OCTOBER 2021 | VOL 179 | NO 7

# NATERIALS & PROCESSES

21st CENTURY STEEL TAKES FLIGHT

ADDITIVE MANUFACTURING

P. 19

Novel Additive Technologies for Metallic Glass



35

14

Hydrogels for 3D Bioprinting



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Thermo-Calc Software

**Empowering Metallurgists, Process Engineers and Researchers** 

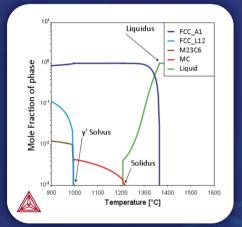
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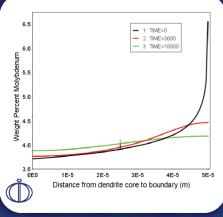
 Predict how actual vs nominal chemistries will affect property data

#### **Temperature Effect**

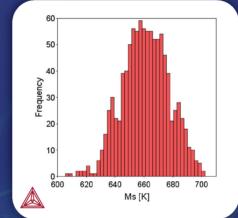


Phase stability vs. temperature for Ni-Base Alloy 282

#### Homogenization



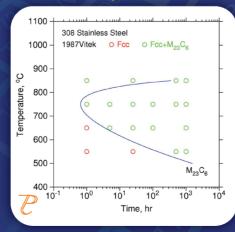
Homogenization of casting segregation in Ni-Base alloy 713



**Composition Effect** 

Calculated Ms temperatures for 410 stainless composition spec range

#### Precipitation

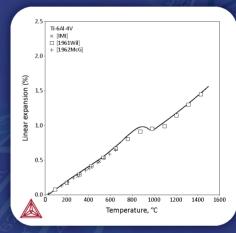


Time temperature precipitation of M<sub>23</sub>C<sub>6</sub> in 308 stainless steel

#### Base Decisions on scientifically supported models

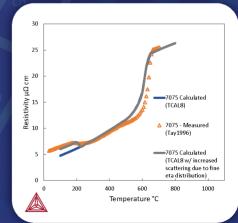
- Accelerate materials development while reducing risk
- Troubleshoot issues during materials processing

#### **Thermophysical Data**



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

#### **Electrical Resistivity**



Calculated electrical resistivity of aluminum alloy 7075

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### RIDING THE WAVE OF SYSTEM-IN-PACKAGE (SIP)

#### OCTOBER 31 - NOVEMBER 4, 2021 | PHOENIX, ARIZONA

#### BE SURE TO ATTEND THIS YEAR'S PANEL AND KEYNOTE SESSIONS!

#### Panel Session: Overcoming the Challenges in System-in-Package Failure Analysis

Discussion points will include SiP fault isolation and failure analysis gaps and challenges, SiP in Advanced Driver-Assistance Systems (ADAS), Electrostatic Discharge (ESD) in SiP, and Wafer Level Chip Scale Package (WLCSP) for SiP devices. Distinguished field experts are invited to serve on the panel to share their viewpoints and solutions based on their broad work experiences and diverse backgrounds, hopefully inspiring an engaged conversation among the panelists and audience.



#### Keynote Session: Tuesday, November 2

Dr. Ravi Mahajan, Intel Fellow, will talk on "Advanced Packaging for Heterogeneous Integration" including the value of packaging as an HI platform, key features in leading edge 2D and 3D technologies, and challenges for failure analysis and debug.



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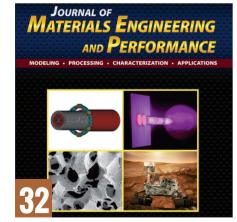
#### DITIVE MANUFACTURING OF METALLIC GLASSES: **CURRENT TECHNOLOGIES AND PATHS TO INFUSION**

#### Punnathat Bordeenithikasem

The high heating and cooling rates of additive manufacturing make it a good fit for metallic glasses, whose high elasticity, strength, hardness, and corrosion resistance are used in high-performance applications such as aerospace, defense, and biomedical devices.

### **On the Cover:**

A main rotor gearbox for next-generation Bell Helicopters was manufactured using Ferrium 64 steel developed by QuesTek Innovations LLC.



#### **ADDITIVE MANUFACTURING** HIGHLIGHTS

An overview of featured articles from the Journal of Materials Engineering and Performance focuses on additive manufacturing.



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



**STRESS RELIEF** Coca-Cola tests its new paper bottle in European markets.

# MATERIALS & PROCESSES

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Ferrium C64 is the culmination of a rigorous design process that bypassed extensive physical experimentation delivering a carburizable steel with unmatched capability.

#### **23 GELATIN METHACRYLATE HYDROGELS FOR 3D BIOPRINTING**

#### Pete Gabriel L. Ledesma, Kai-Hung Yang, and Roger J. Narayan

Because the photopolymerization of gelatin methacrylate can be spatially and temporally controlled, it can be fabricated into hydrogels well suited to a variety of biomedical applications.

#### **28 SUSTAINABLE MATERIALS AND THE CIRCULAR ECONOMY**

#### Tamil Selvan Sakthivel, Seeram Ramakrishna, and Sudipta Seal

The circular economy and sustainability are multifaceted concepts and thus should be pursued through collective advancements across various scientific fields.

#### **TRENDS**

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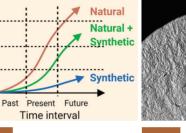
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Availability on Market







#### **35** SMST NEWSWIRE

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.

DVANCED MATERIALS & PROCESSES | OCTOBER 2021

### MATERIALS & PROCESSES

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### **A BRILLIANT FUTURE FOR ADDITIVE**



Additive manufacturing (AM) continues to impact the materials science landscape. Relativity Space recently announced the ability to 3D print a rocket in days instead of years. The process uses significantly fewer parts while optimizing quality and time performance. They're doing something right. But we can also insert other company names and applications and discover further astounding results. AM is a game changer across the board. Yet, more is being asked of the technology every day.

A recent survey conducted by Shapeways (a digital manufacturing platform provider) paints a picture of the state of 3D printing in the U.S. More than 300 mid-sized manufacturers, representing a swath of the automotive, aerospace, robotics, medical device, and industrial sectors, were polled. In rearview mirror mode, the survey shows that over the past three years, the top five customer expectation changes have included: higher demand for quality (53%); increased requests for sustainable solutions (47%); more frequent design changes (45%); desire for higher-level customization (43%); and continued cost reductions (40%). Looking forward, 84% expect the technology to continue increasing their revenues while decreasing costs. The envelope is being pushed.

In response to that push and moving beyond traditional AM sectors, the biomedical industry is making advances of its own. In this penultimate issue of *AM&P* for 2021, Pete Ledesma of the University of the Philippines brings us up to date on how hydrogels can be used in 3D bioprinting to seal leaks in liver and lung tissue. Applications for additive manufacturing are seemingly endless. In fact, our lead story indicates that your next piece of sports equipment may be made of additively manufactured metallic glasses.

If these articles whet your appetite for more AM research, check out the papers from our *Journal of Materials Engineering and Performance* selected for you by ASM Past President William Frazier, FASM, on page 32 and learn how to get involved with other AM efforts within the Society.

Also in this issue, and winning this year's ASM Engineering Materials Achievement Award, QuesTek Innovations provides an update on their novel high-performance carburizable steel. Their Ferrium C64 steel was birthed via integrated computational materials engineering (ICME). Primary applications include lighter-weight transmission gears with increased power density. But the Ferrium C64 story does not stop there. The alloy has now headed into the AM space with the company's engineers producing C64 powders that will maintain the superior performance of AM parts.

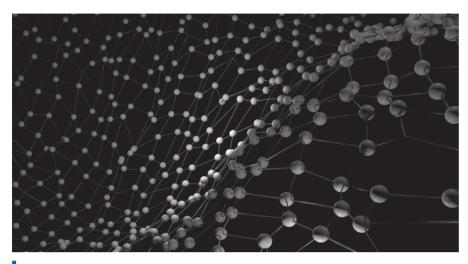
QuesTek also participated in a popular Additive Manufacturing Panel at IMAT, moderated by ASM Vice President David Williams, FASM. Attendees at IMAT and Heat Treat also enjoyed top-notch keynotes from materials engineering leaders at Tesla and Ford describing how electric vehicle development is breaking all the standard molds and forcing new ways of thinking. In Tesla's case, they are moving beyond typical additive to envision new casting techniques. Hint: Imagine one giant cast to replace numerous smaller components.

After these thought-provoking St. Louis sessions, attendees were then doubly rewarded with views of the magnificent Clydesdales while sipping Anheuser-Busch brews during the networking social. Nice way to savor the old world while contemplating the brilliant future.

bonne Mille

joanne.miller@asminternational.org

# **RESEARCH TRACKS**



A new type of piezoelectric molecular crystal is capable of self-healing. Courtesy of CC0 Public Domain.

#### SELF-HEALING PIEZOELECTRIC CRYSTAL

Researchers from the Indian Institute of Science Education and Research in Kolkata, the Indian Institute of Technology Kharagpur, and RWTH Aachen University in Germany discovered a type of piezoelectric molecular crystal capable of autonomous self-healing. Previous efforts to develop self-healing materials have yielded some results including polymers, gels, and other materials that can heal themselves to some degree after an injury. To date, these technologies have shared one thing in common-they are all soft. In the new study, scientists tackled the challenge of developing a self-healing hard material. In this case, that meant figuring out how to get a dense material, made of molecules arranged in a regular way, to heal when ripped apart.

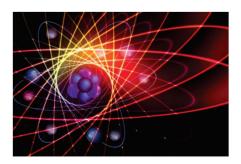
The work involved studying piezoelectric molecular crystals, which are capable of converting mechanical energy into electricity. The team reasoned that the inherent properties of such crystals should lend themselves to self-healing due to their attractive forces. After extensive trial and error, the researchers settled on bipyrazole organic crystals and grew sample crystals in tiny needle shapes, 2-mm long by 0.2-mm wide. Next, they applied just enough pressure to make them break and then watched as they bounced back into straight needles, with no evidence of the break remaining. Testing of the crystal with a polarization microscope system showed that the material had truly healed. The team says such crystals could find use in optical and nanoprobing devices, or perhaps in video screens, enabling smartphones to heal themselves after being dropped. *www.rwth-aachen.de.* 

#### CANDIDATE SUPER-CONDUCTOR ANNOUNCED

Since receiving a \$25 million grant in 2019 to become the first National Science Foundation Quantum Foundry, researchers at the University of California, Santa Barbara (UCSB) affiliated with the foundry have been working to develop materials that can enable quantum information-based technologies. Applications include quantum computing, communications, sensing, and simulation, among others. Now, UCSB materials engineering professor Stephen Wilson and multiple co-authors, including key collaborators at Princeton University, are studying a new material developed in the Quantum Foundry as a candidate superconductor that could be useful in future quantum computation.

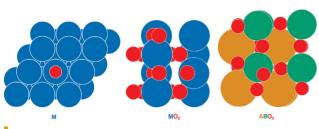
The group's most recent paper, "Discovery of Unconventional Chiral Charge Order in Kagome Superconductor  $KV_3Sb_5$ " is published in *Nature Materials*. Wilson says that the peculiar nature of the self-organized patterning of electrons is the focus of the current work.  $KV_3Sb_5$  was discovered to be a rare metal built from kagome lattice planes, and one that also superconducts. Some of the material's other characteristics led researchers to speculate that charges in it may form tiny loops of current that create local magnetic fields.

The role of Wilson's group was to make the material and characterize its bulk properties. The Princeton team then used high-resolution scanning tunneling microscopy to identify what they believe are the signatures of such a state, which "are also hypothesized to exist in other anomalous superconductors, such as those that superconduct at high temperature, though it has not been definitively shown," according to Wilson. If KV<sub>2</sub>Sb<sub>5</sub> turns out to be what it is suspected of being, it could be used to make a topological qubit useful in quantum information applications. ucsb.edu.



A new material developed in the Quantum Foundry could be useful in future quantum computation. Courtesy of Pixabay/ CC0 Public Domain.

# MACHINE LEARNING A



From left, diagrams show an oxygen atom bonding with a metal, metal oxide, and perovskite. A new machine learning model could help engineers design these catalysts for more sustainable fuel production, among other uses.

#### MACHINE LEARNING IMPROVES CATALYSTS

In a new study, researchers at the University of Michigan used machine learning to predict how the compositions of metal alloys and metal oxides affect their electronic structures. "We're learning to identify the fingerprints of materials and connect them with the material's performance," says chemical engineering professor Bryan Goldsmith. A better ability to predict which compositions are best for guiding certain reactions could improve large-scale chemical processes from hydrogen production to chemical manufacturing.

One of the main approaches to predicting how a material will behave as a potential mediator of a chemical reaction is to analyze its electronic structure, specifically the density of states. Usually, the electronic density of states is described with summary statistics—an average energy or a skew that reveals whether more electronic states are above or below the average. "That's OK, but those are just simple statistics. With principal component analysis, you just take in everything and find what's important," explains Goldsmith.

Principal component analysis is a classic machine learning method. The team used the electronic density of states as input for the model be-

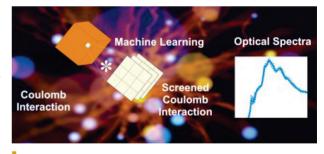
cause it is a good predictor for how a catalyst's surface will adsorb atoms and molecules that serve as reactants. The model links the density of states with the composition of the material—accurately reflecting correlations already observed, as well as turning up new trends.

The team's model simplifies the density of states into two principal components: One covers how the atoms of the metal fit together; the other covers the number of electrons the surface metal atoms can contribute to bonding. From these two variables, the model can reconstruct the density of states in the material. *umich.edu*.

#### SIMPLIFIED MODELING SPEEDS SIMULATIONS

Although computer simulations can help explore light-matter interactions, modeling materials that feature multiple types of structures is a complex job. Now, a research team at the DOE's Argonne National Laboratory has found a way to streamline this task. Using a data-driven approach based on machine learning, the team was able to simplify the solution of the quantum mechanical equations that describe how light is absorbed by a solid, liquid, or molecule.

The key insight was recognizing that not all terms of the quantum mechanical equations need to be computed in the same way. Some could be calculated from simpler quantities, remarkably speeding up the overall simulation. These protocols can lead to big savings when it comes to simulations that may take hours or even days on high-performance computers. The new technique allows simulations of absorption spectra of complex systems to run between 10 and 200 times faster than previous methods. Researchers are now looking at applying these shortcuts and recycling protocols to electronic structure problems not only related to light absorption, but also to light manipulation for quantum sensing applications. anl.gov.



Machine learning can circumvent explicit calculation of certain material behavior to accelerate simulations of optical properties of complex materials at finite temperature.

#### BRIEF

**Cornell University,** Ithaca, N.Y., is partnering in a \$36 million grant from the **Toyota Research Institute,** Los Altos, Calif., for its Accelerated Materials Design and Discovery collaborative university research program, which seeks to use artificial intelligence to discover new materials that could help achieve emissions-free driving. *cornell.edu*.





By adding carbon, hydrogen, and oxygen atoms to hard-to-evaporate metals like tungsten and platinum, researchers efficiently transformed the elements into thin films. Courtesy of Bharat Jalan MBE Lab, University of Minnesota.

#### THIN FILM EFFICIENCY

Researchers at the University of Minnesota Twin Cities College of Science and Engineering, Minneapolis, developed and patented a new technology that enables more efficient manufacturing of a "stubborn" group of metals and metal oxides into thin films. Many metals and their compounds must be made into thin films before they can be used in technological products like electronics, displays, fuel cells, or catalytic applications. "Stubborn" metals, however-which include elements like platinum, iridium, ruthenium, and tungsten, among others-are very difficult to convert into thin films because they require extremely high temperatures to evaporate.

Now, the cheaper, safer, and simpler production technology can evaporate these metals at significantly lower temperatures—fewer than 200°C instead of several thousands. By designing and adding organic ligands to the metals, the researchers were able to substantially increase the materials' vapor pressures. Not only is their new technique simpler, but it also makes higher quality materials that are easily scalable. *cse.umn.edu*.

#### **CERTIFYING STRUCTURES**

One of the most significant scientific and engineering problems in the design of promising structures is the lack of verified mathematical models describing the behavior of polymer composite materials in complex products. Now, a collaborative group of researchers led by Skoltech, Russia, created and tested the model of a thermoplastic composite material chaotically reinforced by short glass fibers. They demonstrated the effectiveness and applicability of this model on strength calculations of promising composite valves and safety devices for portable tanks for road, rail, and maritime transportation of chemical substances.



Material model for the design of pressure vessel service equipment made from polymer composites. Courtesy of Skolkovo Institute of Science and Technology.

According to the researchers, the model makes it possible to significantly reduce the conservatism of strength calculations in the design and customization of structures—minimizing subsequent manufacturing costs while fulfilling necessary safety and quality requirements. The results indicate that mathematical models of the behavior of composite materials are applicable to virtual tests of structures instead of expensive full-scale tests. *www. skoltech.ru/en.* 

#### **LITHIUM BATTERY RESEARCH**

Scientists from South Dakota State University, Brookings, along with collaborators from the University of Texas at Arlington, will work to determine how lithium metal improves battery performance through a three-year, nearly \$450,000 National Science Foundation grant. Understanding how a new material helps increase the stability as well as the storage capacity of lithiumion batteries can help researchers better develop high-performance energy storage devices. This fundamental scientific knowledge will provide guidelines for researchers using this material to develop next-generation batteries to power electric cars and store renewable energy.

Electric cars, for instance, now have a range of around 300 miles. However, according to the researchers, if they can develop high-performance storage devices that are the same weight but can store more energy, the range could be expanded up to 600 miles.

#### BRIEF

A team of international researchers across several institutions synthesized a new carbon that is reportedly the hardest and strongest amorphous material created to date. They subjected fullerenes to very high temperatures and pressures and produced what they call **AM-III carbon**, a type of glass with crystals in it. The material is yellowish, has no defined structure, and is very strong—scoring 113 gigapascals on the Vickers hardness test, higher than some diamonds and approximately 10 times as hard as steel. *https://doi.org/10.1093/nsr/nwab140*.

8

Using lithium metal in place of graphite as the anode material can increase the battery's energy storage capacity, but, over time, the lithium foil tends to form needle-like dendrites. These tiny metal particles can pierce the separator and cause a short circuit, explain researchers.

"Dendrite growth on the electrodes and unstable solid-electrolyte interphase formation have created safety concerns in lithium-ion batteries and hindered practical applications," researcher Yue Zhou said. In previous funded research, Zhou used plasma processing to apply an additional coating to protect the lithium metal anode and thereby prevent dendrite formation. In addition, he combined graphite and silicon oxide to make an ultrathin film that prevents dendrite growth and enhances ion transport. This also creates an artificial interfacial layer between

the electrolyte and the lithium metal electrode that increases stability and improves battery cell performance.

"How the protective layer interacts with the electrochemical process of the lithium metal anode is not well understood," Zhou says. "We want to understand the physical and chemical properties that lead to these desirable properties." What the researchers learn will help improve the safety of electric vehicles and advance efforts to develop devices that can store renewable energy. *sdstate.edu*, *utexas.edu*.



Doctoral student Jyotshna Pokharel assembles a lithium-ion battery in which lithium metal is used in place of graphite as the anode material. Courtesy SDSU.

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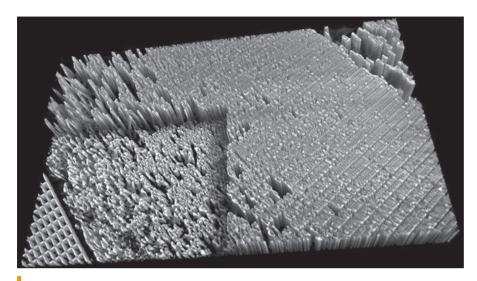
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# TESTING CHARACTERIZATION



This plate with 16-nanometer-wide features was imaged in resolutions of less than 10 nanometers, allowing scientists to see the tiny defects in its shape. Courtesy of Vincent De Andrade.

#### **HIGHER RESOLUTION X-RAYS**

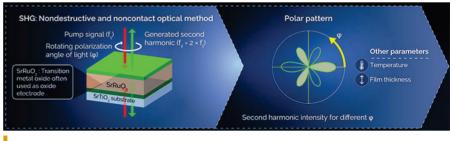
Researchers led by the DOE's Argonne National Laboratory, Lemont, Ill., created a new method for improving the resolution of hard x-ray nanotomography. The team constructed a high-resolution x-ray microscope using the powerful beams of the Advanced Photon Source (APS) and developed new computer algorithms to compensate for issues encountered at tiny scales, such as sample drift and deformation. Using the new method, the team achieved a resolution below 10 nanometers.

The capabilities of this instrument and technique will improve with a continuing research and development effort on optics and detectors and will benefit from the in-progress upgrade of the APS. When complete, the upgraded facility will generate high-energy x-ray beams that are up to 500 times brighter than those currently possible, and further advances in x-ray optics will enable even narrower beams with higher resolution. "After the upgrade, we will push for eight nanometers and below," the researchers say. "We hope this will be a powerful tool for research at smaller and smaller scales." anl.gov.

#### ANALYZING WITH OPTICS

A team of scientists at the Gwangiu Institute of Science and Technology (GIST) in Korea developed a promising all-optical approach to analyze crystal structures in the bulk, interface, and surface of materials. The researchers employed their strategy to investigate thin films of SrRuO,, a metal oxide widely used as an electrode and in oxide engineering. The technique they used, called optical second-harmonic generation, is a non-contact and nondestructive optical method that involves shining a pulsed laser onto a material and measuring the generated second harmonic light, which reveals information about crystalline symmetries. By changing the thickness and temperatures of SrRuO, samples, the scientists could suggest the structural phase diagram of the bulk, surface, and interface of the films.

An important result is that it appears the crystallographic symmetries of the surface and interface, at least



A study of SrRuO<sub>2</sub> thin film symmetries through second harmonic generation (SHG). Courtesy of GIST.

#### BRIEF

Smithers, Akron, Ohio, announces that its chemical and physical testing laboratories are now Nadcap accredited for

in SrRuO<sub>3</sub>, are closely related to the magnetic state and may even reorient the material's magnetic ordering. This opens up a possible avenue for realizing multifunctional devices. According to the researchers, "It may be possible to leverage the magnetic properties and symmetries at the interface and surface to design ultrathin nanodevices with many degrees of freedom or control parameters, which will be useful for applications such as healthcare patches, ultrathin quantum devices, sensors, and more." www.gist.ac.kr/en/main.html.

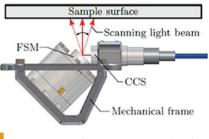
#### SURFACE MEASUREMENT SYSTEM

Researchers from the Vienna University of Technology in Austria created a lightweight optical system for 3D inspection of surfaces with micronscale precision that can operate in the vibration-prone environment of industrial manufacturing plants. The new measurement tool could greatly enhance quality control inspection for high-tech products including semiconductor chips, solar panels, and consumer electronics such as flat panel televisions. Because vibrations make it difficult to capture precision 3D measurements on the production line, samples are periodically taken for analysis in a lab. However, any defective products made while waiting for results must be discarded.

To create the tool, researchers combined a compact 2D fast steering mirror with a high precision 1D confocal chromatic sensor. These sensors can precisely measure displacement, distance, and thickness using the same principles as confocal microscopes but in a much smaller package, making them more suitable for the production floor. They also developed a reconstruction process that uses the measurement data to create a 3D image of the sample's surface topography. The 3D measurement system is compact enough to fit on a metrology platform, which serves as connection to a robotic arm and compensates for vibrations between sample and measurement system through active feedback control.

The teams' co-leader Daniel Wertjanz explains, "By manipulating the optical path of the sensor with the fast-steering mirror, the measurement spot is scanned quickly and precisely across the surface area of interest."

The researchers are now working to implement the system on the metrology platform and incorporate it with a robotic arm. This will allow them to test the feasibility of robot-based precision 3D measurements on freeform surfaces in compromised environments such as an industrial production line. *www. tuwien.at/en.* 



A compact 2D fast steering mirror (FSM) was combined with a high precision 1D confocal chromatic sensor (CCS) to create a lightweight measurement system. Courtesy of Daniel Wertjanz/TU Wien.



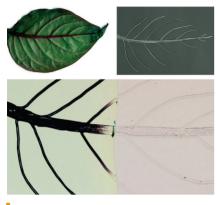
#### New! *Heat Treating Proceedings* Added to ASM Digital Library

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### **PROCESS TECHNOLOGY**



Manufacturing process of a vascularized structure using a 3D-printed template that mimics vascular networks of a leaf. Courtesy of Mayank Garg.

#### **SMART TECH BREAKTHROUGH**

A new polymerization technique to simultaneously cure and vascularize high-performance materials in a matter of minutes, instead of days, was developed by researchers at the University of Colorado, Fort Collins. Inspired by biological systems like human blood vessels or plant leaves, the scientists created microchannels inside structural polymers and composites, imparting multifunctional properties to the host structure such as self-healing and thermal regulation. The research breakthrough shortens the manufacturing cycle from two days of processing under a vacuum at high temperatures to five minutes at room temperature and ambient pressure—without any resources like ovens or pumps.

Internally created heat from fron-

#### BRIEFS

Service Heat Treating,

Milwaukee, recently finalized a multiyear plant expansion of nearly 40,000 sq ft, doubling its space. The company also added another batch furnace and four new nitriding furnaces. *serviceht.com.*  tal polymerization solidifies the host while deconstructing the template into gas, leaving behind a vascular network. Combining the two steps into one eliminates the need for an oven and provides greater control to engineers and scientists to explore advanced biological functions and more complex systems. The discovery paves a new future for efficient smart technology, impacting the aerospace, wind, and automotive sectors. Such industries are increasingly using composite materials in structural parts due to their low density, excellent mechanical properties, and corrosion resistance.

However, composites are prone to internal damages that are not easy to detect. Using vascularized structures allows for delivering beneficial fluids to the damaged site for healing. According to the researchers, these self-healing features will enhance the reliability and longevity of structural composites. *colostate.edu*.

#### **UTILIZING DEFECTS**

Scientists at the Johns Hopkins Applied Physics Laboratory, Laurel, Md., are working to gain a better understanding of different defects and their influence on the mechanical performance of additive manufacturing (AM) materials. The researchers deliberately introduced the two natural types of processing defects, lack of fusion and keyhole, into samples to determine how they influence parts' mechanical properties.

Results showed that while high amounts of each defect are unfavorable,

**Pennex Aluminum,** Wellsville, Pa., completed a \$23 million expansion of their Leetonia, Ohio, facility and plans to hire 70 new employees by year's end. The company specializes in aluminum fabrication and extrusions for the automotive, construction, HVAC, landscaping, and off-road vehicle markets. *pennexaluminum.com.* 

it is more favorable to be in the keyhole domain—at a similar concentration of defects—than in the lack of fusion domain. The team also discovered that microstructural refinement around a keyhole defect can counteract the weakening effect of the defect. Even up to 4-5% porosity in the keyhole domain results in the same yield strength as a part with negligible porosity, a target metric many mechanical engineers use to design parts.

"We modified the laser processing conditions to simulate natural faults in the process and generated three similar amounts of defects in the keyhole and lack of fusion domains," explain the scientists. "Then, we scanned and quantified material from each processing condition using x-ray computed tomography to map the defect size and distribution, comparing samples containing these resultant defects in monotonic tension testing to determine the preferred defect domain for a given amount of defects."

"This work is a critical step in laying the foundation to enable qualification of AM parts in the future," the researchers say. "A general understanding of the influence of the effects of processing conditions on the resulting microstructure and properties of a material and component will provide the scientific basis to enable protocols for safe implementation of additively manufactured parts." *jhu.edu*.



Taylor Bean is a new saw operator at Pennex Aluminum, Leetonia, Ohio.

# EMERGING TECHNOLOGY



Soft circuits can bend, fold, twist, and stretch. Courtesy of Virginia Tech.

#### **UNBREAKABLE ELECTRONICS**

Researchers from Virginia Tech, Blacksburg, created a new type of soft electronics that integrate skin-like circuits to increase durability and flexibility. Their soft and stretchy circuit design could pave the way for devices that are self-healing, reconfigurable, and recyclable. Current consumer devices, such as phones and laptops, contain rigid materials that use soldered wires running throughout. The soft circuit developed by the Virginia Tech team replaces these inflexible materials with soft electronic composites and tiny, electricity-conducting liquid metal droplets, which are initially dispersed in an elastomer as electrically insulated drops.

"To make circuits, we introduced a scalable approach through embossing, which allows us to rapidly create tunable circuits by selectively connecting droplets," explain the researchers. "We can then locally break the droplets apart to remake circuits and can even completely dissolve the circuits to break all the connections to recycle the materials, and then start back at the beginning." The circuits are soft and flexible, like skin, continuing to work even under extreme damage. If a hole is punched in these circuits, the metal droplets can still transfer power. Instead of cutting the connection completely as in the case of a traditional wire, the droplets make new connections around the hole to continue passing electricity. The circuits will also stretch without losing their electrical connection, as the team pulled the device to over 10 times its original length without failure during the research.

"We're excited about our progress and envision these materials as key components for emerging soft technologies," they say. "This work gets closer to creating soft circuitry that could survive in a variety of real-world applications." *vt.edu*.

#### **SEEING IN THE DARK**

Making the invisible visible, an international research team developed a first-of-its-kind prototype technology that could revolutionize night vision. The team, led by The Australian National University, says their new ultracompact, thin-film technology, based on nanoscale crystals, has applications for defense and standard use in consumer glasses. They also say the work of police and security guards—who regularly employ night vision—will be easier and safer, reducing chronic neck injuries from existing bulky night-vision devices.

The technology is extremely lightweight, cheap, and easy to mass produce, making it accessible to everyday users. Currently, high-end infrared imaging tech requires cryogenic freezing to work, and is costly to manufacture, while the new tech works at room temperature. It incorporates the use of metasurfaces to manipulate light in new ways. According to the researchers, this is the first time anywhere in the world that infrared light has been successfully transformed into visible images in an ultrathin screen. "It's a really exciting development and one that we know will change the landscape for night vision forever." www.anu.edu.au.



Lead researcher, Dr. Rocio Camacho Morales, says her team has made the "invisible, visible." Courtesy of Jamie Kidston/The Australian National University.

#### BRIEF

**Rice University,** Houston, is building a 266,000-sq-ft facility to replace its historic Abercrombie Engineering Laboratory. The new space will contain the headquarters for **The Welch Institute for Advanced Materials,** founded in 2020 with a \$100 million gift from the Robert A. Welch Foundation. The institute will develop next-generation materials for energy systems, space systems, biomedical applications, and more. *rice.edu.* 

### SUSTAINABILIT



Mobile phone featuring a self-sanitizing cover produced from a material made by extracting chitin from squid bones. Courtesy of NTHU MSE, Taiwan.

#### EXTRACTING PIEZOELECTRIC MATERIALS

Using common waste products, a research team from National Tsing Hua University in Taiwan created two composite piezoelectric materials. One is a new type of catalyst extracted from discarded rice husks and can treat industrial wastewater 90 times guicker than photocatalysts now in use. The scientists used the other material, extracted from discarded squid bones, to produce a self-sanitizing transparent film suitable for use as a cover on mobile phone screens, elevator buttons, door handles, and more.

The team's catalyst material can be injected into a factory's wastewater pipeline wherein the pressure generated by the water flow helps to purify the pollution without needing any light, overcoming a challenge faced by current photocatalysts. This new type of quartz composite piezoelectric material can also cope with the difficult-to-treat dyes present in wastewater produced by textile factories. In addition to wastewater treatment, the researchers say, this material can be used to produce hydrogen, which can be collected and used to generate energy. The material is also reusable and biodegradable, providing a type of wastewater treatment which is inexpensive, convenient, effective, and environmentally friendly. The research team's guartz composite material has already received patents in Taiwan and the United States.

The second material, made by extracting chitin from squid bones, is another new composite piezoelectric material suitable for producing a transparent film which undergoes self-sterilization whenever it's touched, making it highly suitable as a screen cover for various items in public places. The chitin used to make this new material can also be extracted from shrimp shells, crab shells, and cuttlefish bones, and it can be further manufactured using bionic technology. Self-sterilizing films made from this new material could play a key role in fighting the spread of infectious diseases. nthu-en.site.nthu. edu.tw.

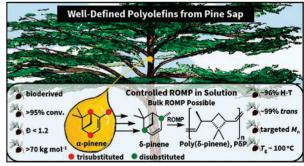
#### PLASTICS FROM **PINE SAP**

Researchers found a new way to source plastic from renewable resources. A team of scientists from Florida State University, Tallahassee, discovered a plastic material derived from pine sap, paving the way for

other new sustainable materials development. The gateway discovery, according to the researchers, is a significant step in the right direction for new biobased materials. Alpha-pinene, the most abundant molecule produced from pine sap, is notoriously difficult to turn into plastics. It's primarily found in turpentine-based cleaners and solvents. Lead researcher Mark Yarolimek first synthetically modified the alpha-pinene to make the compound known as delta-pinene.

"I put alpha-pinene through a series of chemical reactions, multiple purifications, and some trial and error, which eventually proved successful in converting it to delta-pinene," explains Yarolimek. "Once we obtained purified liquid delta-pinene, I converted that into the resultant plastic, poly-delta-pinene, through one final chemical reaction."

The team is also concerned with the chemical recyclability of these new materials. That may mean developing decomposition processes via a chemical stimulus in the future. fsu.edu.



A new process converts tree sap into plastic. Courtesy of ACS Macro Lett.

#### BRIEF

Orion Engineered Carbons S.A., Houston, is working with RISE Research Institutes of Sweden to develop and

### ADDITIVE MANUFACTURING OF METALLIC GLASSES: CURRENT TECHNOLOGIES AND PATHS TO INFUSION

Punnathat Bordeenithikasem\* NASA Jet Propulsion Laboratory California Institute of Technology, Pasadena

The high heating and cooling rates of additive manufacturing make it a good fit for metallic glasses, whose high elasticity, strength, hardness, and corrosion resistance are used in high-performance applications such as aerospace, defense, and biomedical devices. n recent years, additive manufacturing (AM), or 3D printing, has been at the forefront in the world of materials science and engineering. AM is a part fabrication methodology by which the part is produced by adding material layer-by-layer until the final designed geometry is achieved<sup>[1,2]</sup>. Numerous AM technologies have since been developed that use feedstock material of varied shape and chemistry. Industry and academia are diligently working to increase the variety of materials that can be fabricated using AM.

Metallic glasses (MGs) are alloys that do not have a crystal structure. This is generally achieved by rapidly quenching the alloy melt such that the atoms do not have sufficient time to crystallize, hence bypassing the crystallization process. Though, in theory, any metal could be made amorphous if the quench rate is sufficiently fast, most MG compositions are designed with intrinsically slow crystallization behavior such that amorphous parts could be made using experimentally achievable cooling rates. This amorphous microstructure is the reason MGs have properties that are different from conventional alloys. For example, most MGs possess high elasticity, strength, hardness, and corrosion resistance, which have inspired using MGs in high-performance applications like spacecraft hardware, biomedical implants, and sports equipment<sup>[3,4]</sup>.

Due to the fast cooling rate requirements, there are strict dimensional limits in casting or melt spinning MGs, only allowing for relatively thin and simple geometries to ensure sufficient cooling throughout the part to avoid crystallization. This hinders the ability for MGs to be made into net shape, hence reducing their versatility. Attempts have been made to circumvent this limitation, most notably through thermoplastic forming (TPF)<sup>[5]</sup>. However, the tooling, temperature control, and limited compatible alloy compositions have prevented the proliferation of the TPF of MGs. Another method to fabricate MG without the dimensional restraints due to the requisite cooling rates is through AM. Most AM processes employ high heating and cooling rates in the sequential layering process and therefore it is a superb technology to fabricate MG components<sup>[6-8]</sup>.

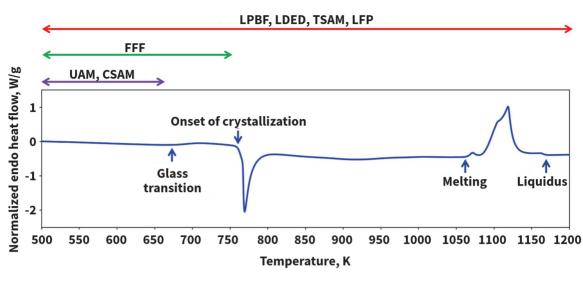
#### **CURRENT AM TECHNOLOGIES**

Though MGs were first reported in the 1960s<sup>[9]</sup>, only recently has the global supply chain matured sufficiently to source MGs in a variety of chemistries and geometries as shown in Fig. 1<sup>[10]</sup>. These form factors include powders, pellets, foils, sheets, plates, wires, and cylinders. This has allowed the development of multiple MG AM technologies based on different feedstock shapes and temperature ranges (Fig. 2). Feedstock material in both amorphous and crystalline forms could be transformed through AM to produce fully amorphous parts or metal matrix composites, which is controlled by adjusting the build parameters.

Approximately 70% of the scientific literature covering 3D printing of MGs describes laser powder bed fusion (LPBF), where a laser (or multiple lasers) is selectively exposed onto a powder bed to consolidate regions of interest<sup>[6]</sup>. Then a new layer of powder is applied on top and the process repeats until the final geometry is achieved. LPBF produces highly accurate parts using powder as feedstock, which could be sourced with relative ease and is available in multiple chemistries. LPBF has enabled bulk MG parts produced out of any alloy system with no thickness limitation. However, the limitations present in LPBF of conventional metals are also present when printing MGs such as porosity, high residual stresses, introduction of oxygen and other defects during powder production and the printing process, scalability of part sizes, and overall cost<sup>[11,12]</sup>. Additionally, with printing MGs, the temperature range within the heat affected zone of subsequent layers must be controlled to not induce unwanted crystallization



**Fig. 1** — Photographs of metallic glasses in different forms: (a) wires, (b) pellets, (c) rods and cylinders, (d) powder, (e) cast plate, and (f) sheets and foils. Images reproduced from Bordeenithikasem<sup>[10]</sup>.



**Fig. 2** — Diagram of the temperature ranges of various MG AM technologies that are discussed in this article, overlaid with a differential scanning calorimeter measurement of an arbitrary MG to illustrate key thermal events.

#### that will deteriorate the integrity of the component.

Another related technology is laser directed energy deposition (LDED), where feedstock powder is fed through nozzles and then ejected into the melt pool or path of the laser to build the part. LDED of MGs is most commonly used to fabricate MG composites for wear-resistant applications<sup>[13]</sup>. The resolution of LDED is worse than LPBF but it allows for intermixing of different powders, supplied via different nozzles and hoppers, to tailor the desired composition and even produce compositional gradients<sup>[14]</sup>. Microstructural gradients from amorphous to crystalline are also possible. LDED is also used to clad MG alloys to a crystalline substrate at desired locations.

Thermal spray additive manufacturing (TSAM) is another technology that uses powder as feedstock. Using a conventional high-velocity oxygen-fuel (HVOF) gun, the powder is thermal sprayed at high temperature onto the substrate in layers. The morphology of each layer is controlled using stencils or masks<sup>[12,15]</sup>. TSAM produces parts with relatively high porosity but extremely high cooling rates, allowing for the production of MG compositions that are difficult to be made amorphous otherwise. The process also has a high-volume throughput, allowing parts to be built quickly. Cold spray additive manufacturing (CSAM) is a complementary technology to TSAM where amorphous powders are ejected at high velocities but at temperatures below the glass transition<sup>[16]</sup>. Each layer adheres due to the mechanical deformation of the sprayed powder.

Sheets and ribbons, generally fabricated through melt spinning, are another form factor of MGs that are readily available in varied chemistries. Laser foil printing (LFP) is a similar process to LPBF that uses sheets or foils as feedstock instead of powder<sup>[10]</sup>. LFP produces parts with lower oxygen content than powder-based processes due to the much lower exposed surface area in the sheet production process. However, the technology is less mature than LPBF and LDED; most LFP components were fabricated on custom-built laboratory-scale equipment.

Ultrasonic additive manufacturing (UAM) also utilizes MG sheets as feedstock. In UAM, an ultrasonic horn is used to consolidate sheets of material at or near room temperature. The high frequency relative motion along the interface causes the materials to fuse without exposing them to elevated temperatures, avoiding issues like crystallization and high residual stresses<sup>[17]</sup>.

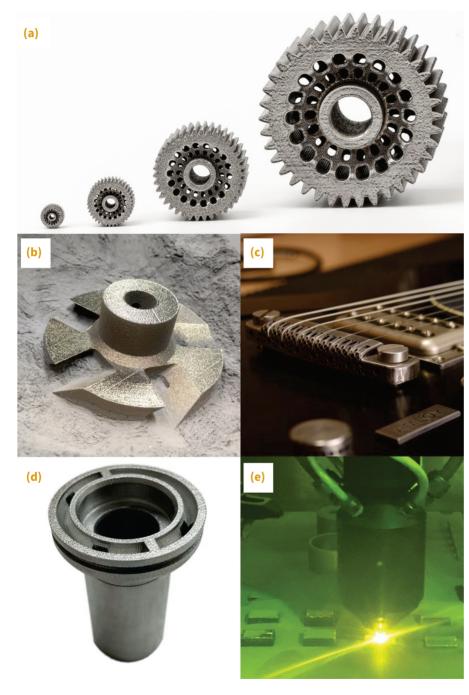
Fused filament fabrication (FFF) takes advantage of the TPF process in MGs and the existing infrastructure behind AM of polymers. MG wires or rods could be extruded through a heated nozzle and then printed in the same

manner as filament-based 3D printing of thermoplastics<sup>[18]</sup>. However, the modest availability of MG compositions with suitable thermal stability and viscosity in addition to the relative difficulty to source MG wire and rods have limited the maturation of FFF.

#### PATH TOWARD TECHNOLOGY INFUSION

Additive manufacturing of MGs has made substantial strides toward technology infusion and commercialization. The assortment of AM technologies applied to MGs can produce parts that cannot be achieved in other materials. Although the AM process introduces porosity and contaminants into the resultant part, the wear properties of the 3D printed MG is comparable to the traditionally cast MG<sup>[11]</sup>. Applications that require high hardness and wear resistance, such as gears and excavation tools for extraterrestrial robotic exploration, were the first prototyped with LPBF, which have shown promising results (Figs. 3a and b)<sup>[19]</sup>. Wear-resistant casings for watches and electronics have also been prototyped. Capitalizing on the elasticity of MGs, guitar bridges that are more efficient at transmitting vibrations were demonstrated (Fig. 3c). The bridge is designed such that the sounds mimic traditional materials used in guitar bridges, such as brass, but with improved performance and part longevity provided by MGs. Another application that utilizes the MG's elasticity is the expansion sleeve (Fig. 3d). The low stiffness and high elasticity allow the MG expansion sleeve to deform more easily than a traditional expansion sleeve, therefore offering better performance. Moreover, the expansion sleeve is designed such that the part is produced in one piece instead of multiple components that require subsequent assembly. The low stiffness, high elasticity of MGs in addition to ease of design of AM have also inspired researchers to create orthopedic implants with integrated porosity to facilitate bone growth<sup>[20]</sup>.

LDED was used by researchers to demonstrate the vast range of micro-



**Fig. 3** — Images of functional components produced by additive manufacturing of metallic glass (MG). (a) MG gears fabricated using LPBF; (b) MG excavation tool bit designed for the excavation of extraterrestrial ice, printed with LPBF; (c) MG guitar bridge made using LPBF; (d) MG expansion sleeve produced with LPBF; (e) LDED system producing multilayer claddings of MG composites. Parts in images (a-d) were manufactured by ©Heraeus AMLOY. Image (e) is reproduced from Bordeenithikasem<sup>[13]</sup>.

structures achieved by altering the processing parameters when fabricating MG composites, and hence tailoring the mechanical properties (Fig. 3e)<sup>[13]</sup>. LDED could be used to selectively clad different regions of parts with MG composites for surfaces that require higher wear resistance. LDED and TSAM could also be used to fabricate Fe-based MG systems that have attractive magnetic properties enabled by the amorphous structure.

Despite advances in AM of MGs, the infusion into practical applications is an uphill battle. With rapidly advancing technology and business ecosystems around all aspects of AM, MGs have to compete directly with any other material that can be 3D printed. Although MGs possess attractive mechanical properties mentioned above, MGs have low plasticity and poor fatigue limit (exacerbated by porosity and other AM defects), are more expensive than conventional alloys, and have limited post processing options after printing, i.e., MGs could not be heat treated using hot isostatic pressing (HIP), a commonly used technique for metal AM parts to relieve stresses and reduce porosity. Research efforts are underway in the community to address these issues.

As with conventional metal AM, industries with low part count and high-performance requirements such as space, aerospace, defense, and biomedical devices, would be most suitable for 3D printing of MGs. Further research is needed to develop alloys specifically for the AM process for both monolithic MGs and MG composites. The main challenge is to identify use cases that leverage the unique properties of MGs and would outperform any other material in a one-to-one comparison. ~AM&P

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# FERRIUM C64: A 21st CENTURY **STEEL TAKES FLIGHT**

Ferrium C64 is the culmination of a rigorous design process that bypassed extensive physical experimentation-delivering a carburizable steel with unmatched capability.

hen QuesTek Innovations formed in 1997, inefficient, costly, and time-consuming trial and error methods for materials design were the industry norm. The company's engineers believed there was a better way-using the power of mechanistic-based computationа al approach to shift from physical experimentation to virtual simulation. By successfully implementing this transformative methodology over the past 25 years, the company's unique approach to integrated computational materials engineering (ICME) has reduced development costs and lead time, improved existing materials and processes, and enabled design and development of novel materials. QuesTek emerged as a leader in this area when its high strength, corrosion-resistant Ferrium S53 steel became the first ICMEdesigned material to receive aerospace AMS and MMPDS qualifications and fly as a safety critical landing gear on the U.S. Air Force T-38 platform in 2010<sup>[1]</sup>.

The company also deployed the accelerated insertion of materials (AIM) process using expected chemistry and thermal process variation along with computational mechanistic models of alloy strength to create simulated data and estimate S53's A-Basis 1% design minimums using only three full-scale data heats. Traditionally, full experimental generation of an A-Basis design minimum would require data from 10 full-scale heats of material<sup>[2]</sup>.

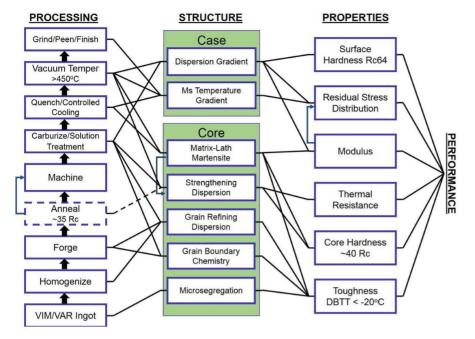
Another standout among Ques-Tek's recent developments is Ferrium C64, a commercially available steel designed for high-performance gearing. The alloy also serves as an enabling technology for future rotorcraft power transmission shafts, gears, and other mission-critical applications, such as those requiring weight savings, fatigue resistance, and temperature stability. The material, Fe-3.5Cr-7.5Ni-16.3Co-1.75Mo-0.2W-0.11C, is the culmination of a rigorous design process that bypassed extensive physical experimentation-delivering a carburizable steel with unmatched capability. This article discusses the material design process for Ferrium C64.

#### **ANATOMY OF A MATERIAL DESIGN**

A systems-based methodology employing ICME technologies was used to design a novel alloy composition. Fundamental to the design process was the development of models predicting the interplay between processing, microstructure, and key material properties. The systems design chart for Ferrium C64 shown in Fig. 1 illustrates

Carpenter Technology's 22 metric ton vacuum induction melter where Ferrium C64 is manufactured. Courtesy of Carpenter.





**Fig. 1** — Systems design chart for Ferrium C64 highlights critical processing-structure-property relationships.

key components of each subsystem with linkages representing a modellable dependency<sup>[3]</sup>.

The genesis of the Ferrium C64 alloy design was the establishment of aggressive property goals as defined by the U.S. Navy in a Small Business Innovation Research (SBIR) proposal solicitation in 2005 seeking to replace traditional steels such as AISI 9310 or Alloy X53. Specifically, this called for a surface carburizable steel—up to 64 HRC—with improved core properties and increased temperature resistance.

As such, during the design process, a double vacuum melted secondary hardening, martensitic steel was identified as the most promising material class to achieve these lofty goals. Further, the material was designed to be carburized to develop ultrahard surface properties. Thermo-Calc software was used to execute the mechanistic, thermodynamics-based approach to calculate key microstructural aspects. These microstructure predictions fed proprietary structure-property models to complete the material design framework as follows:

*Optimal material composition:* With the material processes and microstructural concepts identified, Ques-Tek's materials design engineers set forth to develop the precise and optimal material composition. In effect, Ferrium C64 comprises two material designs: one composition within the core material and a second composition for



Rack of gears undergoing vacuum heat treatment at Solar Atmosphere's Souderton, Pa., facility. Courtesy of Solar.

the case. The variation between the core and case evolves during the carburization process, resulting in a case exhibiting an enriched carbon content for hardness.

*Processing:* The specific alloying additions were identified and chosen to establish a microstructure consisting of a martensitic matrix with an optimized strengthening carbide dispersion that derives from a solutionize, quench, and age heat treatment processing route. Overall alloy content was constrained by the processstructure linkages outlined in Fig. 1. Maximum solution temperature was limited by a combination of process capability within the manufacturing base and the desire to maintain a fine grain structure in the final product.

The design called for high temperature vacuum carburization as a key processing step that would combine solutionizing and carburization into a single operation. Next, a sufficiently high martensite start (Ms) temperature was necessary to ensure nearly full martensitic transformation upon quenching from the solution treatment (the overall alloy content was designed to ensure suitable Ms temperature). Lastly, the strengthening carbide phase fraction and its nucleation and growth parameters had to be optimized to ensure a reasonable aging temperature and time.

Alloying additions: Each elemental alloving addition was designed to play a crucial role in either the material matrix or precipitate dispersion. Ni alloying was incorporated to improve matrix toughness, while Co was included to restrict dislocation recovery during heat treatment, resulting in a martensitic matrix with high dislocation density. This dislocation network provides heterogeneous carbide nucleation sites that help increase the carbide nucleation driving force to ensure a fine strengthening carbide dispersion. While the C content sets the overall phase fraction of the strengthening carbide, the number density and precipitate size within the matrix plays a crucial role regarding mechanical properties. Carbide forming Mo, Cr, V, and W contents were designed to achieve a 3-nm-sized carbide volume fraction after aging that optimizes the carbide strengthening effect. Additionally, the carbide forming elements are significantly overbalanced compared to the core carbon content to enable greater carbide formation in the higher carbon containing case region.

Further, the aging treatment was selected based on the tradeoff between nucleation driving force, inversely proportional to temperature, and growth kinetics, which is proportional to temperature. In summary, the carbide forming elements were balanced to ensure high nucleation driving force with tailored growth kinetics to yield a carbide dispersion within a feasible manufacturing process in both the core and case.

In the end, the ICME-enabled design process yielded a novel alloy composition and process to achieve aggressive property targets by identifying and predicting the role of each element in the material microstructure<sup>[4,5]</sup>.

#### **COMMERCIAL DEBUT OF C64**

A critical factor in the qualification and commercialization of C64 steel was establishing strong partnerships across the entire supply chain from the earliest development stages. These included:

- Specialty steel producer and current licensee Carpenter Technology
- Forgers such as Scot, ATI Ladish, and Larson
- Heat treater Solar Atmospheres
- Machining vendors Forest City Gear, Delta Gear, Triumph Group, and many others
- Superfinisher REM Chemical

In addition, a significant amount of fatigue data was developed at the Gear Research Institute and NASA Glenn Research Center.

### RIGOROUS QUALIFICATION PROCESS

To facilitate end-user adoption, Ferrium C64 underwent the rigorous SAE Aerospace Materials Specification qualification process (designated as AMS 6509) for developing S-Basis minimum design allowables. Compared to former best-in-class gear steels such as AISI 9310 (AMS 6265) or Alloy X53 (AMS 6308), the C64 data showed a targeted step-change in combination of strength, toughness, and surface hardness. Reports from industry are that C64 allows for ~20% increase in power density or a ~20% reduction in component weight at the same power.

Wrought Ferrium C64 steel has been supplied to many industries

ranging from aerospace, oil and gas, and high-performance racing to hand tools and agriculture. Military applications to date are in partnership with the major U.S. helicopter OEMs for Future Attack Reconnaissance Aircraft (FARA) and Joint Multi-Role (JMR) Future Vertical Lift programs.

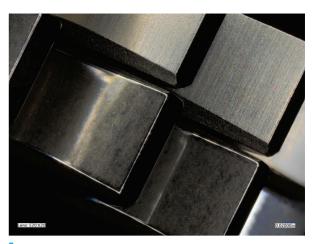
#### IMPROVED MILITARY AIRCRAFT SAFETY

Under the U.S. Armyfunded Future Advanced

Rotorcraft Drive System (FARDS) program, Bell Helicopter evaluated Ferrium C64 steel for next-generation helicopter transmission components. A main rotor gearbox was manufactured using Ferrium C64 steel along with several other novel technologies. This demonstration gearbox was operated under a loss-of-lubrication test condition, which survived >80 minutes of operation without failure in the C64 components. This result is illustrated by post-test images of the gear shaft and input pinion in Fig. 2, showing only minor scuffing on the gear teeth and no failures. Existing best performing helicopter gearboxes last <25 minutes due to the limited temperature stability of strengthening carbides in typical gear steels<sup>[6]</sup>. The high temperature stability designed into Ferrium C64's strengthening carbides enables it to perform at the elevated temperature of a gearbox oil-out condition where other steels fail.

#### ADDITIVE MANUFACTURING PROGRESS

QuesTek is actively transitioning Ferrium C64 into laser powder bed fusion additive manufacturing processes. Initial success based on multiple industrial scale atomization runs and printing on EOS M 290 equipment has shown equivalent static mechanical properties versus wrought C64 bar. Initial axial fatigue and single tooth bending fatigue results are also promising.



Ferrium C64 steel FZG type test gear chemically processed by REM Chemical in as-ground (top of image) and after isotropic superfinishing (bottom). REM can achieve Ra ranges in  $1-6 \mu$ in for C64. Courtesy of REM.



**Fig. 2** — Ferrium C64 input shaft (top) and gearshaft (bottom) imaged after 85 minutes of loss-of-lubrication testing in Bell Helicopter's Future Advanced Rotorcraft Drive System program. Results show minor scuffing on the gear teeth but no signs of surface damage or failure. Courtesy of Bell.

C64 is already one of the most developed high-performance steels for high hardness applications in additive manufacturing, and a new multi-year project is underway for it to be fully "additive ready" by 2023. Additionally, powder blown directed energy deposition and binder jet AM trials are planned. C64 powder and print



**Fig. 3** — Single tooth bending fatigue gears made of Ferrium C64, processed via additive manufacturing on an EOS M 290 machine, heat treated and superfinished.

parameters are commercially available today for entities interested in printing gears or other high hardness applications, such as tools and dies where maraging steel, tool steel, or stainless steel are not ideal material choices.

Alongside additive manufacturing, QuesTek is further enhancing its ICME offerings. The company's ICME cloud-based software suite, currently under development, will bring physics-based models and ICME technologies directly to industry. The new suite will enable efficient modeling and design across all alloy systems to resolve the most pressing challenges and support the design of new materials. ~AM&P

**Note:** Co-authors include Chris Kantner, Jeff Grabowski, and Kerem Taskin of QuesTek Innovations LLC.

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# GELATIN METHACRYLATE HYDROGELS FOR 3D BIOPRINTING

#### Because the photopolymerization of gelatin methacrylate can be spatially and temporally controlled, it can be fabricated into hydrogels well suited to a variety of biomedical applications.

Pete Gabriel L. Ledesma, University of the Philippines Diliman Kai-Hung Yang, North Carolina State University Roger J. Narayan, FASM,\* University of North Carolina and North <mark>Carolina State University</mark>

ydrogels are made of a 3D network of crosslinked hydrophilic polymers that can absorb and retain large amounts of water and still remain insoluble<sup>[1]</sup>. Using different chemical and physical crosslinking strategies, numerous hydrogels have been developed to address a variety of biomedical needs such as 3D cell culture, tissue engineering, controlled drug delivery, and biosensing, among others<sup>[2-6]</sup>. In order to be suitable for these biomedical applications, hydrogels need to mimic the native extracellular matrix (ECM). The ECM is a fibrous 3D network of proteins and polysaccharides that provide cells and tissues with mechanical support, signaling cues, as well as a medium for growth and interaction. In this regard, hydrogels based on naturally occurring polymers have an advantage over synthetic polymers due to their inherent biocompatibility<sup>[7]</sup>.

One such material is gelatin methacrylate (GelMA)—a photopolymerizable hydrogel derived from gelatin. Gelatin is a biopolymer that is a product of the hydrolytic degradation of the triple helical tropocollagen rod. Upon heating (> 40°C), gelatin becomes soluble in water and takes on a randomly coiled structure. At a sufficiently high concentration, cooling of the solution produces a transparent gel as the gelatin partially recovers its helical structure  $\ensuremath{^{[8-10]}}$  .

Of particular interest is the addition of methacrylate groups to the amino  $(-NH_2)$  and hydroxyl (-OH) side chains of gelatin, which impart to it the property of photo-crosslinking. Upon exposure to light and with the presence of a photoinitiator, a hydrogel (i.e., Gel-MA) that is stable at body temperature (37°C) is formed. The photopolymerization of GelMA can be spatially and temporally controlled. As a result, it can be fabricated into hydrogels with purposeful architecture for a variety of biomedical applications<sup>[8,9]</sup>.

#### GeIMA HYDROGEL Preparation

Although various protocols have been reported for the synthesis of Gel-MA, they are essentially variations of the general method that was first reported by Van den Bulcke et al.<sup>[8,11,12]</sup> In brief, the production of GelMA is a two-step process that involves the derivatization of gelatin by methacrylic anhydride (MA), followed by a crosslinking process.

The reaction of methacrylic anhydride with gelatin in a phosphate buffer (pH = 7.4) solution at 50°C attaches methacryloyl units to the -NH<sub>3</sub> and -OH groups of gelatin. Diluting the reaction mixture (~5X) with phosphate buffer stops the reaction. To eliminate low molecular weight impurities, the solution is then dialyzed against deionized water. This dialyzed solution can be freeze dried and refrigerated until use. Finally, to produce the GelMA hydrogel, a water soluble photoinitiator is added and the solution is irradiated with visible light, ultraviolet light,  $\gamma$ -radiation, or an electron beam to initiate the photopolymerization process. The most common photoinitiators are 2-hydroxy-1-[4-(2hydroxyethoxy) phenyl]-2-methyl-1-propanone (Irgacure 2959) and lithium acylphosphinate salt (LAP)<sup>[8,11,12]</sup>.

By changing the amount of MA added, different degrees of methacryloyl substitution can be achieved; this approach produces GelMA with diverse physical properties. In addition, the rate of MA addition and conditions of mixing (e.g., pH and temperature) led to changes in the degree of methacryloyl substitution<sup>[13,14]</sup>.

#### GeIMA BIOPRINTING Modalities

Bioprinting can be achieved through a variety of techniques. These can be broadly classified into extrusion bioprinting, inkjet bioprinting, stereolithography, and laser-assisted bioprinting. In extrusion bioprinting, a piston, screw, or pneumatic pressure pushes a material filament through a nozzle and deposits it onto a print surface<sup>[15]</sup>. In inkjet bioprinting, a thermal, piezoelectric, or electromagnetic source produces bubbles that collapse and eject droplets of materials<sup>[16]</sup>.

In stereolithography (SLA), a 3D structure is formed out of a vat of resin using polymerization from exposure to light from a laser or lamp. A single beam or 2D pattern of ultraviolet or visible light is directed at the resin. This controlled light interacts with the dissolved radical-generating photoinitiators and polymerizes the resin in a specific pattern. Then, the printing platform is moved to allow unpolymerized resin to flow over the top of the structure, enabling repetition of the photopolymerization process for the next layer<sup>[17]</sup>.

In laser-assisted bioprinting (LAB) or laser-induced forward transfer (LIFT), a laser is pulsed into an energy-absorbing material that is attached to a thin sheet of cell-laden ink. The top layer absorbs the energy from the laser pulse to induce a phase change and deformation of the energy-absorbing material. This process results in the ejection of ink onto the print surface<sup>[18]</sup>.

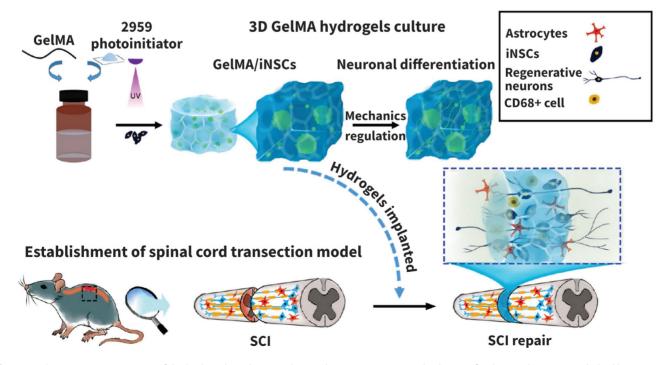
#### CELL-LADEN GeIMA APPLICATIONS

Gelatin methacrylate hydrogels have found a number of uses in tissue engineering and regenerative medicine. In neural cells, GelMA has been fabricated as nerve guidance conduits using digital light processing to support cell survival, proliferation, and migration<sup>[19]</sup>. For spinal cord injuries, 3D biomimetic GelMA hydrogels with induced neural stem cells (iNSCs) have been shown to promote cell regeneration. In vitro, the iNSCs encapsulated in GelMA (Gel-MA/iNSCs) survived and differentiated well. As shown in Fig. 1, in vivo mouse spinal cord transection models show that GelMA/iNSCs significantly promote functional recovery. The material also decreases inflammation by reducing activated CD68<sup>+</sup> cells<sup>[20]</sup>.

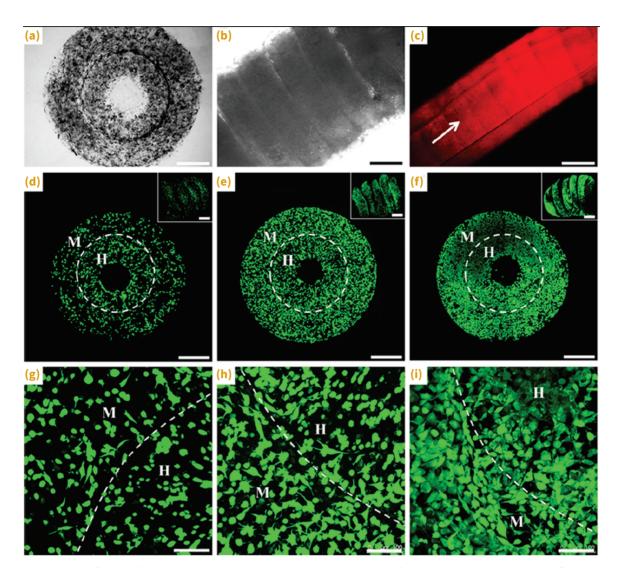
In cardiac tissue engineering, scaffolds and patches have been made based on GelMA hydrogels. For example, a hydrogel composite of reduced graphene oxide and GelMA (rGO-GelMA) demonstrates greater cell viability, proliferation, and maturation versus pure GelMA. This tissue construct can provide a high-fidelity in-vitro model for drug studies and basic cell biology research for understanding cardiac tissue development and other processes<sup>[21]</sup>. Conductive and adhesive cardiopatches have also been made by electrospinning GelMA and conjugating a choline-based bio-ionic liquid. These cardiopatches can minimize cardiac remodeling and preserve normal heart function by providing mechanical support and restoring electromechanical coupling at myocardial infarction sites<sup>[22]</sup>.

In skeletal muscles, acellular GelMA hydrogels have been printed and crosslinked in situ into the defect sites of mice with volumetric muscle loss injury. The GelMA scaffold exhibits proper adhesion to the surrounding tissue and promotes growth of remnant skeletal muscle cells<sup>[23]</sup>. In vascular networks, GelMA hydrogels have been used as an embedding scaffold for bone marrow-derived mesenchymal stem cells and human blood-derived endothelial colony-forming cells. In vitro, these 3D constructs generate an extensive capillary-like network<sup>[24]</sup>.

In bone tissue, a composite hydrogel containing GelMA and hydroxyapatite has been prepared into a biomimetic osteon, a type of bone



**Fig. 1** — Schematic representation of the hydrogel synthesis and animal experiment. A mixed solution of GelMA and iNSCs crosslinked by a photoinitiator under UV irradiation was developed. After generating the complete transection mouse SCI model, the scaffold was transplanted into the injury site. iNSCs = induced neural stem cells; and SCI = spinal cord injury. Courtesy of L. Fan et al.<sup>[20]</sup>



**Fig. 2** — Characterization of osteon-like double-ring modules: (a, b) Phase-contrast images of a single unit and an assembly; (c) fluorescent images under the rhodamine (red) perfusion; (d-f) confocal images of the cell-laden micropatterned unit for 1, 4, and 7 days, (insert) related assembly; and (g-i) higher magnification of confocal images. M = MG63s; H = human umbilical vascular endothelial cells. Cell seeding density:  $5 \times 106$  cells mL<sup>-1</sup>. Scale bars: 500 (a-f) and 100 µm (g-i). Courtesy of Y. Zuo et al.<sup>[25]</sup>

structure. In this bone scaffold, an inner ring with encapsulated human umbilical vascular endothelial cells (HUVECs) act as the blood vessel tubule. The outer ring with encapsulated human osteoblast-like cells serve as the bone<sup>[25]</sup>. As shown in Fig. 2, an osteon-like unit with a double-ring morphology was created; it features a 2 mm outer diameter, 1.2 mm middle diameter, and a 500 µm inner diameter. Human umbilical vein endothelial cells are located in the inner ring of the structure and MG63 osteoblast-like cells are located in the outer ring. The structure exhibits high cell densities seven days after cell encapsulation.

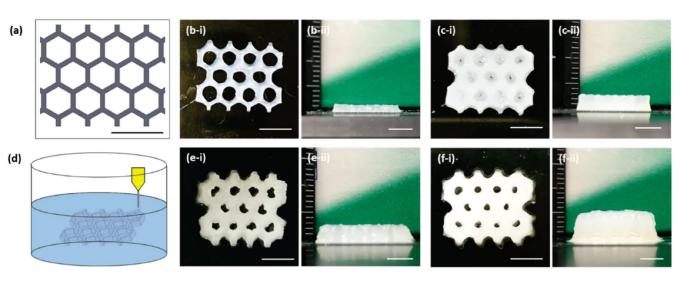
For cartilage regeneration, oligomers of dopamine methacrylate (ODMA)

have been intercalated into GelMA hydrogel chains (ODMA-GelMA) to create a scaffold. These ODMA-GelMA hydrogels have been shown to promote in vivo mesenchymal stem cell adhesion and growth as well as cartilage regeneration after chrondoitin sulfate or TGF- $\beta$ 3 loading<sup>[26]</sup>.

In epidermal tissue engineering, GelMA hydrogels have been found to support keratinocyte growth, differentiation, and stratification into a reconstructed multilayered epidermis. These keratinocyte laden hydrogels can be used as epidermal substitutes and substrates for in vitro skin models<sup>[27]</sup>. GelMA hydrogels have also been used as scaffolds for wound healing. By electrospinning GelMA into 3D fibers, structures have been created that support cell growth, adhesion, migration, and proliferation. Using these materials, formation of cutaneous tissues is accelerated<sup>[28]</sup>.

In liver tissue, composites of GelMA, alginate, and cellulose nanocrystals have been constructed as bicellular liver lobule mimetic structures using microextrusion bioprinting. Figure 3 shows honeycomb architectures with 0.48 mm wall thickness that were created using the hybrid ink. This hybrid hydrogel can be used in tissue engineering and for the culture of liver cells for drug discovery research<sup>[29]</sup>.

In lung tissue, GelMA has been fabricated as a highly adhesive, biocompatible, and biodegradable sealant that can stop air leaks and support tissue



**Fig. 3** — Printability of the hybrid bioink: (a) Schematic of the liver lobule-mimetic honeycomb structure; (b-i, c-i) top and (b-ii, c-ii) side views of the freeform printed structures with a height of 1.8 and 3.4 mm, respectively; (d) schematic illustration of embedded printing of the honeycomb structure; and (e-i, f-i) top and (e-ii, f-ii) side views of the embedded printed structures with a height of 3.75 and 6.8 mm, respectively. Scale bars: 5 mm. Courtesy of Y. Wu et al.<sup>[29]</sup>

regeneration. In vivo experiments involving small and large animal models show that GelMA effectively seals lung leakages with improved performance versus fibrin glue, poly(ethylene glycol) glue, and sutures only<sup>[30]</sup>.

Based on this set of promising results, additional applications of bioprinted GeIMA for tissue engineering are expected over the coming years. ~AM&P

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# SUSTAINABLE MATERIALS AND THE CIRCULAR ECONOMY

The circular economy and sustainability are multifaceted concepts and thus should be pursued through collective advancements across various scientific fields.

Tamil Selvan Sakthivel and Sudipta Seal, FASM,\* University of Central Florida Seeram Ramakrishna, FASM,\* National University of Singapore

n December 2015, multiple nations signed the landmark Paris Agreement to intensify actions needed for a sustainable low-carbon future in order to combat extreme weather. If the nations fulfill their promises, the global economy is anticipated to gain \$400 trillion by 2100; if they fail, it will cost an estimated \$600 trillion<sup>[1]</sup>. The primary objective of sustainability efforts and the circular economy (CE) approach is to enable a low-carbon, modern society in which the technologies and business models least compromise the needs of future generations. In other words, intelligent product design could reduce waste generation and resource needs for greater sustainability.

Micro and nanotechnologies are akin to modern societal development. The worldwide science and engineering community should accept responsibility for developing emerging technologies to realize the circular economy, which is characterized by a pollution-free environment and low-carbon resources for the survival and sustainability of life on Earth.

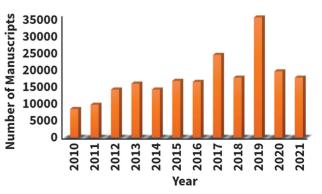
Developing a circular economy around materials at various scales (nano to micro) is the focus of the hour, desire of the day, and need of the century. The University of Central Florida (UCF) and the National University of Singapore (NUS) jointly organized a three-day workshop on the circular economy and sustainability of nanomicro materials<sup>[2]</sup>. At the same time, the Ministry of Environment and Water Resources of Singapore released the Zero Waste Masterplan. It defines Singapore's objective to promote recyclability to 70% by 2030, contrasted with the current  $60\%^{[3]}$ .

Ongoing global and national activities proclaim the circular economy as the new focal pathway toward sustainable methods of creation and utilization. Skeptics question the methodology, despite its accomplishments in catalyzing conversations about product design (e.g., life span, reparability), choices for sharing products, and repositioning products as services<sup>[4]</sup>. Figure 1 indicates the total number of existing pieces of literature on "circular economy and sustainability," showing incremental research interest in the last 10 years (January 2010 - August 2021).

The CE approach has gained steady momentum and popularity and has established four material pillars as its foundation: regenerating resources, maximizing resources' lifetimes, mining resources from waste, and substituting with materials from renewable sources. In parallel, materi-

als science and engineering research has started engaging artificial intelligence and data analytics as new tools. Therefore, a new platform is to be established in the emerging areas of artificial intelligence and data science-driven education and research, as well as a repositioning of the manufacturing sector to utilize the opportunities of cyberphysical space (i.e., the fourth industrial revolution). Combining nanotechnology with the circular economy would provide a platform for sharing scientific knowledge related to reuse, recycle, redesign, remanufacture, reduce, and recover (the 6 Rs), in addition to life cycle engineering and life cycle assessment of sustainable materials, thus minimizing damage to the environment and human health.

This circular model would remove waste and raise efficiency by focusing on: the reuse and recycling of materials; product design to highlight longevity and restoration; and the production of new plans of action, including the sharing economy and the expansion of local closed-loop frameworks (Fig. 2). The advantage of adopting the CE model consists of additional business opportunities through new product creation and enhanced resource effectiveness, diminished dependence on uncommon materials and different products, and



**Fig. 1** — Bibliography review of the keyphrase "circular economy and sustainability" (data collected from Google Scholar, January 2010 - August 28, 2021).

the formation of highly skilled occupation development.

A 2015 McKinsey and Ellen Mac-Arthur Foundation joint investigation, "Growth Within: A Circular Economy Vision for a Competitive Europe," estimates that for the European Union (EU) alone, progressing to a circular economy could include \$1.8 trillion in value by 2030, reduce essential material utilization by 53% by 2050, and lower CO<sub>2</sub> emissions by 83% by 2050. The CE presents a truly win-win circumstance for the planet and individuals to benefit<sup>[5]</sup>.

#### **SIGNIFICANT RESULTS**

The CE has received expanded consideration in academic research and industrial manufacturing, with a focus on closed-loop value and supply chains, including circular business models and product design. The Circular Economy and Sustainability meeting highlighted the unique added values of green nanostructured materials and products in reducing waste materials throughout product and material life cycles, from production to post-consumption. Researchers illustrated the utility of such nano-micro materials through the use of material byproducts or recycled materials as starting materials. Beyond addressing zero waste/waste neutrality objectives, researchers also illustrated green materials and practices to reduce energy requirements. Academic and industrial specialists alike energetically shared a common set of grand challenges in the development of a successful circular economy, including: needed advancements in nano-micro material choice and product-process design; the use of computational methods in optimizing industrial methods to join the ends of a product's life cycle; and enabling products and materials to be used for longer periods.

The technological advances in resource recovery and recycling for materials sustainability have been discussed by several researchers. Specifically, the National Science Foundation's Industry/University Collaborative Research Center on Resource Recovery & Recycling described the technological developments made to convert valuable production wastes into functional manufactured materials for industrial applications. For example, a gas-based reduction process can be used to recover < 60% of high-value magnetite from metallic iron waste<sup>[6]</sup>. Researchers have also highlighted the educational and entrepreneurial dimensions of chemical sustainability by broadly developing the fundamental science and applications of green oxidation catalysis to advance water purification and the environmental, materials, and synthetic spaces<sup>[7]</sup>. Furthermore, it was emphasized that the details of recent and upcoming subcommittee projects will mainstream sustainability among materials researchers. This will include strategies to enhance awareness and understanding of sustainability among materials researchers and training the next generation of materials scientists to incorporate sustainability in their work<sup>[8]</sup>.

The merits of product life cycle assessment (LCA) were discussed in relation to true sustainability or the circularity of various nano-products. For example, a life cycle assessment study by the Danish Environmental Protection Agency advised that a polypropylene bag, a paper bag, and a cotton bag ought to be utilized 37, 43, and 7100 times, respectively<sup>[9]</sup>. Natural biomass-sourced biodegradable polymers provide an innovative option in contrast to single-use plastic sacks. Industry and

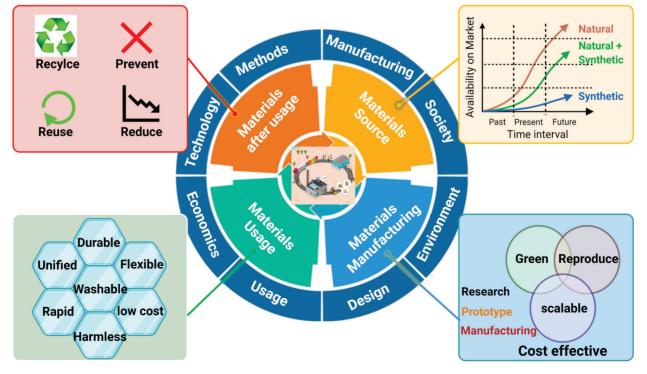


Fig. 2 — Schematic diagram of a circular economic model for sustainable materials development.

academic researchers also stressed that consumers should consciously lower their plastics usage by implementing the 3Rs—reduce, reuse, and recycle—in their day-to-day activities<sup>[10]</sup>.

Another LCA investigation suggests that importing fresh meat such as pork from long distances adds to multiple-times higher CO, emissions than bringing it in from nearby places<sup>[11]</sup>. Likewise, about 60% of the energy consumed in transporting food to Southeast Asia (i.e., Singapore) goes toward fresh air-freighted meats and fish, which represents 3.7% of the food consumed<sup>[11]</sup>. Perhaps sourcing food from neighboring nations or producing it locally will reduce the carbon impact using a nanotechnology approach. It is further recognized that plant protein-based or vegetarian-refined meats have a lower carbon impression than animal-sourced meat.

Subsequently, a panel discussion emphasized these issues, along with the necessity of public inclusion and involvement in addressing the challenges<sup>[12]</sup>. Panel members stressed the development of standards and guidelines as a framework for the successful development of a circular economy in which researchers and industries can amend their present work models, aligning individual endeavors with the overarching common good. Another suggestion was to introduce circular economy principles to ISO standards that can be followed for manufacturing industry business models. It was further mentioned that the circular economy needs to advance the utilization of as many biodegradable materials as would be prudent in manufacturing products (i.e., biological nutrients), so they can return to nature without causing ecological damage toward the end of their useful life.

Panelists also talked about the idea that sustainability concepts are not integrated into materials science or other science and engineering curricula. A suggestion was to integrate the fundamental educational framework into the technical knowledge around sustainability, including system thinking, analytical thinking, leadership, and a framework to tackle challenging and complex problems. The panel also discussed the essential role that nanomaterial technologies play in addressing key issues facing our present and future society. In particular, panelists shared the dire need for a sustainable supply of resources (e.g., energy, water, and products) and infrastructure (e.g., transportation, buildings, and health services) in relation to technological progress.

The conference further highlighted that the greatest volume of greenhouse gases is produced by the industrial sector, suggesting substantial value in focusing green/sustainable efforts on modifying industrial practices. For example, cooperation or networking among manufacturing industries could allow waste-to-value materials exchange at scale with the industry. Intel and Ricoh shared their separate endeavors in embracing manageable procedures and plans of action for accomplishing circularity<sup>[10,13]</sup>.

Intel, a U.S.-based pioneer in microelectronics fabricating, has executed sustainable advancements over its extensive environment of manufacturing, innovation improvement, and worldwide supply chain<sup>[13]</sup>. The company improved the wet procedure packing industry in Southeast Asia by reducing the use of unsafe chemical consumables and water with greener options and a dry procedure, respectively. The annual size of the digital universe (i.e., the information humans make and duplicate) is anticipated to arrive at 180 zettabytes by 2025 due to the huge progression of information. This brings about increasing requirements for energy-productive processing and memory, with a lower asset impression. Groundbreaking innovations in this domain are essential for the future of our digital universe, and Intel is currently researching this area<sup>[13]</sup>.

Ricoh, a printer product company from Japan, is enlisting the 3Rs currently to improve its resource preservation to 50% by 2030. Ricoh advancements also incorporate efforts to decrease the size and weight of products and packaging materials, repair utilized items, reuse parts in great condition, and IN PARTICULAR, PANELISTS SHARED THE DIRE NEED FOR A SUSTAINABLE SUPPLY OF RESOURCES (E.G., ENERGY, WATER, AND PRODUCTS) AND INFRASTRUCTURE (E.G., TRANSPORTATION, BUILDINGS, AND HEALTH SERVICES) IN RELATION TO TECHNOLOGICAL PROGRESS.

reuse the plastic and metal waste while understanding their connections with a typical eco-framework<sup>[10,13]</sup>.

Based on the findings from the CE meeting, the authors explained in the *European Business Review* how a circular economy is beneficial to humankind for a sustainable future in the long run, as well as the different opportunities that the industrial and research sectors will unveil with this transition<sup>[14]</sup>. The deliberations of the conference also led to a textbook on the circular economy for university students and beginners in the field<sup>[15]</sup>.

#### CONCLUSION

The United Nations' Sustainable Development Goals stand as a model for academic researchers and industrial manufacturers to adjust their interests and approaches in improving the state of the planet. The concepts of the circular economy and sustainability are multifaceted and thus ought to be taught using a multidisciplinary educational plan and pursued through collective advancements across various scientific fields. Infusing the principles of sustainable practice into the lifestyles of the younger generation will further support the proposed and discussed challenges to realize sustainable, livable cities and societies in the future. ~AM&P

Acknowledgment: The authors would like to acknowledge the timely support and encouragement received from the National Science Foundation (NSF Award # 1929899); Agency for Science, Technology and Research, University of Central Florida; and the National University of Singapore.

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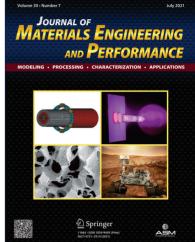


### ADDITIVE MANUFACTURING HIGHLIGHTS IN *JMEP*

he July and September issues of the *Journal of Materials Engineering and Performance (JMEP)* were special issues focused on additive manufacturing. The issues were guest edited by William E. Frazier, FASM, Pilgrim Consulting LLC and editor of *JMEP*; Rick Russell, NASA; Yan Lu, NIST; Brandon D. Ribic, America Makes; and Caroline Vail, NSWC Carderock. These highlighted articles were selected by Dr. Frazier to showcase the varied topics covered in the special issues. *JMEP* is available online at https://link.springer.com/journal/11665/.



Frazier



#### DEVELOPMENT OF BIOMEDICAL IMPLANTS THROUGH ADDITIVE MANUFACTURING: A REVIEW

#### M. Vignesh, G. Ranjith Kumar, M. Sathishkumar, M. Manikandan, G. Rajyalakshmi, R. Ramanujam, and N. Arivazhagan

Additive manufacturing (AM) is an expeditiously developing technology for the manufacturing of biomedical implants. But there are some issues related to metal AM including desired dimensional accuracy, preferred surface quality, strength, etc. Based on these shortcomings, the following properties are chosen for this review article: mechanical and metallurgical behavior. The main objective is to review the above-stated properties for 3D printed biomedical implants manufactured through laser additive manufacturing, friction stir additive manufacturing, paste extruding deposition, and selective laser melting techniques and its future scope of AM processes.

#### FLAW IDENTIFICATION IN ADDITIVELY MANUFACTURED PARTS USING X-RAY COMPUTED TOMOGRAPHY AND DESTRUCTIVE SERIAL SECTIONING

#### Zackary Snow, Jayme Keist, Griffin Jones, Rachel Reed, Edward Reutzel, and Veeraraghavan Sundar

In additive manufacturing, internal flaws that form during processing can have a detrimental impact on the resulting fatigue behavior of the component. In this study, the authors compare XCT scans and automated flaw recognition analysis of the corresponding data to results obtained from an automated mechanical polishing-based serial sectioning system. The results point to the need to recognize the limitations of XCT and for supplementary XCT scan quality metrics in addition to the voxel size.

#### DESIGN OF AN INNOVATIVE OXIDE DISPERSION STRENGTHENED AL ALLOY FOR SELECTIVE LASER MELTING TO PRODUCE LIGHTER COMPONENTS FOR THE RAILWAY SECTOR

#### Roberto Sorci, Oriana Tassa, Alessandro Colaneri, Alessandro Astri, Daphne Mirabile, Simon Iwnicki, and Ali Gökhan Demir

The railway industry can take advantage of additive manufacturing processes from several perspectives such as the production of spare parts on demand or the use of lightweight structures for vibration and noise control. Selective laser melting (SLM) is a metal AM process with industrial maturity where material development can open new prospects for the railway industry. In order to respond to such requirements, this study proposes a framework to study a new material from concept to the processability and finally to the preliminary mechanical characterization of alloy for SLM. The results showed that samples produced by SLM were characterized by > 1% porosity. Compared to the reference Al alloy, an increase up to 20% in microhardness was achieved for ODS samples made by the SLM process.



## **CONNECT** GET ENGAGED, GET INVOLVED, GET CONNECTED

Join the conversation about additive manufacturing. ASM International is exploring the launch of a technical community or committee on additive manufacturing to coordinate ASM activities in this area and to provide new collaboration and networking opportunities for members involved in additive technologies. William E. Frazier, FASM, is leading this effort. If you are interested in getting involved, please visit ASM Connect and complete a brief volunteer interest form, https://bit. ly/3ESZaO4.

## AN EXPERIMENTAL STUDY ON MECHANICAL, THERMAL AND FLAME-RETARDANT PROPERTIES OF 3D-PRINTED GLASS-FIBER-REINFORCED POLYMER COMPOSITES

### Ashish R. Prajapati, Harshit K. Dave, and Harit K. Raval

Fused deposition modeling (FDM) is one of the most common 3D printing processes. Currently, several modifications have been applied to the FDM process where it can manufacture fiber-reinforced polymer composites (FRPC). This paper aims to analyze the mechanical, thermal, and flameretardant properties of FRPC manufactured by the FDM process. Polymer composite filament (a mixture of nylon polymer and short carbon fibers) is used as a matrix, while fiberglass and high-strength, high-temperature fiberglass are used as a fiber reinforcement. A MarkTwo 3D printer from Markforged is employed to create the FRPC parts. The change in different mechanical properties, namely, tensile and impact of polymer composite matrix by adding

different fibers, is studied. In addition, thermogravimetric analysis, x-ray diffraction and flammability testing are also carried out to analyze the thermal properties of composites.

## MELT POOL AND HEAT TREATMENT OPTIMIZATION FOR THE FABRICATION OF HIGH-STRENGTH AND HIGH-TOUGHNESS ADDITIVELY MANUFACTURED 4340 STEEL

### Matthew A. Ryder, Colt J. Montgomery, Michael J. Brand, John S. Carpenter, Peggy E. Jones, Anthony G. Spangenberger, and Diana A. Lados

This study focuses on 4340 steel fabricated using laser powder bed fusion (LPBF); 42 laser power and scan speed combinations have been systematically investigated to optimize the melt pool geometry and ensure fully dense parts. The AM material is compared with a wrought 4340 equivalent and studied in both as-fabricated and heat-treated conditions. Two heat treatments were designed and used to optimize the materials for strength and toughness, respectively. Microstructure, tensile, and fractographic studies are conducted to assess build integrity and establish processing-structure-performance relationships. Tensile properties of the AM materials in all studied conditions were equivalent or better than the comparable wrought materials. The high performance of the AM materials was attributed to the absence of both manganese sulfide inclusions and rolling-induced banding, typically found in the wrought materials.

## VAT-PHOTOPOLYMERIZATION-BASED CERAMIC MANUFACTURING

### Xiangjia Li and Yong Chen

This article presents a detailed account of the processes involved in vat-photopolymerization-based fabrication of ceramics, namely bioceramics, structural ceramics, piezoelectric ceramics, optical ceramics, and polymerderived ceramics. Information and methods of material preparation, curing characteristics, greenpart fabrication, property identification, process design and planning, and quality control and optimization are introduced. The article also provides information on postprocessing techniques, namely debinding and sintering, as well as on the phenomenon of shrinkage and compensation. ~AM&P

## ASM HANDBOOKS ON Additive Manufacturing

In 2020, the Society published ASM Handbook, Volume 24, Additive Manufacturing Processes, its first full volume on additive technologies. Two additional AM volumes are in development:

- Volume 23A, *Additive Manufacturing in Biomedical Applications,* planned for release in 2022
- Volume 24A, *Additive Manufacturing Design and Application* (publication date TBD)

Contact David Vargas at david. vargas@asminternational.org and Amy Nolan at amy.nolan@asminternational. org for details on contributing to Volume 24A. Coverage includes reference papers and case histories in the areas of Design, Metals Process Development (PBF, DED, binder jet), Economic and Business Considerations, Material Characterization, In-Situ Monitoring, and Applications (Aviation, Spaceflight, Medical, Automotive and Heavy Machinery, Oil/Gas and Maritime, Construction, Energy, Electronics).



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THE OFFICIAL NEWSLETTER OF THE INTERNATIONAL ORGANIZATION ON SHAPE MEMORY AND SUPERELASTIC TECHNOLOGIES

# LASER TREATMENT OF ORTHODONTIC NITALLOY DEVICES

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### EDITORIAL OPPORTUNITIES FOR SMST NEWSWIRE IN 2022

The editorial focus for the *SMST NewsWire* in 2022 reflects shape memory and superelasticity technologies for biomedical, actuator applications, and emerging markets.

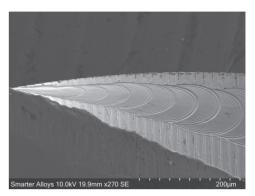
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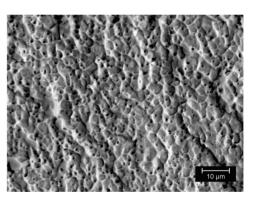
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## LASER PROCESSING FOR Shape Memory Alloys IN Dentistry



## **10** FATIGUE FRACTURE OF NITINOL



## **DEPARTMENTS**

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## **ABOUT THE COVER**

These SmartArch orthodontic archwires, made with copper NiTi, are fabricated by laser processing technology to deliver biomechanically optimized pseudoelastic stress to each tooth. Courtesy of Smarter Alloys.

## SMST NEWS WRE GUEST EDITORIAL

## FROM A STUDENT'S PERSPECTIVE

Can you hear me? Oh, I was double muted." "All I see is your desktop wallpaper. Nice tiger, by the way." "Would everyone, please, mute their mics?"

This last year has been a wild ride for everyone, first converting to virtual and, with extreme caution, slowly reverting to normalcy. I realized a great dynamic will be apparent with the next round of students when consider-



Gantz

ing what is "normal." In my experience, only my graduate schooling was virtual, thus providing a "normal" undergraduate experience. However, the up-and-coming graduates or green professionals will have a split or mostly virtual experience, depending on the local jurisdiction. What is considered "normal" behavior, customs, and routines will vary greatly from student to student. Acknowledging this dynamic should refocus attention to the evaluation process criteria. Doing so may help achieve greater diversity when considering who is an exemplary student, an inspiring researcher, and an uplifting work-family member. As an example, the virtual setting has allowed the option of virtual internships for students who ordinarily may not have considered applying due to financial, family, or health reasons. Virtual internships or other virtual activities have broadened the type of applicants to include the full spectrum between traditional and nontraditional students. Noting the benefits is especially important when considering how inclusion is a fervent and critical topic to the representation of every organization and association.

The intention behind my message is to give credit to those working diligently and with great decorum toward making student outreach opportunities more accessible. I had my fair share of conference experiences during my undergrad years, but as time goes on, the option to travel has become more complicated. I am a returning student, above the typical age range, a parent, and a spouse to someone whose occupation has been affected by the pandemic.

During these trying times, I know my professional progress would have become stagnant if not for the connections made within SMST, the broader shape memory community, and their associations. Within SMST, I will have the privilege of acting as student board member for two consecutive years. During this experience, including the 2019 conference and the CASMART challenges, I have met prominent professionals who continue to pave a path for the next generation in shape memory. More recently, I was offered a virtual internship with NASA to dissect mechanisms influencing shape memory properties. The achievements reflect not only my own efforts but, also, to those continuing to extend their hands, my mentors.

Here is a question for your consideration: How are you paving a path or building a bridge for the next generation? Whether you are a teacher looking to assist your students, or you're considering graduate school, or you are looking for recruitment opportunities, consider sharing the following resources and events hosted by ASM International and the SMST organization. Through the SMST Community on ASM Connect, we can share, boast, and highlight information and resources, or casually share coffee break banter. Students can join the platform-among many other benefits—if they are a member. Visit www.asminternational.org/ students. Another opportunity for student involvement is the CASMART 5th Design Challenge. For the first time, the challenge is available for two consecutive years to offer students a chance to present their work at the SMST May 2022 conference. Visit the event site at asminternational.org/ web/smst-2022. The upcoming year is exceptionally exciting with the celebration of 60 exciting years of Nitinol. Use these tools to stay connected, make connections, and help the community grow!

I hope you find motivation in your research and in those whose careers you will elevate. Please enjoy this issue of the SMST NewsWire.

#### **Faith Gantz**

SMST 2020-2022 Student Board Member

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## SHAPE MEMORY SOCIETY NEWS SMST NEWS

## SMST MEMBERS NAMED 2021 ASM FELLOWS AND AWARDEES

SMST is pleased to announce that several of its members have joined the newest Class of Fellows or have been named a recipient of a 2021 ASM Award. Congratulations to all!

### **2021 CLASS OF ASM FELLOWS**



**Prof. Peter M. Anderson, FASM** Professor The Ohio State University Columbus

"For outstanding contributions in the development of dislocation theory in engineered shape memory alloys, multilayers, and nanocrystalline metals and

as an educator and mentor, who promotes diversity within the University engineering undergraduate and graduate programs and department."

### **2021 ASM AWARD PROGRAM RECIPIENTS**

### Engineering Materials Achievement Award QuesTek Innovations LLC, Evanston, Ill.

Jeff Grabowski, **Chris Kern,** Dr. Thomas S. Kozmel, Prof. Gregory B. Olson, FASM, **Dr. Jason T. Sebastian,** and Kerem Taskin. SMST members names are bold.

"For the design and commercialization of novel highperformance carburizable steels enabling more durable, lighter weight transmission gears with increased power density."



Kern



Sebastian

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

### SMST SEEKS BOARD NOMINATIONS

SMST is seeking nominations for one vice-president/ finance officer to serve a two-year term, and two directors to serve three-year terms. Terms begin October 1, 2022. Any member of SMST in good standing is encouraged to nominate themselves or another member for one of these positions. The incumbents may also seek reappointment by notifying the SMST Awards and Nominations Committee Chair, Jeremy Schaffer at jeremy\_schaffer@fwmetals.com. For more information on eligibility and how to submit a nomination, visit the SMST website as smst.asminternational.org.

## 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 2022-2023 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 February 15, 2022.** For more information on eligibility and benefits visit smst. asminternational.org.

### SMST ANNOUNCES NEW OFFICERS AND BOARD MEMBERS

The Shape Memory & Superelastic Technologies (SMST) board, at the recommendation of the SMST Awards and Nominations Committee, named a new member **Timofei Chekalkin** to serve on the SMST Board for the 2021-2024 term; reappointed is **Andreas Undisz** to serve on the SMST Board for the 2021-2024 term; and reappointed is **Faith Gantz** to serve as Student Board Member for a one-year term 2021-2022. Continuing on the board are **Othmane Benafan** (president), **Adrian McMahon** (vice president), and **Jeremy Schaffer** (immediate past president). Continuing on the board as members are **Ashley Bucsek**, **Tom Duerig**, **FASM, Parikshith Kumar, Jochen Ulmer,** and **Martin Wagner.** Retiring from the board is Frederick Calkins and Aaron Stebner. Alan Pelton and Darel Hodgson will continue to be advisors on the board.

**Timofei Chekalkin** is a team leader at the R&D Center of TiNiKo Co. (formerly Kang & Pand Medical Co.) in Ochang, South Korea. Most of his career has focused on developing, testing, processing, and researching the properties of wrought and porous Nitinol used in clinical applications. He collaborates directly with colleagues from the medical device industry and academia to design appropriate Nitinol products and optimize material properties for their specific applications. He received his diploma (1999) and

## SMST NEWSWRE

## **SHAPE MEMORY SOCIETY NEWS**

Ph.D. (2007) in solid state physics from the National Research Tomsk State University, Russia. He then worked as a research associate at the Research Institute of Medical Materials (Tomsk) for porous/solid NiTi-based shape memory implants research. In 2009, he joined the Lab of Shape Memory Materials at the Research Tomsk State University, first as a guest scientist and later as a senior research fellow. In 2015, he was invited to chair the R&D Center of Kang & Pand Medical Co. Chekalkin's research interests include thermodynamics and phase equilibria of NiTi-related alloy systems, experimental procedures, and in situ devices and equipment with FEM-based technique simulation tools. He has authored and co-authored more than 40 papers, four book chapters, 11 monographs, and eight patents.

Andreas Undisz studied materials science at Friedrich Schiller University (Jena, Germany). He completed his Ph.D. thesis on "Optimization of Structural and Functional Properties of NiTi Alloys for Medical Application" in 2009. In 2010, he was conferred a Feodor Lynen-Fellowship by Alexander von Humboldt-Foundation to proceed with his research at Massachusetts Institute of Technology. At MIT, Undisz broadened his research background toward biology and biomechanics. After his return to Friedrich Schiller University, he continued his research on NiTi alloys as head of the Metals and Biology Group and received the Habilitation for his research on the variability of natural NiTi surfaces. In 2020, Undisz accepted an appointment as full professor in the field of electron microscopy and microstructural analysis at Chemnitz University of Technology (Germany). His teaching activities include lectures, seminars, and lab courses on materials science and microstructural analysis, materials for medical application, and electron microscopy.

Faith Gantz is a Ph.D. student of materials science and engineering at the University of North Texas. Her research is focused on processing and characterization of shape memory alloys with the goal of improving actuation fatigue life. She has support from the NASA ULI program in developing high temperature shape memory alloys for aerospace applications. Gantz's previous projects include collaborating with the Dallas Museum of Art Ceramic on their Metal Work Collection and with the Forging Foundation on high-entropy alloy (HEAs) research. The research produced by laser coating HEAs onto forging die heads earned her the Charles W. Finkl Scholarship sponsored by the Forging Industry Educational and Research Foundation. She was introduced to working with shape memory alloys in her junior year and has continued working with these alloys for her master's thesis and Ph.D. As an undergraduate, Gantz had the opportunity to present her research on the effects of Ni in NiTiHf SMAs during thermo-mechanical processing at SMST 2019 where she won 2nd place in the poster competition. An internship at NASA Glenn Research Center (GRC) came shortly after, where she worked primarily on their shape memory alloys database. After receiving her master's, Gantz received another internship at GRC for two terms focused on studying the mechanism behind the hysteresis in shape memory alloys.



Chekalkin



Gantz



Benafan



McMahon



Schaffer



Undisz





Duerig



Kumar



Ulmer



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## SELECTIVE LASER PROCESSING FOR FUNCTIONALLY GRADED SHAPE MEMORY ALLOY MEDICAL DEVICES: PROCESS AND APPLICATIONS IN DENTISTRY

Novel methods for altering the mechanical properties of functionally graded orthodontic medical devices leads to new applications.

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unctional grading of shape memory alloys offers design flexibility that expands the application space of these materials. Several technologies have been employed to modify the composition and/or mechanical properties over volume; however, significant defects are introduced during these processes that limit their commercial application, including cracking, porosity, oxidation, and lack of control<sup>[1-6]</sup>. In addition to the added defects, these processes are expensive and difficult to scale. Recent progress in additive manufacturing technologies has shown promise in realizing functional grading of complex shapes and work continues to overcome obstacles to commercial scale processing<sup>[7]</sup>.

The authors recently described the use of a laser process to selectively vaporize constituent elements from binary Nickel-Titanium (NiTi) alloys<sup>[8]</sup>. Selective vaporization enables local control of mechanical properties of NiTi-based shape memory alloys by shifting the austenitemartensite transformation temperature range. During the process, pulsed laser energy rapidly increases the local temperatures, partially vaporizing each constituent element. Owing to its relatively lower vapor pressure, nickel

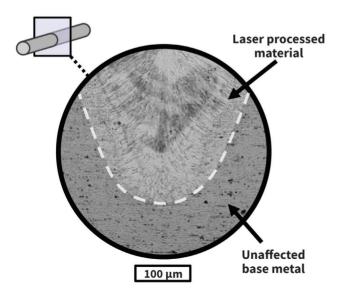


Fig. 1 — Metallurgical cross-section of laser processed wire.

evaporates faster than other elements. Subsequent rapid cooling and solidification forms a columnar dendritic microstructure with a decreased bulk nickel content, and corresponding higher transformation temperature. In contrast, the original base material surrounding the processed section remains mostly unchanged as shown in Fig. 1. The high spatial resolution of the laser process enables tight spatial control over the stiffness of a wire across very short distances. Because the transformation temperature is inversely related to the pseudoelastic plateau stress, the effect of laser processing would be to locally decrease the stiffness. In shape memory actuation applications, the effect is a multi-stage actuation. The process has been described as Multiple Memory Material.

The purpose of this article is to highlight recent applications of selective vaporization to enable functional grading in medical devices for the dental industry.

## **APPLICATIONS**

**Orthodontic archwires:** Since the earliest discussion of NiTi as a functional material in the 1960s, there has been significant interest in its use for biomechanical applications<sup>[9]</sup>. By 1971, the first-ever application of NiTi in medical devices was being investigated in an orthodontic archwire<sup>[10]</sup>. The first NiTi wires were fully cold worked and did not exhibit pseudoelasticity; however, the load-deflection rate was lower than stainless steel<sup>[11]</sup>. A NiTi archwire with pseudoelastic properties, ideal for providing continuous forces for long range tooth movement, was not realized until 1985<sup>[12,13]</sup> when modern NiTi alloys with finely tuned thermomechanical processing enabled the close control of transformation temperature.

The need for functionally graded archwires was recognized in 1981<sup>[14]</sup>, before widespread use of NiTi in orthodontics, with the theory that varying the material properties along the archwire, rather than the wire cross-section, could be used to optimize delivery of orthodontic forces. The resulting archwire could deliver ideal force proportions based on equalized stress distributions in the periodontal ligament

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(PDL)<sup>[15]</sup>. In contemporary orthodontics, the available force from an initial archwire targets resolving the malocclusion in smaller teeth first; thus, a progression in archwire size is required to achieve force levels to move larger teeth. In theory, a functionally graded archwire with stiffness that varies in the mesio-distal direction could produce the ideal available force for each tooth.

Previous attempts at fabrication of functionally graded archwires include welding of segmented wires and selective heat treatment. Fabrication of segmented wires using advanced orthodontic alloys is not trivial. Laser welding<sup>[16]</sup>, impact butt welding<sup>[17]</sup>, and resistance welding<sup>[18]</sup> techniques have been attempted; however, the mechanical properties in the weldments were degraded with a structural weakness that caused the wire to break. The direct electric resistance heat treatment (DERHT) process was used to develop continuous functionally graded archwires<sup>[19,20]</sup>. These archwires were heat treated selectively along the arch to achieve a low stiffness in the anterior section, progressing to a higher stiffness in the posterior section with a transition zone between them<sup>[21,22]</sup>. The process can be expensive and time consuming, and difficult to accurately control, causing high variation in the output<sup>[23]</sup>. Furthermore, due to conduction of heat through the material, there is low property gradient resolution and a limit of only three segments in an archwire is possible. The benefits of the selective laser vaporization method compared to the selective heat-treatment approach outlined above are that a higher resolution of stress gradients and greater range of force levels are possible. While each approach maintains the ideal load-deflection rate and large activation ranges of pseudoelastic NiTi, the former can vary the forces on a tooth-by-tooth basis, while the latter is limited to a gradually decreasing stiffness from the posterior to anterior segments.

A functionally graded archwire (0.016 in. diameter) was fabricated using the selective vaporization laser process to treat a segment between each tooth. Wire segments were produced and tested for force availability in a simulated orthodontic bracket setup. It was found that 10 different wire stiffness conditions deliver target forces at each tooth bracket. The available forces at each bracket for each wire stiffness condition are shown in Table 1, along with the austenitic transformation temperatures for the respective segment. The locations of the segments are shown in Fig. 2. Stress-strain results for each segment, presented in Fig. 3, show how the laser process depresses the pseudoelastic stress plateau differently for each condition. Results of clinical observations using a laser processed wire suggest that treatment kinetics are accelerated during the initial treatment phase while avoiding potential side effects that can delay tooth movement progress<sup>[24,25]</sup>. Orthodontic treatment time can be reduced by  $50\%^{[26]}$ , or 3 to 4 months^{[27]} during the leveling and aligning phase.

**Endodontic files:** Like orthodontic archwires, the files used for cleaning the root canal during endodontic procedures have evolved from stainless steel to predominantly

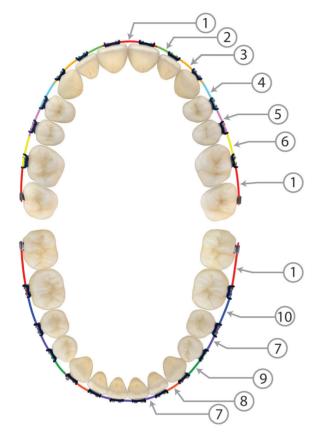
			Available force, N		Transformation T, °C		
Arch	Segment	Condition			A <sub>s</sub>	Ap	A <sub>f</sub>
Upper	Central incisor – Central incisor	1	U1 1.26		6	17	28
	Central incisor – Lateral incisor	2		U2 0.94	0	17	39
	Lateral incisor - Cuspid	3	U3 1.20		0	15	33
	Cuspid – 1st Premolar	4		U4 1.23	0	15	30
	1st Premolar – 2nd Premolar	5	U5 1.18		0	20	38
	2nd Premolar – 1st Molar	6			0	14	30
	1st Molar – 2nd Molar	1		U6 1.68	6	17	28
Lower	Central incisor – Central incisor	7	L1 0.81		1	22	37
	Central incisor – Lateral incisor	7		L2 0.80	1	22	37
	Lateral incisor - Cuspid	8	L3 1.03		1	22	36
	Canine – 1st Premolar	9		L4 1.08	0	15	32
	1st Premolar – 2nd Premolar	7	L5 1.13		1	22	37
	2nd Premolar – 1st Molar	10			0	14	30
	1st Molar – 2nd Molar	1		L6 1.79	6	17	28

## TABLE 1 – SUMMARY OF LASER PARAMETER CONDITIONS, FORCE AVAILABLE PER TOOTH, AND THERMAL ANALYSIS RESULTS PER SEGMENT

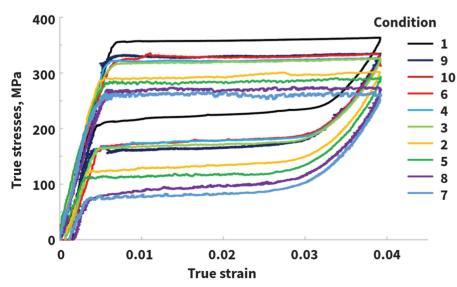
\*Note: A<sub>s</sub>, A<sub>p</sub>, and A<sub>f</sub> are the austenitic transformation start, peak, and finish temperatures, respectively. U1-6 corresponds to the tooth sequence from maxillary central incisor to first molar; the same is applicable for L1-6 in the mandibular arch.

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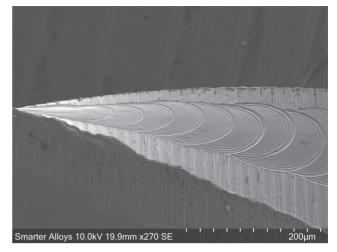
comprise pseudoelastic NiTi. Root canal shaping requires files with high strength, flexibility, and resistance to torsional fracture<sup>[28]</sup>. During the shaping procedure, cyclic stresses occur along the file length from rotation with bending inside the canal. The fatigue life depends on the degree of curvature of the canal<sup>[29]</sup>. Heat treatments are widely used to



**Fig. 2** — Location of stiffness conditions in the laser-processed archwire.



**Fig. 3** — Experimental tensile test results for straight wire samples: as-received base-metal (condition 1) and laser-processed samples (conditions 2 - 10).



**Fig. 4** — SEM image of laser processed NiTi endodontic file showing location of laser processing along the flute of the file.

enhance the fatigue life of endodontic files<sup>[5]</sup>; however, this comes at the expense of torsional strength and cutting power<sup>[28,30]</sup>. Cyclic fatigue accounts for approximately 50% of file failures compared to 30% failure due to excessive torsional stress by taper lock of the tip<sup>[31]</sup>. A functionally graded endodontic file can tailor the local properties to optimize stress/ strain distribution and achieve balanced performance in fatigue life, torsional strength, and cutting efficiency.

Attempts at functional grading of NiTi files include a differential heat treatment intended to create a more flexible zone near the distal tip. As in the example above, differential heat treatments are low resolution, meaning that it is difficult to isolate the heat to certain regions with the outcome of more homogeneous structure. The high spatial resolution and high power density of the laser enables tight control over the stiffness of a wire across very short distances. The laser

> process can be used to locally shift the transformation temperature, both along the length of a file, and across the section. This enables a more flexible stiffness profile to be programmed into a file while maintaining the harder edges necessary for cutting efficiency and torsional strength.

NiTi endodontic files were fabricated with psuedoelastic base metal wire. The laser process was applied to the fluted core of the file (see Fig. 4) to program differential material properties across the section. The cutting edge is unaffected austenite, while the core material transformation temperature is shifted above body temperature to improve flexibility and fatigue life. Figure 5 describes the change in

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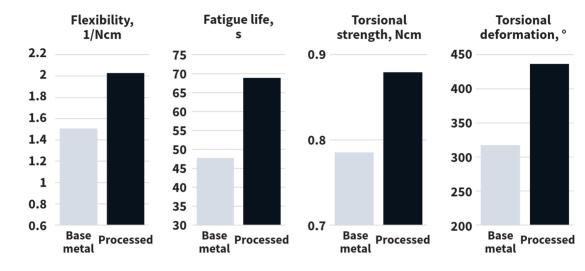


Fig. 5 – Effect of selective vaporization laser processing on the mechanical properties of a pseudoelastic NiTi endodontic file.

mechanical properties of a functionally graded size small endodontic file due to the laser processing. Bend test results show that the resilience of the unaffected shaft remain unchanged, while the processed tip section showed a 35% increase in flexibility. The torsional deformation at failure increased by 37% and the cyclic fatigue life increased by 44%.

## CONCLUSION

Functional grading of NiTi for differential mechanical properties in a monolithic device has been accomplished through selective laser vaporization. The process has been implemented and scaled to commercial levels for dental devices. Future applications of the technology in the medical device space may include suture anchors, orthopedic staples, and scoliosis correction devices. Beyond the medical device industry, commercial applications are being developed in actuation (i.e., automotive) and energy generation.

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## **FATIGUE FRACTURE OF NITINOL**

Nitinol fatigue fracture surfaces generally exhibit similar features to most engineering alloys, including a flat fracture plane, ratchet marks, radial lines, beachmarks, and striations,

Louis G. Malito,\* Matthew L. Bowers,\* Paul L. Briant,\* Gabriel S. Ganot,\* and Brad James\* Exponent Inc., Menlo Park., Calif.

itinol's shape memory and superelastic behaviors are both enabled by a solid state, diffusionless phase transformation between the "parent" high temperature (an ordered B2 or CsCl-type structure) and a "daughter" low temperature (a monoclinic B19' structure). Phraseology from steel metallurgy is customarily adopted, referring to the higher temperature parent phase known as austenite, A, and the lower temperature daughter phase known as martensite, M.

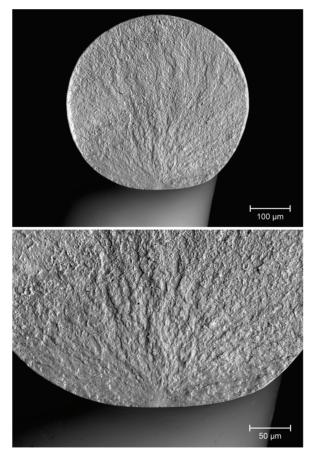


Fig. 1 — BSE micrographs of a fractured wire-form component that was fatigue tested and failed at 15.6 million cycles at 0.45% strain amplitude and 1.0% mean strain. Top: Micrograph of the overall fracture surface of the wire-form part. One can observe "feathering" lines pointing to a fatigue crack origin at the bottom of the image where the peak strain was located during cycling. Bottom: Highermagnification micrograph of the fatigue crack origin.

The phase transformation occurs through shear stress by the formation of crystal twinning in the material. Heat is released (exothermic) during the  $A \rightarrow M$  transformation and is absorbed (endothermic) during the reverse  $M \rightarrow A$  transformation. While the names austenite and martensite are derived from the steel phases of the same names to denote the diffusionless phase transformation that relates the two, the similarities end there. Nitinol's phase transformation is affected by deviatoric stresses while martensite in steel is affected by hydrostatic stresses. Unlike Nitinol, the diffusionless phase transformation in steel is irreversible due to the volume change between the phases.

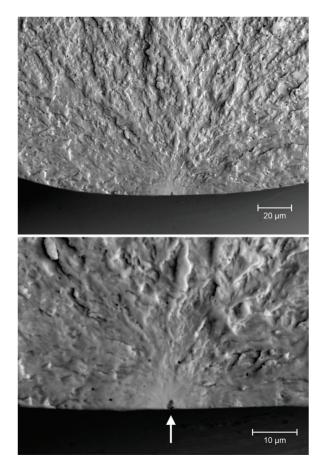


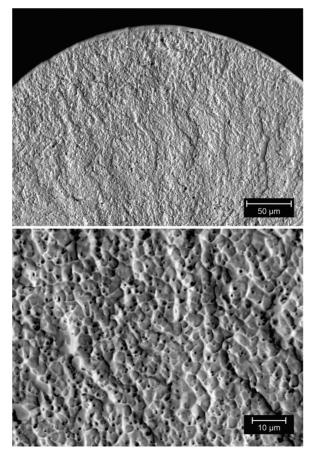
Fig. 2 — High-magnification micrographs of a surface nonmetallic inclusion that acted as an initiation site for the fatigue crack in the wire-form component from Fig. 1.

\*Member of ASM International

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In simple terms, the shape memory effect is exhibited when a phase transformation to martensite is thermally induced, deformed while in the martensitic state, and is then heated to revert it to its parent phase and shape. Superelasticity refers to the reversion of martensite to its parent phase during the release of the deforming stress (without heating). The occurrence of these phenomena is dictated by the temperatures at which the phases in the material are stable, as both thermal and strain energy play a critical role in stabilizing martensite.

Fatigue fracture should always be considered as a possible failure mechanism for Nitinol implants due to the cyclic nature of physiological loading. Nitinol fatigue fracture surfaces generally exhibit similar features to most engineering alloys, including a flat fracture plane, ratchet marks, radial lines, beachmarks, and striations when possible. Specifically, Nitinol fatigue fracture surfaces often exhibit radial lines and "feathering" more frequently than other engineering alloys. Also, due to Nitinol's tension-compression asymmetry, Nitinol can experience fatigue crack initiation and arrest from residual tensile stresses because of high device crimp strains.



**Fig. 3** — SEM micrographs detailing the fast fracture overload area of the fractured wire-form component from Fig. 1. Microvoid coalescence is observed on the fracture surface in this area.

## FATIGUE FRACTURE AND STRIATIONS IN NITINOL

A hallmark of fatigue in Nitinol, as with some other metals, is the presence of striations on the fracture surface. Fatigue striations are produced as a result of incremental sharp crack advancement and subsequent blunting. For a typical work-hardening metal, the initially sharp crack tip is blunted by the plastic zone ahead of it, but the crack tip will sharpen again during subsequent compression. In the case of Nitinol, the region ahead of the advancing crack tip contains deformed martensite, which also serves to repeatedly blunt the crack tip during each cycle<sup>[1]</sup>.

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Fatigue striations are often absent on Nitinol fatigue fracture surfaces due to a few factors. First, striation spacing may be too small to be resolved using common scanning electron microscopes due to small cyclic crack advancement during high-cycle fatigue, especially near crack initiation<sup>[1]</sup>. Second, fatigue striations may be worn away due to repeated crack closure during cycling. Striations in Nitinol fatigue fractures are commonly observed late in the crack growth process.

## FRACTURES INITIATED BY NEAR-SURFACE NONMETALLIC INCLUSION

Nitinol fatigue fractures often originate from near-surface nonmetallic inclusions<sup>[2-4]</sup>. These nonmetallic inclusions are natural byproducts of the physical melting process<sup>[5]</sup> and are further fractured and distributed by forging and drawing operations. Nevertheless, these nonmetallic inclusions, when co-located with critical high cyclic strains, can act as crack-initiating features despite being well within the specifications of ASTM F2063<sup>[6]</sup>. The existence of an inclusion at the fracture origin of a Nitinol device does not necessarily indicate that the inclusion was a defect (or that the device was defective). Any current Nitinol device will likely contain millions of inclusions. The chance of an inclusion, no matter how small, being near the critical cyclic strains (particularly in the absence of another, larger stress-concentrating feature) is relatively high.

Figures 1 through 3 are a collection of BSE micrographs of a Nitinol wire fatigue fracture surface that included a near-surface nonmetallic inclusion. The bent wire-form component was being fatigue tested in a phosphate-buffered saline (PBS) bath at 37°C and 60 Hz. The sample was cycled at 1% mean strain and 0.45% strain amplitude (on the upper end of the elastic portion of austenite, just into the stressinduced martensite plateau). The strains for the fatigue test were determined by finite element analysis. The component fractured after approximately 15.6 million cycles and was removed from the test for investigation. From inspection of the fracture surface in Fig. 1, radial lines point to an origin at the bottom of the fracture surface. Higher magnification of these

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radial lines (Figs. 1 and 2) show a "feathering" fracture morphology that is common in Nitinol fatigue. The overall crack front becomes locally subdivided into a number of different planes, resulting in a microscopic structure of multiple parallel crack paths or lines as observed in Figs. 1 and 2<sup>[7,8]</sup>.

The crack propagated in a semi-circular or "thumbnail" shape approximately one third of the way (166  $\mu$ m deep) through the cross section of the wire before final fracture (bottom of Fig. 1). Figure 2 is a magnification step series focusing on the bottom portion of the overall fracture surface from Fig. 1. The originating nonmetallic inclusion is 2.1 µm in radial length and is located within a region of higher tensile strain (as determined by finite element analysis). No visible fatigue striations were observed at the imaged magnifications in Figs. 1 and 2. The lack of observed striations is further corroborated by smooth features consistent with crack closure on the surface (see Fig. 2). Finally, Fig. 3 depicts the overload or "fast" fracture portion of the sample, where microvoid coalescence was observed.

## **FATIGUE FRACTURE AND CRACK ARREST** FROM HIGH COMPRESSIVE BENDING LOADS

Nitinol may be more susceptible to damage from high compressive strains/stresses than other materials with more symmetric tension-compression behavior<sup>[9-12]</sup>. These high residual tensile strains/stresses from compression can lead to the formation of shear cracks and subsequent overload fractures in Nitinol<sup>[11,12]</sup>. This phenomenon can also affect fractographic interpretation of fatigue. If compressive strains induced during crimping into the catheter are high enough, a residual tensile stress field can form after device deployment/expansion and can help to initiate fatigue cracks over time. The residual tensile stress fields are mostly confined within surface bending fibers and have relatively shallow depths. However, if fatigue cracks initiate on the compressive bending surface (typically at a bend inner curve), fatigue

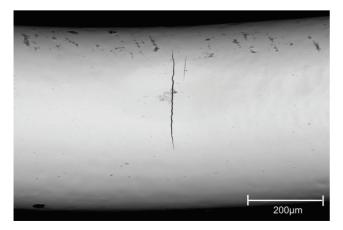


Fig. 4 — SEM micrograph that shows fatigue cracks at the intrados of a sample intentionally subjected to large compressive bending strains during a mock crimping exercise.

crack progression can arrest when the crack front reaches the edge of the relatively shallow residual tensile stress field and enters the compressive stress field. The process of fatigue crack initiation, growth, and then arrest from a residual tensile stress as a result of far-field compressive loads is a commonly observed phenomenon in ductile engineering

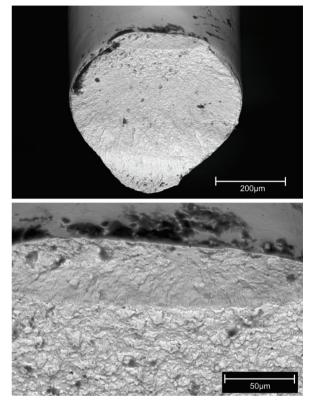


Fig. 5 — SEM micrographs of the lab-fractured Nitinol wire apex specimen. Top: Overview of the entire wire fracture surface. Bottom: Higher-magnification micrograph of the arrested fatigue crack originating on the apex intrados.

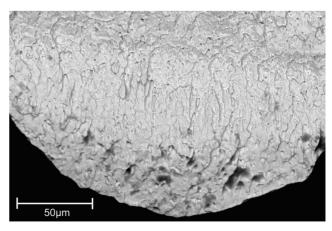


Fig. 6 — SEM micrograph of the final fracture area of the wire apex sample on the tensile surface (extrados). A shear lip is observed at the bottom of the fracture surface. Note that the elongated microvoids "point" back to the origin, consistent with ductile bending overload fractures in Nitinol.

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## FEATURE SMST NEWSWRE

metals<sup>[13,14]</sup>. These fatigue cracks on the compressive surface in ductile engineering materials will arrest as a result of crack closure effects from crack progression outside the residual tensile zone and into the compressive stress field<sup>[13,14]</sup>. The extent to which these residual tensile stress fatigue cracks grow is directly correlated to the magnitude of the far-field compressive load; i.e., higher compressive loads will result in longer crack arrest distances<sup>[12,13]</sup>.

Figures 4-6 are a collection of BSE micrographs from a fractured Nitinol wire apex fatigue specimen in which crack initiation, growth, and arrest occurred on a bend intrados (inner curve) that was intentionally damaged by compressive bending. The sample was first crimped to 12% (tensile) strain on the extrados (outer curve) with a corresponding -15% (compressive) strain on the inner curve. The sample was then allowed to expand and was cycled at a strain amplitude of 1.2% and a mean strain of 1.5% (outer curve) to 10 million cycles. All loading strains mentioned for this sample were all calculated from FEA. This wire apex sample survived fatigue testing and was subsequently inspected for cracks on the intrados. Arrested fatigue cracks are visible in Fig. 4, which is an SEM micrograph of the wire apex inner bend surface. The sample was then manually fractured to expose the surface of the arrested fatigue crack. An overview of the fracture surface is shown in Fig. 5. An arrested thumbnail crack, measuring approximately 44 µm in radial depth, was present at the apex intrados. Further inspection of the arrested crack in Fig. 5 reveals characteristic signs of Nitinol fatigue fracture at this location including "feathering" lines that radiate back to a near-surface nonmetallic inclusion at the origin. Unique to this fracture surface, the arrested fatigue crack transitions into the overload area at a sharply defined ridge. Lastly, a shear lip with microvoid coalescence is observed at the area of final fracture in Fig. 6. ~SMST

**Note:** This article is an excerpt from "Fractography of Nitinol," *Fractography*, Volume 12, *ASM Handbook*, to be published in 2022. Visit the ASM Digital Library for more information, dl.asminternational.org.

**For more information:** Louis G. Malito, senior engineer, Exponent Inc., 149 Commonwealth Dr., Menlo Park, CA 94025, 650.688.7018, Imalito@exponent.com, exponent.com.

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## SMST NEWSWRE

## SMJ HIGHLIGHTS

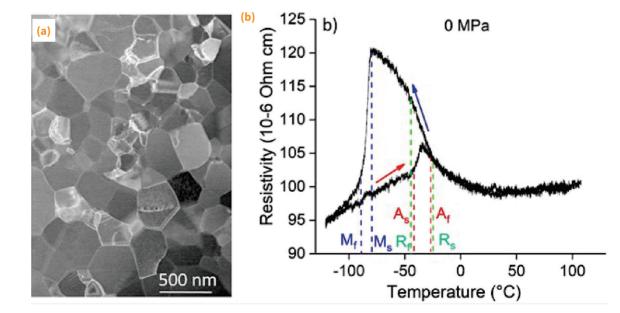


Shape Memory and Superelasticity: Advances in Science and Technology (SMJ) is the official journal of the International Organization on Shape Memory and Superelastic Technologies (SMST), an affiliate society of ASM International. The journal publishes original peer-reviewed papers that focus on shape memory materials research with contributions from materials science, experimental and theoretical mechanics, physics with cognizance of the chemistry, underlying phases, and crystallography. It also provides a forum for researchers, scientists, and engineers of varied disciplines to access information about shape memory materials. These articles, from our published 2021 issues, were selected by Shape Memory Editor-in-Chief Huseyin Sehitoglu. *SMJ* is available through springerlink.com. For more information, visit asminternational.org/web/smst.

#### March 2021

### LATTICE DEFECTS GENERATED BY CYCLIC THERMO-MECHANICAL LOADING OF SUPERELASTIC NITI WIRE Ondřej Tyc, Luděk Heller, and Petr Šittner

Abstract cyclic instability of stress-strain-temperature functional responses of Nitinol is presumably due to the plastic deformation accompanying martensitic transformation proceeding under external stress. To obtain systematic experimental evidence of this, the authors performed a series of cyclic thermomechanical loading tests (10 cycles) on superelastic Nitinol wires with nanocrystalline microstructure, evaluated accumulated unrecovered strains, and analyzed permanent lattice defects created during the cycling by TEM. The accumulated unrecovered strains and density of lattice defects increased with increasing temperature and stress, at which the forward and/or reverse transformation proceeded. It did not correlate with the temperature and stress applied in the test as such. If the martensitic transformation proceeded at low stress (< 100 MPa), the cyclic stress-strain-temperature responses of the wire were found to be almost stable (only marginal accumulated unrecovered strain and few isolated dislocation loops and segments were generated during the thermomechanical cycling). This was the case in thermal cycling at low stresses or in cyclic shape memory test. If the forward and/or reverse martensitic transformation proceeded under large external stress (> 250 MPa), the responses were very unstable (large accumulated unrecovered strains and high density of dislocations and deformation bands). A scheme allowing for estimating the cyclic instability of functional behaviors of various Nitinol wires in wide range of thermomechanical loading tests was introduced (Fig. 1).



**Fig. 1**— (a) Microstructure of the virgin austenitic 15 ms Nitinol wire. (b) Electrical resistivity thermal cycle across transformation range. There are no lattice defects observable by TEM in the recrystallized microstructure.

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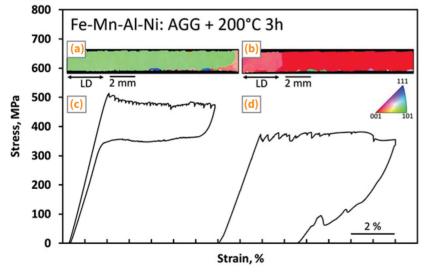
## SMST NEWSWRE

#### June 2021

### DYNAMICS OF PHASE FRONTS DURING HIGH-DRIVING-FORCE TRANSFORMATION OF SHAPE MEMORY ALLOY WIRES

#### Asaf Dana, Hiroshi Sekiguchi, Koki Aoyama, Eilon Faran, Klaus-Dieter Liss, and Doron Shilo

The reverse martensitic transformation proceeds through several subprocesses at various time and length scales. The authors recently studied the transformation kinetics in the large thermodynamic driving-force regime. They induced a rapid heating pulse in a shape memory alloy wire and tracked its evolution by multiframe time-resolved x-ray diffraction at synchrotron radiation with simultaneous stress measurements. The study identified three stages occurring at different times on the microsecond-scale and at different length scales. Specifically, the transformation was shown to occur initially in a thin layer near the surface, and only later in the bulk of the wire. They explain the obtained experimental results by modeling the evolution of the phase transformation using a continuum approach. Theoretical approaches are discussed and model fitting to experimental results provides insight into the kinetic relation between the velocity of the phase front and the driving force. Results support a scenario in which a cylindrical phase front propagates inward along the wire radius. The propagation of such a high-specific energy front releases energy faster than low-energy fronts forming under low driving forces.



**Fig. 2** — Superelastic behavior of Fe–Mn–Al–Ni tensile samples with different grain orientations. EBSD orientation map plotted for LD of the near-<101> sample (a) and the corresponding stress–strain curve up to 7% applied strain; (b) EBSD orientation map plotted for LD of the near-<001> sample; (c) the corresponding stress–strain curve up to 8% applied strain (d).

#### September 2021

### EFFECT OF CRYSTALLOGRAPHIC ORIENTATION AND GRAIN BOUNDARIES ON MARTENSITIC TRANSFOR-MATION AND SUPERELASTIC RESPONSE OF OLIGO-CRYSTALLINE Fe-Mn-AI-NI SHAPE MEMORY ALLOYS A. Bauer, M. Vollmer, and T. Niendorf

In situ tensile tests employing digital image correlation were conducted to study the martensitic transformation of oligocrystalline Fe-Mn-Al-Ni shape memory alloys in depth. The influence of different grain orientations, i.e., near-<001> and near-<101>, as well as the influence of different grain boundary misorientations are in focus of the present work. The results reveal that the reversibility of the martensite strongly depends on the type of martensitic evolving, i.e., twinned or detwinned. Furthermore, it is shown that grain boundaries lead to stress concentrations and, thus, to formation of unfavored martensite variants. Moreover, some martensite plates seem to penetrate the grain boundaries resulting in a high degree of irreversibility in this area. However, after a stable microstructural configuration is established in direct vicinity of the grain boundary, the transformation begins inside the neighboring grains eventually leading to a sequential transformation of all grains involved (Fig. 2).

#### September 2021

### PHASE STABILITY OF THREE Fe-Mn-AI-Ni SUPER-ELASTIC ALLOYS WITH DIFFERENT AI:NI RATIOS

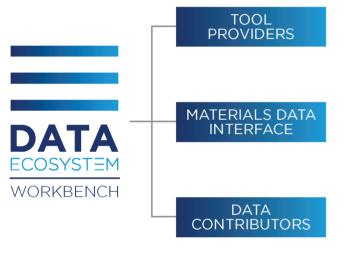
J.M. Vallejos, M.F. Giordana, C.E. Sobrero, and J.A. Malarría

Fe-Mn-Al-Ni superelastic alloy is a potential candidate for diverse engineering applications due to its outstanding properties and low material costs. Recent studies suggest that slight changes in the chemical composition severely affect superelastic response, phase stability, and grain growth kinetics. In this paper, we found that the Al stabilizes the parent  $\alpha$  phase at high temperature and promotes the formation of  $\beta$  precipitation at lower temperature. An alloy with a 3:1 ratio between Al and Ni produces homogeneously distributed ß precipitates with high phase fraction in the alpha matrix after quenching from 1200°C. The presence of these precipitates stabilizes the  $\alpha$  phase, lowers the martensitic transformation temperature and gives the alloy a fully reversible stress-induced martensitic transformation behavior without the need to apply an aging step. In alloys with lower Al content the  $\beta$  precipitation produced during quenching is severely restricted and pseudoelasticity is impaired.



Data Ecosystem uses advanced analytical tools such as machine learning to accelerate materials and engineering innovation, making the results accessible to the entire STEM community.

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World-renowned materials science innovators will come together to contribute materials data for the advancement of the STEM community.



Q4 2021 Building the foundation based on expert Member insights.



Q1 2022 Launch of prototype interface and creation of business unit within ASM and launch of Data Management education courses.



Q3 2022 Additional features, products, and upgrades to user interface.



Q1 2023 Significant enhancements made to interface and additional features added; additional education courses in Data Management curriculum.

### Website coming soon!

For more information, please contact onlineDBsales@asminternational.org



ASMNEWS



## ASM MATERIALS EDUCATION FOUNDATION ANNOUNCES 2021 SCHOLARSHIP WINNERS

### William Park Woodside Founder's Scholarship

The William Park Woodside Founder's Scholarship was established in 1996, by a gift from Mrs. Sue Woodside Shulec in honor of her grandfather. William Park Woodside founded our Society as the Steel Treaters Club more than 100 years ago and later served as president of ASM. The scholarship was established to support an ASM student member studying materials science and engineering at the junior or senior level who demonstrates strength in leadership, character, and academics. Tuition of up to \$10,000 for one academic year and a certificate of recognition are awarded to the recipient.



#### **Baylie Phillips**

Montana Technical University

Baylie Phillips is a rising junior at Montana Technological University, majoring in metallurgical and materials engineering with minors in chemistry and mathematics. She plans to have multiple careers in the fields of aerospace, environmental, and post-sec-

ondary education. This summer, Phillips was an intern at the Massachusetts Institute of Technology's Materials Research Laboratory Summer Scholars program, where she studied additive manufacturing of net-shaped single crystal turbine blades. In the fall, Phillips secured an undergraduate research project studying metal matrix composites. She will also be working on Montana's NSF EPSCoR CREWS project to investigate selenium absorption and removal in continuous-flow metal recovery systems to improve the state's water quality. Upon graduation, she plans to pursue a master's degree in materials science.

## The Lucille and Charles A. Wert Scholarship

The Lucille and Charles A. Wert Scholarship was established in 2006 through a generous bequest by the couple. It serves as an expression of their commitment to education and the materials science and engineering community. Tuition of up to \$10,000 for the academic year is awarded through this scholarship.



Liam Stack South Dakota School of Mines & Technology

Liam Stack is a junior metallurgical engineering student at the South Dakota School of Mines and Technology (SDSMT). He maintains a 4.0 GPA and is involved in a variety of clubs and activities, such as Circle K Interna-

tional, undergraduate research with the materials science department, and serving as vice president of the SDSMT Material Advantage Chapter. After graduation, Stack plans to pursue a master's degree in materials science and then work in aerospace materials development.

#### George A. Roberts Scholarships

The George A. Roberts Scholarships were established in 1995 through a generous contribution from Dr. George A. Roberts, FASM, past president and retired CEO of Teledyne, to the ASM Foundation as an expression of his commitment to education and the materials science and engineering community. Scholarships are awarded to outstanding undergraduate members of ASM at the junior or senior level who demonstrate exemplary academic and personal achievements, and interest and potential in metallurgy or materials science and engineering. Five scholars



will receive a certificate and check for \$6000 toward educational expenses for one academic year.

**Natalie Crutchfield** University of Central Florida

Natalie Crutchfield is a senior in the materials science and engineering department at the University of Central

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## HIGHLIGHTS 2021 SCHOLARSHIP WINNERS

Florida. She has participated in research since her freshman year, working in biomaterials, computer modeling, regenerative medicine, and tissue engineering. She recently participated in a Research Experience for Undergraduates program funded by the National Science Foundation at the University of Georgia, where she worked with Professor Handa to publish a paper on biomaterials research. After graduation, Crutchfield plans to pursue a Ph.D. in materials science and engineering and work to develop medical devices and therapies.



#### Sarah Gonder

McMaster University

A senior in the materials science and engineering program at McMaster University, Sarah Gonder has a passion for materials innovation, decarbonization, and improving equity in engineering. After receiving an NSERC USRA,

she has continued her research under Dr. Bosco Yu for the past 16 months. She has explored computational and cellular materials through developing algorithms to generate impact absorption architectures for additively manufactured helmets. With the support of AIST's Samson Canadian Steel Intern Scholarship, she is currently contributing to global decarbonization initiatives in the ferrous pyrometallurgy group at Hatch Ltd. Serving as president of McMaster's MSE Society and supervising an undergraduate researcher in Dr. Yu's group has highlighted Gonder's passion for mentorship. She aspires to enter project management after completing a master's degree in Europe.



#### Giovanna Janes

University of British Columbia

Giovanna Janes is entering her third year of materials engineering at the University of British Columbia, where she is an All-American varsity track athlete as well as an Academic All-Canadian. This summer, she worked with Deacon Industrial Design,

modeling structural elements and developing a soon-to-be patented mechanism in civil engineering. As a member of the Strive business and engineering team, Janes helped develop more efficient methods for delivering and collecting recyclable materials. She is hoping to specialize in biomaterials and advanced textiles while continuing her passion for helping others by working to create adaptive devices and prosthetics.



## Kaitlyn Khachadoorian

North Carolina State University

Kaitlyn Khachadoorian is a senior studying materials science and engineering at North Carolina State University. After completing two collaborative internships working on materials analysis and root-cause investigation as a defectivity design engineer at ASML,

she is motivated to drive advancements in the semiconductor industry. Khachadoorian is an All-American and Scholar Athlete on the NC State Cheerleading Team, as well as a member of the Honors Program, Grand Challenge Scholars Program, and University Ambassadors, and chairperson of the NC State Material Advantage Chapter.

#### **Owen Murdock**

The Ohio State University



Owen Murdock is a senior majoring in welding engineering at The Ohio State University (OSU). He has received recognition and awards from numerous organizations, including ASM, AWS, NACE, and the Ohio House of Representatives. In 2021, Murdock was selected

as a DAAD RISE scholar in Germany, where he assisted in doctoral materials science and engineering research at RWTH Aachen University. OSU granted approval for Murdock to complete his senior year as a full-time researcher with BMW (Munich), coinciding with his senior capstone project. He plans to continue this research for his master's thesis under the tutelage of Dr. Peter Mayr at the Technical University of Munich.

#### Acta Materialia Scholarships

The Acta Materialia Scholarships were established in 2017 by Acta Materialia through its Board of Governors, as an expression of commitment to education and the materials science and engineering community. Two scholars will each receive a certificate and check for \$5000 toward educational expenses for one academic year.



### Emma Arterbury

Colorado School of Mines

Emma Arterbury is a rising junior at Colorado School of Mines, studying metallurgical and materials engineering. She has performed undergraduate research under the supervision of Robert Cryderman in the Advanced Steel

Products and Processing Research Center at Mines for the past year, discovering a passion for problem solving, meticulous record keeping, and out-of-the-box thinking. Arterbury aspires to obtain a Ph.D. in metallurgical and materials engineering and become a professor.

**Cassidy Sjovall** 

South Dakota School of Mines & Technology

Cassidy Sjovall is entering her junior year at South Dakota School of Mines & Technology, studying metallurgical engineering. She enjoys the combination of aspects of mechanical engineering and chemistry that are

involved with metallurgical engineering. Sjovall maintains a 4.0 GPA while serving as vice president of her school's chapter of the Society of Women Engineers, treasurer of the SDSMT Baja SAE team, and participating in Material Advantage events. She recently finished a co-op with Nucor-Yamato Steel Co.

### David J. Chellman Scholarship

The David J. Chellman Scholarship was established in 2014 by Mrs. Arline Denny in honor of her husband, a long-standing Senior Technical Fellow with Lockheed Martin Corp. and ASM Life Member who enthusiastically served on the AeroMat Conference Organizing Committee for more than 25 years. The scholarship is an expression of his commitment to education and the materials science and engineering community, and is awarded based on academic merit and financial need. Tuition of \$2500 for the academic year is awarded through this scholarship.



### Gladys Durán Durán

Instituto Tecnologico de Morelia

Gladys Durán Durán is enrolled in her final year at Instituto Tecnologico de Morelia, majoring in materials engineering and specializing in metallurgy and welding. In addition to metallic glasses field research work, she has experience speaking at national and

international congresses, winning third place in the undergraduate speaking competition at MS&T20. She participated in the Cast in Steel 2021 Competition organized by the Steel Founders' Society of America, and she served as chair of the student chapter of Morelia that achieved recognition as Student Chapter of Excellence by the Material Advantage program. Durán Durán is fascinated with materials science and its role in aerospace and green energy development.

### Ladish Co. Foundation Scholarships

Established in 2011, the Ladish Co. Foundation Scholarship is awarded to an outstanding undergraduate member of ASM who has demonstrated exemplary academic and personal achievements, as well as interest and potential in metallurgy or materials science and engineering. Students must be a Wisconsin resident and must attend a Wisconsin university to qualify. Two scholars were selected this year, and will each receive a certificate and check for \$2500 toward educational expenses for one academic year.



### River Carson

University of Wisconsin-Madison

River Carson is a senior in materials science and engineering at the University of Wisconsin-Madison, with a second major in mathematics. His plan is to pursue a Ph.D. focused on x-ray diffractometry or another area where he can use his background in

mathematics. During the first year of the COVID-19 pandemic, Carson took a gap year to work as a potter and use his knowledge of materials science to focus on the development of a thermal-shock-resistant clay cookware body. Now returned to his position as president of the Material Advantage/American Foundry Society, UW-Madison Student Chapter, he hopes to solidify the recent establishment of the club forge that was disrupted by the pandemic.

### **Griffin Tong**

#### University of Wisconsin-Madison

Griffin Tong is a junior in the materials science and engineering program at the University of Wisconsin-Madison. As an undergraduate research assistant in the John H. Perepezko group, he focuses his studies on high-temperature structural materials and heat treatment. He is also dedicated to supporting his peers as co-president of the Material Advantage Chapter at UW-Madison and as an Undergraduate Learning Center tutor. Tong's goal is to earn his Ph.D. and advance the field as a researcher and educator.

### **Outstanding Scholar Awards**

The Outstanding Scholar Awards were established to recognize students who have demonstrated exemplary academic and personal achievements, as well as interest and potential in metallurgy or materials science and engineering. Three awards of \$2000 each are funded by the ASM Materials Education Foundation.



## Emmie Benard

Arizona State University

Emmie Benard is a rising junior at Arizona State University (ASU), studying materials science and engineering. She has worked on fabricating and characterizing photodetectors for medical imaging in the Radiological Instrumentation Laboratory at the Uni-

versity of California, Santa Cruz where she obtained second authorship on a review article. She is also a researcher in the Quantum Materials and Applications Laboratory at ASU, synthesizing and characterizing nanomaterials. Benard works as an undergraduate teaching assistant for an intro-

## HIGHLIGHTS DESIGN COMPETITION

ductory materials engineering class at ASU and has been on the Dean's List four consecutive times. She has received multiple scholarships through Medtronic, a local biomedical engineering company in Phoenix, and she intends to concentrate on materials chemistry and polymer synthesis as she continues her education.



#### **Jennifer Johnson** South Dakota School of Mines & Technology

Jennifer Johnson is currently a junior in metallurgical engineering at South Dakota School of Mines and in the accelerated master's program for materials science and engineering. She has also attained a certificate in

engineering management from the Industrial Engineering Department. Since her freshman year, Johnson has conducted undergraduate research focused on sustainable mineral processing from mechanochemical processing applications to reduce water usage in mining. She serves as president of Material Advantage, treasurer of SME, an active member of the welding club, and student ambassador for SDSMT. Following graduation, Johnson plans to work in research and development for the improvement of metals and materials.



#### Katrina Santos

Arizona State University

Katrina Santos is in her last year as an undergraduate student, studying materials science and engineering at Arizona State University. In 2018, she and four of her classmates were awarded first place in the ASM Inter-

national Domesday Competition at the MS&T Conference. Santos' interests are currently related to the semiconductor industry. Her work at Intel Corp. as a process engineer intern focuses on chemical vapor deposition using vertical deposition furnaces. She plans to work toward a master's degree in materials science.

### Edward J. Dulis Scholarship

The Edward J. Dulis Scholarship was established in 2003 and is awarded to an outstanding undergraduate member of ASM at the junior or senior level who demonstrates exemplary academic and personal achievements, as well as interest and potential in metallurgy or materials science and engineering. One scholar will receive a certificate and check for \$1500 toward educational expenses for one academic year.



### Zachary Katz

McGill University

Entering his fourth year of study in materials engineering at McGill University, Zachary Katz has focused on the field of biomimetic and bioinspired materials. Through co-op and intern positions with the National

Research Council of Canada, he has contributed to research on tough, architected ceramics. Of these projects, his work on crania-inspired suture structures won the undergraduate award during ASM's Student Night academic poster and video competition. Katz serves as the composites and engine sub-team leader with the Baja team and is involved in student council.



## 2021 Undergraduate Design Competition Winners Announced

The ASM Materials Education Foundation and Design Competition Committee are pleased to announce the winners of the 2021 Undergraduate Design Competition. First prize goes to **Michigan Technological University** for "Design of an Antimicrobial Beta Brass Alloy for Improved High Temperature Deformation Processing." Team members include Lauren Bowling, Anna Isaacson, Maria Rochow, Sidney Feige, and advisor Paul Sanders. The team will receive \$2000, along with \$500 to the department to support future design teams.

Second prize goes to **Virginia Polytechnic Institute and State University** for "Biodegradable Polysaccharide Blend to Replace Synthetic Toothbrush Bristles." Team members include Thomas Canfield, Ryan Friedman, Sara McBride, Kaavya Nimmakayala, advisor Abby Whittington, and industry collaborator Vincent Mascia. The team will receive \$1500. Third prize goes to **University of Florida** for "Breast Tissue Expander Port Material Redesign." Team members include Leah Buch, Anna-Sophia Hadley, Brendan O'Donnell, Chris Orozco, Martin Vivas-Gonzalez, graduate student coach David Christianson, and advisor Michele Manuel. The team will receive \$1000.

## EXECUTIVE DIRECTOR CORNER **HIGHLIGHTS**

## **ASM Constitutional Amendments Approved**

The proposed amendments to the ASM Constitution were approved by the ASM Membership at the 108th Annual Meeting on September 13, 2021. These amendments were outlined in the May/June issue of *ASM News* and were editorial in nature. However, four high-level changes were approved that include:

- The reintroduction of a second vice president filling the role of Senior Vice President.
- The inclusion of Article VII defining the Affiliate Society's relationship with ASM.
- Providing for eligibility of Past Presidents of ASM Affiliate Societies to be nominated for ASM Vice President.
- The title of Managing Director was updated to Executive Director.

Additionally, the ASM Board of Trustees reviewed and approved amendments to the **ASM Rules for Government** last updated in 2011. Both the updated and approved **ASM Constitution** and the **Rules for Government** can be found at asminternational.org/about/governance.

## Nomination Deadline for the 2022 Class of Fellows is Fast Approaching

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

Criteria for the Fellow award include:

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

**Deadline for nominations for the class of 2022 is November 30, 2021.** To nominate someone, visit the ASM website to request a unique nomination form link. Rules and past recipients are available at asminternational. org/membership/awards/asm-fellows or by contacting Christine Hoover, 440.671.3858 or christine.hoover@ asminternational.org.

### ASM Nominating Committee Nominations Due

ASM International is seeking members to serve on the 2022 ASM Nominating Committee. As noted in the ASM Constitution, approved at the ASM Annual Meeting on September 13, 2021, the committee will select a nominee for 2022-2023 senior vice president, (who will serve as president in 2023-2024), a vice president (who will serve as president in 2024-2025), and three nominees for trustee. Candidates for this committee can only be proposed by a Chapter through its executive committee, an ASM committee or council, or an affiliate society board. **Nominations are due December 15.** For more information, contact Leslie Taylor at 440.338.5472, leslie.taylor@asminternational.org or visit asminternational. org/about/governance/nominating-committee.

## EXECUTIVE DIRECTOR

#### IMAT and Heat Treat Recap

As we start the 4th quarter, I want to take this opportunity to thank our incredible volunteers throughout the world and the Operations team at Materials Park. ASM is continuing to grow and fight through the effects of



Aderhold

the pandemic due to the hard work and dedication of these two groups. I'd also like to welcome our two new "Chief Volunteers" Dr. Judith Todd as our new president and Dr. David Williams as our new vice president. I look forward to continued growth and success under their leadership.

Recently, the IMAT and Heat Treat Conference and Exhibition took place on September 13-16, in St. Louis. While the current environment surely presented challenges, the support from the ASM community allowed for a successful step forward in providing networking and knowledge dissemination-two critical values of our Society. Overall, the event saw participation from more than 1000 attendees and 300 exhibitors. Dr. Sandra Magnus, former NASA astronaut, kicked off the conference speaking at the Women in Materials Engineering breakfast on Monday morning. Key talks from both Tesla and Ford were highlights touching on critical aspects related to materials processing and the impact on the heat-treating industry. The final night of IMAT/ HT was capped by a very well attended networking event at Anheuser-Busch. ASM would like to thank the programming committees, conference chairs, attendees, speakers, exhibitors, and the community at large for their support. As we look to the future, we will continue to listen to our members and enhance our offerings to find the right balance of in-person and virtual events.

Lastly, the Materials Education Foundation recently completed their Summer Materials Camps. Due to the pan-

## HIGHLIGHTS TECHNICAL COMMUNITIES

demic, the camps this year were virtual. However, the camps reached more than 400 teachers and 100 students. Since 2000, the Materials Camps have reached almost 18,000 students! Many of these students decide to pursue STEM-based careers as a result of their experiences with our camps. If you would like to learn more or even sponsor a camp, please visit asmfoundation.org.

As always, thank you for your support of ASM!

Ron Aderhold, Acting Executive Director, ASM International ron.aderhold@asminternational.org

## Growing Technical Engagement at ASM International

ASM International has been a pillar of technical excellence supported by our authors, editors, peer reviewers, subject matter experts, speakers, and members who have provided content for successful handbooks, conferences, journals, magazines, and education courses. As we adjust to changes in tech-



nology and the advances in the dissemination of information, ASM must adapt as a society and meet the growing need for materials related knowledge while reinventing how it is distributed and consumed.

ASM is able to publish quality, reviewed, technical content because of the experts in our membership who dedicate their time to volunteering and sharing their expertise with the larger community. To help foster this sharing of knowledge, ASM has embarked on a reimagined volunteer committee structure, which will enable our membership to find technical areas of community where they can choose to get involved and work alongside others who share that same passion.

In short, ASM's new volunteer structure allows for the quick formation of technical areas of interest both informally and formally. If a group of volunteers would like to communicate and discuss a technical topic in more of an ad hoc format, ASM Connect allows for the formation of a Technical Community for a specific topic where spontaneous or planned discussion can occur and both experts and those seeking knowledge can communicate together online.

After it is formed, a Technical Committee would operate with a defined mission, leadership, meeting cadence, and goals. Technical Committees can form based on a group of volunteers' desires to gather around a specific technical topic and work toward deliverables such as programming at a conference, working on a Handbook, or building an education course. Technical Committees can form directly with enough volunteer support and Technical Communities may desire to transition to a more formal committee structure over time.

To engage with or create a Technical Community, visit ASM Connect at https://connect.asminternational.org/ home. To review current open volunteer opportunities with current ASM Committees or to submit interest in forming a new Technical Committee, visit https://connect.asminternational.org/volunteeropportunities/aboutvolunteering.



### 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 careercenter.asminternational.org.

## HISTORICAL LANDMARKS **HIGHLIGHTS**

## ASM HISTORICAL LANDMARKS: CELEBRATING 50 YEARS

#### Seeking Nominations for Milestone Year

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

The 2022 Award marks the 50th anniversary of ASM's recognition of Historical Landmarks throughout the world. Be a part of this special anniversary by submitting a nomination for the 2022 Historical Landmark award.



Nominations for designation of historical landmarks may be submitted by chapters, affiliate societies, committees, councils, award selection committees or by individual members.

**Nomination submission deadline is February 1, 2022**. For more information or to request a unique nomination link, visit asminternational.org/how-to-nominate-for-asm-awards.



First ASM Historical Landmark named—Electric Furnace, Crucible Specialty Metals Division, Colt Industries (1972)—Syracuse, N.Y.



ASM International Headquarters Building and Geodesic Dome (2009)—Materials Park, Ohio.



Eiffel Tower (1989)—Paris.



Liberty Bell (2006)—Philadelphia.



Delhi Iron Pillar (2013)—New Delhi.

## HIGHLIGHTS FROM THE FOUNDATION

## FROM THE FOUNDATION

#### Message from New Chair

As we welcome fall and the changing seasons, the ASM Materials Education Foundation is changing seasons as well. After serving for three years as the ASM Foundation chair, Glenn Daehn is now graciously passing the torch to me. I am proud to take on this very important role and look forward to continuing the growth of the Foundation.



Keough

My enthusiasm comes from knowing that the ASM Foundation has the best tools in the box for reaching teachers and students, and for increasing the number of students choosing the materials field: makers, engineers, managers, and educators. The Foundation is focusing efforts to increase participation in the ASM Materials Camps and associated year-round programs that benefit teachers and students. To learn more about the ASM Materials Education Foundation's activities and mission, visit their website at asmfoundation.org. Without the dedication of our Master Teachers, volunteers, and staff, many students would not know what materials science and engineering is and how it is applied to our everyday world. Our Master Teachers offer knowledge, enthusiasm, and assistance to help teachers implement our content in their classrooms. And teachers continue to receive support throughout the school year. Teachers and students share their testimonies of how our programs changed their perspective about materials science, increased what they already knew, and opened the door to more opportunities in materials science and engineering. Many students have chosen their career paths because of the exposure to our Materials Camps.

I am very excited to be the new Chair of the ASM Materials Education Foundation during this critical time for the organization and look forward to working with everyone involved as we continue the path of hands-on, minds-on programs for teachers and students. We are exciting and cultivating the next generation workforce in the materials field.

> John "Chip" Keough, FASM Chair, ASM Materials Education Foundation

#### 2021 Recipients of the Kishor M. Kulkarni Distinguished High School Teacher Awards

The ASM Materials Education Foundation and the Teacher Award Committee proudly announce the selection of two Ohioans, **Briana Richardson** of Washington High School and **Scott Spohler** of Global Impact STEM Academy as recipients of the 2021 Kishor M. Kulkarni Distinguished High School Teacher Award.

Established in 2007, the Kishor M. Kulkarni Distinguished High School Teacher Award recognizes the accomplishments of one high school science teacher who has demonstrated a significant and sustained impact on precollege age students. Following the pandemic year (2020) when nominations for this award were put on hold, Dr. Kulkarni decided to grant two awards this year. The award includes \$2000 for each recipient.





#### **IMS Salutes Corporate Sponsors**

The International Metallographic Society (IMS) relies on corporate financial support to maintain its excellent awards program. IMS extends sincere appreciation to the following companies for their support.

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#### IMS Sponsors:

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From left, Richardson and Spohler.

## EMERGING PROFESSIONALS HIGHLIGHTS



#### **HTS Award Deadlines**

#### ASM HTS/Bodycote Best Paper in Heat Treating Contest

The ASM Heat Treating Society established the Best Paper in Heat Treating Award in 1997 to recognize a paper that represents advancement in heat treating technology, promotes heat treating in a substantial way, or represents a clear advancement in managing the business of heat treating.

The award, endowed by **Bodycote Thermal Process-North America**, is open to all students, in full time or part time education, at universities (or their equivalent) or colleges. Students who have graduated within the past three years and whose paper describes work completed while an undergraduate or post graduate student are also eligible. The winner will receive a plaque and a check for \$2500. Paper submission deadline is March 1, 2022.

To view rules for eligibility and paper submission, visit the Heat Treating Society website at hts.asminternational. org and click on Membership & Networking and Society Awards. For additional information, or to submit a nomination, contact Mary Anne Jerson at 440.671.3877 or email maryanne.jerson@asminternational.org.



ASM INTERNATIONAL

#### Seeking Nominations for EDFAS Awards

The ASM Electronic Device Failure Analysis Society (EDFAS) established two awards to recognize the accomplishments of its members. The awards will be given at ISTFA 2022. Nominate a worthy colleague today!

#### **EDFAS Lifetime Achievement Award**

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

#### **EDFAS** President's Award

The EDFAS President's Award shall recognize exceptional service to EDFAS and the electronic device failure analysis community. Examples of such service include committee service, service on the board of directors, organization of conferences or symposia, development of education courses, and student and general public outreach. While any member of EDFAS is expected to further the Society's goals through service, this award shall recognize those who provide an exceptional amount of effort in their service to the Society.

**Nomination deadline for both awards is March 1, 2022.** For rules and nomination forms, visit the EDFAS website at edfas.org, click on Membership & Networking and then Society Awards, or contact Mary Anne Jerson at 440.671.3877 or maryanne.jerson@asminternational.org.



**Emerging Professionals** 

### EMERGING PROFESSIONALS

## Using Electrochemistry to Understand Materials Degradation

### Ho Lun Chan and Debashish Sur

From the Iron Age to the Bronze Age to our current Steel Plus Superalloy Age, corrosion has been a daunting barrier that restrains the lifespan and application of metallic materials. The first paper on corrosion was published by French chemist L.J. Thenard in 1818 describing the electrochemical nature of corrosion<sup>[1]</sup>. After nearly 200 years of research and development, we are at the time when electrochemical measurements can be performed from the nano to macro-scale in either atmospheric or high-temperature conditions. Nevertheless, electrochemistry encompasses its own complex array of theories that need to be coupled with analytical measurements (e.g., SEM, XPS, Raman) so that the underlying properties that define corrosion and its degradation can be revealed. Here is a brief overview:

1) Open-circuit potential (E<sub>oc</sub>):

 $E_{oc}$  is probably the simplest measurement to make when exposing a metal to a solution medium. It directly reflects the metal stability in the corrosive over time. The potential value itself is defined by a mix of thermodynamic and kinetic factors, and therefore, it should never be ignored.

2) DC electrochemistry:

DC potential (i.e., driving force) or current (i.e., reactivity) is applied on a corrodent either at a fixed scan rate or magnitude, and may be considered as an accelerated corrosion test. ASTM standards (e.g., G59-97, G61-86) provide guidance on performing and analyzing these experiments.

3) Electrochemical impedance spectroscopy (EIS):

AC potential or current is applied on the corrodent at a range of frequency. Result can be represented through

## HIGHLIGHTS CHAPTERS IN THE NEWS

impedance spectra similar to how an AC circuit is analyzed. This typically allows the calculation of instantaneous corrosion rate and passive film thickness. EIS when coupled with x-ray photoelectron spectroscopy (XPS) analysis can provide oxide metal cation compositions. This enables a synergistic understanding of chemical as well as physical properties of passive films in alloys ranging from binary to complex concentrated alloys<sup>[2]</sup>.

4) Mott-Schottky technique:

This method is often used to understand the semiconductor properties of oxides, i.e., whether the oxide is p-type or n-type, the concentration of ionic donors. Knowing this information helps researchers to recognize the predominant defect pair in their material (e.g., p-type = cation vacancy + anion interstitial), and to relate point defects to electrochemical corrosion behavior, which remains a topic of research today.

#### **References:**

- 1. L.J. Thenard, Annales de Chimie et de Physique, 8, 1818.
- 2. A.Y. Gerard, et al., Acta Mater., 198, p 121-133, 2020.

## WOMEN IN ENGINEERING

This profile series introduces materials scientists from around the world who happen to be females. Here we speak with Ingrid M. Padilla Espinosa, postdoctoral researcher, University of California, Merced.



#### What part of your job do you like most?

I love doing research because it Padilla Espinosa challenges me. I get to learn new things

all the time, and also every day feels different. Plus, I often get to experience the excitement of "Eureka moments." In my current position I work with graduate students, and it is very satisfying to see them achieving their goals.

#### What is your engineering background?

I have a bachelor's degree in mechanical engineering, and a master's and doctorate in nanoengineering.

#### What attracted you to engineering?

Love for mathematics and curiosity, but my greatest motivation was the possibility to create and modify devices that could benefit hundreds of people.

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

I always wanted to be an engineer, but I considered getting into a theater program as a second career.

#### What are you working on now?

I'm doing research on the plastic deformation of nanoparticles and their size, shape, and orientation dependency, using computational modeling.

#### Are you actively engaged with ASM or its affiliates?

Yes, I recently became engaged with the Inclusion, Diversity, Equity, and Awareness (IDEA) Committee.

#### Finish this sentence: Women in materials engineering are...

Changing the world by bringing new perspectives that lead to innovative solutions in all fields.

#### **Hobbies**?

Traveling, meeting people, outdoor activities, reading historical novels.

#### Favorite motto or quote?

"Everyone you will ever meet knows something you don't." – Bill Nye

#### Last podcast listened to?

The TED Radio Hour on The Food Connection, highly recommended.

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

## CHAPTERS IN THE NEWS

### **Philadelphia Awards First Centennial Scholarship**

One hundred years is a significant milestone for any professional association and worthy of a celebration. In honoring the past, the Philadelphia (Liberty Bell) Chapter decided to look to the future with the creation of a Centennial Scholarship Fund.



The inaugural Centennial Scholarship (2021) winner, Erika Garro, was Garro awarded \$5000 from the Chapter. Garro

is a student at Drexel University with a 3.93 GPA. After her 2023 graduation, she'll have both a B.S. and M.S. in materials science and engineering. Garro is active in Drexel's Material Advantage Chapter as its membership chair. She is now completing a co-op assignment as an engineering assistant with Exponent Failure Analysis Associates Inc.

The Philadelphia Chapter also presented special awards to local high school students participating in the Delaware Valley Science Fair that includes Eastern Pennsylvania, New Jersey, and Delaware High School students

## MEMBERS IN THE NEWS **HIGHLIGHTS**

whose projects featured materials or materials science in their development, testing, or analysis. The following winners were awarded special medallions and monetary prizes:

- Jabe Cox 10th grade Upper Darby H.S., Drexel Hill, Pa. "Bioplastics"
- Kehbuma Nukuna 11th grade The Charter School of Wilmington, Del. – "The Effects of Strengthening Polymers on the Infrastructural Lifespan of Industrial Concrete"
- Cameron Schmidt 12th grade Central Bucks H.S., Doylestown, Pa. – "Usability of Recycled ABS via Filament Extrusion for 3D Printing Applications"

#### **Chennai Celebrates Platinum Jubilee**

On the occasion of India celebrating its 75th Independence Day on August 15, the ASM International Chennai Chapter and ASM Material Advantage Chapters (IIT Madras, NIT Trichy, and SRM IST) organized two special events. First, they held a one-day online workshop on "Frontiers in Materials" on August 14. On August 15, a Platinum Jubilee Independence Day Special Lecture was given by Dr. U. Kamachi Mudali, now an ASM trustee. He presented "Platinum Jubilee and Beyond: Materials Demand and Issues." The events were attended by budding engineers, students, research scholars, practicing engineers, academicians, and scientists.

#### MA Bangladesh Holds Symposium

The Material Advantage (MA) Bangladesh University of Engineering and Technology (BUET) Chapter organized an online interactive symposium on July 15-18 entitled "The Material Symposium 2021: Bridging the Experiential Gap" with a goal of connecting students with distinguished individuals in materials and metallurgy. A variety of experience was provided by the 13 invited speakers, researchers, and industry professionals. Session topics included: Materials 4.0, Opportunities in Bangladesh and Abroad, Scopes Beyond Engineering, Women in Materials Science, Bioma-



A slide from Zi-Kui Liu's presentation.

terials, Machine Learning, and Additive Manufacturing. The first keynote speaker was Zi-Kui Liu, FASM, the Dorothy Pate Enright Professor at Pennsylvania State University and director of the Phases Research Laboratory. Alphons A. Antonysamy (CEng FIMMM), a principal materials scientist of additive manufacturing at GKN Aerospace UK, was the second keynote speaker. Each session had audiences of more than 200 students.

#### **Rocky Mountain Presents Nano Lecture**

Dr. Suveen N. Mathaudhu from the metallurgical and materials engineering department of the Colorado School of Mines was the speaker for the Rocky Mountain Chapter's technical meeting, held virtually on September 2. He presented a talk entitled "There's Plenty of Room at the Bottom...for Ultrastrong Nanostructured Mg-Alloys." He covered the latest strategies for synthesis and



Mathaudhu

fabrication of these alloys—despite having the lowest density of all structural metallic materials—and opportunities for this emerging class of materials.



We look forward to celebrating your future success!

## MEMBERS IN THE NEWS

### Lewandowski Receives Distinguished Professorship

John J. Lewandowski, FASM, the Arthur P. Armington Professor of Engineering II in the department of materials science and engineering at Case Western Reserve University in Cleveland, was recently awarded Distinguished University Professorship in recognition of the culmination of his 35 years of achievements. It is the highest honor the university bestows



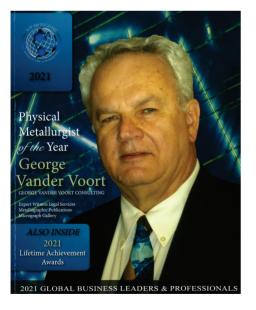
Lewandowski

## HIGHLIGHTS MEMBERS IN THE NEWS

on a member of its professoriate. Lewandowski's research is globally renowned, with his work appearing in more than 345 journal publications and 25 edited books and review papers. Lewandowski also serves as the director of the Advanced Manufacturing and Mechanical Reliability Center, which he originally founded as the Mechanical Characterization of Materials Center in 1987. He has received more than 160 grants, totaling nearly \$40 million in research funds, and has worked with some of the leading industries in the field of materials, including Howmet, General Electric, Boeing, Pratt & Whitney, Lincoln Electric, and Timken. Currently, his research efforts include focusing on the relationship between processing, structure, property, and performance using advanced manufacturing techniques.

#### Vander Voort Twice Honored

**George F. Vander Voort, FASM,** received two new accolades to add to his many previous awards. He was named "Top Consultant of the Year" from the International Association of Top Professionals, leading to his photo being projected onto the Reuters Building in Times Square on August 13. In addition, the Top 100 Registry just published its 2021 issue of "Global Business Leaders & Professionals" with his portrait on the cover and a two-page biography. The registry gave him the moniker "Physical Metallurgist of the Year," and included three of his color micrographs of meteorites in the publication.



#### **AFECO Wins Energy Innovation Challenge**

**Shri Prakash Maladkar,** managing director at AFECO Heating Systems in Kolhapur, India, represented his company in accepting recognition from the United Nations Industrial Development Organization (UNIDO) as winner of the Facility for Low Carbon Energy Deployment Challenge. Maladkar and the company won the award for their product, AL-THERMOS, an energy efficient aluminum holding furnace. The furnace consumes 75% less electricity than conventional versions. The challenge was conducted by UNIDO, the Bureau of Energy Efficiency, and the Confederation of Indian Industry, to recognize technologies that are energy efficient and help reduce the carbon footprint. The company received \$50,000 from UNIDO as part of the award.



#### **Olivetti Wins MIT Bose Award**

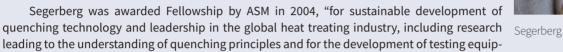
**Elsa Olivetti,** the Esther and Harold E. Edgerton associate professor in materials science and engineering at the Massachusetts Institute of Technology (MIT), is the 2021 winner of the MIT Bose Award for Excellence in Teaching. The award recognizes her teaching style, which is characterized by dedication, care, and creativity. In addition to her teaching duties, Olivetti is also co-director of the MIT Climate and Sustainability Consortium.



Olivetti

## IN MEMORIAM

Sören Segerberg, FASM, passed away July 27 at age 78. Born in 1943, he graduated from the materials science and engineering program at KTH Royal Institute of Technology, Stockholm, Sweden, in 1968 with a degree in metallurgical science. He worked for 25 years at the Institute for Engineering Technology Research (IVF, today RISE) in Gothenburg, Sweden. He is the author or co-author of more than 30 R&D reports. Segerberg was a leading expert in the quenching area and was one of the initiators of the development and sale of IVF's Quenchotest/ivf SmartQuench for quality control of cooling curve measurement. After 35 years on the market, this equipment remains an international leader with customers and agents in about 40 countries. Segerberg was also a contributor to the development of ASTM D6200 and ISO 9950.



ment for gas and liquid quenchant characterization." He was a member of the international heat treatment association IFHTSE as well as ASM and its cooling committees and work groups. He was also a member of the German heat treatment association AWT and one of the first recipients of the Karl-Wilhelm-Burgdorf Prize.

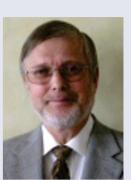
A memoriam session for Sören Segerberg will be arranged at the 27th IFHTSE Congress in Salzburg, Austria, September 5-8, 2022.



Samuel Pershing Grant, 100, of Wesley Chapel, N.C., passed away May 24, 2019, at his residence. He was born on October 22, 1918, in Chattanooga, Tenn., and served in the United States Army Corps of Engineers during World War II in Italy and North Africa as a Second Lieutenant. He earned a master's degree in physics from Georgia Tech and worked as a nuclear engineer at Carolina Power & Light. He was a member of the ASM Carolinas Central Chapter. In remembrance, Prof. M. Grace Burke, FASM, thanked "the late great Sam Grant" during her Henry Clifton Sorby lecture at IMAT 2021 in St. Louis.

Grant

Word has been received at ASM Headquarters of the death of Life Member Abraham A. Sheinker, 81, of Youngstown, Ohio. He was an ASM member for 59 years and a member of the ASM Warren Chapter.



# STRESS RELIE

## **COCA-COLA'S PAPER BOTTLE GETS TEST RUN IN EUROPE**

The Coca-Cola Company is testing its first paper bottle in markets in Hungary this year. A run of 2000 bottles of the plant-based beverage AdeZ will be offered in the pioneering package via e-grocery retailer Kifli.hu. The bottle is made of a paper shell with 100% recyclable plastic lining and cap.

The paper bottle project, which is being co-developed by Coca-Cola's R&D team in Brussels and The Paper Bottle Company (Paboco), a Danish startup supported by ALPLA and BillerudKorsnäs, in cooperation with Carlsberg, L'Oréal, and The Absolut Company, is moving into the consumer testing phase to measure the package's performance and shopper response to the format. Coca-Cola and Paboco unveiled the first-generation prototype, which consists of a paper shell with a recyclable plastic lining and cap, a year ago.

The technology developed by Paboco is designed to create 100% recyclable bottles made of sustainably sourced wood with a bio-based material barrier capable of resisting liquids,  $CO_2$ , and oxygen, and is suitable for beverages, cosmetics, and other liquid goods. The ultimate goal is a bottle that can be recycled as paper.



Coca-Cola's paper bottle prototype is made of a paper shell with 100% recyclable plastic lining and cap. Courtesy of The Coca-Cola Company.

The innovation supports The Coca-Cola Company's World Without Waste sustainable packaging target to collect and recycle a bottle or can for each one it sells by 2030, while substantially reducing use of virgin packaging materials and using only 100% recyclable packaging materials. Achieving this vision requires investment in innovation and collaboration with partners to drive collection, recycling, and sustainable design.

In addition to beverages, the packaging is suitable for holding other liquid goods such as beauty products. If successful, the next iteration of its prototype with Coca-Cola would consist solely of paper without the plastic liner.

## STATEMENT OF OWNERSHIP, MANAGEMENT, CIRCULATION, ETC.

Required by the Act of 23 October 1962, Section 4369, Title 39, United States Code, showing the ownership, management, and circulation of *Advanced Materials & Processes*<sup>®</sup>, publishes eight issues per year: January, February/March, April, May/June, July/August, September, October, and November/December at 9639 Kinsman Road, Materials Park, Ohio 44073, USPS # 762-080. Annual subscription rate is \$499.

The publisher and editor are Scott D. Henry and Joanne Miller, respectively, both of 9639 Kinsman Road, Materials Park, Ohio, 44073. The owner is ASM International<sup>®</sup>, Materials Park, Ohio, which is a not-for-profit educational institution, the officers being; President and Chair of the Board, Judith A. Todd; Vice President and Trustee, David B. Williams; Immediate Past President and Trustee, Diana M. Essock; Secretary and Acting Executive Director, Ron Aderhold; Treasurer and Trustee, John C. Kuli; Trustees, Burak Akyuz, Ann Bolcavage, Elizabeth N. Hoffman, Navin J. Manjooran, Toni Marechaux, U. Kamachi Mudali, James E. Saal, Priti Wanjara, and Ji-Cheng Zhao; Student Board Members Shruti Dubey, Andrew J. Ruba, and David Scannapieco. There are no known bondholders, mortgagees, and other security holders owning or holding 1% or more of the total amount of bonds, mortgages, or other securities.

The issue date for circulation data below is July/August 2021. The average number of copies of each issue during the preceding 12 months is: (a) Total number of copies printed: 4,901; (b) Paid and/or requested circulation: (1) Paid/requested outside-county mail subscriptions: 4,123; (2) Paid in-county subscriptions: 0; (3) Sales through dealers and carriers, street vendors, counter sales, and other non-USPS paid distribution: 439; (4) other classes mailed through the USPS: 0; (c) Total paid and/or requested circulation: 4,562; (d.1) Free distribution or nominal outside-county: 40; (d.3) Free distribution by mail: 59; (e) Total free distribution: 99; (f) Total distribution: 4,661; (g) Copies not distributed: 341; (h) Total: 5,002; (i) Percent paid: 91. The actual number of copies of single issue published nearest to filing date is: (a) Total number of copies printed: 3,243; (b) Paid and/or requested circulation: (1) Paid/requested outside-county mail subscriptions: 2,883; (2) Paid in-county subscriptions: 0; (3) Sales through dealers and carriers, street vendors, counter sales, and other non-USPS paid distribution: 295; (4) other classes mailed through the USPS: 0; (c) Total paid and/or requested circulation: 3,178; (d.1) Free distribution or nominal outside-county: 0; (d.3) Free distribution by mail: 55; (e) Total paid and/or requested circulation: 3,178; (d.1) Free distribution or nominal outside-county: 0; (d.3) Free distribution by mail: 55; (e) Total free distribution: 55; (f) Total distribution: 3,233; (g) Copies not distributed: 407; (h) Total: 3,640; (i) Percent paid: 90.

I certify that the statements made by me above are correct and complete. Scott D. Henry, Publisher

## MATERIALS & PROCESSES **EDITORIAL PREVIEW**

## **NOVEMBER/DECEMBER 2021**

**Materials Testing & Characterization** 

#### **Highlighting:**

- Emerging Characterization Techniques
- Iron-based Archaeometallurgy in North Africa
- Advanced Testing Methods

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**Emerging Analysis Methods** 

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# BORRNSHOP



The 2022 Cadillac Blackwing V-series includes a binder-jet printed knob shifter. Courtesy of Azoth.

## **3D PRINTED KNOB SHIFTER IS FIRST TO HIT PRODUCTION**

The first metal 3D printed part made with binder jetting is now being used in production on a General Motors vehicle. The 3D printed medallion is produced by Azoth and sits on the manual shifter knob of the new 2022 Cadillac Blackwing V-series models, which also feature additional 3D-printed parts developed by General Motors.

"Binder jetting enables quantities at the speeds and quality levels demanded by automotive production," says Azoth's general manager and cofounder Cody Cochran. "We use a disruptive process that is more efficient than other technologies. We can print the prototype of a part every five or six days, while other processes and technology can take three to six months building molds, retooling, and going through multiple suppliers."

"I believe this is a breakthrough application for the 3D printing industry and a major win for metal binder jetting, proving this is a production ready process," he said.

In a release issued last year, Cadillac said that by leveraging additive manufacturing, it was able to reduce costs and waste when developing the manual transmission. *azoth3d.com*. to demonstrate 3D printing using a material simulating regolith, such as that found on the surfaces of planetary bodies such as the Moon. The results could help determine the feasibility of using regolith as a raw material and 3D printing as a technique for on-demand construction of habitats and other structures on future space exploration missions. *nasa.gov.* 

### A LIQUID-CRYSTAL INK FOR DIRECT-INK-WRITING

Cholesteric liquid crystals are an ideal material for healthcare applications because they mimic iridescent materials found in nature. But until recently it has been difficult to use them in advanced, rapid production methods.

Researchers from the department of Chemical Engineering and Chemistry at Eindhoven University of Technology, in collaboration with TNO, DSM, Brightlands Materials Center, and SABIC have created a nature-inspired liquid crystal elastomer-based ink that can be 3D printed on a surface via direct-ink-writing (DIW). DIW is an extrusion-based 3D printing approach where an ink is dispensed from a small

NASA SENDS REGOLITH 3D PRINTER TO ISS

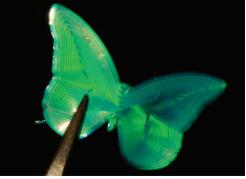
NASA's recent resupply mission to the International Space Station (ISS) included a machine to test 3D printing of regolith, which is loose rock and soil. The Redwire Regolith Print project will work in tandem with ManD, an existing printer system, nozzle onto a surface on a layer-by-layer basis.

The new liquid crystal ink has several key properties. First, the light reflective properties of the ink rely on the precise helical alignment of molecules throughout the material which requires fine tuning of the printing process. Second, the molecules in the ink can self-assemble into such structures that display colors similar to natural iridescent materials, like those in butterfly wings. Third, the new ink has greater viscosity than previous inks, which makes it suitable for DIW printing.

"To successfully print the new ink with DIW, we varied parameters like print speed and temperature. And to get the ink to print properly, we also made an ink containing low-molecular weight liquid crystals," says lead author and Ph.D. candidate Jeroen Sol.

Researchers were able to control the nanoscale molecular alignment very accurately by varying the print speed. This allowed them to have greater control over the appearance and light reflecting properties of the material.

Given that the new liquid crystal ink can be printed with DIW, it could be used in future printing procedures for personalized medical devices such as thin wearable biosensors that interact visually and colorfully with the wearer. www.tue.nl/en/.



Researchers were able to use a new liquid-crystal ink to print synthetic butterfly wings. Courtesy of Eindhoven University of Technology.





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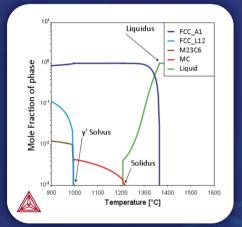
## Do you rely on handbook data? What if the materials data you need doesn't exist?

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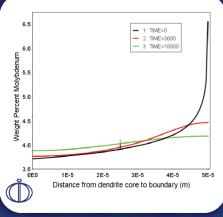
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## **Temperature Effect**

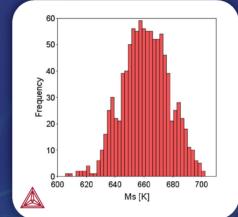


Phase stability vs. temperature for Ni-Base Alloy 282

## Homogenization



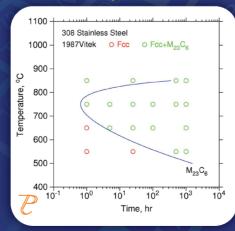
Homogenization of casting segregation in Ni-Base alloy 713



**Composition Effect** 

Calculated Ms temperatures for 410 stainless composition spec range

## Precipitation

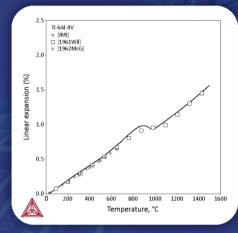


Time temperature precipitation of M<sub>23</sub>C<sub>6</sub> in 308 stainless steel

### Base Decisions on scientifically supported models

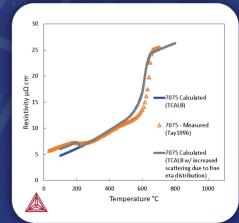
- Accelerate materials development while reducing risk
- Troubleshoot issues during materials processing

## **Thermophysical Data**



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

## **Electrical Resistivity**



Calculated electrical resistivity of aluminum alloy 7075

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