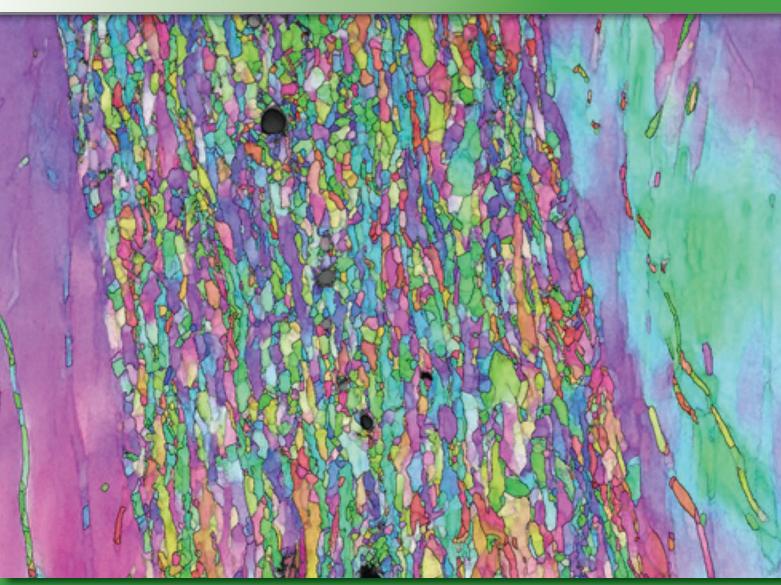


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# **ASM's Reengineered Website Has Arrived!**

Our new website has been redesigned with you in mind! We've listened to your feedback and are excited to share our new site with you. Improvements include:

- Sleek and modern design.
- Revamped navigation makes it easier to find content with less clicks.
- "Browse Categories" in our Material Resources section lets you filter ASM's huge collection of content by resource type, subject, and publication date.
- Enlarged text makes the website easier to read and provides an intuitive look and feel.
- Streamlined Shopping Cart and Checkout process.

#### Have a look around!

- Enhanced Membership Points program lets you earn points for product purchases and use them to download content for free!
- Social networking capabilities in our Membership Directory lets members upload their profile pictures to aid in networking event recognition.
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<ul> <li>✓ Verv sample</li> <li>✓ Verv</li></ul>	12	ADOULASIM	Committees & Expositions Communities		•
Physical metallurgy of steel heat treatment		<ul> <li>✓ View sample</li> <li>∴ View TOC</li> <li>☆ Email a friend</li> </ul>	Fundamentals and P         Hardcover       Product code: 05344G       ISBN: 978-1-627X         Classified as:       Additive Manufacturing       Erzang         Price:       \$249.00       Member Price: \$189         INISTOCK       Information about shipping / delivery         Description       Delivery Options         Coverage on heat treating in the ASM Handbook, Volume 4A, Steel Heat Treating volumes on heat treating. Volume 4A introduct.	Classifications Dev Series is being expanded into several volumes, and ng Fundamentals and Processes is the first of multiple es the basics of steel heat treating and provides in-depth	
			Physical metallurgy of steel heat treat	ment	







Jacquet-Lucas Award •

Transmission Kikuchi Diffraction •

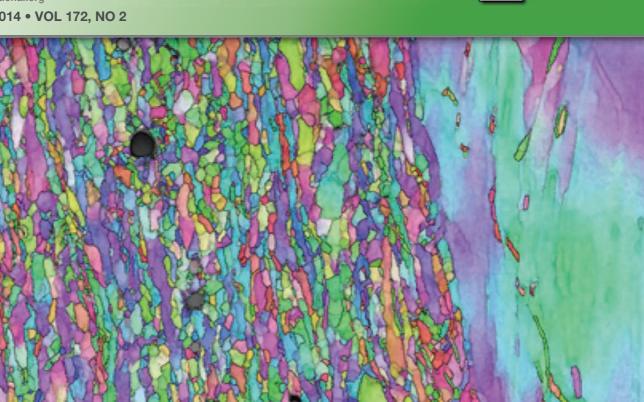
Additive Manufacturing of Titanium •



## Microscopy/Metallography Advances

TAM BLOW INCLUDED IN THIS ISSUE

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#### ON THE COVER:

Large scale transmission Kikuchi diffraction orientation map showing a nanocrystalline shear band cutting across coarse grains in an Al-Mg-Cu alloy. Scale is 2 µm. Courtesy of the Australian Centre for Microscopy & Microanalysis, The University of Sydney. www.sydney.edu.au.

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**13** Transmission Kikuchi Diffraction in the Scanning Electron Microscope: **Orientation Mapping on the Nanoscale** 

contents

Patrick W. Trimby and Julie M. Cairney By changing a setup and using electron transparent samples, hundreds of laboratories worldwide have access to a characterization technique that measures grain sizes and orientations in nanocrystalline materials.

#### **JACQUET-LUCAS AWARD Extrusion Welding in a Magnesium Alloy** (AM30) Hollow Section Extrudate

Nabeel H. Alharthi and Wojciech Z. Misiolek This entry won the prestigious Jacquet-Lucas Award for Best in Show at the 2013 International Metallographic Contest held in conjunction with the M&M 2013 conference in Indianapolis.

Additive Manufacturing of Titanium Alloys

Although the widespread use of titanium alloys is constrained by high costs, powder metallurgy techniques such as additive manufacturing represent an economical approach to fabricating titanium

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#### **METALLURGY LANE** Wrought Iron: A Metal for the Ages Charles R. Simcoe

Early humans had no knowledge of iron. Unlike gold, silver, and copper, iron does not exist as a native metal. Here we explore the early days of wrought iron production.

#### **International Thermal Spray and** Surface Engineering

B. Dutta and F.H. Froes

components.

The official newsletter of the ASM Thermal Spray Society (TSS). This quarterly supplement focuses on thermal spray and related surface engineering technologies.

#### **ASM News**

The monthly publication about ASM members, chapters, events, awards, conferences, affiliates, and other Society activities.

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# materials witness

### It's complicated

iving in Cleveland in the heart of winter offers ample time for reflection on a wide range of topics, from the economy and materials science breakthroughs to matters closer to the heart. With sub-zero temperatures blanketing much of the country lately, I'm sure many of you can relate. The arctic cold snap in early January sent windchill temps to below -35°F here, though I was fortunate to be in balmy Alexandria, Va., during that stretch visiting NIST in Gaithersburg, Md., and NASA in Washington, D.C. I'll take 7° over -35° any day.



Both NIST and NASA are busy working on interesting projects, which we will report on in the next few months. Many of their efforts support the Materials Genome Initiative and big plans are underway with regard to testing and characterizing various materials in new and comprehensive ways. It may sound trite, but the stuff of life—materials—used to be so much simpler. As we report in this month's *Market Spotlight*, a new study from Yale University's School of Forestry and Environmental Studies reminds us that less than a century ago, fewer than 12 materials were in widespread use—wood, brick, iron, copper, gold, silver, and a few plastics.

Contrast that with today's complex multi-material products such as the latest computer chips, superalloy turbine blades, and ever more complicated vehicle technology. The scary part? Many of these wonder materials contain elements that are becoming scarcer and harder to source. The bright spot is that well trained scientists and engineers will be in high demand to develop alternative and substitute materials.

Speaking of vehicle technology and modern materials, the aluminum versus steel drama continues to play out. Consider Ford's big splash at the 2014 North American International Auto Show, held last month in Detroit. Ford's new F-150 pickup truck, which goes on sale later this year, uses more high-strength steel than ever, making it stronger and lighter than previous models. More interesting though, high-strength, military-grade aluminum alloys are used throughout the body for the first time. Up to 700 lb were shaved away, helping the F-150 "tow more, haul more, accelerate quicker and stop shorter, and improve efficiency," say company sources. Cost will be an issue though: Aluminum is more expensive than steel and factory retooling costs need to be accounted for if and when automakers make the switch from steel to aluminum.

In steel's corner at the Auto Show was the 2014 North American Truck of the Year award that went to the Chevrolet Silverado—featuring a large amount of advanced high-strength steel, "which offers significant lightweighting benefits at a lower cost," says Ron Krupitzer of The Steel Market Development Institute. "It's an exciting time in the automotive industry, as automakers evaluate vehicle components for lightweighting potential to meet industry needs, while also balancing customer preferences and safety expectations. It's no surprise that in the case of the Silverado, those solutions were advanced steel."

Ultimately, consumers will vote with their purchasing power as automakers figure out ways to meet increasingly stringent fuel economy mandates. Look for a report next month on NIST's efforts in this area via their Center for Automotive Lightweighting. In the meantime, try to savor the time for reflection and solitude offered by Old Man Winter.

7. Richard

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2013 ISTFA Photo Contest Color Winner

Image description: Leakage between two circuits. Thinned the die from the silicon side then removed layer by layer until Bump level. Found Dendrite between two bumps!

By: Ty Kha | Company: Intel Corporation



# Study explores substitutes for 62 scarce metals

new study from Yale University's School of Forestry and Environmental Studies, New Haven, Conn., investigates potential substitutes for 62 metals as some of these materials are becoming harder to source. The study, "On the Materials Basis of Modern Society," was recently published in the *Proceedings of the National Academy of Sciences* and reaches a few dire conclusions. The authors state, "The situation need not inspire panic, but should stimulate more diligent and comprehensive approaches to the balance between supply and demand across the entire periodic table." For a dozen different metals, potential substitutes for their main applications are either inadequate or do not exist.

The report begins with a stark reminder that not even a century ago, fewer than 12 materials were in widespread use—wood, brick, iron, copper, gold, silver, and a few plastics. Contrast that to today's multi-material products, for example, where a modern computer chip contains more

than 60 different elements

selected by product design-

ers and materials scientists

for enhanced performance.

Another example looks at

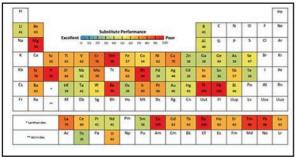
the increased diversity of

superalloy metals used in

aircraft turbine blades.

These nickel-rich alloys

were developed with vari-



Elements shown in red, such as rhenium and lead, appear to have very poor substitution potential, which will likely have a negative impact on product performance and price.

ous alloying elements over the years to achieve greater corrosion resistance and stability at higher temperatures. The result is increased engine operating temperatures and higher efficiency, made possible by a complex mix of materials. Researchers contend that other modern products followed similar performance evolutions, i.e., faster computers and better medical images, but worry about suitable substitutes as some elements are becoming scarcer.

The Yale study also points to other studies on this topic, such as the 2008 National Research Council's Minerals, Critical Minerals, and the U.S. Economy. Further, it explores a few examples where substitute materials were successfully used. For example, when the cobalt supply was threatened in the 1970s due to a civil war in Zaire, scientists at the General Motors Research Laboratories and colleagues developed cobalt-free substitute magnets that worked well. Another case involves a rhenium shortage that impacted the superalloys used in gas turbines. Scientists at the General Electric Research Laboratories developed alternative alloys containing little or no rhenium. However, the study points out that these may be exceptions. For example, some widely used metals such as copper, chromium, manganese, and lead have no good substitutes available for their major uses. Others that have very low substitute performance potential include rhenium, rhodium, lanthanum, europium, dysprosium, thulium, ytterbium, yttrium, strontium, and thallium. The authors assert that substitution by a different material is likely to decrease product performance, raise prices, or both. For more information, visit www.pnas.org/cgi/doi/10.1073/pnas.1312752110.



#### Single crystal clarifications

Thank you to all who sent emails regarding my article on the development of single crystal superalloys (September 2013). Four items need to be clarified or corrected: 1) Fan materials today include titanium alloys and composites; 2) Herb Hershenson was hired to run the chemistry group and later hired Bill Goward to oversee coatings, oxidation, and corrosion; 3) the Air Force Office of Scientific Research contract on the study of single crystal superalloys originally had Gerry Leverant as the principal investigator. Shortly after Gerry left Pratt & Whitney, the Statement of Work was revised to focus on the mechanism of Re strengthening as well as the identification of lower and upper limits of Re to provide enhanced creep resistance without the risk of forming undesirable phases; and 4) the Navy contract to run single crystal turbine blades in the PT6 was actually under the Naval Air Propulsion Center, with Joe Glatz as program manager.

Tony Giamei

#### Seeking dome details

I am a civil engineering student at the University of Ghent in Belgium. We have a project-based assignment for a course in spatial structures and are very interested in the geodesic dome, as in your Facebook page cover photo. We must explain how the forces are transmitted into the structure and also need to create a scale model. Can you provide some information about the structure, such as dimensions, construction details, or other information about the principles of the design?

Björn Noyelle, via ASM's Facebook page

[The design of Materials Park was the vision of William Hunt Eisenman, ASM's managing director from 1918 through 1958, Cleveland architect John Terence Kelly, and inventor R. Buckminster Fuller. Within the park, the geodesic dome was engineered and created by R. Buckminster Fuller. It is the largest dome of its kind and was one of his favorites. Made of extruded aluminum pipe, the open-work structure stands 103 ft high and 250 ft in diameter, weighs 80 tons, and has more than 65,000 parts. The dome stands on five pylons, two of which rise up from courtvards set into the building.-Eds.]

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



## Stay-strong jeans last longer

DSM, the Netherlands, joined forces with Levi Strauss & Co., San Francisco, to create jeans with a blend of strength, durability, and style. Dyneema, an Ultra High Molecular Weight PolyEthylene (UHMWPE) fiber, can be found in Levi's 501 Warrior and Trooper style jeans. Its use brings a 25% performance improvement in abrasion resistance and strength over traditional jeans of the same weight. Unlike other high-strength fibers, Dyneema is soft and supple, so jeans retain their comfort and fit, say company sources. In addition, both Levi and DSM say they are committed to reducing their carbon footprint by creating sturdy, long-lasting products. *www.dyneema.com*.

#### **Electric car is 3D printed**

The two-passenger Urbee (urban electric with ethanol as backup) is designed to use the least energy possible, getting more than 200 mpg on the



The two-passenger Urbee is the first prototype car ever to have its entire body printed using an additive process.



Sadik Khan, Sahadat Hossain's doctoral student, views a failing slope along U.S. Route 287.



Dyneema, an UHMWPE fiber, is being used in Levi's 501 Warrior and Trooper style jeans to increase durability.

highway and 100 mpg in the city. And now, it is the first prototype car ever to have its entire body printed using an ad-

ditive process. Jim Kor, president and senior designer of Kor Eco-Logic, wanted to make Urbee aerodynamic and as "green" as possible throughout the design and manufacturing processes.

Together with Stratasys, Eden Prairie, Minn., the team selected ABS as the material of choice and began to build the car. The fullscale door and side panels were completed first. "These were big panels," says Kor. "The parts fit together perfectly." The remaining body panels are currently being built by Stratasys. "Just to make the first car was quite an achievement," notes Kor. "With our second prototype, we will design to the Stratasys printer capabilities. We want to exploit the full capacity of the machines." That means designing both the interior and exterior. It also means using plastic only where it is needed. *www.stratasys.com*, *http://korecologic.com*.

#### **Recycled plastic pins fortify highway slopes**

A University of Texas at Arlington civil engineering researcher won a \$1 million state transportation department (TxDOT) contract to install pins made from reclaimed and recycled plastic along some of the region's busiest highways to shore up clay soils that support the roads. Sahadat Hossain, an associate professor of civil engineering, demonstrated the technique as a cost effective and efficient solution to failing soil slopes as part of the project during the last few years. The study indicates that the cost of slope stabilization and repair can be reduced by more than 50% when using these recycled plastic pins compared to conventional methods.

The pins are roughly 4 in. wide by 4 in. deep and 8-12 ft in length. An assistant professor and expert in analyzing material cracks and fractures collaborated on the project and developed a numerical model to help TxDOT field staff determine where to place the pins to ensure soil stability. The reinforced sections along U.S. Route 287 have held up much better than the untreated areas along that road, researchers found. Hossain and his team are also working on sustainable pavement base and sub-base materials as part of this project. *For more information: Sahadat Hossain*, 817/272-3577, hossain@uta.edu, www.uta.edu.



## **METALS** POLYMERS **CERAMICS**

## briefs

Research led by **University of Nebraska-Lincoln** found that using a small amount of graphene oxide as a template improves carbon nanomaterials, promising to improve composite materials. The graphene oxide nanoparticles are incorporated as a template to guide the formation and orientation of continuous carbon nanofibers. A group led by chemist SonBinh Nguyen of **Northwestern University**, Evanston, III.,

synthesized the graphene oxide, and the resulting carbon nanofiber structure has an orientation similar to fibers with enhanced properties. These graphene-based nanofibers are now being tested for enhanced properties and to improve the technique. www.unl.edu, www.northwestern.edu.

**REFORM** (resource-efficient factory of recyclable manufacturing composite) is a project funded by the European Commission and focuses on the manufacture of composites to develop cleaner, more efficient technologies for composites manufacturing. These composites are produced with a polymer base and reinforced with fiber, allowing fabrication of lighter components that maintain or improve mechanical properties. The methods considered are rolling, machining (cutting and edge finishing), assembly, and recycling. Tecnalia, Spain, is working alongside 13 partners and is gearing its work toward machining composite materials, using waterjet as well as conventional cutting. http://reform.eu.com, www.tecnalia.com/en.

#### New design aims toward crash-free cars

Volvo, Sweden, hopes to achieve a goal of zero vehicle fatalities or serious injuries in their models by 2020. The new scalable product architecture (SPA) significantly improves protection in worst-case scenarios and creates innovative features to help drivers avoid accidents. The safety cage in the original XC90 contained 7% hot-formed boron steel, while the new version features more



The XC90 safety cage is built using the new scalable product architecture and features more than 40% hot-formed steel, which translates into significantly improved strength without adding mass or weight.

than 40% hot-formed steel, which improves strength without adding mass or weight. Cars built on the new SPA have smart belt pre-tension systems that help keep occupants in the vehicle before and during collisions. For example, a rearward-facing radar detects rear impact, so safety belts tighten in advance to keep occupants in place. Camera, radar, and sensor technologies are extended to detect more objects around the car and offer support at higher speeds and in more situations, such as at intersections. The new features also auto brake for large animals and pedestrians when driving in the dark. *www.volvocars.com*.

#### New alloys for use in aerospace components

IBC Engineered Materials Corp., Wilmington, Mass., is working directly with Lockheed Martin's F-35 electro-optical targeting system (EOTS) engineering, design, and quality teams in Orlando to develop components to demonstrate the technical and commercial viability of their Beralcast alloys as alternatives to parts made from beryllium-aluminum alloys. Several advanced prototype castings are being used to evaluate the new alloys and castings for critical structural and sub-system aerospace components. Beralcast alloys can be used in virtually any high performance application requiring complex, lightweight, and high-stiffness parts. The new alloys can be substituted for aluminum, magnesium, titanium, and metal matrix composites, as well as pure beryllium or powder metallurgy beryllium-aluminum. Beralcast's principal alloys are more than three times stiffer than aluminum with 22% less weight and can be precision-cast for simple and complex 3D stability. *www.ibcadvancedalloys.com*.

#### Lowering titanium's cost for lightweight products

A novel method for extracting titanium significantly reduces the energy required to separate it from its tightly bound companion, oxygen. Zhigang Zak Fang, professor of metallurgical engineering at the University of Utah, Salt Lake City, and colleagues note that while titanium is the fourth most common metal in the Earth's crust, the high-en-

Walter Voit, assistant professor of materials science and engineering and mechanical engineering at the **University of Texas**, Dallas, was awarded \$1 million to create medical devices with greater control of prosthetics in wounded soldiers. He created shape memory polymers that can respond to the body's environment and become less rigid when implanted in the body. These polymers are implanted when they are rigid and then flex toward the stiffness of the tissue. The medical devices should survive implantation in the body for more than one year. For more information: Walter Voit, 972/883-5788, walter.voit@utdallas.edu, www.utdallas.edu.

ergy, high-cost method used to extract it prevents its use in broader applications.

The most common technique, called the Kroll process, used to extract the metal from titanium oxide was invented in the 1930s and has undergone slight improvements. The method requires temperatures above 1800°F and is expensive. Fang's team was able to eliminate the energy-intensive steps of the Kroll process. In the lab, they successfully tested a new series of reactions for isolating titanium that halves the temperature requirements of the conventional method and consumes 60% less energy. For more information: Zhigang Zak Fang, 801/581-8128, zak.fang@ utah.edu, http://powder.metallurgy.utah. edu.

#### Shielding stops neutrons cold

When faced with the challenge of protecting sensitive scientific equipment and computers from radiation, engineers at the DOE's Thomas Jefferson National Accelerator Facility, Newport News, Va., came up with three innovative products that could soon find their way to nuclear power plants, particle accelerators, and other radiation-generating devices around the world. The technologies form a system for shielding that is less expensive, lighter, and less bulky than standard products and are easily manufactured using existing techniques. They consist of recipes for a lightweight concrete that is  $4 \times$  better at slowing down neutrons than ordinary concrete, a boron-rich concrete that absorbs neutrons using less material, and a thin, boron-rich paneling for use in space-restricted areas. Their system, while using clever new techniques to shield against neutrons, works on the same principles as those currently in use. It consists of a concrete layer to slow down neutrons, a material to absorb them, and a thin lead layer to halt any residual radiation. www.jlab.org.



Three new products for shielding against neutrons consist of a boron-rich paneling for use in space-restricted areas (front), boron-rich concrete that absorbs neutrons using less material (left), and a lightweight concrete that is 4 × better at slowing down neutrons than ordinary concrete (right). Courtesy of DOE's Jefferson Lab.

A unique solar panel design could provide less expensive sustainable power that is more efficient and requires less manufacturing time. It was developed by a team led by scientists at the **University of Pennsylvania** and **Drexel University**, both in Philadelphia. The tests were conducted, in part, at the Advanced Photon Source at the DOE's **Argonne National Laboratory**, Ill. The new class of ceramic materials has three main benefits: It can produce a solar panel thinner than today's silicon-based market leaders, it uses less expensive materials than those used in today's high-end thin-film solar panels, and it is ferroelectric, a key trait for exceeding the theorized energy-efficiency limits of today's solar cell material. www.drexel.edu, www.upenn.edu, www.anl.gov.



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## **TESTING** CHARACTERIZATION

## briefs

Crane Engineering, Plymouth, Minn., acquired Safety Engineering Associates, Madison, Wis., a vehicle testing and accident reconstruction services provider. Safety Engineering clients will now have access to Crane's chemical, materials, metallurgical, and micro-imaging laboratories, including its in-house 3D laser scanner and computed tomography resources. www.cranengineering.com, www.safetyengineering.com.

MTS Systems Corp., Eden Prairie, Minn., held a grand opening of its vehicle system engineering laboratory in collaboration with Chery Automobile Co. Ltd. in Wuhu, China. The Chery-MTS lab is a center for system and full-vehicle testing applications, as well as development of new testing technologies. Chery is the leading domestic automotive manufacturer and car exporter in China. www.mts.com.

M+P Labs, Schenectady, N.Y. and Greenville, S.C., changed its name to Lucideon effective February 1. M+P works predominantly in the energy, aerospace, and healthcare sectors. The Lucideon group includes UK-based materials technology company Ceram, and international sustainability, verification, and certification specialist CICS (Complete Integrated Certification Services Ltd.). www.lucideon.com.

#### Mini but mighty magnet enables advanced spectroscopy

Scientists at Rice University, Houston, pioneered a tabletop magnetic pulse generator that reportedly does the work of a room-sized machine. RAMBO (Rice advanced magnet with broadband optics) allows researchers to run spectroscopy-based experiments on materials in pulsed magnetic fields up to 30 tesla. RAMBO was created in collaboration with Hiroyuki Nojiri at the Institute for Materials Research at Tohoku University, Sendai, Japan, and has windows that allow researchers to directly send a laser beam to the sample and collect data at close range.

"We can literally see the sample inside the magnet," explains physicist Junichiro Kono. "We have direct optical access, whereas if you go to



A palm-sized coil is the heart of RAMBO, a Rice-built tabletop system to expose experiments to high magnetic fields. Courtesy of Jeff Fitlow/Rice University.

a national high magnetic field facility, you have a monster magnet, and you can only access the sample through a very long optical fiber. You cannot do any nonlinear or ultrafast optical spectroscopy. RAMBO finally gives us the ability to combine ultrastrong magnetic fields and very short and intense optical pulses."

RAMBO's unique configuration reportedly allows for the best access in a powerful magnetic field generator meant for scientific experimentation. Researchers can collect realtime, high-resolution data in a system that couples high magnetic fields and low temperatures with direct optical access to the magnet's core, says Kono. In addition, the unit can run a new experiment in a 30-tesla field every 10 minutes (or less for smaller peak fields), as opposed to waiting the hours often required for field generators to cool down after each experiment at large laboratories. *For more information: Junichiro Kono, 713/348-2209, kono@rice.edu, www.rice.edu.* 

#### **Canadian 3D microscope explores defect formation**

A new 3D x-ray computed tomography (CT) microscope at the University of British Columbia's Okanagan campus lets researchers see inside the internal structure of materials and explore 3D images magnified 1000 times. According to assistant engineering professor André Phillion, this is the first step towards lighter and stronger materials that can be used in aerospace, energy, and manufacturing. The microscope is B.C.'s first high-resolution CT scanner, and one of only five in Canada. For those in manufacturing, this opens the window to determine how defects form and how they can lead to failure, says Phillion, who cites the basic aluminum alloy automobile wheel as an example.

The research is expected to not only improve manufacturing processes, like the casting of aluminum alloys for wheels, but also enhance performance and extend component lifetimes. "We are currently looking at a wide range of materials—metals, composites,

> paper-based products—and trying to decipher images that are enormously complex," says Phillion. "The end goal is to make products that are lighter and have fewer defects, and to also find new uses for traditional materials." *For more information: André Phillion, 250/807-9403, andre.phillion@ubc.ca, www.spsl.ok.ubc.ca.*

#### Testing liquid cells boosts battery research

Chongmin Wang of Pacific Northwest National Laboratory (PNNL), Richland, Wash., and colleagues used transmission electron microscopes (TEMs) to watch how the ebb and flow of positively charged ions deforms electrodes in batteries. Recent work funded through the DOE's Joint Center for



André Phillion demonstrates the small sample size to be imaged by UBC's new 3D x-ray CT microscope.

Energy Storage Research (JCESR) showed that sodium ions leave bubbles behind, potentially interfering with battery function. But up to this point, TEMs have only been able to accommodate dry battery cells.

Working with JCESR colleagues, Wang led development of a wet battery cell in a TEM at DOE's Environmental Molecular Sciences Laboratory on the PNNL campus. The team built a tiny battery with one silicon elecOfficial ceremonies held in December 2013 marked the formal rebranding of **Sherry Laboratories**, Daleville, Ind., under the **Element Materials Technology** name. Acquired by Element in March 2013, Sherry adds nearly 300 specialized staff and more than 10 facilities to Element's global laboratories. Sherry will continue to provide testing services to the aerospace and defense, oil and gas, transportation, power generation, environmental, microbiological and food, and petrochemical sectors. www.element.com.

trode and one lithium metal electrode, both contained in a bath of electrolyte. When the battery was charged, the silicon electrode swelled, as expected. However, under dry conditions, the electrode is attached at one end to the lithium source and swelling starts at just one end as ions push their way in. In the study's liquid cell, lithium could enter the silicon anywhere along the electrode's span, and swelled all along its length at the same time.

The total amount the electrode swelled was about the same, though, whether researchers set up a dry or wet battery cell, suggesting that either condition could be used to study certain aspects of battery materials. With regard to the elusive solid electrolyte interphase layer, Wang says they couldn't see it in this initial experiment. In the future, they will try to reduce the thickness of the wet layer by at least half to increase the resolution.

"This layer is perceived to have peculiar properties and to influence charging and discharging performance," explains Wang. "However, we do not have a concise understanding or knowledge of how it forms, its structure, or its chemistry. We expect the liquid cell will help us uncover this mystery layer." *For more information: Chongmin Wang*, 509/371-6268, *www.pnnl.gov.* 

Bombardier Aerospace, UK, selected Moog Industrial Group, East Aurora, N.Y., to supply an aerospace test system to provide fullscale structural testing on the CSeries aircraft advanced carbon fiber wing. The test system is being commissioned at Bombardier's facilities in Belfast, Northern Ireland, and will be used to carry out fatigue



testing of composite wing structures. www.moog. com/industrial.

Bombardier CSeries FTV1 during flight testing. Courtesy of Moog.

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### briefs

Researchers at DOE's **SLAC National Accelerator Laboratory** and **Stanford University**, Calif., say they built the first battery electrode that heals itself, potentially opening a new and commercially viable path for making next-generation lithium ion batteries. The secret is a stretchy polymer that coats the electrode, binds it together, and spontaneously heals tiny cracks that develop during battery operation. www6.slac.stanford.edu.

industry



Stanford researcher Chao Wang holds a solid piece of the self-healing polymer used to protect silicon battery electrodes. Courtesy of Brad Plummer/SLAC.

The U.S. Army Research Laboratory extended UC Santa Barbara's Institute for Collaborative Biotechnologies

(ICB) contract, providing an additional \$48 million over three years to support research inspired by biological systems. ICB supports basic research in six themes inspired by nature, such as the structure of sea sponges and adhesion of gecko feet. Themes range from understanding complex biological systems on a cellular level to engineering synthetic materials inspired by natural models. www.ucsb.edu.



A composite of multiwalled carbon nanotubes and carbon black serves as the matrix for in situ nucleation and growth of Pt nanocrystals. Courtesy of Peter Allen/UCSB.

#### Modeling metamaterials makes strides

A team led by Ecole polytechnique fédérale de Lausanne (EPFL), Switzerland, found a way to create computational models that can be applied to a range of metamaterials—artificial materials with properties not normally found in nature. Metamaterials are made of microscopic pieces of common materials like metals or plastics, which are arranged in precise repeating patterns, giving them unusual properties. For example, patterns designed at subwavelength sizes can affoct and manipulate light or sound unusus in v



Metamaterials have properties not normally found in nature and are useful in advanced optics.

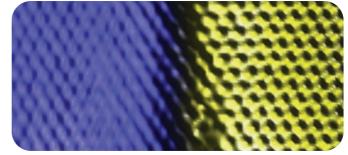
fect and manipulate light or sound waves in unconventional ways.

The best way to predict the full spectrum of metamaterial properties is through computer modeling, which requires some mathematical abstraction. A team led by Jan Hesthaven developed a computational approach that seeks to improve modeling efforts. An approach called the discontinuous Galerkin method, a class of numerical methods for solving differential equations, as well as a set of differential equations known as Maxwell's equations that describe how electromagnetic waves propagate in space and time, are used to model metamaterials. In order for a computer model to work, equations must be translated from continuous functions into discrete or noncontinuous ones.

Building on previous efforts, researchers developed a new way to solve Maxwell's equations specifically for metamaterials. The new method can greatly improve computer modeling of these materials, allowing faster discovery, design, and manufacturing of new forms and structures, says Hesthaven. *www.epfl.ch.* 

#### New approach to create 2D hybrid materials

Researchers at the DOE's Oak Ridge National Laboratory (ORNL), Tenn., and the University of Tennessee, Knoxville, created a new technique to form a 2D, single-atom sheet of two different materials with a seamless boundary. The team combined two com-



Colorized scanning tunneling microscope image shows a single-atom sheet composed of graphene (blue) combined with hexagonal boron nitride (yellow). Courtesy of ORNL.

pounds, graphene and boron nitride, into a single layer only one atom thick.

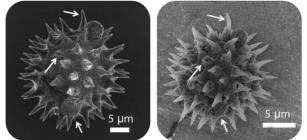
One method to combine materials into heterostructures is epitaxy, in which one material is grown on top of another so both have the same crystalline structure. To grow the 2D materials, the team directed the growth process horizontally instead of vertically. Graphene was first grown on copper foil, etched to create clean edges, and then boron nitride was grown using chemical vapor deposition. Instead of conforming to the structure of the copper base layer as in conventional epitaxy, the boron nitride atoms took on the crystallography of the graphene.

Not only did the technique combine the two materials, it also produced an atomically sharp boundary, a 1D interface, between them. This method can be applied to other combinations of 2D materials, assuming the different crystalline structures are similar enough to match one another, according to ORNL's An-Ping Li. *For more information: An-Ping Li,* 865/576-6502, *apli@ornl.gov, www.ornl.gov.* 

## SURFACE ENGINEERING

#### Magnetic pollen replicated for tailored adhesion

Researchers at the Georgia Institute of Technology, Atlanta, created magnetic replicas of sunflower pollen grains using a wet chemical, layer-by-layer process that applies highly conformal iron oxide coatings. The replicas possess natural adhesion properties inherited from the spiky pollen particles while gaining magnetic behavior, allowing for tailored adhesion to surfaces. Researchers



SEM images of a pollen particle (left) coated with iron oxide and a replica of the same particle (right) after firing at 600°C to remove the organic material and crystallize the iron oxide. Arrows point to features preserved by the process. Courtesy of Brandon Goodwin and Ken Sandhage.

chose particles from the sunflower (Helianthus annuus). The sunflower pollen grains are nearly spherical, but covered with spikes that can entangle with the hairs on bees' legs, or adhere to surfaces via van der Waals forces at nanometer-scale distances, explains Professor Ken Sandhage.

The burr-like pollen particles were washed with chloroform, methanol, hydrochloric acid, and water to clean the surfaces and expose hydroxyl groups for chemically attaching their coating. Iron oxide was applied using an automated, layer-by-layer surface sol-gel process developed earlier for coating diatom shells made of silica. Reaction of the iron oxide precursor with the hydroxyl groups on the surface of the pollen particles results in highly conformal coatings. *For more information: Ken Sandhage, 404/894-6882, ken.sandhage@mse.gatech.edu, www.gatech.edu.* 

#### Tougher dies for automotive manufacturing

Manufacturers need processes that increase the lifetime of dies and reduce setup times, such as laser metal deposition. A universal, reproducible process for practical industrial use is lacking, however. This deficiency was remedied by researchers from the Fraunhofer Institute for Production Technology IPT, Germany. They rebuilt a conventional five-axis milling machine so that it could be used to alloy forming dies automatically via laser. The machine can be embedded into the current manufacturing process and increase the lifetime of dies by more than 150%. The new process also improves the quality of components and makes



Using laser metal deposition makes the forming die from Mühlhoff Umformtechnik GmbH more robust. Courtesy of Fraunhofer IPT.

it possible to plan setup times with greater precision.

In addition to the laser metal deposition machine, another key part of the system is the integrated CAx software, which allows all the requisite laser surface treatment processes to be controlled in a clear and reproducible manner. All necessary process parameters are transmitted to the machine without the need for any interface. Processes can be simulated in detail and optimized in advance of actual processing operations. *www.ipt.fraunhofer.de/en.html.*  industry NeWS

## briefs

Abakan Inc., Miami, completed Phase I and II of its PComP nanocomposite coatings growth and expansion strategy, which involved installing additional powder microencapsulation equipment to double throughput and added more nanoparticle production equipment and sintering furnaces to expand production to 18 tons per year. Phases III and IV are scheduled to expand production to 60 and then 180 tons per year throughout this year. Further growth and expansion plans call for the acquisition of up to 10 U.S.-based thermal spray production businesses that serve the growing oil and gas sector. www.abakaninc.com.

New coatings developed with European Union support enable the release of healing agents in response to changes in the environment. The novel nanomaterials and coating technologies were developed by the EU-funded project Multi-level protection of materials for vehicles by "smart" nanocontainers (MUST).

Miniature containers (nanocontainers) were loaded with healing agents and incorporated into multilayer coatings. Changes in the environment release the agents and help transport them throughout the coating. Some of MUST's top performers include pre-treatments and primers for corrosion inhibition in automobile and aircraft components. http://cordis.europa.eu.

Alcoa, Pittsburgh, developed a proprietary surface finishing technology that enables consumer electronics companies to use aerospace-grade aluminum to make thinner, lighter, and stronger mobile devices. ProStrength Finishing Technology is a process that allows mobile device manufacturers to design clear or color-anodized durable surfaces using high strength, heat treatable aluminum alloys employed in the aerospace and defense industries. Using high strength alloys could reduce the thickness and weight of a device by more than 50%. Being able to use aerospace-grade alloys promotes the trend toward ever thinner and lighter mobile devices. www.alcoa.com.



## briefs

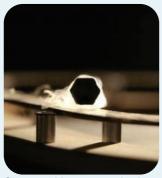
U.S. Department of Energy,

industry

Washington, D.C., announced more than \$13 million for five projects to strengthen domestic solar manufacturing and speed commercialization of efficient, affordable photovoltaic and concentrating solar power technologies. As part of the DOE's SunShot Initiative, these awards will help lower the cost of solar electricity, support a growing solar workforce, and increase U.S. competitiveness in the global clean energy market. www1.eere.energy.gov/solar/suns hot/index.html.

## Researchers at **The University of British Columbia,** Canada,

discovered a universal electronic state that controls the behavior of high-temperature superconducting copper-oxide ceramics. The work reveals the universal existence of so-called "charge density waves"-static ripples formed by the self-organization of electrons in the material's normal state. These ripples carry the seeds out of which superconductivity emerges. The findings suggest the existence of a universal chargeordering that is common to all cuprate materials, and uncover its connection to the emergence of superconducting behavior. The work also proves that two techniques-resonant x-ray scattering and scanning tunneling microscopy-can be used to probe the mysteries of charge density waves. www.ubc.ca.



Copper-oxide superconducting pellet levitating over a magnetic track. Courtesy of UBC Science.

#### Thermoelectric materials near production scale

Researchers at Fraunhofer Institute for Physical Measurement Techniques IPM, Freiburg, Germany, show that half-Heusler compounds—highly suitable materials for thermoelectric processes—can be produced significantly more efficiently and cost-effectively than before. "Half-Heusler compounds fulfill almost all of the necessary criteria," explains project director Benjamin Balke at the University of Mainz. "The alloys consist of a wide range of materials, nickel being one, and are much more environmentally friendly than



Individual components of thermoelectric modules are only a few millimeters in size. They are cut from specific alloys such as half-Heusler compounds. Courtesy of Fraunhofer IPM.

previous materials, possess good thermoelectric properties, and withstand high temperatures." Researchers achieved a ZT value of 1.2, which corresponds to the best published values for half-Heusler compounds so far, according to the team. It is crucial for industrial applications to attain the efficiency values achieved in the lab during actual mass production. *www.ipm.fraunhofer.de/en.html*.

#### Could rainfall power cell phones?

Georgia Tech, Atlanta, researchers are developing a family of power generators that take advantage of the triboelectric effect to produce small amounts of electricity for portable devices and sensors. Professor Zhong Lin Wang is using the triboelectric effect to create surprising amounts of electric power by rubbing or touching two different materials together. He believes the discovery could power mobile devices such as sensors and smartphones by capturing the otherwise wasted mechanical energy from sources such as walking, wind currents, vibration, ocean waves, or cars driving by.

"The fact that an electric charge can be produced through triboelectrification is well known," Wang explains. "What we have introduced is a gap separation technique that produces a voltage drop, which leads to a current flow in the external load, allowing the charge to be used. This generator can convert random mechanical energy from our environment into electric energy." Wang and his research team report that a square meter of single-layer material can now produce as much as 300 W. They have found that the volume power density reaches more than 400 kW/m<sup>3</sup> at an efficiency of more than 50%. *For more information: Zhong Lin Wang, 404/894-8008, zhong.wang@mse.gatech.edu, www.nanoscience.gatech.edu.* 

#### Test facility to improve wind turbines

Premature failures of mechanical systems have a significant impact on the cost of wind turbine operations and the total cost of wind energy. The DOE's National Renewable Energy Laboratory (NREL), Golden, Colo., added a new 5-MW Dynamometer Test Facility at its National Wind Technology Center (NWTC). The facility is capable of testing drive-trains up to 5 MW—large enough to test virtually any land-based turbine—and employs dynamically variable loading capabilities that allow researchers to better simulate turbine field conditions. A dynamometer system replaces the rotor and blades of a wind turbine and allows researchers to control the turbine drivetrain's mechanical and electrical systems

while simulating normal and extreme operating conditions. The facility incorporates a non-torque loading system into the testing regimen, a hydraulic device that allows for simulation of both the rotational and bending loads that a wind turbine rotor places on a drivetrain. *www.nrel.gov/wind*.

A visitor looks at the high-speed driveshaft on the new dynamometer at the NWTC's 5-MW Dynamometer Test Facility. Courtesy of Dennis Schroeder.



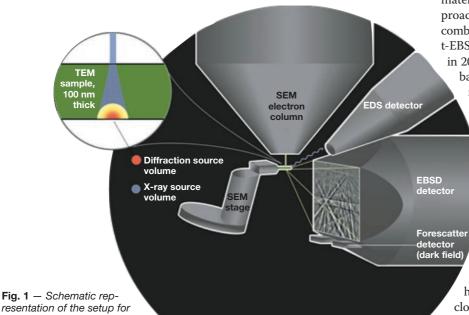
## Transmission Kikuchi Diffraction in the Scanning Electron Microscope: Orientation Mapping on the Nanoscale

Patrick W. Trimby Julie M. Cairney The University of Sydney, Australia

> By changing a setup and using electron transparent samples, hundreds of **laboratories** worldwide have access to a characterization technique that measures grain sizes and orientations in nanocrystalline materials.

lectron backscatter diffraction (EBSD) is one of the most established microanalytical techniques for characterizing microstructures of crystalline materials. EBSD application has grown dramatically since the first commercial systems became available in the early 1990s, as today most materials characterization laboratories have access to an EBSD system. Reasons for its popularity include speed, ease of use, and the extensive range of applications in which it can be used. The primary reason, however, is that EBSD provides unique information at an impressive spatial resolution. On a field emission gun scanning electron microscope (FEG-SEM), EBSD can measure phase and orientation from domains as small as 100 nm<sup>[1]</sup>. The exact value depends on the material in question, but EBSD can effectively characterize materials with grain sizes in the sub-micrometer range.

The Hall-Petch relationship, observed in the early 1950s, in which yield strength in materials was demonstrated to be inversely related to grain size<sup>[2,3]</sup>, has inspired materials scientists to produce finer grained materials. As grain size approaches the nanocrystalline range (i.e., under 100 nm), a material's physical properties change dramatically and are often superior to conven-



tional microcrystalline materials. Not only do strength and hardness increase, but fatigue resistance, diffusivity, and even magnetic properties are shown to improve<sup>[4]</sup>. The small grain sizes of these materials pose a problem for materials characterization: With mean grain sizes in the 100 nm range, EBSD, even on a FEG-SEM, does not have the necessary spatial resolution. The solution has been to turn to the transmission electron microscope (TEM), in which the higher electron beam energy and low beam spread through electron transparent samples combine to offer subnanometer resolution.

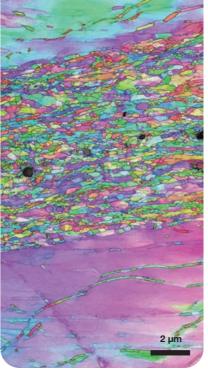
In the last few years, a simple yet innovative alternative strategy was proposed by a group of researchers at the National Institute of Standards and Technology (NIST), Boulder, Colo., led by Robert Keller and Roy Geiss. They demonstrated that a conventional EBSD system in a FEG-SEM could be used to collect diffraction patterns from sub-10 nm domains in an electron transparent sample<sup>[5]</sup>. They named it transmission EBSD, or t-EBSD. By simply changing the setup and using electron transparent samples (such as those used for TEM studies), hundreds of laboratories worldwide have access to a characterization technique that is able to measure grain sizes and orientations in nanocrystalline materials. Realizing the potential of this approach, at the University of Sydney we started combining automated orientation mapping with t-EBSD and published some of our first results in 2012<sup>[6,7]</sup>. Because the technique involves no backscattering of electrons, we proposed a more scientifically accurate name: Transmission Kikuchi diffraction, or TKD.

#### **TKD** methodology

Setting up for automated TKD analysis is relatively simple—samples need to be thin, typically under 200 nm in thickness (although for many materials, below 100 nm is optimal). Standard TEM sample preparation techniques such as electropolishing or ion-beam milling are usually sufficient. The sample is then mounted in the SEM chamber in a horizontal or sub-horizontal orientation, close to the level of the top of the EBSD detector, as shown schematically in Fig. 1. The electron beam, usually set to the highest accelerating voltage (30 kV for most SEMs), is focused onto the sample and the Kikuchi diffraction pat-

resentation of the setup for TKD in the SEM. The inset highlights the source volumes for electron diffraction and x-ray excitation within an electron transparent sample.

Fig. 2 — Large scale TKD orientation map showing a nanocrystalline shear band cutting across coarse grains in a naturally aged ECAPdeformed Al-Mg-Cu alloy.



tern is projected from the bottom surface of the sample onto the EBSD detector to the side. Standard commercial EBSD software indexes the diffraction pattern: Indexing speeds are similar to conventional EBSD, between 20 and 100 analyzed points per second, which allows orientation maps to be collected by stepping the electron beam across the sample surface on a regular grid of points. The horizontal sample



orientation is also ideal for simultaneous chemical analysis using energy dispersive x-ray spectroscopy (EDS), providing elemental distributions on the submicron scale. The measurement spacing (step size) for automated TKD analyses is routinely between 2 and 20 nm.

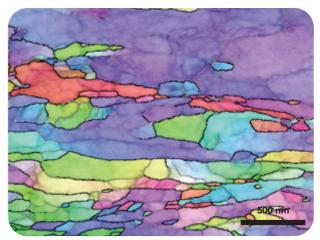
The spatial resolution of the TKD technique depends on a number of factors. The electron beam is scattered as it interacts with the sample, and some of these scattered electrons will impinge on crystal lattice planes at the necessary Bragg angle for diffraction. The diffracted electrons form the Kikuchi bands that make up the diffraction pattern. However, those electrons that are scattered in the upper part of the sample are likely scattered further before exiting towards the detector, meaning they will not contribute significantly to the diffraction pattern. The end result is that the diffraction pattern originates close to the lower surface of the sample (Fig. 1 inset), enabling effective analysis even when grain size is significantly smaller than sample thickness. Many of the first results using TKD demonstrate effective spatial resolution for various materials between 2 and 10 nm<sup>[5, 7, 8]</sup>.

Results were collected at the Australian Centre for Microscopy and Microanalysis using a Zeiss Ultra FEG-SEM equipped with an Oxford Instruments Nordlys Nano EBSD detector, an X-Max 20 EDS detector, and running AZtec v2.2 software.

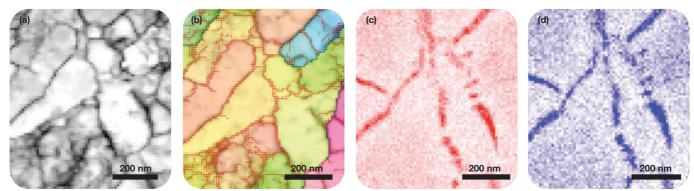
#### **Application examples**

The following examples all come from a series of Al-Mg-Cu alloys subjected to severe plastic deformation using equal channel angular pressing (ECAP). Samples underwent varying aging processes prior to deformation, resulting in significant variations in the final microstructures. All samples were subjected to 4-pass ECAP at room temperature.

Figure 2 shows a large area (>200  $\mu$ m<sup>2</sup>) orientation map from a naturally aged sample collected using TKD, with a step size of 7.5 nm. Full recrystallization has not taken place, with large grains at the top and bottom of the analysis area separated by a broad, nanocrystalline shear band. The large grains are significantly deformed, with many low angle



**Fig. 3** — High resolution TKD orientation map of part of the shear band shown in Fig. 2. Low angle boundaries ( $2-10^{\circ}$ ) are marked in gray, high angle boundaries (>10°) in black.



**Fig. 4** — Corresponding TKD and EDS element maps of an artificially aged ECAP-deformed AI-Mg-Cu alloy. (a) TKD diffraction pattern quality map. (b) TKD orientation map, with low angle boundaries (0.5-10°) in red, high angle boundaries (>10°) in black. (c) Mg distribution map. (d) Cu distribution map.

boundaries, and contain some intriguing ribbons of recrystallized nanocrystalline grains, yet little grain refinement has occurred. Closer inspection of the shear band shows a grain size in the region of 100-500 nm, although some grains are as long as  $2-3 \mu m$ . Contrary to interpretations of TEM images, grains are generally elongated and contain many low angle boundaries, shown by gray lines in Fig. 2.

A more detailed investigation of part of one such shear band is shown in Fig. 3. Here, the TKD step size is 5 nm and the elongated nature is clear, especially in the lower part of the area. Despite the presence of grains that extend across the whole field of view ( $2.5 \mu m$ ), mean grain diameter in this area is 208 nm (118 grains), compared to 170 nm (2064 grains) measured from the complete shear band shown in Fig. 2. Analysis using conventional EBSD would be impossible due to both the small grain size and highly deformed nature of the crystal lattice.

A detailed TKD orientation map of part of an artificially aged Al-Mg-Cu alloy is shown in Fig. 4a. Here, the EBSD detector collected high resolution diffraction patterns, enabling significantly improved orientation accuracy (better than  $\pm$  0.2°); the longer dwell time at each point improves the x-ray count, enabling high quality chemical mapping simultaneous to TKD measurements. The red lines in Fig. 4b mark low angle boundaries above 0.5° misorientation, while black lines represent high angle boundaries (>10°). The maps in Figs. 4c and 4d show the distribution of Mg and Cu respectively.

Several features of the microstructure are prominent: The very fine scale cell structure, dominated by low angle boundaries (the field of view is only 750 nm across), plus clear segregation of Mg and Cu into the boundaries. A closer look at the chemical segregation is even more intriguing—only some boundaries show any segregation, and there is no clear preference towards segregation at either high or low angle boundaries. The ability to correlate crystallographic and chemical data with exceptional spatial resolution (the Cu- and Mg-enriched zone across these boundaries is, in places, only 2 pixels or 20 nm across) and, potentially, to cover large numbers of grains is unique to integrated TKD-EDS mapping in the SEM and is proving an invaluable technique to unlocking the secrets of these deformed Al alloys.

#### Conclusions

Occasionally a new technique emerges that can dra-

matically enhance our ability to characterize materials on the micro- or nano-scale. The recent "discovery" of TKD in the SEM is different: It is a technique that uses instrumentation that is both widely available and easily used, yet promises remarkable improvements in spatial resolution compared to existing techniques. The examples shown in this article illustrate the power and versatility of the technique. Even on a light metal such as an Al-Mg-Cu alloy, TKD permits fast orientation mapping with spatial resolution better than 10 nm, and chemical mapping using simultaneous EDS with resolution on the order of 20-30 nm. This allows the routine characterization of true nanocrystalline materials in the SEM, offering grain size, boundary, texture, phase, and chemical measurements on a scale otherwise unachievable without using a TEM.

Potential applications of TKD for materials characterization are wide ranging. Up until now the focus has been primarily on bulk nanocrystalline materials in which the grain size is below the limit of conventional EBSD, but the improved spatial resolution also enables analysis of highly deformed structures<sup>[8]</sup>. Ongoing research using TKD has provided orientation data on materials as diverse as superconductors, solar cells, fuel cells, nanoparticles, and even iron oxides in magnetotactic bacteria. The impact of this new and exciting technique will only increase in the coming years.

For more information: Patrick Trimby, Australian Centre for Microscopy & Microanalysis, The University of Sydney, Madsen Building F09, NSW, 2006, Australia, 612/9351-7561, patrick.trimby@sydney.edu.au, http://sydney.edu.au/acmm.

#### Acknowledgements

The authors wish to thank Morgan Tort (The University of Melbourne, Australia) for providing the samples and permitting the authors to show results, and Deirdre Molloy for helping with Fig. 1.

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#### **JACQUET-LUCAS AWARD**

# **Extrusion Welding** in a Magnesium Alloy (AM30) Hollow Section Extrudate

Nabeel H. Alharthi\* Lehigh University

Bethlehem, Pa. King Saud University Riyadh, Saudi Arabia

Wojciech Z. Misiolek, FASM\* Lehigh University Bethlehem, Pa.

> This entry won the prestigious Jacquet-Lucas Award for Best in Show at the 2013 International Metallographic Contest held in conjunction with the M&M 2013 conference in Indianapolis.

he number of magnesium extruded parts used in the automotive industry is increasing due to their lightweight and high specific mechanical properties that facilitate reduced fuel consumption. However, technical challenges in the deformation processing of magnesium alloys exist and include low formability, which is attributed to their hexagonal close packed (hcp) crystal structure. The extrusion process is a promising metal forming process because it applies the hydrostatic compression state of stress in the deformation zone, enabling improved workability.

Producing hollow section profiles, such as the double hat extrudate shown in Fig. 1, uses a porthole die as shown in Fig. 2. When a magnesium alloy billet is extruded through the porthole die, material is separated by eight die arms into strands, which are then joined together under high temperature and high pressure conditions in the die's welding chamber. This joining process occurs as solid-state bonding and is known as extrusion welding. The main objective of this work is to better understand the integrity and microstructure of extrusion welding in a magnesium alloy (AM30) hollow section extrudate (See Table 1).

#### Metallographic sample preparation

Preparing metallographic samples of mag-

Fig. 2 - Porthole die

used for the double

hat extrusion.

nesium alloys is challenging because the alloys are relatively soft and can be easily scratched during preparation. In addition, twin deformation can occur during polishing and will be visible after etching. Further, some intermetallic particles always contaminate the polishing cloths and can cause additional scratches. Water should be avoided during the final polishing process because magnesium alloys have a tendency to stain when exposed to waterbased liquids.

Due to the extrusion profile symmetry, only three locations (A, B, and C) are shown in Fig. 1, which are equivalent to all eight extrusion welds that were metallographically investigated. Specimens were sectioned transversely to the extrusion direction, mounted, ground, and polished using standard metallographic specimen preparation techniques. Specimens were then chemically polished using 100 mL methanol, 12 mL hydrochloric acid, and 8 mL nitric acid for 10 seconds. Finally, specimens were etched using a solution of acetic picral (4 g picric acid, 5 mL acetic acid, 10 mL H<sub>2</sub>O, 100 mL ethanol) for 5 seconds<sup>[1]</sup>.

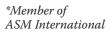
#### Results

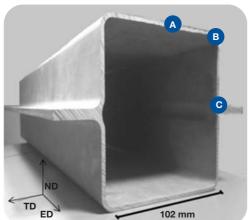
Figures 3, 5, and 6 are each a collage of polarized light optical microscopy (LOM) images in locations A, B, and C, respectively, and show

the different types of extrusion welding.

Figure 3 shows the difference in grain sizes between the middle of the image where the extrusion welding occurred and the rest of the extrudate near the edge of the image represents a lessmodified alloy microstructure in the welding chamber. Figure 4 is a magnified image captured in the middle of the specimen in location A (see Fig. 3), which

shows good extrusion weld integrity with no visible weld line. On the other hand, Figs. 5 and 6 show different types of extrusion welding with a curved weld line. The presence of the line is most likely due to oxidization of the strands' surfaces or insufficient pressure and/or temperature. These welding lines could be straight or tortuous, depending on the velocities of the two





**Fig. 1** — Investigated extrusion welding locations of the AM30 profile.

TABLE 1 - NOMINAL CHEMICAL COMPOSITION OF ALLOY AM30 (wt%)						
Mg	AI	Mn	Zn	Fe	Ni	Cu
Balance	3.4	0.33	0.16	0.0026	0.0006	0.0008

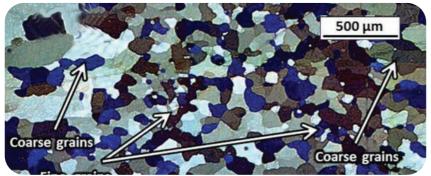
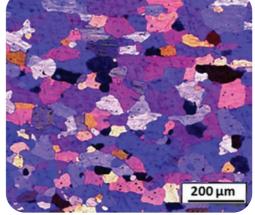


Fig. 3 – Polarized light optical microscopy (LOM) mosaic image of location (A) of the extrudate.



**Fig. 4** — Fine grain structure as evidence of good weld integrity in location (A) seen at higher magnification than in Fig. 3.

neighboring strands and their surface roughness and applied stresses.

Figure 7 shows some coarse grains along the extrusion weld lines. In contrast, necklacing of small grains appears along the extrusion weld lines in Fig. 8, which could represent newly recrystallized grains.

#### Conclusions

Three different samples representing various extrusion weld locations of a hollow section magnesium alloy AM30 extrudate were investigated. The following microstructural features were observed by using polarized LOM:

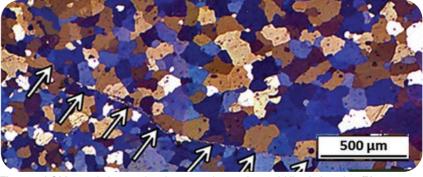
- Two types of extrusion welding were observed—one without a weld line, which indicates preferred extrusion welding, and the other with a weld line, which could represent a potential defect.
- Straight and tortuous weld lines occurred at extrusion welding locations with coarse and fine grains surrounding the weld line.

Further investigations are needed to learn the material chemistry of the weld line. More in-depth analysis will determine the nature of the potentially recrystallized small grain around the weld line. ⊃

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ig. 5 – LOM mosaic image showing curved extrusion weld line in location (B).

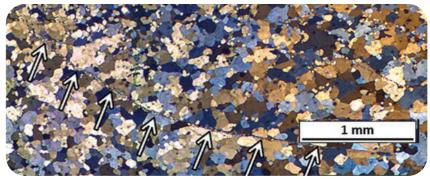
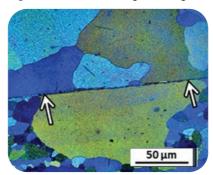


Fig. 6 - LOM mosaic image showing curved extrusion weld line in location (C).

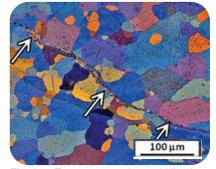


**Fig. 7** — Straight extrusion weld line in location (B).

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**Fig. 8** — Tortuous extrusion weld line in location (C).

## Additive Manufacturing of Titanium Alloys

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> Although the widespread use of titanium alloys is constrained by high costs, powder metallurgy techniques such as additive manufacturing represent an economical approach to fabricating titanium components.

itanium alloys are among the most important advanced materials and are key to improved performance in both aero-

space and terrestrial systems, due to an excellent combination of specific mechanical properties and outstanding corrosion behavior<sup>[1]</sup>. However, widespread use is constrained by the high cost of titanium alloys compared to alternative materials<sup>[1]</sup>.

The high cost of producing conventional titanium components has spurred numerous investigations into potentially lower cost processes, including powder metallurgy (PM) near-net-shape techniques such as additive manufacturing (AM)<sup>[1]</sup>. This article reviews AM with an emphasis on the "work horse" titanium alloy, Ti-6Al-4V. AM is receiving significant attention from numerous organizations including the U.S. Navy, as it envisions use aboard carriers with parts able to be rapidly fabricated for immediate use by

battle groups<sup>[2]</sup>. Various approaches to AM, along with examples of components made by different AM processes, are presented. The microstructures and mechanical properties of Ti-6Al-4V produced by AM are also discussed and compare well with cast and wrought products. Finally, the economic advantages of AM compared to conventional processing are presented.

#### Additive manufacturing overview

All AM technologies are based on the principle of slicing a solid model into multiple layers and building the part up layer by layer following the sliced model data. Following ASTM classification, AM technologies for metals can be broadly classified into two categories: directed energy deposition and powder bed fusion (Table 1). Several technologies fall under each category as branded by different manufacturers. While powder bed fusion

## TABLE 1 – ADDITIVE MANUFACTURING TECHNOLOGIES FOR PROCESSING TITANIUM AND ITS ALLOYS

AM Category	Technology	Company	Description
Directed Energy Deposition (DED)	Direct Metal Deposition (DMD)	DM3D Technology LLC (formerly POM Group)	Laser and metal powder used for melting and depositing with a patented closed loop process.
	Laser Engineered Net Shaping (LENS)	Optomec Inc.	Laser and metal powder used for melting and depositing.
	Direct Manufacturing (DM)	Sciaky Inc.	Electron beam and metal wire used for melting and depositing.
Powder Bed Fusion (PBF)	Selective Laser Sintering (SLS)	3D Systems Corp. (acquired Phenix Systems)	Laser and metal powder used for sintering and bonding.
	Direct Metal Laser Sintering (DMLS)	EOS GmbH	Laser and metal powder used for sintering, melting, and bonding.
	Laser Melting (LM)	Renishaw Inc.	Laser and metal powder used for melting and bonding.
	Laser Melting (SLM)	SLM Solutions GmbH	Laser and metal powder used for melting and bonding.
	LaserCUSING	Concept Laser GmbH	Laser and metal powder used for melting and bonding.
	Electron Beam Melting (EBM)	Arcam AB	Electron beam and metal powder used for melting and bonding.

\*Member of ASM International technologies enable construction of complex features, hollow cooling passages, and high precision parts, they are limited by the build envelope, single material per build, and horizontal layer construction ability. In comparison, directed energy deposition technologies offer larger build envelopes and higher deposition rates, but their ability to construct hollow cooling passages and finer geometry is limited. Direct metal deposition (DMD) and laser engineered net shaping (LENS) technology can deposit multiple materials in a single build and add metal to existing parts.

Commercially available AM technologies melt powder or wire feedstock using either laser or electron beam heat sources. Laser-based systems operate under inert atmosphere (for titanium processing) in contrast to the vacuum environment of electron beam systems. While vacuum systems are more expensive, they offer low residual stress compared to laser-based systems, and electron-beam-processed parts can be used without stress relieving operations. Heat source effects on mechanical properties are discussed in more detail further in this article.

#### **Powder bed fusion**

Powder bed fusion technologies place a layer of metal powder on the build platform and then the powder is scanned with a heat source, such as a laser or electron beam, to either partially or completely melt the powder in the path of the beam and resolidify and bind it together as it cools (ASTM specification F2924-12a and -13 for Ti-6Al-4V and Ti-6Al-4V ELI grades, respectively). Layer-by-layer tool path tracing is governed by the CAD data of the part being built. Figure 1 shows a schematic explaining the steps involved in this process:

- A substrate is fixed on the build platform.
- The build chamber is filled with inert gas (for laser processing) or evacuated (for electron beam processing) to reduce the chamber's oxygen level to the desired level.
- A thin layer of metal powder (20-200 mm thick, depending on the technology and equipment) is placed on the substrate and leveled to a predetermined thickness.
- The laser or electron beam scans the powder bed surface following the tool path precalculated from the CAD data of the component being built.
- This process is repeated for the following layers until the build is complete.

#### **Directed energy deposition**

Directed energy deposition technologies work by injecting material into a meltpool rather than scanning a powder bed (AMS specification 4999A for Ti-6Al-4V). Figure 2 shows a schematic of DMD technology (laser-based metal deposition). Steps for the directed energy deposition process include:

• A substrate or existing part is placed on the work table.

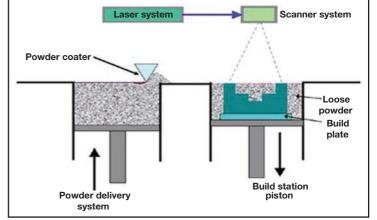
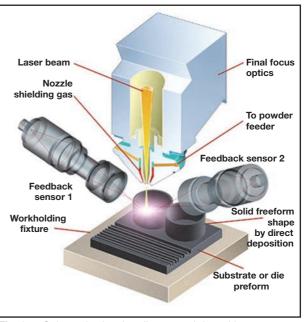


Fig. 1 — Schematic showing powder bed fusion technology. Courtesy of Jim Sears.



**Fig. 2** — Schematic showing direct metal deposition technology. Courtesy of DM3D Technology.

- Similar to powder bed fusion, the machine chamber is closed and filled with inert gas (for laser processing) or evacuated (for electron beam processing) to reduce the chamber's oxygen level to the desired level (AMS 4999A specifies below 1200 ppm). The DMD process offers local shielding and does not require an inert gas chamber for less reactive metals than titanium, such as steels, Ni alloys, and Co alloys.
- At the start of the cycle, the process nozzle with a concentric laser or electron beam is focused on the part surface to create a meltpool. Material delivery involves powder traveling through a coaxial nozzle (laser) or through a metal wire with a side delivery (electron beam). The nozzle moves at constant speed and follows a predetermined toolpath created from the CAD data. As the nozzle (tooltip) moves away, the meltpool solidifies and forms a metal layer.

TABLE 2 – COMPARISON OF VARIOUS ADDITIVE MANUFACTURING TECHNOLOGIES				
Item/Process	Laser-based PBF (DMLS)	Electron beam-based PBF (EBM)	Laser-based directed energy deposition (DMD)	
Build envelope	Limited	Limited	Large and flexible	
Beam size	Small, 0.1-0.5 mm	Small, 0.2-1 mm	Large, can vary from 2-4 mm	
Layer thickness	Small, 50-100 μm	Small, 100 μm	Large, 500-1000 μm	
Build rate	Low, cc/h	Low, 55-80 cc/h	High, 16-320 cc/h	
Surface finish	Very good, Ra 9/12 μm, Rz 35/40 μm	Good, Ra 25/35 μm	Coarse, Ra 20-50 μm, Rz 150-300 μm, depends on beam size.	
Residual stress	High	Minimal	High	
Heat treatment	Stress relief required, HIP'ing preferred.	Stress relief not required, HIP'ing may or may not be performed.	Stress relief required, HIP'ing preferred.	
Chemistry	ELI grade possible, negligible loss of elements.	ELI grade possible, loss of Al needs to be compensated for in powder chemistry.	ELI grade possible, negligible loss of elements.	
Build capability	Complex geometry possible with very high resolution. Capable of building hollow channels.	Complex geometry possible with good resolution. Capable of building hollow channels.	Relatively simple geometry with less resolution. Limited capability for hollow channels.	
Repair/Remanufacture	Possible only in limited applications (requires horizontal plane to begin remanufacturing).	Not possible	Possible; capable of adding metal on 3D surfaces under 5+1-axis configuration, making repair solutions attractive.	
Feature/metal addition on existing parts	Not possible	Not possible	Possible; depending on dimensions, ID cladding is also possible.	
Multi-material build or hard coating	Not possible	Not possible	Possible	

• Successive layers follow the same principle and build up the part layer-by-layer.

Table 2 provides a comparison of capabilities, benefits, and limitations of various AM technologies used for producing titanium parts<sup>[3-6]</sup>.





**Fig. 3** — Hydraulic manifold built using EBM technology. The part was built at Oak Ridge National Laboratory (ORNL). Courtesy of ORNL.

**Fig. 4** — Medical implant application. Courtesy of Jim Sears.

#### **Titanium AM applications**

Extensive exploration regarding use of AM titanium parts in aerospace and medical applications is underway. Other potential AM applications include the chemical and defense industries. While powder bed fusion technologies are suitable for smaller, complex geometries with hollow unsupported passages/structures, directed energy deposition is better suited for larger parts with coarser features requiring higher deposition rates.

Use of finer powder grains combined with smaller laser/electron beam size achieves a superior surface finish on the as-built parts from the powder bed fusion technologies compared to directed energy deposition technologies. However, the majority of AM parts require finish machining for most applications. Beyond building new parts, the ability of directed energy technologies to add metal onto existing parts makes it possible to apply protective surface coatings, remanufacture and repair damaged parts, and reconfigure or add features to existing parts.

#### **Complex geometry**

A small beam size and small layer thickness, along with support of the powder bed, allow powder bed fusion technologies (such as EBM, DMLS, or SLS) to produce complex geometries with high precision and unsupported structures. Figure 3 shows an example of a hydraulic manifold mount for an underwater manipulator built using EBM technology. Building the integrated mount and manifold with internal passageways in a single operation eliminates fabrication of multiple parts and costs much less. A quality surface finish eliminates the need to machine finish all surfaces except seal surfaces and threading of screw holes. Generally, the PBF technique achieves a better surface finish than the DED approach, although demanding applications such as aerospace require finish machining. Figure 4 shows a biomedical implant built with a Ti-6Al-4V alloy using DMLS technology. These technologies also make it possible to build patient-specific custom implants.

#### Adding features to existing parts

Directed energy deposition technologies, such as DMD and/or LENS, can add metal to 3D surfaces to allow additional features to be added to existing parts

and/or blanks, which is not possible using the PBF approach. Adding features to a forged or cast preform, as opposed to machining such features, can result in the most cost-effective manufacturing process, where a significant reduction of preform size and weight can be achieved by eliminating the need for a machining allowance. Examples include various casings and housings in jet engines where flanges and bosses can be added on cast or forged cylindrical preforms. To illustrate, Fig. 5 shows a feature added to a titanium fan casing for an aerospace engine.

#### Remanufacturing

One of the application areas best suited to directed energy deposition techniques is remanufacturing and repair of damaged, worn, or corroded parts. Due to the ability to add metal to select locations on 3D surfaces, these technolo-

gies can be used to rebuild lost material on various components<sup>[7-9]</sup>. Closed loop technologies, such as DMD, achieve a minimum heat affected zone (HAZ) in the repaired part, which helps retain its integrity.

Figure 6 shows cross-section microstructures of the DMD area of a remanufactured turbine blade. Excellent process control during DMD leads to a fully dense microstructure as observed in the vertical cross-section. A layer thickness of roughly 0.1-0.2 mm was applied and a minimal HAZ occurs in the as-deposited blade. The DMD vision system plays a significant role in this type of remanufacturing application. An integrated, calibrated vision system allows automatic identification of part location in the machine coordinate system, resulting in precise processing. Other titanium components that can be repaired using DMD include housings, bearings, casing flanges, and landing gears.



Fig. 5 — Fan case produced by adding features with AM (laser-aided directed energy deposition) to a forged perform. Courtesy of Jim Sears.

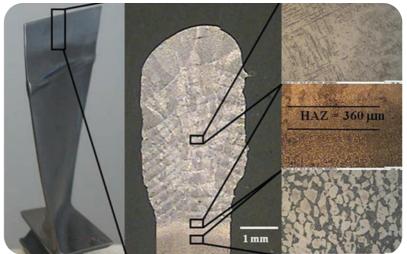


Fig. 6 — DMD repair of turbine components. Left, repaired vane; middle, macro cross-section; and right, microstructures (top to bottom shows the clad, interface, and base material). Courtesy of DM3D Technology.

#### **Microstructure and mechanical properties**

The Aerospace Materials Specification SAE AMS4999A covers Titanium Alloy Direct Products Ti-6Al-4V Annealed. This calls for a post-build annealing treatment of 1025°F (550°C). If a hot isostatic pressing (HIP'ing) treatment is used, it should be at no less than 14.5 ksi (100 MPa ) within the 1650°-1750°F (899°-954°C) temperature range for 2-4 hours followed by a slow cool to below 800°F (427°C). Minimum tensile properties should be UTS 124-129 ksi (855-889 MPa, depending on direction), YS 110-116 ksi (758-800 MPa), and elongation of 6%.<sup>[10]</sup>

Typical microstructures of as-built material using the DMD process and after subsequent HIP'ing and aging are shown in Fig. 7. The as-built microstructure shows the typical martensitic structure expected for Ti-6Al-4V cooled rapidly from the beta phase field, while

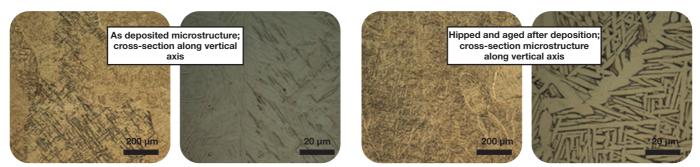
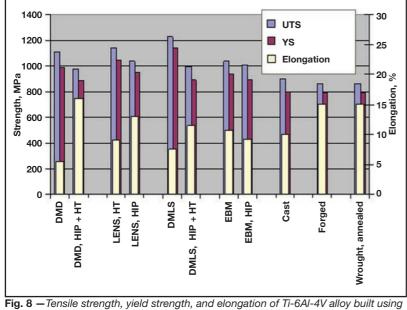
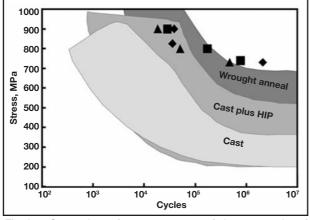


Fig. 7 — Microstructure of DMD-built Ti6AI4V before and after HIP'ing. Courtesy of DM3D Technology.

the HIP'd and aged material shows the expected grain boundary of alpha and intergranular coarse alpha plates. This microstructural transition from as-deposited to the HIP'd-aged condition is also reflected



various AM processes: DMD (direct metal deposition)<sup>[11]</sup>, LENS (laser engineered net shaping)<sup>[12]</sup>, DMLS (direct metal laser sintering)<sup>[6]</sup>, EB (electron beam melting)<sup>[6]</sup>, HIP (hot isostatic pressing), HT (heat treatment).



**Fig. 9** — Comparison of room temperature fatigue properties of Ti-6AI-4V processed by AM versus conventionally fabricated Ti-6AI-4V.  $\blacksquare$ ,  $\blacklozenge$ , and  $\blacktriangle$  represent properties in the three orthogonal directions, x, y, and z respectively. Courtesy of EADS/Jim Sears.

through their tensile properties. While tensile strength and yield strength is a little lower after HIP'ing and aging, ductility improves significantly as a result of the microstructure changing from martensitic to a trans-

> formed beta (precipitated alpha) structure. Asbuilt electron beam processed material contains a similar microstructure, although martensite is replaced by a lamellar alpha phase.

> Tensile properties of Ti-6Al-4V fabricated by a number of additive manufacturing techniques are shown in Fig. 8. All processes achieve strength levels superior or comparable to conventional material (cast, forged, and wrought annealed). As-built materials in laser-based processes such as DMD, LENS, and DMLS exhibit less ductility due to formation of the martensite phase. However, ductility can be improved through subsequent HIP'ing and/or heat treatment. As a result of reduced residual stress, EBM-processed Ti-6Al-4V achieves greater ductility when compared to laser-processed Ti-6Al-4V. Fatigue properties were tested using many different cycles. In general, as-built Ti-6-4 offers fatigue resistance similar to cast and wrought material, even without HIP treatment, as shown in Fig. 9.

#### Additive manufacturing economics

Among the main benefits of powder bed fusion processes is their ability to create hollow structures and therefore achieve weight savings. The aerospace industry, where weight savings can make significant impacts, is actively looking into AM processes. A case study involving a seat buckle for commercial passenger jets is a prime example of this capability<sup>[13]</sup>.

A lightweight seat buckle with hollow structures was designed based on an extensive finite element analysis study to ensure adequate strength against shock loading. The part was produced using a DMLS Ti-6Al-4V alloy. Replacing a conventional steel buckle with a hollow AM titanium buckle achieves weight savings of 85 g per buckle, a 55% weight reduction. Applying this across an Airbus A380 with 853 seats results in weight savings of 72.5 kg. According to the project sponsor, Technology Strategy Board, UK, this weight savings translates to 3.3 million liters of fuel savings over the life of the aircraft, equivalent to \$3 million, while the cost of making the buckles using DMLS is only \$256,000.

The direct manufacturing ability of AM technologies also helps reduce manufacturing costs in the case of high buy-to-fly ratio parts. For example, researchers at Oak Ridge National Laboratory built a Ti-6A-4V Bleed Air Leak Detect (BALD) bracket for the Joint Strike Fighter (JSF) engine using EBM technology (Fig. 10)<sup>[14]</sup>. Traditional manufacturing from wrought Ti-6Al-4V plate costs almost \$1000/lb due to a high (33:1) buy-to-fly ratio as opposed to just over 1:1 for the AM-built part. Estimated savings through AM is approximately 50%.

Direct deposition techniques such as DMD can not only be used to create parts, but these technologies can also be used for remanufacturing, repair, and/or feature building on existing parts. Damaged aerospace titanium components such as bearing housings, flanges, fan blades, casings, vanes, and landing gears can be rebuilt using these technologies at 20-40% of the cost of new parts[11]. Worn flanges in jet engine casings have been rebuilt using DMD at less than half the cost of new parts. Extensive work is also underway to investigate the feasibility of using such technologies to salvage components that are mismachined during conventional manufacturing. Successful realization of these efforts will have a significant impact on the titanium manufacturing industry. While most of the commercial activities in the AM industry are concentrated in the U.S. and Europe, significant efforts are underway in other parts of the world as well, including China<sup>[15]</sup>.

#### Conclusions

Significant advances in additive manufacturing technologies over the past few years have led to the production of fully functional parts using titanium and its alloys. While powder bed fusion technologies offer the ability to build hollow near-net shapes with finer resolution, directed energy-based technologies offer the ability to add features to existing parts and remanufacture and repair damaged parts, besides building parts directly from CAD data. Most studies reveal that the properties of AM material are as good as, or superior to, conventionally fabricated titanium alloys. Matching the correct AM technology to the application, along with proper design optimization, can achieve significant savings by reducing both weight and scrap. The aerospace and medical industries have so far been the largest users of titanium AM materials, while other industries, such as automotive, are beginning to exploit the benefits of AM titanium alloys as well.

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**Fig. 10** — BALD bracket for Joint Strike Fighter (JSF) built using EBM technology. Courtesy of ORNL.

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# Metallurgy Lane

## Wrought Iron: A Metal for the Ages

Early humans had no knowledge of iron. Unlike gold, silver, and copper, iron does not exist as a native metal. Here we explore the early days of wrought iron production.

ll iron, except for occasional meteorites, is tied up in compound form. Therefore, its discovery must have been fortuitous as humans built fires and placed stones within the fire pits to maintain heat. People learned that iron could be reduced from iron-rich stones at high temperatures in an atmosphere of burning wood. This type of iron was not melted, but was formed in a solid state as stone was slagged off, leaving a mass of iron particles at the bottom of the pit. The iron was essen-

Wrought iron was used to make body armor, as seen in this exhibit at The Metropolitan Museum of Art, New York. Courtesy of Mattes. tially carbon-free because it never reached a temperature in which carbon could be dissolved. As is widely known, carbon is the element in iron that

produces steel, which can then be heat treated to achieve higher strength levels. Iron particles can also be consolidated by hammering hot or cold, as is done with particles of gold, silver, and copper.

As time went on, special fire pits were built to increase the amount of iron that could be produced. Bellows were used to raise the temperature by blowing air into the fire. With further refine-



The Hudson River Chain was used during the Revolutionary War to prevent British naval vessels from sailing upriver. The Great Chain, shown here, was constructed in 1778 with 750-lb links forged in Orange County, N.Y. Courtesy of Wikimedia Commons.

ment, this process was used for many centuries to produce what we call "wrought iron." During the thousands of years that humans have used iron, wrought iron has been the dominant form, as it could be worked hot or cold to produce weapons, armor, knives, and many items for everyday life.

#### **Evolution of wrought iron**

By the Middle Ages, blowing engines and larger furnaces increased temperatures until the iron melted. The molten iron dissolved the carbon from the fuel until it reached 3-4% of the iron. When this iron cooled, it was brittle and could not be worked either hot or cold and could only be cast into molds that formed the shape of the finished article. This "cast iron" could be used for cannons, cannon balls, utensils, and eventually stoves, lamp posts, and other products, yet it could not replace wrought iron.

Demand for wrought iron greatly increased with the advent of the Industrial Revolution. At the same time, a new process called "puddling" was developed in England during the late 18th century by Henry Cort and others. The puddling process was carried out in a reverberatory furnace, in which the fire is separate from the "charge," which was cast iron from the blast furnace rather than iron ore. Heat and gases from the fire passed over the cast iron, both melting it and removing the carbon. Cast iron melts at roughly 2100°F and is dissolved into the slag. As carbon is removed, the melting point of the carbon-free iron increases to 2800°F, higher than the furnace temperature. The iron then precipitates as solid particles that fall through the slag and onto the hearth as a mixture of iron and slag. This mixture is then agitated with a paddle to form a semisolid ball, which is handled as ordinary wrought iron and made in batches of approximately 500-600 lb.

In the first half of the 19th century, iron production increased rapidly, from about 50,000 tons at the beginning to more than 500,000 tons by 1850 with primary demand from the railroads. Railroading started in the U.S. in 1830 with roughly 10 miles of track outside of Charleston, S.C., and somewhat more laid by the Baltimore and Ohio Company. By 1840, 3000 miles of track had been constructed. By 1850, 9000 miles were complete. During the 1850s,

Metallurgy Lane is a new series authored by **ASM life member** Charles R. Simcoe, developed to share the early history of the U.S. metals and materials industries along with key milestones and developments. Simcoe, a World War II veteran, holds a degree in metallurgical engineering from Purdue University and spent his career in physical metallurgy R&D at Westinghouse Atomic Power Division, Battelle Memorial Institute, **Armour Research** Foundation. and Simonds Steel Co.



The Menai Suspension Bridge, UK, designed by Thomas Telford and completed in 1826, features large amounts of wrought iron. Courtesy of Bencherlite.



Workers building the Aquia Creek and Fredericksburg, Va., railroad. Wrought iron production reached its peak during construction of the intercontinental railroads. Courtesy of the U.S. National Archives.

another 20,000 miles were added in the U.S.—before even a single rail was laid west of the Mississippi.

As the railroads connected major Eastern cities and pushed into the Midwest, demand for iron kept moving west along with much of the U.S. population. For example, Chicago had one rail line and a population of 29,000 in 1850. By 1860, it had 11 rail lines and 109,000 inhabitants. Demand for iron created a boom in iron making west of the Allegheny Mountains in Pennsylvania as well as in Kentucky and southeast Ohio, which was home to more than 50 blast furnaces that supplied iron throughout the Civil War.

#### John Fritz and other iron pioneers

An early pioneer from this period of iron making is John Fritz. Born on a farm in eastern Pennsylvania in 1822, Fritz was apprenticed to the blacksmith trade at age 16. After working in several iron plants, he had enough experience to take charge of a new facility in Johnstown, Pa., which later became well known as the Cambria Iron Company. An entrepreneur by the name of Daniel Morrell had leased this unfinished Johnstown facility, in which two previous sets of investors had gone broke trying to build an integrated mill to produce railroad rails. Once fully developed, this mill would use coke in its blast furnaces and incorporate both puddling furnaces and rolling mills.

Fritz's first job was to make sure all machinery was operating properly and could handle the flow of iron in its progression from the blast furnace to puddling furnaces to squeezers and finally to the rolling mills. He discovered that the local ores were not suitable for making rails, which required persuading the financially strapped principals to buy better ore and pay the freight charges to bring it to Johnstown. However, this did not solve the problem of excessive scrap production during rolling. The mill was losing money and Fritz concluded that the current rolling method was part of the problem.

The standard rolling method was to pass a hot billet through the rolls and with pivoted tables on the output side, the semi-finished rail was lifted and shoved across the top roll to the entrance side where it was again passed through a second groove in the rolls to further reduce the metal to a particular rail shape. The process was repeated in successively smaller openings until the final rail was formed. Because rails were cracking during the later rolling stages, Fritz determined that the metal was too cold for the attempted reduction. His solution was to stack a third roll over the two working rolls (with a second set of grooves between the second and third rolls) so that the rail could be reduced in both directions. This helped keep the metal hot due to both the faster reduction time and shorter total rolling time.

Fritz designed and built the first three-high rolling mill in 26 days. It was immediately successful, and soon all rails manufactured in the U.S. were made using this mill design. However, the task of commissioning a new plant, solving numerous mechanical and metallurgical problems, and the continuous lack of adequate finances finally brought Fritz to exhaustion. He wrote in his notes, "After six years of as hard, laborious, faithful, vexatious work as ever fell to the lot of man to do, I decided to leave the scene of my early struggles and try my fortunes elsewhere." Fritz then joined the new Bethlehem Iron Company where he spent the rest of his career.

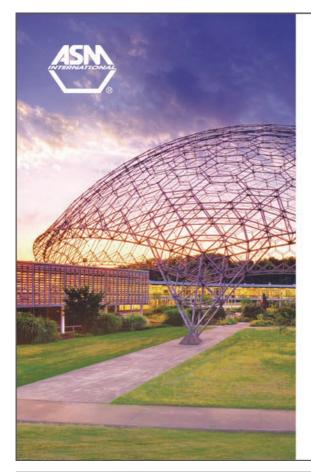
#### Wrought iron production wraps up

Wrought iron production reached its peak during construction of the intercontinental railroads. By 1880, it was being replaced by the new Bessemer process for making steel. Yet substantial production of wrought iron continued for another 30 years, as it was highly valued for toughness and corrosion resistance. For these reasons, the Eiffel Tower was built with wrought iron rather than steel. Although wrought iron is no longer produced, it will be remembered as the primary method for iron making for most of human history.

For more information: Charles R. Simcoe can be reached at crsimcoe@yahoo.com. For more metallur-gical history, visit www.metals-history.blogspot.com.



Iron pioneer John Fritz stands beside an 800,000-lb Riehle testing machine, 1909. Courtesy of Lehigh University.



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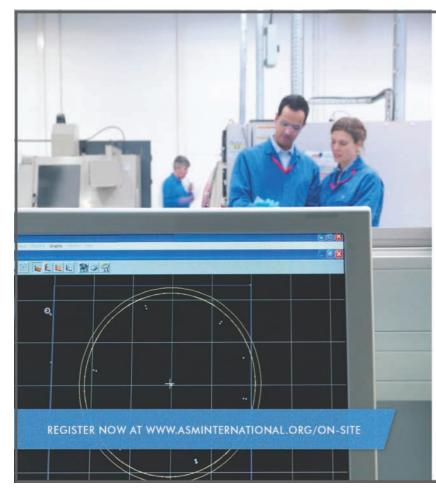
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FEBRUARY 2014 • VOL 9 • ISSUE 1





THE OFFICIAL NEWSLETTER OF THE ASM THERMAL SPRAY SOCIETY

# **Thermal Spray Coatings in the Aerospace Industry/ Military**

JTST Highlights Society

Society News

tss.asminternational.org

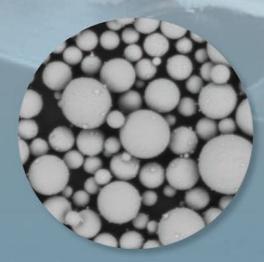






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#### THE OFFICIAL NEWSLETTER OF THE ASM THERMAL SPRAY SOCIETY

Nanocoating Provides Superior Corrosion Protection Via Two-Coat System

- 6 Accepted Practices to Test Bond Strength of Thermal Spray Coatings
- 9 Method Measures Ceramic Coating Stress and Damage on Turbine Blades

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- 12 JTST Highlights

#### About the cover

The Teslan coating from Tesla NanoCoatings is being tested in a number of field demonstration projects including the fuel tanks seen here. www.teslanano.com.

#### **Editorial Opportunities for iTSSe in 2014**

The editorial focus for iTSSe in 2014 reflects established applications of thermal spray technology such as power generation and transportation, as well as new applications representing new opportunities for coatings and surface engineering.

May	Energy/Power Generation Industries
August	Automotive Industry/Industrial Applications
November	Emerging Technologies

To contribute an article to one of these issues, please contact the editors c/o Julie Kalista at Julie.Kalista@asminternational.org. To advertise, please contact Kelly Thomas, Kelly.Thomas@asminternational.org. Editors Robert Gansert Rajan Bamola Managing Editor Julie Kalista Art Director Barbara L. Brody Production Manager Joanne Miller

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Luc Pouliot, President

Charles M. Kay, Immediate Past President

Christian Moreau, Vice President

Douglas G. Puerta, Secretary/Treasurer

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CONTENTS

## Transitioning to 2014



am happy to report that our final event of 2013, the Reliability, Durability and Performance Assessment of Thermal Spray Coatings conference, was hosted by GE Global Research and turned out to be an unparalleled success with more than 150 attendees, 20 distinguished speakers, and 19 tabletop exhibits. That model, a well known organization hosting a TSS event, is one that we certainly want to revisit in the future, both for the credibility that it brings to the given event (which translates into higher attendance) and for the positive impact on the financials. I actually had preliminary discussions with representatives from OEMs and equipment manufacturers, and they were all enthusiastic about the idea.

#### ITSC 2014 — Not Fiction: Thermal Spray the Key Technology in Modern Life!

The International Thermal Spray Conference and Exhibition takes place this year May 21-23 in Barcelona. The threeday exposition features a conference, poster session, exhibition, education courses, young professional competition, social events, and much more. At ITSC you will find information about equipment for thermal spraying, research and specialist institutes, applied research, and the latest innovations conveniently located in one big forum. The exposition will be held in the Palau de Congressos de Catalunya, in northwest Barcelona.

ITSC is a golden opportunity for the global thermal spray community to meet, network, exchange key information, and conduct business. The conference officially opens with an awards ceremony and plenary lectures. Look for them on Wednesday, May 21. The exposition and poster session also kickoff on that date.

#### **TSS committee chairs named for** 2013-2014 term

The ASM Thermal Spray Society Board appointed chairs to each of its committees for the 2013-2014 term. Luc Pouliot, Tecnar Automation Ltd., Quebec, Canada, continues as president of TSS and Charles M. Kay, ASB Industries, Barberton, Ohio, still serves as TSS immediate past president and chair of the Nominating Committee. Robert C. Tucker, Jr., FASM, The Tucker Group, continues as chair of the Journal of Thermal Spray Technology Committee. Atin Sharma, Sulzer Metco, Westbury, N.Y., serves as chair of the Membership, Marketing and Outreach Committee. Christian Moreau, Concordia University, Montreal, serves as vice president of TSS and chair of the TSS Program Committee. Greg Wuest, Sulzer Metco, Westbury, N.Y., continues to serve as chair of the TSS Safety Committee while Andre Mc-Donald, University of Alberta, Edmonton, Canada, was named chair of the TSS Training Committee. Timothy N. McKechnie. Plasma Processes Inc., Huntsville, Ala., continues to serve as chair of the TSS Awards Committee.



Tucker

Moreau





Wuest



McDonald If you are interested in serving on an affiliate society com-

mittee, please contact the respective committee chair or Sarina Pastoric at sarina.pastoric@asminternational.org or fax 440/338-6614.

The ITSC awards banquet takes place on Thursday, May 22. As part of the exhibition, numerous companies from all over the world display their latest developments and related applications. The conference program offers more than 200 presentations from distinguished speakers covering several topics ranging from fundamental science to industrial applications. Whether you are a thermal spray engineer/technician, a TS scientist, a student, or a supplier of TS equipment and materials, you do not want to miss ITSC. Vamos a Barcelona!

#### North American Cold Spray Conference 2014: Covering the World of Cold Spray

This year the NACSC will be held September 16-17 in Bromont, 40 minutes south of Montreal. Attendees will gain a basic understanding of the cold spray process, follow global R&D programs on cold spray technology, receive firsthand information on industrial applications, and be able to network with international experts. Advancements in cold spray technology are helping to expand the commercial and academic applications of this technically superior metal deposition process.

#### Aerospace Coatings Conference 2014: Development and Manufacturing Trends for the 21st Century

The third TSS Aerospace Coatings Conference will be held in Hartford, Conn., October 8-9. This symposium brings together thermal spray professionals involved in a wide range of responsibilities. By attending the event, you'll be able to understand future coating and process requirements, learn about cost reduction improvements related to quality and reliability factors, and gain an appreciation of existing thermal spray coatings for the aerospace industry. Job shop sprayers, engineers, technicians, end-users, suppliers of equipment and material, academicians, researchers, material scientists, marketing companies, and entrepreneurs will all benefit from this event. Gain key perspectives with exciting presentations from invited presenters including Rolls Royce, Sulzer Metco, PWA, Curtiss-Wright Surface Technologies, LLC Surface Technologies, Delta Airlines Inc., Lufthansa Technik AG, Naval Aviation Department, Pratt and Whitney, KLM Engineering and Maintenance, and other leading organizations.

Your regular and active participation in those events is very important and will ensure that we can keep organizing such world-class meetings in the future. I thank you in advance and look forward to seeing you there.

> Luc Pouliot, president, Thermal Spray Society



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#### JTST editorial team expands, names new lead editor

Armelle Vardelle was named lead editor of the Journal of Thermal Spray Technology (JTST) by Christian Moreau, editor-in-chief of JTST, and Robert C. Tucker, Jr., chair of the JTST Editorial Committee. Vardelle succeeds

Basil Marple, who retired Vardelle

from the position after holding it since its creation in 2010. As lead editor, Vardelle will be in charge of papers for special issues, including the annual special double issue of JTST that contains a selection of expanded papers originating from the International Thermal Spray Conference (ITSC). Professor Vardelle is co-chair of the doctoral program in science and engineering of materials, mechanics, energy and aeronautics and co-chair of the department of materials science and engineering at the University of Limoges, France.

Moreau and Tucker also announced two new associate editors on the JTST editorial team: Margaret Hyland and Georg Mauer. Hyland is deputy dean, faculty

of engineering, and professor, chemical and materials engineering, University of Auckland, New Zealand. Mauer has been head of the thermal spray technology research group at Forschungszentrum Jülich, Germany, Institute of Energy and Climate Research, IEK-1: Material Synthesis and Processing since 2008. Hyland and Mauer join current JTST associate editors, Seiji Kuroda, Kendall Hollis, and Chang-Jiu Li.



Moreau



Tucker





Hyland



Mauer



Nominations for ASM Thermal Spray Society Board

Society Board positions. In accordance with the TSS Rules for Governance, the Nominating Committee is

particularly seeking nominees for vice president and one director from all segments of the thermal spray

community. Nominees must be a member of the ASM Thermal Spray Society and must be endorsed by

five TSS members. Board members whose terms are expiring may be eligible for nomination and possible re-election on an equal basis with any other nominee. Nominations must be received by March 1,

2014. Nomination forms are located on the ASM TSS website at http://tss.asminternational.org. Direct

questions to Charles M. Kay, ASM TSS Nominating Committee Chair, cmkay@asbindustries.com.

The ASM TSS Nominating Committee is currently seeking nominations to fill two ASM Thermal Spray

#### Hollis



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**TSS NEWS** 





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### Solicitations for Student Members to the TSS Board

The ASM Thermal Spray Society is seeking applications for the two student board member positions. Nominations are due by March 1, 2014. Students must be a registered undergraduate or graduate during the 2014-2015 academic year, studying or involved in research in an area closely related to the field of thermal spray technology. To apply, submit an application package consisting of:

- Current resume/CV
- Two-page essay (typed and double-spaced in English) addressing your interest in participating in the program, including:
  - What experiences led to your interest in the program?
  - What qualities, characteristics, and skills do you possess that will make you a strong candidate to serve as a student representative on the TSS Board?
  - What do you hope to gain from this program?
- Students also must submit two letters of recommendation from faculty.

Applications will be reviewed by the TSS Nominating Committee, and candidates approved by the TSS Board. Selected participants will be notified by June 1, 2014, and they will begin their nonvoting, one-year term as student representatives on the TSS Board of Directors in October 2014. Student representatives must attend one regularly scheduled TSS Board meeting held in the U.S. each year, with expenses for travel, hotels, and meals paid for by ASM-TSS; and must participate in two interim TSS Board teleconferences. Student representatives will receive a one-year complimentary membership (worth \$25) in Material Advantage, the program that provides student membership to ASM, TMS, AcerS, and AIST. Submit your application package to sarina.pastoric@ asminternational.org.

#### **Education course at ITSC 2014**

Thermal Spray Technology, Processing, and Evaluation May 18-20, 2014 8:00 a.m. – 5:00 p.m.

This course, taking place at the International Thermal Spray Conference and Exhibition in Barcelona, Spain, will be taught by Christopher C. Berndt, FASM, HoF, and Douglas G. Puerta. It provides an understanding of thermal spray processes, presents complex scientific concepts in terms of simple physical models, and integrates this knowledge into practical applications and accepted thermal spray practices. It is not a laboratory course, but provides detailed instruction for the evaluation of thermal spray coatings. Metallographic preparation and the methods used to ensure rigorous and practical evaluation are also covered. Each registrant receives the Handbook of Thermal Spray Technology and a comprehensive set of notes that include the presentation slides. To register, visit www.asminternational.org/education or contact the ASM Member Service Center at memberservicecenter@asminternational.org.



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# Nanocoating Provides Superior Corrosion Protection Via Two-Coat System

Todd Hawkins

Tesla NanoCoatings Inc. Massillon, Ohio



ne of the many threats the U.S. military must constantly defend against is particularly insidious and advances imperceptibly as it silently erodes the integrity of military, municipal, and global infrastructures—corrosion. It is an ever present

and relentless enemy that varies in both intensity and hostility, and combating it requires considerable time and expense.

Based on extensive research, the U.S. Army and Army Corps of Engineers recently coordinated a new front in the war against corrosion by employing potent emerging technologies that promise a more effective, yet lower-cost defense than current protective methods achieve. Considered the military's center of expertise for corrosion prevention based on its responsibility for structures such as locks, dams, and bridges, the Army Corps of Engineers is presently conducting these procedures under the most rigorous conditions because its interests include a vast infrastructure constructed in some of the world's most extreme environments.

#### Challenges

In the past, anti-corrosive approaches used almost exclusively by the military to protect steel structures consisted of a three-part process that was proven to be effective, but costly. This technology, largely unchanged for three decades, begins with a base coat heavily loaded with zinc as the primary defense. However, the density of the zinc results in an inherently unstable and brittle base coat. The second, intermediate coat provides a barrier to moisture and oxygen, reducing the brittleness issue. The third and final coat imparts color and surface finish.

Surface preparation and material application represent the major expenses related to painting large structures. The traditional, three-coat system is both cumbersome and costly. Another drawback is the high lead content, which can create environmental issues during application and often requires extensive precautions when being removed or in preparation for repainting. While not common, issues with adhesion failure related to high zinc levels also can occur. Other problems can arise when inadequate or incorrect surface preparations cause serious and sometimes catastrophic peeling after a relatively short time due to poor bonding between the surface and zinc-rich base layer.

#### Solutions

As a result of these challenges, the Army and the Army Corps worked with Tesla NanoCoatings to develop an enhanced defense against corrosion. Over the past eight years, this included the testing and documentation of a new carbon nanocoating that provides superior corrosion protection through a two-coat system that is applied like paint, acts like plating, and relies on a much lower zinc content to eliminate an intermediate coat.

Lost conductivity resulting from reduced zinc content is offset by highly conductive carbon nanotubes (CNTs) that self-assemble into rope structures. In addition to enhanced conductivity, nanotechnology also imparts  $5-10 \times$  the adhesion strength compared to traditional methods. This builds stability into an inherently unstable primer, making adhesion failure far less likely.

CNTs help create a base layer that is not only highly conductive, but also abrasion-resistant, tough, and flexi-

ble. In addition, it is cost effective as it eliminates an entire application step, features a lifecycle that can last twice as long as traditional coating systems, and requires no special training to apply. It is also compatible with conventional paint application equipment and is formulated with a lower lead content to reduce environmental issues in application and removal.

#### **Results and conclusions**

Based on the results of initial standardized testing, the Army Corps of Engineers deployed the Teslan coating from Tesla NanoCoatings in a number of field demonstration projects such as fuel tanks, pipelines, water towers, and bridges at military installations throughout North Carolina and Washington. Due to the significance of Tesla's technology, the U.S. Army identified its corrosion technology as a "technical solution."

While the Army Corps of Engineers continues to monitor the performance of its present demonstration projects, Teslan has begun test applications in various other industries including petrochemical facilities and oil and gas well sites where corrosion is a common problem. **iTSSe** 

**For more information:** Todd Hawkins is president of Tesla NanoCoatings Inc., 1311 20th St SW, Massillon, OH 44647, 330/837-1840, www.teslanano.com.



Fig. 1 – Teslan 2-coat system, 8.5 total mils.

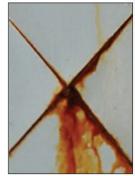
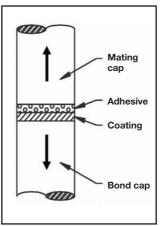


Fig. 2 — Standard 3-coat system, 12.9 total mils.

# Accepted Practice to Test Bond Strength of Thermal Spray Coatings

The ASM Thermal Spray Society (TSS) Accepted Practices Committee on Metallography released a new document on the accepted practice to test the bond strength of thermal spray coatings. of ASTM C633 requirements and provides examples of best practices confirmed by hundreds of tests performed wordwide, adopted by numerous industrial standards, and requested to comply by international technical standardization and certification organizations such as ISO, AS, SAE, and Nadcap.

sufficient adhesion bond between the coating and substrate is critical for the functionality of the coated part. Coating integrity and durability directly depends on the cohesion bond strength. ASTM C633 "Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings" is the baseline and mandatory process to follow for tensile testing of bond strength of thermal spray coatings. The test is required as a condition of approval for new coatings and their suppliers, and is the core qualification test for coatings for aviation, oil and gas, automotive, power, marine, and many other industries. This practice clarifies details



**Fig. 1** — Samples assembled for bond test and applied tensile load.

#### Test scope

The ASTM C633 test applies tensile stresses to coated systems consisting of a coated sample glued to another cylindrical sample as shown in Fig. 1. Alternatively, the coating could be applied on a cylindrical "button," which is glued between two cylindrical samples as shown in Fig. 2. The load is applied in a perpendicular direction to the interface between the coating and substrate. The amount of tensile load is gradually increased from 0, which results in sample failure-the coating pulled out from the substrate or fractured in two pieces inside the coating. The coated surface is always a flat circle with a diameter of 1 in. + 0/-0.005 in., and sample dimensions are standardized by

Bond test step-by-step

- Inspect coating quality on bond caps. (Coating chipping, cracks, delamination, separation, or overspray are not allowed.)
- Prepare mating caps; grit blast their flat surface. (Do not grit blast coating surface).
- Apply a layer of glue on coating surface.
- Place coated samples with applied glue layer into V-groove of curing fixture (see example of V-groove gravity fixture in Fig. 3).
- Add mating cap in the fixture with the grit-blasted surface facing the glue layer on coated sample.
- Apply compression pressure to assembled samples. Note: Fig. 3 doesn't show the additional weight on the top of assembled sample to keep them in compression during glue curing (see below under requirements).
- Place fixture with assembled and compressed samples in preheated oven.
- Keep samples under pressure in heated oven until glue cures; cool samples and release pressure.
- Carefully remove excess glue from each assembly with grinding. Recommended abrasive size for grinding paper or wheel is not coarser than 120 mesh; grinding direction is parallel to glued surface; interface damage/removal of sample material(s) not allowed.
- Place assembled samples in grips of tensile machine, gradually apply tensile load, and record failure load.
- Measure diameter (D) of sample failed face, and calculate the failure area as follows:  $3.14 \times D^{2}/4$ .
- Bond strength = load of failure/failure area. To obtain representative results, compare properties of coatings deposited and tested at different facilities; test conditions and processes require consistency and standardization.

#### **Coating thickness**

Coating thickness of 0.01 in. (0.25 mm) is recommended if coating porosity is below 2%. If the coating has high porosity, increase coating thickness to 0.015 in. (0.38 mm) and above, as recommended by ASTM C633 to prevent glue penetration through the coating to the substrate, but do not exceed the thickness required for the coated part application. Thicker coatings have lower bond strength due to residual stress accumulation that can lead to nonrepresentative test results.

ASTM 633. Bond strength is calculated as the load at sam-

ple failure divided by the coating failure area.

#### **Coating thickness variation**

The coating thickness should not vary across the surface by more than 0.001 in. (0.025 mm). The coating surface may be finished by grinding or machining when thickness variation is excessive. Other treatment, such as grit blasting, should not be used to level the coating thickness. Coating tapering increases sample misalignment, which results in failure at a lower load.

#### Glue

Polyamide-epoxy FM 1000 Adhesive Film is recommended (mandatory in many industrial specifications) as a bonding glue for the coating tensile test. Advantages include allowing testing of high-porosity coatings (such as abradable and ceramic coatings) without glue penetration to the substrate. Disadvantages include losing its own strength if stored at temperatures of 85°F (29°C) and above, but can work for years if stored in a refrigerator.

#### Curing cycle

Recommended cycle for FM 1000 film: Heat assembled samples in oven to  $340 \pm 10^{\circ}$ F ( $170 \pm 6^{\circ}$ C), and cure them for 90 min.  $\pm 10$  min. at the temperatures at bond line of  $340 \pm 10^{\circ}$ F ( $170 \pm 6^{\circ}$ C). Curing conditions may vary based on manufacturer recommendations and for different types of glue. Control the curing temperature with calibrated thermocouple touching (or bonding to) assembled samples. Class 5 furnace (per AMS2750) with temperature uniformity in the working zone of  $\pm 25^{\circ}$ F is sufficient.

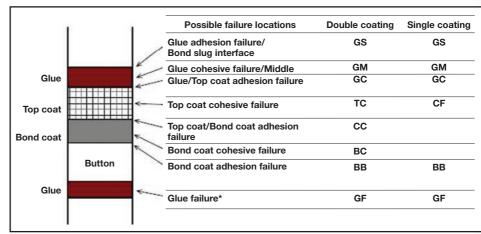


Fig. 2 — Bond test failure map for coating (s) applied on cylindrical button. Courtesy of Sulzer Metco Inc.

#### Fixture

Figure 3 shows a V-grooved gravity fixture. Such a design is working with FM1000 adhesive film. To keep assembled samples in compression during glue curing, one solid steel cylinder (1 in. diam. by 2 in. long) must be placed on top of two assembled samples. Fixture could be made from steel or an aluminum alloy, and should support samples at 30° to vertical position. The horizontal groove in the middle of the fixture shown in Fig. 3 protects it from contact with excessive glue during the curing process.

#### Work in clean and controlled environment

Protect samples from contamination: Do not touch coated surface. Keep coated samples in clean envelopes; plastic is recommended. Use filtered compressed air when removing grit, or dry sample surfaces from liquid degreaser. Protect samples from excessive humidity.

#### Verify alignment

Sample misalignment always leads to low test results. Verify their alignment in the fixture before curing, and after curing and removing excessive glue from the sample sides. Use tensile machine with universal joints to ensure sample self-alignment when tensile load is applied.

#### Control load by applying speed

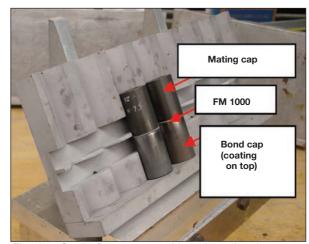
Recommended crosshead speed for tensile load is 0.03-0.05 in./min.

#### Number of coated samples

It is recommended to test five samples and identify the coating bond strength as an average of the five results. For well-established dense coatings with high adhesion and cohesion strength (such as cermets), sample number can be reduced to three for one data point.

#### Test reference samples (glue test)

Always test at least one uncoated reference sample with each set of curing samples to verify/confirm that the glue



**Fig. 3** — Gravity fixture to assemble and cure bond test samples. Picture does not show steel cylinders on top of assembled samples. One steel cylinder (1 in. diam. by 2 in. long) on top of each assembly is sufficient to bond FM1000 to samples

itself has sufficient bond strength (10,000 psi as a minimum for FM1000).

#### Keep records

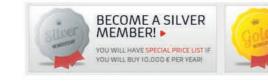
Keep records of all test conditions and file test reports. Bond test reports should include as a minimum: glue strength; coating bond strength; percent of each mode of coating failure (see Fig. 2); test date; operator name/signature; reference on applicable standard(s), specifications, and local instructions documenting test conditions and requirements.

This accepted practice is intended to be used as a baseline for your test processes, but does not replace local test/lab instructions. Additional requirements may apply based on available equipment, testing materials, and customer specifications. Acceptance testing should always be performed in accordance with ASTM C633 (latest revision).





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# Method Measures Ceramic Coating Stress and Damage on Turbine Blades

Researchers are supplying the aerospace industry with innovative technologies to support the next generation of more efficient and reliable aircraft.

Mike Renfro Eric Jordan University of Connecticut, Storrs

> s part of a research project funded by the National Science Foundation and Rolls Royce Inc., scientists developed a novel method of measuring stress and damage in the protective ceramic coatings applied to turbine engine blades to prolong their life. Blades are exposed to some of the most intense heat generated by the engine, and their reliability is crucial to extending engine performance. The thermal barrier coatings that provide insulation between the cooled blade and the hot gases in the engine are highly stressed and age over time eventually resulting in failure.

> Maintenance crews currently rely on visual blade inspections to judge wear and tear along with a routine maintenance schedule based on engine performance data accumulated over time. If the protective ceramic coating is severely damaged internally, cracked, or chipped, the turbine blades can be damaged and even melt under the engine's intense temperatures. A more quantitative approach to determine the remaining life of the coating is desired so that good blades can be returned to service while coatings near the end of their life can be replaced.

> A process that uses laser technology to measure stress and damage in the ceramic coatings by exciting the bonding material between the coating and metallic blades was recently refined by UConn researchers. A low power laser is focused through the coating and the energy from the laser makes the bonding material fluoresce and that fluorescence is stress-dependent. During the process, the wavelength of light emitted by the fluorescence can be measured to determine how strong the bond is, how much viable coating is left, and whether the blade is damaged and prone to failure.

> Previous lab correlation work indicates that when the coating is first made, it is very high stress because the bond between the coating and metal is stronger. As the coating cracks, the stress becomes less and less, and when it becomes too low, it simply flakes off. Determining this stress is key to predicting remaining life. However, applying this technique to actual hardware from an engine exposed new challenges.

Turbine jet engines—because they draw in huge quantities of outside air—are notorious for accumulating a lot of

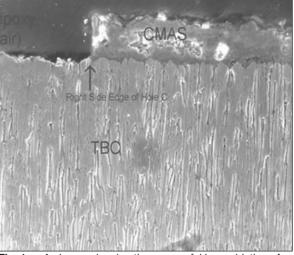


Fig. 1 — An image showing the successful laser ablation of contaminants from the thermal barrier coating on the surface of a turbine engine blade. Courtesy of Michael Renfro/ Surface & Coatings Technology.

dust and other contaminants during performance, which makes testing the stress of engine blade coatings difficult. The contaminant on the turbine blades is highly irregular and in some places it is very thick and in others very thin. Attempting to sand it off would damage the ceramic coating itself and using chemicals would eat through the coating as well.

To get around this, a technique was developed that uses a pulsing laser to blast off the fine layer of contaminants, without damaging the underlying ceramic coating or blades. The laser is focused to a sufficient energy density to ionize the contaminants, ripping them off the surface and making them glow. Each laser pulse strips away about 1  $\mu$ of material at a time. By monitoring the ionization process with a spectrometer, the chemical composition of the removed material can be determined and the point at which all the contaminant is removed can be detected.

After the surface has been cleaned, the stress measurement can be performed. The technique has been developed into an instrument that can automatically clean an area on a blade and perform stress maps across the surface of the blade. It is a unique application of the same technology used by the Mars Rover to determine the chemical composition of rocks, and by art experts to restore valuable paintings to their original condition if they have been touched up or altered over time.

Development of an application for the technology to make it more portable for use while engines are still in service and attached to a plane is underway. **iTSSe** 

For more information: Mike Renfro is UTC associate professor of engineering innovation and associate department head, University of Connecticut, 860/486-5934, renfro@engr.uconn. edu, www.uconn.edu.



The Journal of Thermal Spray Technology (JTST), the official journal of the ASM Thermal Spray Society, publishes contributions on all aspects—fundamental and practical—of thermal spray science, including processes, feedstock manufacture, testing, and characterization. As the primary vehicle for thermal spray information transfer, its mission is to syner-

gize the rapidly advancing thermal spray industry and related industries by presenting research and development efforts leading to advancements in implementable engineering applications of the technology. Articles from the October and December issues, as selected by *JTST* Editor-in-Chief Christian Moreau, are highlighted. The December issue features papers from the 2012 Asian Thermal Spray Conference, organized by guest editor Dr. Seiji Kuroda. The last two articles highlighted below are from this special issue. In addition to the print publication, *JTST* is available online through www.springerlink.com. For more information, please visit www.asminternational.org/tss.



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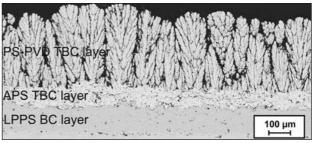




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#### "Improved Thermal Cycling Durability of Thermal Barrier Coatings Manufactured by PS-PVD" S. Rezanka, G. Mauer, and R. Vaßen

The plasma spray-physical vapor deposition (PS-PVD) process shows promise for the manufacture of thermal barrier coatings (TBCs). The durability of PS-PVD manufactured columnar TBCs is strongly influenced by the compatibility of the metallic bondcoat (BC) and ceramic TBC. Earlier investigations show that a smooth BC surface is beneficial for durability during thermal cycling. Further bond improvements between BC and TBC could be achieved by optimizing the formation of the thermally grown oxide (TGO) layer. In the present study, parameters of pre-heating and deposition of the first coating layer were investigated in order to adjust TGO growth. The durability of PS-PVD coatings was improved while maintaining a much higher deposition rate in comparison to EB-PVD. Improved thermal cycling lifetimes more than two times higher than conventionally sprayed TBCs were measured in burner rigs at ~1250°C/1050°C surface/substrate exposure temperatures.



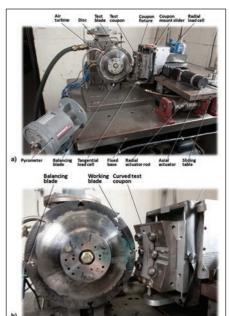
Scanning electron micrograph (back-scattered electron image) of double-layer system with 50 Im APS layer.

#### "Tribological Characterization of Plasma-Sprayed CoNiCrAlY-BN Abradable Coatings"

#### E. Irissou, A. Dadouche, and R.S. Lima

Plasma spray torch parameters were varied to produce a set of abradable coatings exhibiting a broad range of porosity levels (34-62%) and superficial Rockwell hardness values (0-78 HR15Y).

Abradability tests were performed using an abradable-seal test rig, capable of simulating operational wear at different rotor speeds and seal incursion rates (SIRs). Tests determined rubbing forces and quantified blade and seal wear characteristics for slow and fast SIRs. Erosion wear performance and ASTM C633 coating adhesion strength test results are also reported. For optimal abradability performance, coating hardness needs to be lower than 70



(a) General view of the abradable test rig.(b) Close-up view of the test section.



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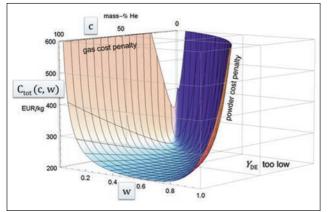
5

GERMAN WELDING

and 50 HR15Y for slow and fast blade incursion rate conditions, respectively. Erosion wear performance, as well as coating cohesive strength, was shown to be a function of coating hardness. Current results define coating specifications in terms of hardness and porosity for targeted applications.

#### **"Fundamental Cost Analysis** of Cold Sprav" **O. Stier**

The cost structure of the cold spray (CS) process is analyzed using a generic cost model applicable to all current types of CS



Dependence of the total costs, Ctot, of cold-sprayed MCrAIY coatings on the He mass fraction c of the propellant gas and the powder-to-gas mass loading ratio w when He is recovered with 85% capture efficiency.

systems and applications. The cost model was originally developed at Siemens and is easy to use and sufficiently accurate. Process costs depend on gas stagnation properties and are discussed. Results indicate that high pressure is generally favorable, He-N<sub>2</sub> blends possess economic potential, and He recovery saves costs in high volume production (even when He-N<sub>2</sub> blends are used). The cost model determines the cost-optimal He concentration of the propellant gas for a given application. CS is, among others, suited to spray bond coatings on gas turbine blades and offers cost-saving potential, as shown.

#### **Education Course:** Introduction to Thermal Spray May 5-6, 2014 **ASM Headquarters** Materials Park, Ohio Instructor: Richard A. Sayman

As the thermal spray profession has changed, so has the need to ensure safe and consistent methods for thermal spray operators. Leaders in the Thermal Spray Society compiled their knowledge and experience to provide a comprehensive, easy-to-understand course. This course provides a strong knowledge and awareness of the five critical areas of thermal spray: Safety, preparation, troubleshooting, spraying, and keeping records, and the required tasks associated with these areas.

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# Affiliate Societies' Committee Chairs Named for 2013-2014 Term

he Boards of the Electronic Device Failure Analysis Society (EDFAS), Heat Treating Society (HTS), International Metallographic Society (IMS), and Thermal Spray Society (TSS) have appointed chairs to each of their committees for the 2013-2014 term. Chairs for ASM Society and General Committees and Councils appeared in the January 2014 issue of *ASM News*. The purpose of each committee is stated on the ASM website, www.asminternational.org. Click on Membership and Committees, followed by Committee Involvement, and then Affiliate Committees.

#### ELECTRONIC DEVICE FAILURE ANALYSIS SOCIETY (EDFAS)



Li

Bodoh



6

Moore

Walraven



Beaudoin



Perdu

**Jeremy A. Walraven,** technical staff, Sandia National Laboratories, Albuquerque, N. M., continues to serve as president of EDFAS.

Henry

**Susan Li**, device analysis lab manager, Spansion Inc., Sunnyvale, Calif., continues to serve as chair of the Education Committee. **Leo G. Henry**, managing engineer, ESD TLP Consulting, Fremont, Calif., continues to serve as vice chair. **Tom Moore**, president, Waviks Inc., Dallas, continues as

...in this issue

- 43 Affiliate Societies' Committee Chairs
- 44 Student Board Member Applications
- **45** Seeking Nominations

chair of the Membership and Nominating Committees.

**Felix Beaudoin**, functional characterization engineer, International Business Machine, Hopewell Junction, N.Y., was named chair of the *Electronic Device Failure Analysis* Magazine Committee.

**Dan J. Bodoh,** FA tools development, Freescale Semiconductor, Austin, Texas, was named chair and **James J. Demarest**, advisory engineer, International Business Machine, Albany, N.Y., was named vice chair of the Events Committee. Mr. Bodoh is general chair of ISTFA 2014.

**Philippe Perdu,** senior expert, CNES, Toulouse, France, continues to serve as chair of the International Growth Committee.

Jones

#### **HEAT TREATING SOCIETY (HTS)**

**Roger A. Jones,** corporate president, Solar Atmospheres Inc., Souderton, Pa., was elected president of HTS.

Thomas E. Clements, Engineering Manager-Metals and Thermal Processes, Caterpillar Inc., is currently serving as HTS immediate past president, as well as chair of the HTS Awards & Nominations and the HTS Finance Committees.

Richard D. Sisson, Jr., FASM, director, Manufacturing and Materials

Engineering, Worcester Polytechnic Institute, Worcester, Mass., continues to serve as chair of the HTS Education Committee. **Michael J. Schneider, FASM**, general manager, Prod. Materials and Metallurgy, The Timken Company, North Canton, Ohio, was named vice chair.

Sisson

Continued on next page

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- 47 Chapter News
- 48 Members in the News



Submit news of ASM and its members, chapters, and affiliate societies to

Joanne Miller, editor, ASM News • ASM International, 9639 Kinsman Road, Materials Park, OH 44073 tel: 440/338-5151, ext. 5662 fax: 440/338-4634 e-mail: joanne.miller@asminternational.org

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Schneider

#### HIGHLIGHTS...ASM Award Nominations





Ahmao



Goldstein

Pape



Rollings

Faulkne

Hills, Mich., continues to serve as chair of the HTS Technology & Programming Committee, and Rozalia Papp, business development specialist, Air Liquide US LP, serves as vice chair.

Aquil Ahmad, retired senior

principal engineer, Eaton Corp.,

West Bloomfield, Mich., was

named chair of the HTS Research

Robert C. Goldstein, director of

engineering, Fluxtrol Inc., Auburn

and Development Committee.

David Rollings, consultant, Lake Villa, Ill., was named chair of the Membership Committee.

Chuck Faulkner, marketing manager, Heat Treatment, Houghton International, Valley Forge, Pa., was named chair of the HTS Expositon Commtitee.

#### INTERNATIONAL METALLOGRAPHIC SOCIETY (IMS)

Richard Blackwell, FASM, general manager, Buehler Canada, serves as president of IMS.

Natalio T. Saenz, technologist, Pacific Northwest National Laboratory, Richland, Wash., serves as IMS immediate past president and chair of the Nominating Committee.

Jaret J. Frafjord, technical director, IMR KHA Portland, Portland, Ore., serves as chair of the Publications Commit-

## **ASM Board of** Trustees

#### Nominations due March 15

It's time to nominate candidates for ASM vice president and trustee. Qualifications include: materials professionals with broad experience in ASM, business, and management; experience in ASM committee and ASM chapter work; managerial experience (budgets and policy making) is most desirable; is an individual or chapter sustaining ASM member; understands the duties and responsibilities required and is willing to serve if elected; understands ASM's strategy and objectives; has knowledge of the field of materials and information technology. Vice presidential nominees must have previously served on the ASM Board of Trustees. Contact Leslie Taylor at 440/ 338-5151 ext. 5500, or leslie.taylor@ asminternational.org. Visit website for rules and nomination form at http://www. asminternational.org/about/governance/ nominating-committee.

tee and IMS vice president.

George Abraham, IV, materials engineer, Allied High Tech Products Inc., Compton, Calif., continues to serve as chair of the IMS Education Committee.

Katherine F. Day, inside sales manager, PCC KLAD LLC, Cypress, Texas, was named chair of the Technology Committee.

Jim Leftwich, business developoment, PSI, Skokie, Ill., was named chair of the Membership, Marketing and Outreach Committee.

#### THERMAL SPRAY SOCIETY (TSS)

See page 2 of *iTSSe* in this issue for the TSS committee chairs.

Blackwell



Saenz



Frafiord



Abraham





l eftwich

If you are interested in serving on an Affiliate Society committee, please contact the respective committee chair directly to discuss further details. You may also leave inquiries with Sarina Pastoric at sarina.pastoric@asminternational.org or fax 440/338-6614.

## IMS Joins ASM, HTS and TSS In Seeking **Student Board Member Applications**

#### We're looking for Material Advantage student members to provide insights and ideas to the ASM, HTS, TSS, and IMS Boards!

We are pleased to announce the continuation and expansion of our successful Student Board Member Programs. In addition to ASM, HTS, and TSS, IMS also seeks Student Board Members. Each Society values the input and participation of students and is looking for their insights and ideas.

#### An Opportunity like No Other!

- All expenses to attend meetings paid for by the respective Society
- Take an active role in shaping the future of your professional Society
- Actively participate in your professional Society's Board meetings
- · Gain leadership skills to enhance your career
- Add a unique experience to your resume
- Represent Material Advantage and speak on behalf of students • Work with leading
- professionals in the field

#### **Opportunities Specific to each Society:** ASM International

- Attend three (3) Board meetings (June 16-18, 2014,
- October 12-15, 2014, and February/March 2015) • Term begins June 2014

#### **ASM Heat Treating Society**

- Attend two (2) Board meetings (October 2014 and Spring 2015)
- Participate in four (4) teleconferences
- Term begins September 2014

#### **ASM Thermal Spray Society**

- Attend one (1) Board meeting in October 2014
- · Participate in two (2) teleconferences
- · Receive a one-year complimentary membership in Material Advantage • Term begins October 2014
- International Metallographic Society
- · Participate in monthly teleconferences • Attend one (1) Board Meeting (August 2-6, 2015)
- Term begins August 2014

# **Seeking Nominations**

## 2014 Bradley Stoughton Award for Young Teachers

Winner receives \$3000 Deadline March 1

This award recognizes excellence in young teachers in the field of materials science, materials engineering, design, and processing.

Are you aware of a colleague who:

• Is a teacher of materials science, materials engineering,

design, processing, and related fields

• Has the ability to impart knowledge and enthusiasm to students

Viola Manuel

- Is 35 years of age or younger by **May 15** of the year in which the award is made
- Is an ASM Member

Nominate them for the 2014 award!

# Honorary Membership

**Deadline February 1** 

Honorary Membership in the Society was established in 1919. It recognizes distinguished service to the materials science and engineering profession, ASM's strategic plan/initiatives, and to the progress of mankind. Honorary Membership is among the most prestigious awards of the Society. Consequently, it is expected that all nominees will be truly outstanding individuals who have significantly furthered the purposes of the Society through

2013 Honorary Membership, Dr. H.M. Mehta

an evidenced appreciation of the importance of materials science and through distinguished service.

## 2014 ASM International Student Paper Contest Deadline April 1

The contest recognizes the best technical paper with a graduate or undergraduate student as first author that is published in an ASM sponsored publication during the

year. The winner will be recognized and presented with their award during the ASM Leadership Awards Luncheon to be held in Pittsburgh during October 12-15 in conjunction with MS&T'14. The award consists of a certificate, \$500 cash prize, and up to \$500 toward expenses to attend MS&T'14. The paper will also be published in one of ASM's publications.

## ASM-IIM Visiting Lecturer for 2014 Deadline February 15

The cooperative Visiting Lecturer program of ASM International and the Indian Institute of Metals (IIM) is seeking lecturers for 2014. Criteria for the 2014 ASM-IIM Visiting Lecturers are as follows:

- ASM members who visit India
- Experience delivering technical presentations of interest to government, industrial, or academic organizations



ASM-IIM Visiting Lecture, Prof. Narsingh Singh, FASM

- Able to lecture on current technological conditions in India
- Available between April 1 and December 31

• Definite travel plans to and from India using own funds The award carries with it an \$800 honorarium to be used for travel expenses within India and a certificate of recognition to be presented at the ASM Leadership Awards Luncheon scheduled for October in Pittsburgh during MS&T'14.

# Engineering Materials Achievement Award

Deadline March 15

The Engineering Materials Achievement 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. The purpose of this award is to seek out and recognize outstanding developments in the application of materials in products or in engineering structures and to honor the organization or individuals responsible for them.



2013 Engineering Materials Achievement Award, David L. Joyce

Do you know of an innovative, cutting-edge scientific achievement that has distinctly impacted industry, technology, and society within

the past 10 years? Consider submitting a nomination for the Engineering Materials Achievement Award.

#### **NOMINATION FORMS & RULES**

Applications, nomination forms, and rules for all of these awards can be found at http://www.asminternational.org/membership/awards

For more information, contact Christine Hoover at 440/338-5151 ext. 5509, or christine.hoover@asminternational.org.



2013 Bradley Stoughton Award for

Young Teachers, Prof. Michele

## From the Foundation: **New Director** Joins ASM

In October, ASM's Materials

Education Foundation welcomed its new Director of Development and Operations, Nichol Campana, who introduces herself in this debut column that will report on Foundation activities every other month.

)rior to joining the ASM Materials Education Foundation, I worked at the American Cancer Society for 10 years and held various positions during my time there. My expertise and background are centered on nonprofit management and fundraising. Since joining the Foundation, I have been focused on developing a fundraising platform to ensure financial success, as well as building the brand and reputation of this wonderful organization. In addition, I plan to utilize my best practice expertise-methods and techniques to produce results, which are used to benchmark and gain knowledge and understanding. I have plans to work with the Foundation staff and Board of Trustees on the implementation of those best practices and benchmarking exercises. ASM Materials Education Foundation asks that you stay tuned for all of our new and exciting initiatives.

I was fortunate enough to be hired in time to attend MS&T 2013, in Montreal, where I had the opportunity to attend the ASM Materials Education Mini-Materials Camp. It was truly a wonderful experience. The instructors were very engaging and the students were receptive and participatory. One experiment in particular stood out, and made me wonder if I would have been a better science student if interactive learning had been part of my education. The experiment utilized a racquet ball and liquid nitrogen and demonstrated the effects of extremely cold temperatures on different materials. The instructor asked what would happen if he dropped the ball-would it break or bounce? To their amazement, the ball shattered and made a loud noise. The students were encouraged to touch the material to feel the effects of temperature change on the material. Like the students, it left me wanting more and filled me with many questions. If you have not previously experienced the ASM Materials Camps, I highly encourage you to attend. I also look forward to meeting many ASM members in my new role with the Foundation. Feel free to contact me at nichol.campana@asminternational.org.

# **IMC:** Revised Rules, Fewer Classes, Larger Prize Money

The International Metallographic Contest (IMC), an annual event cosponsored by the International Metallographic Society (IMS) and ASM International to advance the science of microstructural analysis, just got more enticing. Updates to the rules recently approved by the IMS Board are designed to encourage participation and to simplify the process for participants to submit entries. There are now five different classes of competition-down from 11 classes-covering all fields of optical and electron microscopy:

- Class 1: Optical Microscopy—All Materials
- Class 2: Electron Microscopy—All Materials
- Class 3: Student Entries—All Materials (Undergraduate or Graduate Students Only)
- Class 4: Artistic Microscopy (Color)—All Materials
- Class 5: Artistic Microscopy (Black & White)—All Materials

All of the classes have increased prize money! Best-In-Show receives the most prestigious award available in the field of metallography, the Jacquet-Lucas Award, which includes a cash prize of \$3000.

The international competition showcases the finest work of metallographers, scientists, technicians, and students from around the world. The contest is open to all (except contest judges), including non-members.

For a more complete description of the new rules, tips for creating a winning entry, and judging guidelines, visit www.metallography.net.

# **Certificate Program Graduates**

Congratulations to all who completed their Certificate Program through ASM's Education offerings in 2013! If Oscars were given for a job well done, we'd nominate you! Completing this extra coursework while continuing in your full time position takes tremendous dedication. We're sure it was worth it, knowing that this achievement will make such a positive difference in your career path. Well done!

#### **Extension Diplomas & 2013 Graduates:**

Applied General Metallurgy: Eduardo Bejarano, Matthew Byrne, Susan Cavanaugh, Marcia Clark, Rick Clary, Robert Doggett, Timothy Howard, Brian Krueger, Eric Pintueles, and Jeff A. Stanford Corrosion: Susan Cavanaugh

Ferrous Metallurgy: Susan Cavanaugh, Irene Hardgrove, and James I. Middleton, Jr.

Heat Treating: Dayna Fettig

Non Ferrous Metallurgy: Susan Cavanaugh

Testing & Inspection: Matthew Byrne and Tammy Hankes Welding Metallurgy: Thomas H. Boone, Rick Clary, Robert Doggett, Bhupinder Kataria, and James Rodrigues

ASM Certificate Programs are valuable in furthering knowledge in a chosen field and can lead toward the next career goal. Learn how you, too, can achieve a Certificate in a related field. Contact Liz Halderman, Lifelong Learning Advisor at 800/336-5152 ext. 5707 or liz.halderman@ asminternational.org.

ASM's reengineered website has arrived! Visit the new www.asminternational.org today.



# **Chapter** News

#### **Rhode Island's Spouse Night**

On December 11, the ASM Rhode Island Chapter held its spouse's meeting at the Barker Theatre in Providence. Members and guests of the Chapter were treated to an entertaining evening consisting of the production, *Bell, Book, and Candle.* At the end of the play, the entire cast and crew joined the attendees for wine and a light dinner, making for a delightful evening.



Members and guests of the ASM Rhode Island Chapter gather during the holidays at the Barker Theatre in Providence for their annual spouse's night.

#### **Puget Sound Learns about Kayak Construction**

The Puget Sound Chapter's last meeting of the year is usually more social with a less-technical talk of interest to family members. In keeping with that tradition, the December 2013 topic was on kayak construction. The speaker, Dr. Roy Baggerly, FASM, recently retired from Boeing. His presen-

## VOLUNTEERISM COMMITTEE Profile of a Volunteer



#### *Ron Radzilowski, FASM* Manager - Metallurgical Technology

Severstal North America

Ron Radzilowski is an ASM Fellow and industry veteran with 47 years of work experience—but he brings the energy and enthusiasm of youth to his profession. "I

can't sit around! I don't participate in conferences and symposiums for my own benefit. I do it for my company and for ASM because I owe them a debt of gratitude."

After working in the metals industry for nearly a half century, Radzilowski is still learning, still traveling internationally, and still sharing his expertise—as Manager of Metallurgical Technology for Severstal North America, and on ASM committees for Volunteerism, Emerging Technology Awareness (ETAC), and New Products & Services. He also helps with the summer ASM Materials Camp for Teachers at the University of Michigan.

For Severstal in Dearborn, Radzilowski advises senior management on technical aspects of steelmaking and "technology benchmarking." He enjoys working with entrepreneurial companies to explore new technologies that work with steel in the automotive industry and elsewhere.

Radzilowski has held every officer role for the Detroit ASM chapter and encourages students and young professionals to get involved. He knows that support from a tation included showing a 17-ft kayak and a paddle, both of which he built. He also highlighted the unique techniques and methods used by the Aleut people to construct their kayaks. These methods



suggest the Aleuts understood tribology and used technology to reduce wear and friction.

#### LA and Ravi Honor Golf Organizer

In recognition of his long work with the Scholarship Golf Tournament, Dick Berryman (center, Scholarship Golf Tournament chair and Chapter Council Representative, District II), receives a certificate of appreciation from Ravi



Ravindran, FASM, (right, ASM president) and a package of golf balls from Mike Hahn (left, Chapter secretary) while Chuck Daugherty (back, Scholarship Golf Tournament Committee member) looks on.

company's senior management plays a critical role. He sees two key advantages to ASM: first, as an "information society" at the forefront of disseminating new technology as well as archiving older information—and just as importantly, providing a means to interact at the local and national level. "I meet people and take back ideas for commercialization and share what others are doing—in both technology and business."

About his 2009 election as ASM Fellow, he says, "We should be a model for the young, giving back on the national level, but also as a mentor at the local level, getting younger people involved. As a volunteer leader, Radzilowski's enthusiasm is keeping ASM alive for the next generation.

# **Updated Online Products**

To continue providing the most current, searchable information in the industry, ASM released an update to the **ASM Medical Materials Database** on December 19. This represents the fourth successful and on-time quarterly release of the database for 2013.

The **ASM Handbooks Online** also was updated the same day and now includes the new Volume 5A: *Thermal Spray Technology*.

Check out the new content of both products on ASM's newly redesigned website. Visit www.asminternational.org, select Materials Resources, and then choose from the list of Online Databases.

For a list of upcoming ASM Training Courses, see our ad on page 26 of this issue.

# Members in the News

#### Williams Joins EWI Board

Columbus-based technology innovator EWI recently welcomed **David B. Williams, FASM,** of The Ohio State University (OSU) to its Board of Directors. Williams is dean of the OSU College of Engineering. From 2007 to 2011, he served as the President of the University of Alabama, Huntsville. Prior to 2007,



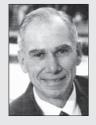
Williams spent 31 years at Lehigh University in Bethlehem, Pa., as an MSE professor. He directed Lehigh's Electron Microscope Lab and the Microscopy School from 1980-88. A native of Leeds, England, Williams received his degrees from the University of Cambridge. In 2013, Williams was selected as the Alpha Sigma Mu Lecturer.

#### **Materials Research Socity Honors Dresselhaus**

Massachusetts Institute of Technology professor emeritus Mildred Dresselhaus received the Material Research Society's Von Hippel Award during the organization's fall meeting in Boston. In her acceptance speech, the awardee recalled that Arthur R. von Hippel embodied scientific curiosity and mentorship and shared her passion for music. Dresselhaus received the award for her pioneering contribu-

## IN MEMORIAM

**George S. Ansell, FASM,** of Tucson, Ariz., died on August 30, 2013. Ansell, born in 1934, was the Colorado School of Mines' 13th president, serving 1984-1998. He earned a bachelor's degree in metallurgical engineering in 1954 and a master's the following year, both from Rensselaer Polytechnic Institute (RPI). After serving in the U.S. Navy, he was ap-



pointed to the Metal Physics Consultant Staff of the U.S. Naval Research Laboratory as a physical metallurgist. Ansell earned a Ph.D. in metallurgical engineering from RPI in 1960 and joined their faculty that same year. He was named Robert W. Hunt Professor and later, dean of the school of engineering at RPI. After his retirement from Mines in 1998, he received an honorary degree and the metallurgy department was renamed the George S. Ansell Department of Metallurgical and Materials Engineering. Ansell was a member of the ASM Rocky Mountain chapter.

**Robert H. McCreery, FASM**, 88, Portland, Ore., passed away on October 13. He was born on September 11, 1925, in Muncie, Ind., served with the U.S. Army during WW II, and earned his B.S. from Purdue University. McCreery worked as a metallurgical engineer at Teledyne Portland Forge, retiring in 1990. He was a licensed professional engineer and a life member of both ASM International and ASME. McCreery also was a past ASM trustee. Author of several technical articles, his topics included nondestructive case depth measurements and effects of reduction on strand cast steel.

Word has been received at ASM Headquarters of the death of Life Member **Robert W. Carter** of Libertyville, Ill. (Chicago Chapter).

tions to the fundamental science of carbon-based and other low-electron-density materials, leadership in energy and science policy, and exemplary mentoring of young scientists. Von Hippel encouraged Dresselhaus to study carbon—espe-



cially graphite—and made her part of his string quartet. Von Hippel died in 2003 at age 105, having worked in the lab until age 90.

#### **Jones Receives Heritage Award**



Heritage Awardee Bill Jones is flanked by MTI presidents, Buster Crossley (left), serving 2011-2012, and Jim Roberts (right), 2012-2013.

William R. Jones, FASM, CEO of Solar Atmospheres Inc., Souderton, Pa., received the Heritage Award at the Metal Treating Institute (MTI) Fall Meeting in Las Vegas on October 12. This prestigious award is given in recognition of an individual's lifetime commitment to the betterment of the commercial heat treating industry with one or more notable accomplishments. Only nine people in the 78-year history of MTI have ever received this award.

#### Spain Joins Alpha Sigma Mu

The Alpha Sigma Mu MSE Honorary Society recently welcomed Spain as its newest chapter affiliate. **Frederick Schmidt, FASM,** the honor society's president, welcomed the new members and commend the University of Oviedo

and the community for contributions to global technology. In his presentation, Schmidt reported there are 58 active chapters in North America at prominent university locations, and active international chapters in Australia, Canada, and Spain. The honor society expects to charter new chapters in Austria (Wels), Brazil, Germany, Hungary, Poland (Krakow), and Russia (St. Petersburg).



Juan Asensio-Iozano Alpha Sigma Mu trustee (left) presents the new charter to Juan-Jose Fernández, president of the new Spanish chapter.





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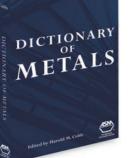
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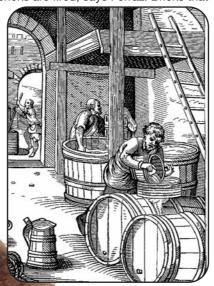


#### Building better bricks with beer

Eduardo Ferraz and colleagues at the Polytechnic Institute of Tomar, Portugal, are mixing brewery grains into clay bricks to enhance their ability to trap heat without compromising strength. With a clay paste containing 5% spent grains, they created bricks as strong as conventional ones, while reducing heat loss by 28%. The grains make the bricks more porous so they trap more air, which increases heat retention. One downfall of the process is the smell. Bill Daidone of the Acme Brick Co., Fort Worth, Tex., says his lab abandoned experiments because the stench of the moist grains was overpowering. However, this problem vanishes once the bricks are fired, says Ferraz. Bricks that

provide insulation without sacrificing strength could be a big boost to the construction industry, according to John Sanders, a scientist at the National Brick Research Center at Clemson University, S.C. http://portal.ipt.pt/portal/ portal/international.

Researchers mix brewery grains into clay bricks to enhance their ability to trap heat without compromising strength.





A new, "greener" dyeing method coaxes already-colored fibers from caterpillars by feeding them dyed leaves. Photo by Shutterstock.

#### Dyed leaf diet yields colored silk

Cultivated silkworms have been spinning luxurious white silk fibers for use in clothing for thousands of years. Current dyeing practices produce wastewater with potentially harmful toxins, so scientists are turning to a new, "greener" dyeing method in which they coax already-colored fibers from the caterpillars by feeding them dyed leaves. Researchers dipped or sprayed mulberry leaves, the silkworm's food of choice, with azo dyes to see which ones, when consumed, would transfer to the silk. Of the seven dyes they tested, three were incorporated into the caterpillars' silk, and none seemed to affect the worms' growth. Scientists noticed that certain dye traits, such as the ability to dissolve in water, affected how well the dye worked. "These insights are extremely important in development of novel dye molecules that can be successfully used in this green method of producing colored silk fabrics," say researchers. www.acs.org.

#### What's the frizz about: New method indexes bad hair days

Beauty experts at Dow Corning, Midland, Mich., developed a verifiable Frizz Index testing method that quantifies hair's frizziness independently of its volume. It represents a breakthrough that gives hair care manufacturers a tool to describe hair tress shape and appearance in a more accurate way, allowing them to formulate new products that will effectively tame frizzy, flyaway hair. "Dozens of frizz control products an etoric abeliate tout their ability to teme unruly beir without unwanted

on store shelves tout their ability to tame unruly hair without unwanted side effects like influencing hair volume or leaving hair feeling greasy or crispy," explains Sylvie Bouzéloc, Hair Care application designer. "Until now, manufacturers never had a way to prove a product's ability to deliver on those claims apart from testing the product's effect on volume. Our new Frizz Index will give them a way to demonstrate product performance to their consumers." *www.dowcorning.com/personalcare.* 

A verifiable Frizz Index testing method that quantifies hair's frizziness independently of its volume was developed by Dow Corning researchers.

# Specimen Name: adidas adiZero Prime SP track shoe with Cal Nano "Lone Star" spike

SUCCESS ANALYSIS

## Vital Statistics:

The adidas Innovation Team and California Nanotechnologies (Cal Nano) worked together to create "the lightest and strongest track shoe in the world." Originally designed for Olympic athletes, shoes are now available on the mass market. Produced using computer-controlled multi-stage progressive

cold forgers, the metal matrix nano composite (MMNC) spike is made of an advanced powder metallurgy composite comprised of micro- and nano-reinforcements embedded in a 6000 series aluminum matrix—a material substantially stronger, stiffer, and harder than aerospace aluminum alloys.

The concept of building an ultra-light, nano-based track shoe Success Factors: originated from the "barefoot" running movement, with the goal of creating a way for athletes to experience the track as closely as

possible without sacrificing performance.

With greater hardness, strength, and wear resistance than conventional steel alloys, the Cal Nano R&D team helped develop an enhanced spike one-third the weight of traditional designs. The nano spike is able to grip onto a thinner shoe, allowing runners to experience a barefoot sensation.

wire Nanocomposite forging system.

Nanocomposite wire.

# About the Innovators:

Integrating nanotechnology within the track shoe design occurred under the guidance of Mic Lussier, director of the adidas Innovation Team, to produce shoes for athletes at the Beijing and London Olympics. Based on several gold medal finishes, the technology was further optimized under the joint direction of Chris Melnyk, CEO of Cal Nano.

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adidas adiZero Prime SP track shoes sport auiuas auizero rrime Sr track snoes spor nano-based spikes that are one-third the weight of steel-based designs.

## What's Next:

Nanotechnology offers application possibilities across a range of industries due to its lightweight, high strength attributes. Automotive components are one example, as federal mandates dictate ever more stringent fuel economy standards. Another promising field is aerospace, with new nano- aluminum, titanium, and superalloys being developed for use in airframes, rocket engines,

- Cal Nano is working with leading universities to develop advanced nano-titanium for use in biomedical implants and also produces nano-enhanced, carbide-based thermal spray powders and coatings for use in extreme environments scram jet engines, and satellite systems.
- such as down-hole applications in the oil and gas industry.

# Contact Details:

California Nanotechnologies 17220 Edwards Rd., Cerritos, ČA 90703 562/991-5355, c.melnyk@calnanocorp.com, www.calnanocorp.com

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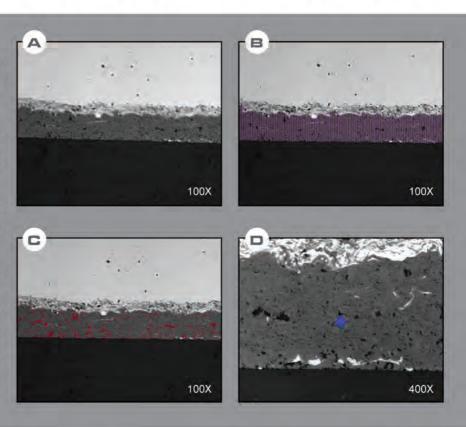
Here is an example that provides a better understanding of how this advanced technology is altering the operation of Clemex CMT.HD (micro-hardness testing system) in measuring a ceramic coating:

A First, the software takes a series of images from the sample under the micro-hardness tester at 100X, as a traditional microscope would do.

■ The layer in the coating that binds the metallic base to the outer ceramic coating is then measured, as is the ceramic coating as a whole (magenta).

C Other parameters are automatically quantified, namely the porosity of the coating, the phase percentage and the roughness (red).

■ Next, the turret of the tester turns automatically to 400X, and Clemex CMT locates the geometric centre of the ceramic coating to conduct the micro-hardness test (blue). This outstanding location ability is the key element in the analysis and enables the generation of a uniform database on the material's micro-hardness.



The example above confirms that Clemex CMT.HD, when equipped with the latest innovations, works like a microscope, with the added difference that it also helps in execute micro-hardness tests.

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