senior experimental metallurgist, Progress Rail, LaGrange, Ill. (nominated by the Chicago Regional Chapter); **Alexandra Merkouriou**, project manager for Project Daedalus, University of Connecticut, Colchester (nominated by the Emerging Professionals Committee); **Dr. Dongwon Shin**, senior R&D staff member, Oak Ridge National Laboratory, Tenn. (nominated by the Alloy Phase Diagram Committee); **Hans Shin**, laboratory director, Pacific Testing Laboratories Inc., Valencia, Calif. (nominated by the San Fernando Valley Chapter); **Dr. John Smugeresky, FASM**, chief technical officer, A M Materials Consultants, Pleasanton, Calif., and senior member technical staff (retired), Sandia National Laboratories, Livermore, Calif. (nominated by the Materials Properties Database Committee); **Prof. Veronique Vitry**, associate professor, University of Mons, Belgium (nominated by the IDEA Committee).

The Nominating Committee will meet on April 14 and its recommended slate of officers will be published in the May/June issue of *ASM News*.

ASM Officers Appoint Members

In accordance with the ASM International Constitution, ASM president **Diana Essock, FASM**, vice president **Dr. Judith Todd, FASM**, and immediate past president **Dr. Zi-Kui Liu, FASM**, appointed nine members to the Nominating Committee from among candidates proposed by chapters, committees, councils, and ASM Affiliate Society boards.

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» HIGHLIGHTS MD CORNER

MD CORNER

Survey Says: Members Value ASM and Want More

Thank you to all who took the time to fill out our recent member survey. This survey is an important tool for our operations team to understand what you are thinking and what areas we should focus on to increase the value you receive from your membership.



Aderhold

We are still analyzing the data, but so far we have learned that our members stay involved because ASM is an organization that is passionate about materials science; stays current with industry trends; provides excellent technical publications and education; and offers networking opportunities both online and at ASM events, conferences, chapter meetings, and through volunteering.

Our technical material remains a core strength for ASM, and the survey suggests that members want greater access to these tools and resources. On that point, I would like to highlight two projects the operations team is working on. For our **Digital Library**, in addition to the *ASM Handbook* series, technical books, and failure analysis content already available, we are adding access to ASM journals, conference proceedings, and magazines.

I'm also happy to announce that we started a project

to redesign the **ASM Website**. Our goal is to provide members with an easy-to-use portal to access the wide variety of products and services that will help maximize the value of being an ASM member. We are now forming a **Web Focus Group** to help us design the new site. If you would like to get involved, go to ASM Connect, and then to Volunteer Opportunities, and let us know.

Being part of the materials community and networking also ranked high in the survey. ASM continues to offer many ways to get involved—by volunteering to serve on a range of committees and councils, joining our thriving online member community **ASM Connect**, or by attending one of our many physical or virtual conferences.

ASM's educational opportunities were also cited as an important part of our member services. Our new digital short courses, along with our in-person classroom and lab courses, local chapter courses, and conference programming, provide many options to keep up with the latest developments in materials science and learn new skills to build your career.

As you can see, there are many ways to take advantage of your ASM membership to help you grow both personally and professionally. As always, I would enjoy hearing about your experiences as a member and what we could do to enhance it. Thank you!

*Ron Aderhold, Acting Managing Director, ASM
ron.aderhold@asminternational.org*

ASM and its Affiliate Societies Seek Student Board Members

We're looking for Material Advantage student members to provide insights and ideas to ASM and its Affiliate Society Boards. We are pleased to announce the continuation of our successful Student Board Member programs. Each Society values the input and participation of students and is looking for their insights and ideas.

Opportunities specific to each Society:

ASM International

- Attend four board meetings (July 26-27, September 12-15 during IMAT, spring and summer 2022)
- Term begins June 1

ASM Electronic Device Failure Analysis Society

- Attend one board meeting (fall 2021)
- Participate in three teleconferences
- Term begins October 1

ASM Failure Analysis Society

- Attend one board meeting (fall 2021)
- Participate in three teleconferences
- Term begins October 1

ASM Heat Treating Society

- Attend one board meeting (fall 2021)

- Participate in two teleconferences
- Term begins October 1

ASM International Metallographic Society

- Attend one board meeting (fall 2021)
- Participate in monthly teleconferences
- Term begins October 1

ASM International Organization on Shape Memory and Superelastic Technologies

- Attend one board meeting (spring 2022)
- Participate in three teleconferences
- Receive a one-year complimentary membership in Material Advantage
- Term begins October 1

ASM Thermal Spray Society

- Attend one board meeting in the U.S. (fall 2022)
- Participate in three teleconferences
- Receive a one-year complimentary membership in Material Advantage
- Term begins October 1

Application deadline extended to April 30. Visit asminternational.org/students/student-board-member-programs for complete form and rules.



Canada Council Award Nominations due April 30

ASM's Canada Council is seeking nominations for its 2021 awards program. These prestigious awards include:

G. MacDonald Young Award – The ASM Canada Council established this award in 1988 to recognize distinguished and significant contributions by an ASM member in Canada. This award consists of a plaque and a piece of Canadian native soapstone sculpture.

M. Brian Ives Lectureship – This award was established in 1971 by the Canada Council of ASM to identify a distinguished lecturer who will present a technical talk at a regular monthly meeting of each Canadian ASM Chapter that elects to participate. The winner receives a \$1000 honorarium and travels to each ASM Canada Chapter throughout the year to give their presentation with expenses covered by the ASM Canada Council.

John Convey Innovation Awards – In 1977, the Canada Council created a new award to recognize sustaining member companies that contribute to development of the Canadian materials engineering industry. The award considers a new product and/or service directed at the Canadian or international marketplace. Two awards are presented each year, one to a company with annual sales above \$5 million, and the other to a company with annual sales below \$5 million.

View sample forms, rules, and past recipients at asminternational.org/membership/awards/nominate.

To nominate someone for any of these awards, email christine.hoover@asminternational.org for a unique nomination link.



VISIT THE CAREER HUB

Matching job seekers to employers just got easier with ASM International's CareerHub. After logging on to the ASM website, job seekers can upload a resume and do searches on hiring companies for free. Advanced searching allows filtering based on various aspects of materials science, e.g., R&D, failure analysis, lab environment, and manufacturing. Employers and suppliers can easily post jobs and set up pre-screen criteria to gain access to highly qualified, professional job seekers around the globe. For more information, visit the CareerHub site. <http://careercenter.asminternational.org/>.

International Metallographic Contest at IMAT

Deadline: September 3

The International Metallographic Contest (IMC), an annual contest cosponsored by the International Metallographic Society (IMS) and ASM International to advance the science of microstructural analysis, will take place on September 12 as part of IMAT 2021. The conference is scheduled for St. Louis, September 13-16. Six different classes of competition—including a new video class—cover all fields of optical and electron microscopy:

Class 1: Light Microscopy—All Materials

Class 2: Electron Microscopy—All Materials

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

Class 4: Artistic Microscopy (Color)—All Materials

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

NEW—Class 6: Video Entry

The new video class is aimed at engaging undergraduate students who may be unable to get into the laboratory due to COVID-19 restrictions.

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

Revealing the hierarchical microstructure of innovative additively manufactured metal parts with conventional light microscopy Class 1

Introduction

Additively manufactured parts are characterized by a peculiar microstructure, originated by the distinctive layer-by-layer process. In case of additive technology based on the localized melting of the metallic feedstock, as laser-based powder bed fusion (LPBF), the resulting microstructure has a hierarchical arrangement, consisting of macro and microscopic features affecting the final properties. Commonly, several advanced metallographic techniques (e.g., FEI-SEM, EBSD, etc.) are adopted in order to reveal the microstructure; however, main microstructural features can be also qualitatively appreciated by means of conventional light microscopy (LM). The present microstructural characterization was conducted on Co28CrFeNi samples, in the as-built condition, produced with a conventional LPBF machine equipped with a continuous wave laser source.

Experimental

Samples were extracted from small Co28CrFeNi blocks along the directions parallel to the building one (V), parallel to the building platform (H). They were embedded in a phenolic resin and subjected to the following metallographic preparation:

- Grinding with SiC papers: 180, 320, 600, 800 and 1200 grit.
- Polishing with polycrystalline diamond suspensions (0.3 and 1 µm) up to a mirror finish.
- Electrochemical etching performed at 4 V for 20 s in a 5 mL HCl, 10 g FeCl₃, 100 mL H₂O solution.

Microstructural analyses were carried out with a Zeiss Imager A1 optical microscope at low (5x) and high (50x) magnification with a 0.5x camera adapter and using polarized light, bright and dark fields.

Low-magnification LM

H cross-section

V cross-section

High-magnification LM

Bright field evidences in detail the solidified melt pools due to successive laser scans, being almost semicircular in V and elongated in the H one.

Dark field, instead, underlines the extremely fine cellular structure inside the solidified melt pools and peculiar of oriented cellular structures belonging to different columnar grains.

By comparison with high-magnification polarized light microscopy it can be found that differently oriented cellular structures belong to different columnar grains.

Conclusions

Conventional light microscopy can facilitate and accelerate the preliminary characterization of the peculiar hierarchical microstructure of innovative additive manufacturing components. With the sole optical microscope, indeed, main microstructural features (columnar grains, melt pools, cellular structure) can be simultaneously investigated. Basing on a proper optical analysis, further advanced analyses can be more successfully planned and targeted on specific aspects.

IMC 2020 Jacquet-Lucas winning poster by Lavinia Tonelli from the University of Bologna, Italy.

» HIGHLIGHTS EMERGING PROFESSIONALS

FROM THE FOUNDATION

Supporting the Farm Team

It is now April, which means professional baseball is getting started—and it feels a little more like normal than last year. When baseball took a hit in 2020 due to the global pandemic, minor league baseball teams unfortunately were forced to shutter their seasons. My son and I discussed what that could mean for the future of baseball.



Wilson

Without minor league teams to hone skills and develop players to eventually reach the major leagues, how would major league teams field that loss? Hopefully, just one “lost” year will not have a significant impact. But what if minor league teams disappeared altogether, permanently? What impact would that have on major league teams fielding a full roster of skilled and well-prepared players?

As director of the ASM Materials Education Foundation, I like to think of our programs as the minor leagues, or a “farm team,” to ASM International. Students who attend ASM Foundation programs—or are taught by teachers who have attended—learn the basics of materials science, about the field, career options, and how to pursue them. These are students who are anxious to attend a university materials science program, plan to become a professional in the field, and then ideally join ASM International to further their careers.

This is why we chose not to shut down our “minor league” programs last summer. This is why we found a way to continue to reach both students and teachers, despite the pandemic. And we are continuing to provide our programs this summer, albeit remotely. The ASM Foundation remains as dedicated as ever to reach and positively impact students who will be sparked by materials science and want to pursue it into the “major leagues.”

As always, thank you to the members, volunteers, and everyone supporting ASM Materials Camps and our other programs. Because of you, students go on to the majors each and every year!

Carrie Wilson

Executive Director

ASM Materials Education Foundation

EMERGING PROFESSIONALS

Embracing Virtual Communication

Jonathan Healy, EPC Co-chair

Now, over a year into the COVID-19 pandemic, many people have settled into what we consider the “new normal”—a world filled with Zoom meetings and particularly devoid of personal interaction. These recent times have underlined the importance of virtual communication. Luckily, a robust set of resources is available for emerging professionals (EPs) to engage with mentors and colleagues alike, both inside and outside of ASM.



Healy

Perhaps the most commonly used of all these platforms is LinkedIn. I believe most of today’s EPs are aware of this resource, although for many, its potential is not fully realized. Aside from making sure to have a completely filled in and up-to-date profile (picture included!), it is crucial for EPs to engage and interact with the platform. Most importantly, don’t be afraid to reach out to that next potential connection. I am always surprised by people’s unwillingness to reach out for a myriad of reasons, when in reality, people are generally willing to lend a helping hand when they can.

Aside from LinkedIn, there are several other resources that have sprung up. On the technical consulting side, we have the Fishbowl app, which is essentially a Reddit-like LinkedIn. On the more research-focused side, we have sites like ResearchGate that not only help colleagues connect, but also serve as a forum to help solve technical challenges as a community.

Last, and most specific to our Society, there is ASM Connect. Hopefully, most members are aware of this platform by now, but for those who aren’t, it is ASM’s members only online community. It takes some of the best parts of the aforementioned sites and specializes them to create one of the largest materials-centric online communities. If you haven’t had the chance yet, check it out and be on the lookout for an upcoming EP community on the site.

As our “new normal” slowly begins to fade back to pre-COVID times, remember these great resources, because there will always be a place for virtual communication.

Get Engaged, Get Involved, Get Connected

Watch for a new Emerging Professionals Community on ASM Connect coming soon. Visit the ASM Connect home page at connect.asminternational.org and get into the conversation!

WOMEN IN ENGINEERING HIGHLIGHTS

WOMEN IN ENGINEERING

This profile series introduces materials scientists from around the world who happen to be females. Here we speak with **Guiru Nash Liu, FASM**, senior experimental metallurgist, Progress Rail Inc., A Caterpillar Company, LaGrange, Illinois.



What does your typical workday look like?

I am very busy every day conducting failure analysis on locomotive/engine components. Often, I am needed on hot and urgent failures from new engines to determine the root cause so that we can improve the process or design to improve the reliability.

What part of your job do you like most?

Finding the evidence that contributed to the failures. It is just like doing a detective's job.

What is your greatest professional achievement?

Since 2000, my colleagues at Progress Rail and I have worked to meet U.S. EPA restrictions for locomotive emissions Tier II, III, and IV engines. With stricter emission requirements, there are increased demands on the materials of construction. It is a testament to our work that today's locomotives are more reliable, fuel efficient, and environmentally friendly than ever before. My greatest contributions are

in finding the failure mechanisms of the locomotive/engine components and providing the solutions to prevent production lines being shut down, ensuring on-time delivery of locomotives, and preventing further failures in the railroad industry. Other engineers have described my failure analysis reports as "always very thorough." I report the facts without speculation and provide the root cause of the failures and the solutions. Whenever engineers need urgent answers to a failure, they seek me out for answers because they trust my findings. My motto is to seek truth from facts. With high quality and fact-oriented root cause analysis results and preventive solutions, I have saved millions of dollars and perhaps lives by preventing future failures.

Are you actively engaged with ASM or its affiliates?

Yes, I have been and continue to be very active with ASM. I have held various positions in the Chicago Chapter including chair, where under my leadership, the chapter started the first local students summer camp nationwide. I have served at the ASM national level on many committees and am a reviewer for *Metallography*, *Microstructure*, and *Analysis* and the *Journal of Materials Science*. I served on the ASM Nominating Committee in 2012 and will serve again in this year. I was inducted into the 2020 Class of ASM Fellows and will receive the 2022 Alan Ray Putnam Service Award.

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



» HIGHLIGHTS INTERNATIONAL WOMEN'S DAY

CHAPTERS IN THE NEWS

Pune Chapter Celebrates International Women's Day

WOMEN ARE THE LARGEST UNTAPPED RESERVOIR OF TALENT IN THE WORLD.

On March 6, to celebrate and salute women in the materials field, a special program was arranged by the ASM Pune Chapter, under the guidance of Udayan Pathak, chapter chair. With 44 enthusiastic attendees, the virtual event coincided well with International Women's Day and March as Women's History Month. Pune's Women's Day Celebration also presented a global platform for highlighting some exceptionally impressive materials professionals.

Dr. Judith Todd, FASM, ASM vice president (2020-2021), department head, P.B. Breneman Chair, and professor of engineering science and mechanics at Penn State University, briefed participants on ASM International's journey in building diversity, equity, and inclusion. She stated that 8 years ago, Diana Essock (current ASM president), FASM, founded and led ASM's Women in Materials Engineering Committee to build a community of women across the society—from students to senior leaders. Some initiatives along the way included an annual women's breakfast; increasing the number of female Fellows, award winners, and board/committee leaders; and the formation of the IDEA Committee (Inclusion, Diversity, Equity and Awareness).

Anuradha Das, head of human resources, Engineering & Research Centre (ERC), Tata Motors Ltd., shared her experiences of increasing the participation of women on the shop floor. She spoke about efforts in creating the brigade of "women in blue" by sourcing and training rural and urban girls. Mukta Kulkarni, managing director, Industrial Enterprises, gave an inspiring talk about how she motivated



Part of Dr. Todd's presentation, this photo represents the growth in women in leadership roles and as awardees. From left, Diana Essock, FASM, introduces 2019 ASM Bronze Medal winner Danielle Cote with David Furrer, FASM, presenting the plaque.

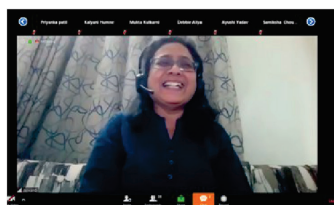
many women to learn finances along with obtaining a formal technical education.

An important part of program was the panel discussion on "Diversity and Inclusion" in which panelists Anuradha Das, Dr. Judith Todd, Dr. Kanyakumari Datta (proprietor, Data Metallurgical Co.), Dr. Sarika Verma (principal scientist/associate professor, AMPRI), and Mangesh Shetye (sr. general manager, Carraro India) expressed valuable insights on the importance of diversity and how to create an inclusive culture at the workplace. The program was hosted by Nutan Niharika, moderated by Jaswandi Gotmare, and concluded with remarks by Debbie Aliya, FASM.

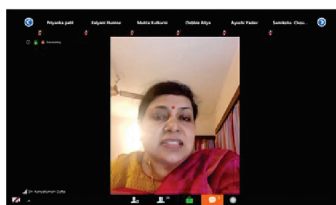
Aliya was invited to speak due to her strong connections to the Pune Chapter and as a role model of U.S. women entrepreneurs. She has traveled to India multiple times, making presentations at the Pune and Chennai Chapters and teaching her famous Failure Workshop, once to a standing-room-only crowd of 100.

The timely and impressive event, hailed as a success by all, serves as a role model not only for women in materials events but also for ASM's global interactions.

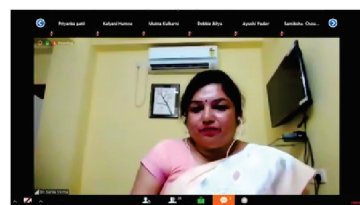
Special thanks to reporter Samiksha Choudhary for her contributions to this piece.



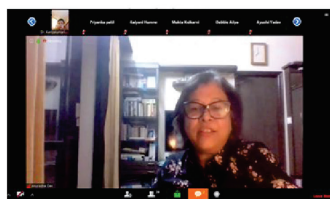
Jaswandi Gotmare



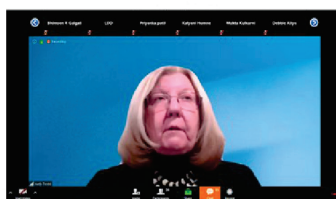
Kanyakumari Datta



Sarika Verma



Anuradha Das



Judith Todd



Mangesh Shetye

The panel discussion was a highlight of the Pune Chapter's Women's Day Celebration.

MEMBERS IN THE NEWS HIGHLIGHTS

Hartford Hosts AM Aerospace Expert

The Hartford Chapter welcomed Eliana Fu, industry manager of aerospace and medical at Trumpf USA, as their speaker for a February 9 virtual meeting. Fu spoke on “Additive Manufacturing Applications for Space Exploration.” The talk covered the technical advantages of AM for aerospace and space exploration, highlighting examples that compared traditional manufacturing to current AM processes along with the benefits of reduced lead time and greater access to space. Future ideas for printing in space were also explored. Educated at Imperial College London with a masters and Ph.D. in materials science, Fu worked at TWI, TIMET, and then SpaceX, before turning her attention to additive manufacturing. Fu has written a book based on her experiences as a female engineer at SpaceX.



Fu

Philadelphia's Eisenman Night

The Liberty Bell Chapter of Philadelphia hosted their annual Eisenman Award Night in a virtual format on February 18. This year's award recipient and presenter was John A. Janiszewski, P.E., FASM, of Hatch/LTK. His intriguing talk was entitled “Old Metallurgists Never Die, They Just Hammer Away: Reflections on 50 Years in the Profession.” In his presentation, Janiszewski reflected on some of the lessons learned, both technical and professional, in his long career in the power and rail industries. He discussed opportunities for materials engineers to make both themselves and their colleagues more effective by understanding how they interact with other technical personnel, how materials applications have evolved over the years, and how important the personal and professional relationships with other materials engineers are to keeping the materials profession moving forward.



Congratulations to this ASM Chapter
celebrating 100 years
of serving local members!

Central Massachusetts

Thank you for your commitment!
We look forward to celebrating your future success!

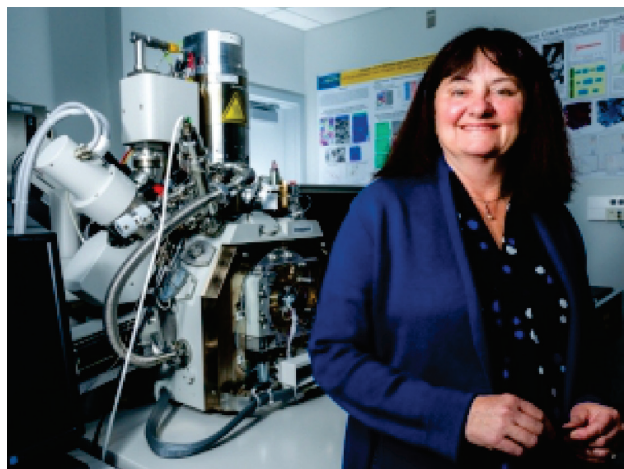
MEMBERS IN THE NEWS

Pollock Makes News with Standout Alloy

Tresa Pollock, FASM, has been making materials science news quite a bit lately. Most recently, her team's work in making a defect-resistant superalloy that can be 3D-printed has made a splash in industry headlines. The professor of materials and associate dean of the College of Engineering at UC Santa Barbara published a paper about the innovation in the journal *Nature Communications*, in collaboration with Carpenter Technologies, Oak Ridge National Laboratory, and other UCSB colleagues. The research was supported by a \$3 million Vannevar Bush Faculty Fellowship that Pollock was awarded from the U.S. Department of Defense in 2017.

Pollock and team describe a new class of high-strength, defect-resistant, 3D-printable superalloys, defined as typically nickel-based alloys that maintain their material integrity at temperatures up to 90% of their melting point. Most alloys fall apart at 50% of their melting temperatures. These new superalloys contain approximately equal parts cobalt and nickel, plus smaller amounts of other elements. The materials are amenable to crack-free 3D printing via electron beam melting as well as the more challenging laser-powder-bed approaches, making them broadly useful for a multitude of printing machines entering the market.

Pollock holds a Ph.D. in materials science and engineering from the Massachusetts Institute of Technology and a B.S. in metallurgical engineering from Purdue University. She was the ASM/TMS Distinguished Lecturer in 2013 and was awarded the ASM Gold Medal in 2014. She currently serves as principal editor of *Metallurgical Transactions A* and *B*.

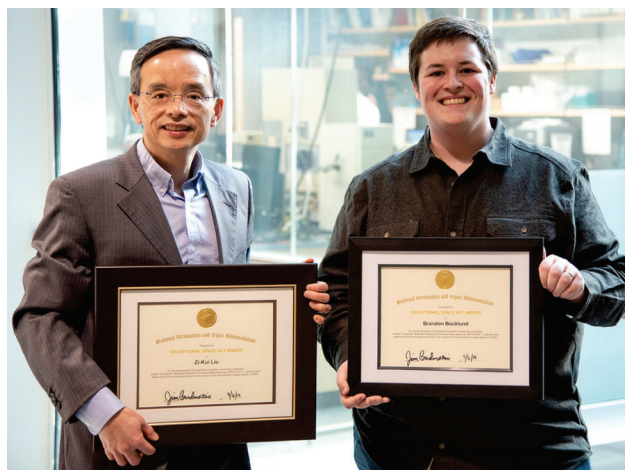


Tresa Pollock in her lab. Courtesy of Matt Perko.

» HIGHLIGHTS MEMBERS IN THE NEWS

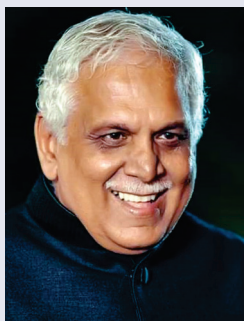
Liu Featured in Industry Publication

Zi-Kui Liu, FASM, was recently interviewed by HappyValley Industry, a publication in the State College, Pa., area. They asked to feature him after hearing that the DOE's Advanced Research Projects Agency-Energy (ARPA-E) had awarded his Penn State team a \$1.2 million grant for the design and manufacturing of ultrahigh-temperature refractory alloys. They were curious about why his research is important on a global scale, how it would change the industry, and what inspired him to pursue this type of research. The ASM past president stated that his project "aims to develop ultrahigh temperature materials for gas turbine use in the aviation and power generation industries through computational design, advanced manufacturing techniques and systematic characterization. High temperature materials such as Ni-based superalloys are the key component of the jet engines that enable today's aerospace industry. Materials for higher operation temperatures are desirable to increase the engine efficiency and thus reduce the pollutants." The full interview can be accessed at <https://bit.ly/2Ngf5QQ>.



Zi-Kui Liu, FASM, and graduate student Brandon Bocklund were part of a team that placed in the annual NASA Software of the Year competition for tools that are part of their ARPA-E project. ASM member Richard Otis (not shown) developed the code for the project.

IN MEMORIAM



Aurora

Word has been received at ASM Headquarters of the death of past trustee **Premkumar "Prem" Aurora**. The very active member and beloved leader from the India Chapter (Mumbai) passed away on March 10. Aurora served on the ASM board from 2017 to 2020. A complete obituary will be included in the next issue of *Advanced Materials & Processes*.

The **ASM Minnesota Chapter** notified ASM Headquarters of the passing of several of their longtime Life Members: **Richard Kielty**, age 82, of Richfield, passed away on March 17, 2020. He was a dedicated metallurgical engineer, having worked at Twin City Testing as well as Materials Evaluation and Engineering. He retired in 2019. He had been an ASM member for 56 years and a member of the Failure Analysis Society.

Marshall W. Nelson, age 91, died on March 23, 2019, in St. Paul. He was born on May 31, 1927. Nelson graduated from Rockford West High School and the Illinois Institute of Technology. As a mechanical engineer, he became a leader in the combustion industry. He founded Marshall W. Nelson and Associates in Milwaukee, Wisconsin, and Eagan, Minnesota. His ASM membership spanned 65 years.

Stanley R. Nelson, P.E., passed away August 14, 2020, at the age of 96. From St. Paul, he was born on December 18, 1923. Nelson was a WWII Veteran having served with the Marine Raiders in the Pacific Theater. An ASM member for 60 years, he was also a member of the Failure Analysis Society and Heat Treating Society.

Robert S. "Bob" Ray, age 83, of Anoka, died in February 2020. He was born in Phelps, Wisconsin, and grew up in the Upper Peninsula of Michigan where he graduated from Bergland High School in 1955. He attended Michigan Tech and graduated in 1962 with a degree in metallurgical engineering. In 1964, he was hired by Federal Cartridge and had a 35-year engineering career with them. Ray was also a member of the ASM Heat Treating Society.

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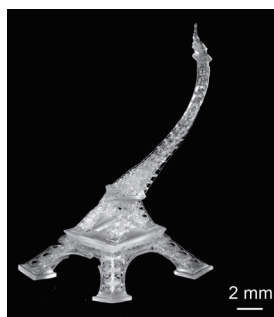
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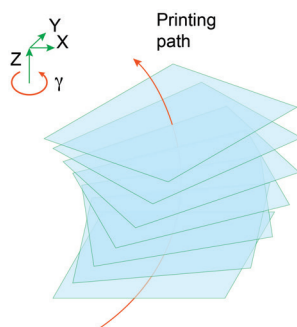
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3D PRINTSHOP



2D Shear + 1D Twist

Tiny Eiffel Tower produced by 3D printing method that can change printing direction on the fly. The printing path is shown on the right. Courtesy of Northwestern University.



polymerize, converting the liquid to a solid. This contributes to the speed and precision of our 3D printing process.”

In a paper published in the journal *Advanced Materials*, researchers demonstrate several applications, including 3D printing a customized vascular stent and printing a soft pneumatic gripper made of two different materials, one hard and

one soft. A double helix and a tiny Eiffel Tower are two other printed examples in the study. northwestern.edu.

‘ON THE FLY’ 3D PRINTING WITH A TWIST

A team of engineers from Northwestern University have developed a new method that uses light to improve 3D printing speed and precision while also, in combination with a high-precision robot arm, providing the freedom to move, rotate, or dilate each layer as the structure is being built.

The method introduces the ability to manipulate the original design layer by layer and pivot the printing direction without recreating the model. This “on-the-fly” feature enables the printing of more complicated structures and significantly improves manufacturing flexibility.

The Northwestern process uses a robotic arm and a liquid photopolymer that is activated by light. Sophisticated 3D structures are pulled out from a bath of liquid resin by a high-precision robot with enhanced geometric complexity, efficiency, and quality compared to the traditional printing process. The arm is used to change the printing direction dynamically.

“We are using light to do the manufacturing,” says Cheng Sun, associate professor of mechanical engineering at Northwestern’s McCormick School of Engineering. “Shining light on the liquid polymer causes it to crosslink, or

3D PRINTING TO FINETUNE PHOTONIC CRYSTAL FIBERS

Fiber optics are conventionally produced by drawing thin filaments out of molten silica glass down to microscale dimensions. By infusing these fibers with long narrow hollow channels, a new class of optical devices termed “photonic crystal fibers” were introduced.

“Photonic crystal fibers allow you to confine light in very tight spaces, increasing the optical interaction,” explains Andrea Bertoncini, a postdoc at King Abdullah University of Science and Technology. “This enables the fibers to massively reduce the propagation distance needed to realize particular optical functions, like polarization control or wavelength splitting.”

One way researchers tune the optical properties of photonic crystal fibers is by varying their cross-sectional geometry or arranging them into fractal designs. Typically, these patterns are made by performing the drawing process on scaled-up versions of the final fiber. Not all the geometries are possible with this method, however, due to gravity and surface tension.

To overcome such limitations, the

group turned to 3D printing. Using a laser to transform photosensitive polymers into transparent solids, the team built up photonic crystal fibers layer by layer. Characterizations revealed that this technique could successfully replicate the geometrical pattern of several types of microstructured optical fibers at faster speeds than conventional fabrications.

Bertoncini explains that the new process also makes it easy to combine multiple photonic units together. They 3D printed a series of photonic crystal fiber segments that split the polarization components of light beams into separated fiber cores. A custom-fabricated tapered connection between the beam splitter and a conventional fiber optic ensured efficient device integration.

“Photonic crystal fibers offer scientists a type of ‘tuning knob’ to control light-guiding properties through geometric design,” says Bertoncini. “However, people were not fully exploiting these properties because of the difficulties of producing arbitrary hole patterns with conventional methods. The surprising thing is that now, with our approach, you can fabricate them. You design the 3D model, you print it, and that’s it.” www.kaust.edu.sa/en.



Photonic crystal fibers are built layer by layer at much faster speeds than conventional methods. Courtesy of Anastasia Serin/KAUST.

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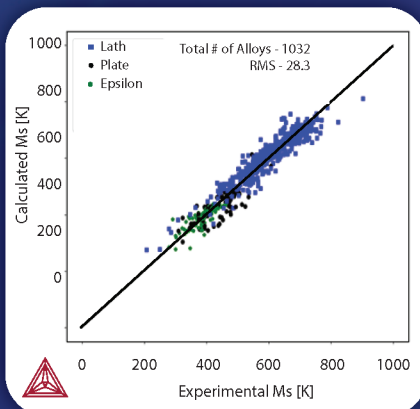
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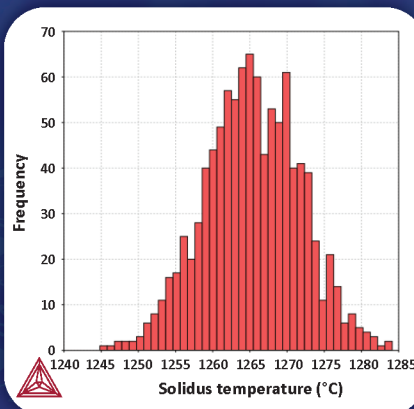
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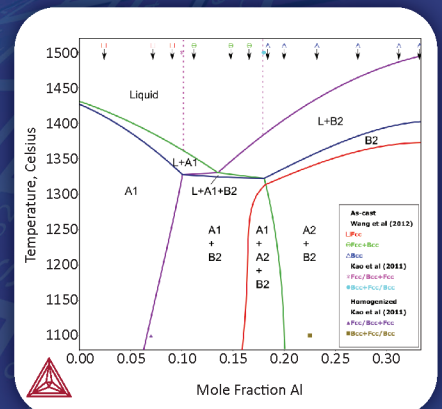
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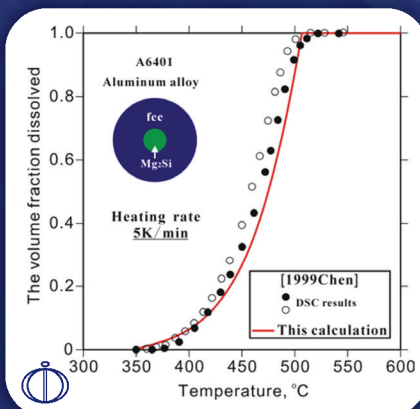
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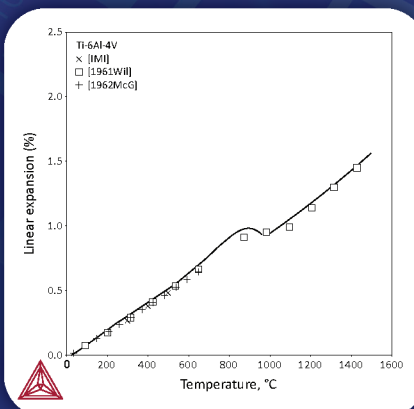
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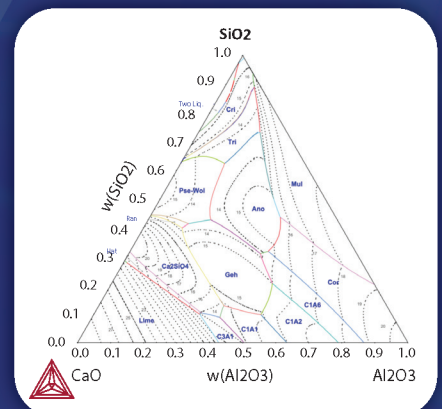
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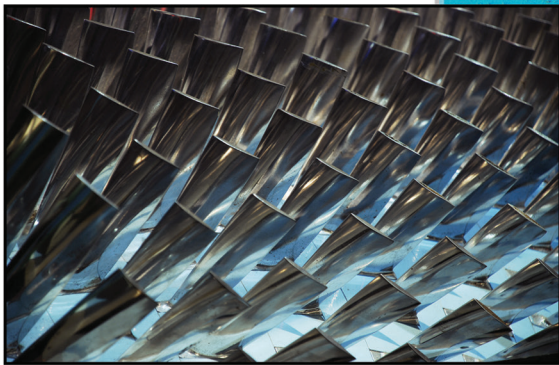
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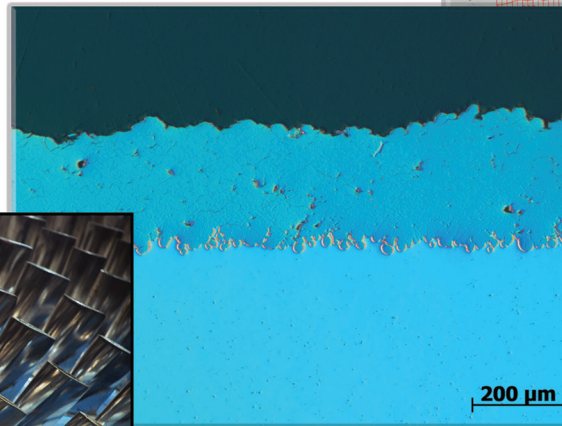
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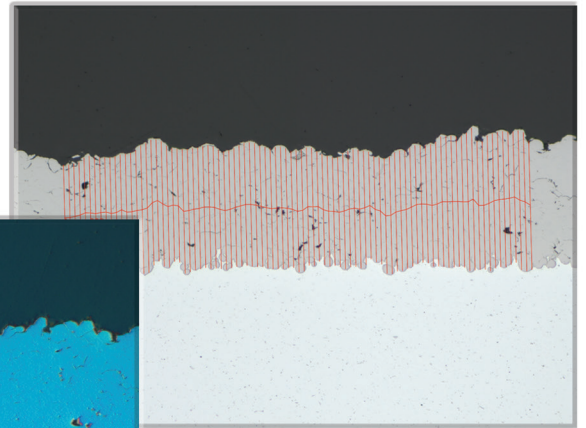
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