

AMERICAN CERAMIC SOCIETY

bulletin

emerging ceramics & glass technology

MAY 2016

Ensuring longevity:

Ancient glasses help predict durability of vitrified nuclear waste

- Measuring architectural glass coatings
- Low-fire enamels on steel appliances
- Thin films for integrated photonics



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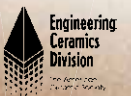
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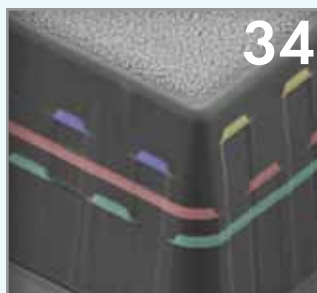


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NSF funds program to accelerate discovery of new materials and tech

A new Materials Innovation Platforms (MIP) program funded by the National Science Foundation recently made its first awards to Pennsylvania State University and Cornell University, with the aim to “significantly accelerate materials research and development,” according to an NSF news release.

The institutions will serve as “platforms” to develop new bulk and thin-film crystalline hard materials through state-of-the-art instrumentation in an environment that “combines multidisciplinary expertise with the best tools available, providing access to the instrumentation, data, and new materials created,” the release explains.

Penn State will focus on developing new materials for next-generation electronics that are faster, use less energy, and can be built on flexible substrates as well as other applications at its new facility, the 2-D Crystal Consortium (2DCC).

Cornell University will focus on the interfaces between oxide-based and 2-D materials with its Platform

for the Accelerated Realization, Analysis, and Discovery of Interface Materials (PARADIM).

“We see the platforms as pushing the frontiers in materials research,” Fleming Crim, NSF assistant director for math-



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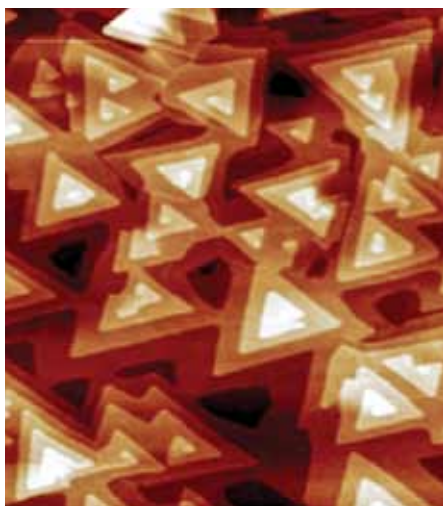
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The surface of a bismuth selenide film shows the triangular layer structure that is characteristic of 2-D chalcogenide materials.

Credit: Juan Revorung, Department of Materials Science and Engineering, Pennsylvania State University

emical and physical sciences, says in the release. “In its first call for proposals, NSF is focusing on crystal growth, because the United States has fallen behind in this area of science after having been a global leader in material synthesis, which is essential for advancing basic materials research and will add to the important investment the foundation is making in mid-scale instrumentation.”

Each MIP program awardee will act as a “nexus of activity” for a focused research theme, where platforms are equipped with user facilities, according to the release. Researchers throughout the U.S. who also engage in these areas of research will have access to the resources, too, to help get their own work on the fast track to development.

“Without question, one of the most exciting aspects to these awards will be to see just how quickly these platforms can accelerate the pace of materials development,” Sean L. Jones, NSF materials research program director, says in the release. For more information, visit 1.usa.gov/22u1dSP. ■

CoorsTek investing \$120M in new advanced materials R&D facility



CoorsTek has big plans for its facility in Golden, Colo.—plans that include a \$120 million R&D facility.

CoorsTek is investing in the future—and it looks like the future has lots of advanced materials in it.

The company recently announced that it is investing \$120 million to build an advanced materials R&D facility in its headquarters city of Golden, Colo.

“This investment will support the rapid development of new materials—helping CoorsTek technology and manufacturing customers solve their toughest challenges with the high-performance properties of advanced ceramics,” according to a CoorsTek press release.

The facility, called the Center for Advanced Materials, will house an R&D hub “outfitted with leading-edge equipment to develop innovative ceramic materials and processes for a variety of next-generation applications,” a comprehensive analytical laboratory, and a materials manufacturing facility “enabling swift commercialization and volume production using the latest technologies in advanced ceramic processing,” the release states.

Learn more about the new center in a CoorsTek video available at youtu.be/kb2QgiVV6YU. ■

Business news

Alcoa’s future value-add company to be named “Arconic” (alcoa.com)...3M unveils state-of-the-art R&D laboratory at global headquarters (news.3m.com)... AGC to build second float-glass production plant in Brazil (agc-glass.eu)... Toyota partners in making wind-power hydrogen for fuel cells (ap.org)... Ames Laboratory scientists join consortium to research lightweight materials (ameslab.gov)... Faraday Future earns first US patent (faradayfuture.com)... PNNL to give helping hand to small green-energy businesses (pnnl.gov)... Alfred engineering school awarded SUNY funds for ceramics scholar in new center (alfred.edu)... Lockheed seeks to lay off up to 1,000 aeronautics workers (lockheedmartin.com)... Rio Tinto completes sale of inter-

est in Bengalla Joint Venture for \$616.7M (riotinto.com)... Saint-Gobain to export refractories from India, set up new R&D center (saint-gobain.com)... LKQ Corp announces agreement to acquire Pittsburgh Glass Works (lkqcorp.com)... Asahi to release cover glass with fingerprint recognition sensor (asahi-glass.com)... Almatris increases production to service nonmetallurgical alumina markets (almatis.com)... Seven Refractories start positively into 2016 (sevenrefractories.com)... 3M and Schuberth to collaborate on next-gen tech, distribute Ceradyne helmets (news.3m.com)... Ceralink now offers fatigue testing for metals (ceralink.com)... NIST announces funding opportunity for NNMI Manufacturing Innovation Institutes (nist.gov) ■

Nanostructured glass eternally stores high volumes of data in 5-D

Researchers at the University of Southampton (United Kingdom) have developed a glass-based 5-D data storage method with incredibly high capacity and a near-unlimited lifetime.

Using femtosecond laser writing, the Southampton researchers create three layers of nanostructured dots, each 5 μm apart, in quartz. The size, orientation, and 3-D position of each nanodot—together accounting for 5-D—store the data. To retrieve the data, a polarizer and optical microscope read the nanostructures by measuring disruptions to light polarized through the glass.

Watch the ultrafast laser in action in a video at youtu.be/OP15blgK5oU.

According to a new Southampton press release, “The storage allows unprece-



Credit: University of Southampton

The Holy Bible stored as 5-D data in glass.

dened properties, including 360 TB/disk data capacity, thermal stability up to 1,000°C, and virtually unlimited lifetime at room temperature (13.8 billion years at 190°C), opening a new era of eternal data archiving.”

Because of the possibility of storing so much data in such a compact package, the technology could be used to archive large collections of data, such as in museums and libraries. To demonstrate that value, the Southampton researchers wrote digital copies of the Universal Declaration of Human Rights, Isaac Newton’s Opticks, Magna Carta, and King James Bible into bite-sized pieces of glass. ■

DOE launches \$40M effort to advance materials research for renewable energy

The United States Department of Energy recently launched a \$40 million effort to improve materials for clean energy solutions. The new national-laboratory-led program—called the Energy Materials Network—will “give American entrepreneurs and manufacturers a leg up in the global race for clean energy,” according a DOE news release.

The Energy Materials Network program will focus on design, testing, and production of advanced materials by facilitating relationships between science and industry to give companies more access to advanced materials innovation resources available at DOE’s national laboratories in an effort to bring these new materials to market faster.

DOE’s Office of Energy Efficiency and Renewable Energy is funding the establishment of four initial national-laboratory-led consortia to “bring

together national laboratories, industry, and academia to focus on specific classes of materials aligned with industry’s most pressing challenges related to materials for clean energy technologies,” according to the release.

- The Lightweight Materials Consortium (LightMat), led by Pacific Northwest National Laboratory, will enable increased vehicle fuel efficiency by designing specialized alloys and carbon-fiber-reinforced polymer composites that can be manufactured on a large scale.
- The Electrocatalysis Consortium (ElectroCat), led by Argonne National Laboratory and Los Alamos National Laboratory, will be dedicated to finding new ways to replace the rare and costly platinum group metals currently used in hydrogen fuel cells with more abundant and inexpensive materials.
- The Caloric Cooling Consortium (CaloriCool), led by Ames Laboratory,



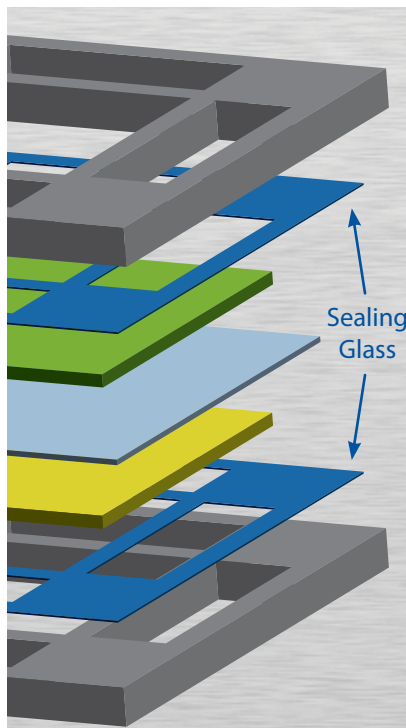
Credit: BibliothequeRemes2, Flickr, CC BY-NC-ND 2.0

A new infusion of funding is seeking to advance materials research for renewable energy.

will leverage the laboratory’s capabilities in the field of “caloric” refrigerant materials to develop, demonstrate, and deploy these innovative cooling technologies.

- One more consortium will be established later this year, according to the release. It will focus on developing new materials to make solar photovoltaic modules more durable and cost-effective.

For more information, visit 1.usa.gov/24pywVO. ■



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NASA developing quieter, more fuel-efficient supersonic commercial jet

NASA's ambitious 10-year New Aviation Horizons plan seeks "to design, build, and fly a variety of flight demonstration vehicles, or 'X-planes.'" The plan will test several significantly redesigned aircraft concepts, which, if successful, could take to the skies around 2020.

"We're at the right place, at the right time, with the right technologies," Jaiwon Shin, associate administrator for NASA's Aeronautics Research Mission Directorate, says in a NASA press release. "The full potential of these technologies can't be realized in the tube-and-wing shape of today's aircraft. We need the X-planes to prove, in an undeniable way, how that tech can make aviation more Earth friendly, reduce delays, and maintain safety for the flying public, and support an industry that's critical to our nation's economic vitality."

The X-planes will test "such things as lightweight composites; quieter, more advanced engines; quieter landing gear and flap mechanisms; shape-changing wing flaps; and bug-resistant coatings," according to a *Gizmag* article. "The agency says that these have the potential to save the air industry \$225 billion dollars over a 25-year period."

But to save money, sometimes you have to spend it. This is precisely why NASA recently announced that it is spending \$20 million to push supersonic jet travel back into commercial reality. NASA's reimagining of the supersonic commercial jet is one of the projects within the New Aviation Horizons plan.

NASA is funding \$20 million over 17 months to develop Quiet Supersonic Technology (QueSST). The funding is going to a team led by Lockheed Martin for preliminary design work on a reimagined supersonic jet. GE Aviation (Cincinnati, Ohio) and Tri Models Inc. (Huntington Beach, Calif.) also are subcontractors on the project.

According to a NASA press release, "The company will develop baseline aircraft requirements and a preliminary aircraft design, with specifications, and provide supporting documentation for concept formulation and planning. This documentation would be used to prepare for the detailed design, building, and testing of the QueSST jet. Performance of this preliminary design also must undergo analytical and wind tunnel validation."

So why does NASA think quiet supersonic jets are suddenly possible?

"The trick to making airplanes quiet is to change the way the air flows around the airplane," Juan Jose Alonso,

professor of aeronautics and astronautics at Stanford University who worked on the X-plane design at NASA headquarters from 2006 to 2008, says in a *Wired* article.

That trick involves eliminating points that stick out from the airplane frame, minimizing

air disruptions and, hence, minimizing shockwaves.

The key to successful design for noise reduction is to attack the problem from several angles, according to the *Wired* article. "That gives them more options, like trying out different nose shapes to minimize the leading edge of a shock wave. They're also looking at putting the air intake on top rather than underneath the engine, and entirely eliminating the forward-facing cockpit window. (Pilots will navigate with the help of video cameras.) It's also possible that the airframe itself might help dissipate shocks rather than form them."

NASA scientists have been using 3-D computer modeling and simulations to show that by incorporating these design concepts, the new supersonic X-plane should be able to reduce the noise level of its booms to a manageable sound level of 65-70 dB. ■

World's blackest material Vantablack now absorbs even more light

The world's blackest material, Vantablack, just got blacker.

The material, which was developed by United Kingdom company Surrey NanoSystems a few years ago, consists of a dense coating of carbon nanotubes that absorbs nearly all light that hits the material's surface. According to the company, "The near total lack of reflectance creates an almost perfect black surface."

But apparently Surrey NanoSystems was not happy with the almost-perfect status of its black material—the company recently revealed that it has slightly improved its blackest black material to be even blacker.

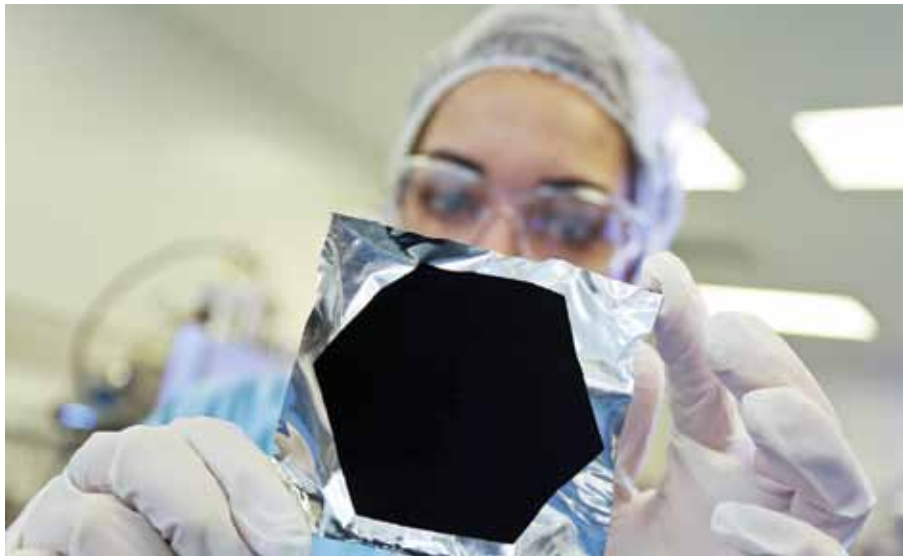
According to the company, the new version of Vantablack absorbs so much light that it cannot even be measured by spectrometers. The company provides no additional information on exactly what it did differently to make the new version of Vantablack its blackest yet, however.

Watch the new material's incredible light-trapping power in the Surrey



Credit: Lockheed Martin

An artist's concept of a possible Low Boom Flight Demonstration Quiet Supersonic Transport (QueSST) X-plane design.



Credit: Surrey NanoSystems

Vantablack coated onto a piece of wrinkled aluminum foil looks more like nothing than anything else.

NanoSystems video at youtu.be/O0CYc_mC3Uo.

But, in general, how does Vantablack suck up such a vast majority of the light that hits its surface?

Surrey NanoSystems grows Vantablack surfaces via chemical vapor deposition of carbon nanotubes that measure just 20 nm in diameter and stretch out in length 14–50 μm. And Vantablack’s thin and long carbon nanotubes are tightly packed—Surrey NanoSystems says that “a surface area of 1 cm² would contain around 1,000 million nanotubes.”

According to the company, the lengths of the tubes in relation to their small diameters and the space surrounding them are what make the material so effective at trapping light, forcing it to bounce around among the nanotubes.

The materials’ nanostructure traps light so well that it barely escapes—less than 0.036% of light is reflected from the surface (of the original Vantablack)—making a coating of the material look more like nothing than like anything else.

And according to an article from *The New York Times* detailing an interview with Surrey chief tech officer Ben Jensen, Vantablack has a production advantage that makes the material promising for diverse applications—growing

carbon nanotubes at lower temperatures.

“Growing carbon nanotubes isn’t new,” Jensen says in the article. “But typically they’ve been grown at a very high temperature: 750 degrees centigrade. That would destroy most underlying materials, so they grew them on things like silicon, diamond, and sapphire, which can stand high temperatures. We’re building on work to grow nanotubes at a lower temperature for microelectronics.”

But Vantablack’s impressive properties do not stop there.

According to a 2015 press release from the United Kingdom’s National Physical Laboratory, which measured the material’s officially recorded light reflectance, “Vantablack has the highest thermal conductivity and lowest mass-volume of any material that can be used in high-emissivity applications. It has virtually undetectable levels of outgassing and particle fallout, thus eliminating a key source of contamination in sensitive imaging systems. It withstands launch shock, staging, and long-term vibration, and is suitable for coating internal components, such as apertures, baffles, cold shields, and microelectromechanical-systems-type optical sensors.” ■

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ACerS Section events and happenings

Stay on top of upcoming events for your Section. Having a Section meeting or event soon? Contact Stephanie Liverani at sliverani@ceramics.org to include yours in the next issue of the *ACerS Bulletin*.

June 8, 2016

Southwest Section: Hilton Birmingham Perimeter Park Hotel – Birmingham, Ala.; ceramics.org/sections/southwest-section ■

ACerS and GOMD announce 2016 lecture awards at May Meeting

ACerS and the Glass and Optical Materials Division will honor its 2016 lecture award recipients during ACerS GOMD meeting, May 22–26, in Madison, Wis. For more information about the award lecturers, visit ceramics.org/glass-optical-materials-division-2016-award-speakers.

Stookey Lecture of Discovery



Griscom

Monday, May 23, 8 a.m.
David L. Griscom, impactGlass research international, *The life and unexpected discoveries of an intrepid glass scientist*

George W. Morey Award



Eckert

Tuesday, May 24, 8 a.m.
Hellmut Eckert, Institute of Physics in São Carlos, University of São Paulo, Brazil & Institute of Physical Chemistry, University of Münster, Germany, *Spying with spins on messy materials: 50 years of glass structure elucidation by NMR spectroscopy*

Norbert J. Kreidl Award for Young Scholars



Li

Tuesday, May 24, Noon
Lan Li, postdoctoral associate in the department of materials science and engineering at Massachusetts Institute of Technology, *Materials and devices for mechanically flexible integrated photonics*

Darshana and Arun Varshneya Frontiers of Glass Science Lecture



Ciccotti

Wednesday, May 25, 8 a.m.
Matteo Ciccotti, professor of mechanics and physics of materials at École Supérieure de Physique et Chimie Industrielles

de la Ville de Paris (ESPCI Paristech, France), *Multiscale investigation of stress-corrosion crack propagation mechanisms in oxide glasses*

Darshana and Arun Varshneya



Dejneka

Frontiers of Glass Technology Lecture

Thursday, May 26, 8 a.m.
Matthew J. Dejneka, research fellow, Corning Glass Research Group, *Chemically strengthened*

glasses and glass-ceramics ■

Engineering Ceramics Division secretary nominations due August 15

The ECD Nominating Committee invites nominations for the incoming division secretary candidate for 2016–2017 to be presented for approval at the ECD Annual Business meeting at MS&T16 and to go on the ACerS annual division officer ballot in spring 2017. Nominations and a short description of the candidate's qualifications should be submitted by **August 15** to Junichi Tatami, Yokohama National University, Japan, chair of the ECD Nominating Committee (tatami@ynu.ac.jp); Hua-Tay Lin, Guangdong University of Technology, China (huataylin@comcast.net); or Vojislav V. Mitic, University of NIS, Serbia (vmitic.d2480@gmail.com). For more information, visit ceramics.org/divisions. ■

In memoriam

*Derek Albon
John Storer-Folt
Raymond P. Heilich*

Some detailed obituaries also can be found on the ACerS website, ceramics.org/in-memoriam.

Names in the news

Edwards named dean at Rochester Institute of Technology



Edwards

ACerS member Doreen Edwards has accepted the position of dean of the Kate Gleason College of Engineering at Rochester Institute of Technology (Rochester, N.Y.) effective July 1, 2016. Edwards has been dean of the Kazuo Inamori School of Engineering at Alfred University (Alfred, N.Y.) since 2009 and acting vice president for statutory affairs at Alfred since 2014. Edwards says the decision to leave AU was difficult, but she is excited about the challenges ahead. "I'm sad to be leaving Alfred University. It's been my academic home for over 18 years. I look forward to maintaining strong personal and professional connections with the faculty, staff and alumni." The full press release can be accessed at bit.ly/154B1nR.

Ballato named academician in World Academy of Ceramics



Ballato

ACerS member John Ballato, a Clemson University professor who specializes in making optical fiber, has been elected an academician in the World Academy of Ceramics. The appointment goes to individuals who have made internationally renowned contributions to the advancement of ceramics, culture, science, and technology. The Academy's total membership is limited to about 200 people worldwide. "It's always humbling to be recognized by your peers," Ballato says in a news release. "This appointment is particularly special because the members come from all over the world. So many others are equally deserving." The full press release can be accessed at bit.ly/1TPC8wc. ■

Students and outreach

Student contests at MS&T16

The following Material Advantage student contests will be held at MS&T this year in Salt Lake City, Utah:

- Undergraduate Student Poster Contest
- Undergraduate Student Speaking Contest
- Graduate Student Poster Contest
- Ceramic Mug Drop Contest
- Ceramic Disc Golf Contest

For more information on any of the contests or student activities at MS&T, visit matstech.org/students, or contact Tricia Freshour at tfreshour@ceramics.org. ■

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CERAMIC AND GLASS INDUSTRY FOUNDATION

The Ceramic and Glass Industry Foundation's first annual report highlights new student-focused initiatives

The Ceramic and Glass Industry Foundation (CGIF) works to attract, inspire, and train the next generation of ceramic and glass professionals. Several programs were launched in 2015 to accomplish these goals and are highlighted in the CGIF's forthcoming 2015 Annual Report.

Introducing young people to the world of ceramic and glass materials is one of the primary goals of the CGIF. Its student outreach programs promote ceramic and glass science and engineering to middle and high school students through various initiatives. CGIF volunteers and staff participate in science fairs and other events nationwide to promote the opportunities and rewards of a career in this field, including the 2016 USA Science & Engineering Festival in Washington, D.C., which was held April 15–17.

The CGIF reaches out to students by providing teachers with ACerS Material Science Kits, which provide supplies and lesson plans for fun, interactive materials science experiments in the classroom.

Another program launched in 2015 is the CGIF University-Industry Network. Designed to encourage schools around the world to continue teaching key concepts in ceramic and glass science, this program provides financial and programmatic resources to key professors at participating universities to give their students more opportunities to develop an interest in the field.

The CGIF University-Industry Network also helps connect students

with industry leaders who utilize ceramic and glass materials. Five schools were selected to pilot the program in the 2015–2016 academic year: Alfred University (Alfred, N.Y.), Clemson University (Clemson, S.C.), The Colorado School of Mines (Golden, Colo.), Pennsylvania State University (State College, Pa.), and Missouri University of Science and Technology (Rolla, Mo.).

To help job and internship seekers find the best ceramic and glass career opportunities, the CGIF launched the Ceramic and Glass Career Center in 2015 (careers.ceramics.org). The site is promoted to all members of ACerS, including students and young professionals. It is the ideal place for employers who utilize ceramic and glass materials to find the most qualified materials students and professionals.

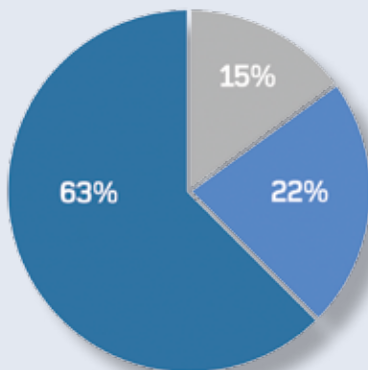
The CGIF also supported student travel grants and exchanges in 2015 to enable students to gain a broad perspective and professional insight into their chosen career path. The CGIF provided travel assistance to four U.S. students to attend the European Ceramics Society (ECerS) Summer School in Madrid, Spain, in June 2015. In June 2016, the CGIF will expand the program by offering travel grants to 15 students to attend the ECerS Electroceramics Summer School June 23–25, in Limoges, France.

The CGIF acknowledges the help and support of its volunteers and donors. For more information or to get involved, contact Marcus Fish, CGIF development director, at 614-794-5863. ■

2015 Program Expenses

■ Student exchanges and travel grants	\$ 6,000
■ Student outreach	\$ 9,000
■ University-Industry Network	\$25,000

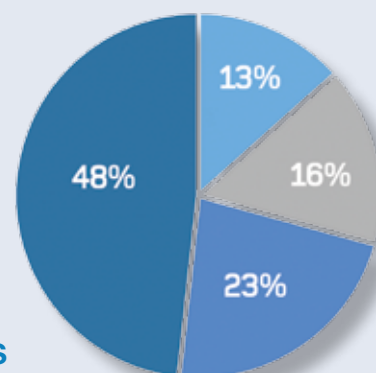
Total program expenses \$40,000



2015 Funding Sources

■ Individuals.....	\$ 87,186
■ Foundations.....	\$103,500
■ Corporations.....	\$146,226
■ ACerS matching gifts	\$314,500

Total funding \$651,412



Awards and deadlines

Deadlines for upcoming nominations

May 15, 2016

NEW! Samuel Geijsbeek PACRIM International Award

This new award recognizes individuals who are members of the Pacific Rim Conference (PACRIM) societies for contributions to the field of ceramics and glass technology that have resulted in significant industrial and/or academic impact, international advocacy, and visibility of the field. Two Geijsbeek awards will be presented at PACRIM 2017. The Geijsbeek Award consists of a certificate and \$1,000 honorarium.

Glass & Optical Materials: Alfred R. Cooper Scholars Award

This award recognizes undergraduate students who have demonstrated excellence in research, engineering, and/or study in glass science or technology. The recipient will receive a plaque, a check for \$500, and free MS&T registration.

Electronics: Edward C. Henry Award

This annual award recognizes an outstanding paper reporting original work in the *Journal of the American Ceramic Society* or the *ACerS Bulletin* during the previous calendar year on a subject related to electronic ceramics. The author(s) will be presented with a plaque and \$500 (split between authors).

Electronics: Lewis C. Hoffman Scholarship

The purpose of this \$2,000 tuition award is to encourage academic interest and excellence among undergraduate students in the area of ceramics/materials science and engineering. The 2016 essay topic is: electronic ceramics for electrical or electromagnetic energy control.

July 1, 2016

Engineering Ceramics Division: James I. Mueller Award

This award recognizes the accomplishments of individuals with long-term service to ECD or work that has resulted in significant industrial, national, or academic impact in the field. Selection can be based on either criterion. The awardee

receives a memorial plaque, certificate, and honorarium of \$1,000. Contact Soshu Kirihara at kirihara@jwri.osaka-u.ac.jp for more information.

Engineering Ceramics Division: Bridge Building Award

This award recognizes individuals outside the United States who have made outstanding contributions to engineering ceramics. The main criteria are the individual's contributions to the field of engineering ceramics, including expansion of the knowledge base and commercial use thereof, or contributions to the visibility of the field on an international stage. Award selection can be based on either criterion. The award consists of a glass piece, certificate, and an honorarium of \$1,000. Contact Andrew Gyekenyesi at Andrew.L.Gyekenyesi@nasa.gov for more information.

Engineering Ceramics Division: Global Young Investigator Award

This award recognizes an outstanding scientist conducting research in academia, industry, or at a government-funded laboratory. ACerS members 35 years of age or younger are eligible for consideration. The award consists of a glass piece, certificate, and \$1,000. Contact Jingyang Wang at jyywang@imr.ac.cn for more information.

September 1, 2016

2017 ACerS Class of Fellows

Nominees need to be at least 35 years old and have been members of the Society at least for the past five years continuously. Be sure to adhere to nomination and support letter length guidelines—nominations that do not conform will be returned. Scanned and faxed signature forms are permitted in lieu of original mailed signature forms. Previously submitted nominations may be updated, as long as they do not exceed length limitations.

Additional information and nomination forms for these awards can be found at ceramics.org/awards. Contact Marcia Stout at mstout@ceramics.org with any questions. ■

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NIST

This article first appears exclusively in the *Bulletin*, and can later be found online on *Ceramic Tech Today*.

Visualizing atoms at grain boundaries: Atom probe tomography gets into oxides

For such a small thing, grain boundaries are big.

“Grain boundaries are important because they dominate the properties of many ceramics—this is inconvenient, because grain boundaries typically make up only a fraction of the volume compared with bulk properties,” Brian Gorman, associate professor of metallurgical and materials engineering at Colorado School of Mines (Golden, Colo.), explains via email.

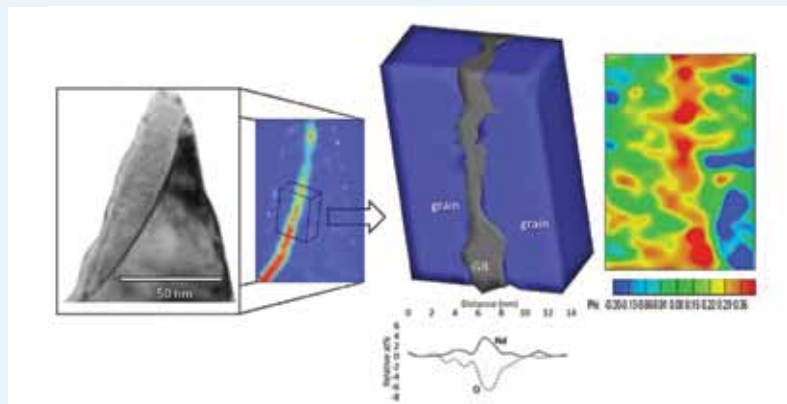
Grain boundaries influence a host of material attributes, including a material’s thermal, electrical, optical, magnetic, and mechanical properties.

Gorman and a team of researchers at Colorado School of Mines and the University of Florida are well on their way to solving grain boundaries’ secrets, however. The team recently achieved unprecedented atom-by-atom visualization of the chemical composition of grain boundaries.

In addition to providing maps of the atoms that reside at grain boundaries, the results provide direct insight into how local charges influence a material’s ionic conductivity—results that could help tune a material’s nanoscale composition to optimize macroscale properties.

The work, recently reported in the *Journal of Materials Chemistry A*, describes how the team used atom probe tomography (APT) to 3-D quantify oxygen and cation compositions at grain boundaries within a polycrystalline material.

APT uses a laser to evaporate atoms from a sharp-tipped sample of material, using a sensitive detector to resolve individual atoms with high detection efficiency. Although APT previously has been used to quantify grain boundaries in metallic materials, its application to oxide materials has been less straightforward.



(left) Brightfield TEM image of the atom probe specimen, overlaid onto an APT data reconstruction locating silicon impurities. (center) 3-D volume reconstruction (and corresponding 1-D composition profile, below) illustrates excess neodymium dopants and substoichiometric oxygen at the boundary. (right) Quantification of 3-D charge distribution and, thus, voltage at the grain boundary.

But not anymore—the team 3-D-quantified impurity cations and oxygen stoichiometry with subnanometer resolution in neodymium-doped ceria, a material with important energy applications.

Beyond this feat of atomic resolution, the team also quantified space charge voltage at grain boundaries and related it to ionic conductivity. In other words, the scientists showed that they can directly correlate structural measurements to material properties. “This ‘closes the loop’ on processing–structure–property relationships,” Gorman says.

The results represent the first time that researchers have measured 3-D oxygen stoichiometry at subnanometer spatial resolution—information that can associate defect chemistry at the nanoscale, which again ties together with macroscale properties, Gorman adds.

But the results did not come overnight.

“In order to accomplish this work, we had to first prove that we could determine oxygen concentrations quantitatively using laser-assisted atom probe tomography. We accomplished this over several years and, using experimental data, tied in to

finite-element models of the atom probe experiment.”

But the scientists did not stop there. They did their due diligence, making sure that they were on the right track.

Gorman continues, “We also had to prove that the oxygen stoichiometry changes we were observing were indeed associated with grain boundaries, so we performed correlative transmission electron microscopy imaging before and after APT experiments—again which we have been working on for more than eight years. Finally, we put together mathematical formalisms to convert 3-D APT data first into charge distributions and then into voltage distributions using a 3-D solution to the Poisson equation.”

Altogether, the results give the team confidence that what they have is the real deal. “We are now very comfortable with the whole process and believe we can analyze grain boundaries in virtually any oxide,” Gorman says.

The paper, published in the *Journal of Materials Chemistry A*, is “Three-dimensional quantification of composition and electrostatic potential at individual grain boundaries in doped ceria” (DOI: 10.1039/C5TA10064J). ■

Go thin or go home: Scientists create world's thinnest lens that could revolutionize consumer tech

When it comes to developing ultrathin lenses, scientists at Australian National University (Canberra, Australia) may have changed the game. The team created what it describes as “the world’s thinnest lens, one two-thousandth the thickness of a human hair,” which could revolutionize flexible computer displays and miniature cameras, according to a recent ANU press release.

Lead researcher Yuerui Lu from ANU Research School of Engineering says the team’s discovery hinged on the remarkable potential of molybdenum disulfide crystals.

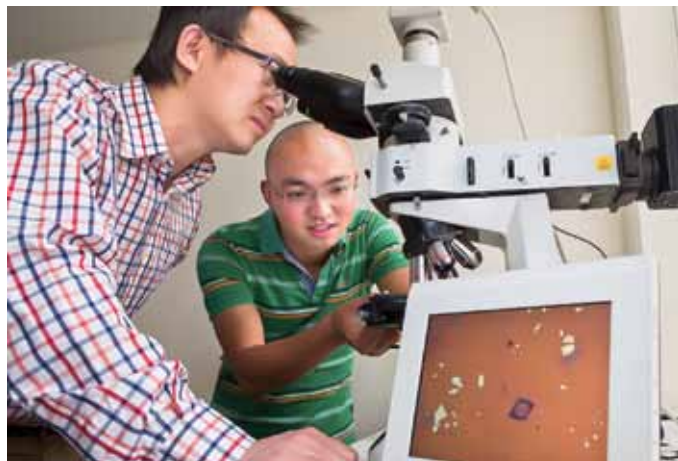
Molybdenum disulfide is a chalcogenide compound with interesting properties for high-tech applications because of its flexible electronic characteristics. “This type of material is the perfect candidate for future flexible displays,” Lu says in the release. “We will also be able to use arrays of microlenses to mimic the compound eyes of insects.”

The team created its new ultrathin lens from a 6.3-nm-thick crystal—nine atomic layers—which it peeled off a larger piece of molybdenum disulfide with sticky tape. The team then made a 10- μm -radius lens using a focused ion beam to shave off the layers, atom by atom, until it fashioned the lens into a dome shape, according to the release.

“Molybdenum disulfide is an amazing crystal,” Lu adds. “It survives at high temperatures, is a lubricant, a good semiconductor, and can emit photons, too. The capability of manipulating the flow of light in atomic scale opens an exciting avenue toward unprecedented miniaturization of optical components and the integration of advanced optical functionalities.”

The team also discovered that single layers of molybdenum disulfide—0.7-nm-thick—had remarkable optical properties, appearing to a light beam to be 38 nm, or 50 times thicker. Known as “optical path length,” this property determines the phase of light and governs interference and diffraction of light as it propagates, the release explains.

For comparison’s sake, molybdenum disulfide crystals’ refractive index—the property that expresses how many times faster light travels in a vacuum than in does in a specific material—has a high



Credit: Stuart Hay, ANU

Yuerui Lu (left) and Jiong Yan (right) from ANU Research School of Engineering examine what they describe as “the world’s thinnest lens, one two-thousandth the thickness of a human hair.” The lens is the purple circle that appears on the screen in the foreground.

value of 5.5. That significantly outshines the refractive index of diamond (2.4) and water (1.3).

Looks like the solution to next-generation ultrathin lenses is crystal clear.

The open-access paper, published in *Light: Science and Applications*, is “Atomically thin optical lenses and gratings” (DOI: 10.1038/lssa.2016.46). ■

Research News

Collaboration boosts potential for CdTe solar cells

Scientists at the National Renewable Energy Laboratory (Golden, Colo.) collaborated with researchers at Washington State University (Pullman, Wash.) and University of Tennessee (Knoxville, Tenn.) to improve maximum voltage available from a cadmium telluride (CdTe) solar cell, which is a key factor in improving solar cell efficiency. The team improved cell voltage by placing a few phosphorus atoms on tellurium lattice sites and then carefully forming ideal interfaces between materials with different atomic spacings to complete the solar cell. This approach improved conductivity and carrier lifetime by orders of magnitude, thereby enabling the fabrication of CdTe solar cells with an open-circuit voltage that broke the 1-V barrier for the first time. For more information, visit nrel.gov.

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This article first appears exclusively in the *Bulletin*, and can later be found online on *Ceramic Tech Today*.

Bringing the bounce: Unusual chemical structure gives new metallic glass material its elasticity

Engineers at the University of Southern California (Los Angeles); the University of California, San Diego; and the California Institute of Technology (Pasadena) have created a new metallic glass material with an unusual chemical structure that makes it incredibly hard and yet elastic, according to a *USC News* article.

The material, called SAM2X5-630, can withstand heavy impacts without deformation, the article explains. And it retains most of its original strength when pushed beyond its elastic limits without fracturing.

SAM2X5-630 falls into the category of “bulk metallic glasses” or BMGs, artificially generated materials that possess disproportionate strength, resilience, and elasticity because of their unusual chemical structure, according to the article.

“Typical metals and metal alloys have an organized, crystalline structure at the atomic level. BMGs are formed when metal and metal alloys are subjected to extreme heat and then rapidly cooled, exciting their atoms into disorganized arrangements and then freezing them there,” the article explains.

To create SAM2X5-630, the team heats powdered iron composite to

630°C (1,166°F) and then rapidly cools it. The team uses a spark plasma sintering process in which the iron compound is powdered, placed in a graphite mold, and zapped with a current under pressure, the article explains. The technique superheats the powdered iron enough to bind it without liquefying it.

Spark plasma sintering saves time and money. “You can produce materials that normally take hours in an industrial setting in just a few minutes,” Olivia Graeve, ACerS member and professor of mechanical engineering at the Jacobs School of Engineering at UC San Diego, says in a UC San Diego news release. Graeve led design and fabrication work on the material.

But, what makes SAM2X5-630 particularly interesting is that it is not wholly a glass. The team found that controlling the exact amount of heat and timing to create the material is the key to its unique properties. If the same iron composite is heated and cooled at even slightly different temperatures or rates, a totally different atomic structure results that does not have the same elastic properties.

According to the UC San Diego news release, a 1.5- to 1.8-mm-thick piece of SAM2X5-630 has a Hugoniot elastic limit of 11.76 ± 1.26 GPa. “By comparison, stainless steel has an elastic limit of 0.2 GPa, while that of tungsten carbide (a high-strength



Transmission electron microscopy image showing various levels of crystallinity embedded in the amorphous matrix of the alloy.

ceramic used in military armor) is 4.5 GPa.”

“It [SAM2X5-630] has almost no internal structure, like glass, but you see tiny regions of crystallization,” Veronica Eliasson, assistant professor at the USC Viterbi School of Engineering and lead author of the research, says in the article. “We have no idea why a small amount of crystalline regions in these bulk metallic glasses makes such a big difference under shock loading.”

The unique qualities of SAM2X5-630 make the material widely applicable for use in protective shields, such as body armor for soldiers and meteor-resistant casings for satellites.

The open-access paper, published in *Scientific Reports*, is “Shock wave response of iron-based in situ metallic glass matrix composites,” (DOI:10.1038/srep22568). ■

Research News

DFG launches German–Russian cooperation in materials research

The German Research Foundation (DFG) and Russian Foundation for Basic Research will fund a new project to establish a transnational German–Russian research group. The group will include 25 participating scientists from TU Dresden and TU Ilmenau in Germany and universities in Yekaterinburg, Moscow, and Perm, Russia. The program’s goal is to create magnetically controlled elastic materials and to use them for customized sensory applications. Russian research groups will contribute expertise in the theory of magnetic hybrid materials. German groups will focus on mechanical and magnetic characterization, microstructure analysis, and technical application of these novel materials. For more information, visit tu-dresden.de.

Three-way catalysts: Cool way to make catalytic converters

Advanced Institute for Materials Research (Tohoku University, Japan) researchers have developed a new, mild-temperature method for producing cerium oxide nanorods. The nanorods show excellent oxygen storage capacity at temperatures below 200°C, making them promising for use as catalysts to control harmful vehicle emissions. The team adopted a method that involves corrosion of ribbons of cerium–aluminum alloys in an alkaline medium. In this reaction, aluminum is leached, whereas cerium is oxidized. Importantly, this reaction occurs under mild conditions, which allow fabrication of fine nanorod structures with diameters of ~5–7 nm. For more information, visit research.wpi-aimr.tohoku.ac.jp.

This 2-D material could upstage graphene in the digital tech game

Graphene has a lot going for it. But now there is a new one-atom-thick material that could boot graphene from its seat as the wonder material to advance electronic tech.

A physicist at the University of Kentucky (Lexington, Ky.), working in collaboration with scientists from Daimler in Germany and the Institute for Electronic Structure and Laser (IESL) in Greece, has discovered a new material “made up of silicon, boron, and nitrogen—all light, inexpensive, and Earth-abundant elements,” according to a university news release. To boot, this material is extremely stable—a property many other graphene alternatives lack.

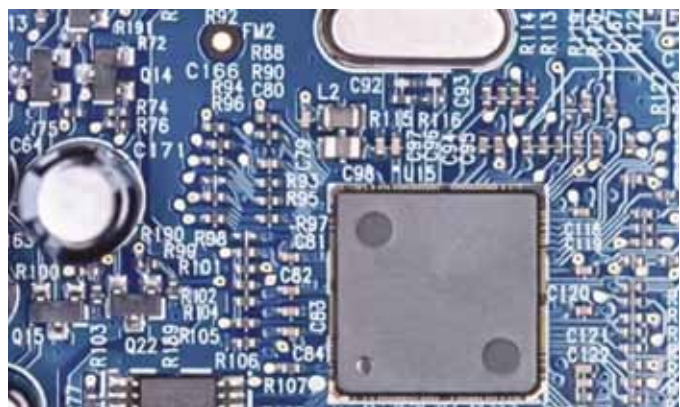
“We used simulations to see if the bonds would break or disintegrate—it didn’t happen,” senior author Madhu Menon, physicist in the University of Kentucky Center for Computational Sciences, says in the release. “We heated the material up to 1,000 degrees Celsius, and it still didn’t break.”

Graphene’s strength and unique properties put the material in a class of its own, but it has a downside: It is not a semiconductor, and, because of that, it has not been able to compete in the digital tech industry, the release explains.

Researchers on the quest to discover new 2-D semiconducting materials have relied on a class of three-layer materials called transition-metal dichalcogenides (TMDCs), which can be made successfully into digital processors. But TMDCs present significant bulk compared with wafer-thin graphene—and they are made of materials that are not readily available or necessarily cheap.

Menon and his colleagues set their sights on finding an alternative that is light, Earth-abundant, inexpensive, and can act as a semiconductor. After many tests and experiments, they uncovered the right combination of silicon, boron, and nitrogen to create a stable structure that was arranged in the same hexagonal molecular pattern as graphene.

Although the new material is metallic, like graphene, it can transform into a semiconductor easily by attaching other ele-



Credit: University of Kentucky; YouTube

Researchers have developed a promising new 2-D material.

ments on top of the silicon atoms—a property that offers the “exciting possibility of seamless integration with the current silicon-based technology,” the release explains.

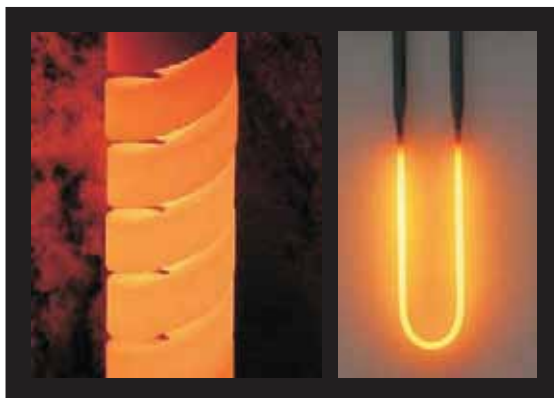
And the attachment of other elements can be used to selectively change bandgap values—a major advantage over graphene when it comes to solar energy conversion and electronics applications, according to the research.

“We are very anxious for this to be made in the lab,” Menon says. “The ultimate test of any theory is experimental verification, so the sooner the better!”

A video featuring the new material and how it could upstage graphene is available at youtu.be/IKc_PbTD5go.

The paper, published in *Physical Review B, Rapid Communications*, is “Prediction of a new graphene-like Si₂BN solid” (DOI: 10.1103/PhysRevB.93.081413). ■

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Atomic vibrations in nanomaterials

Atomic vibrations, or “phonons,” are responsible for how electric charge and heat is transported in materials. Researchers at ETH Zurich in Switzerland have shown for the first time what happens to atomic vibrations when materials are nanosized and how this knowledge can be used to systematically engineer nanomaterials. Their work shows that when materials are smaller than ~10–20 nm, vibrations of the outermost atomic layers on nanoparticle surfaces are large and are important in how the material behaves. For example, the researchers show—using experiment and theory—that surface vibrations interact with electrons to reduce the photocurrent in solar cells. For more information, visit ethz.ch/en/news-and-events. ■

Redesigned micro solid oxide fuel cell may provide more power, less charging, to small consumer electronics

Despite significant advancements in solid oxide fuel cell (SOFC) technology, these power sources continue to be plagued by problems that inhibit their viability for many commercial uses.

One big problem is that many current designs use silicon to support the cell's internal membranes, but these cells eventually suffer from degradation and instability because of thermal expansion mismatch between those materials. This instability limits use of SOFCs in devices that require fast switching between on and off (i.e., most electronic devices).

Researchers at Pohang University of Science & Technology (POSTECH) in South Korea have developed a micro-sized SOFC that sidesteps silicon's problems, instead using a much more thermally and mechanically robust support—porous stainless steel, which significantly improves the cell's thermal and mechanical stability.

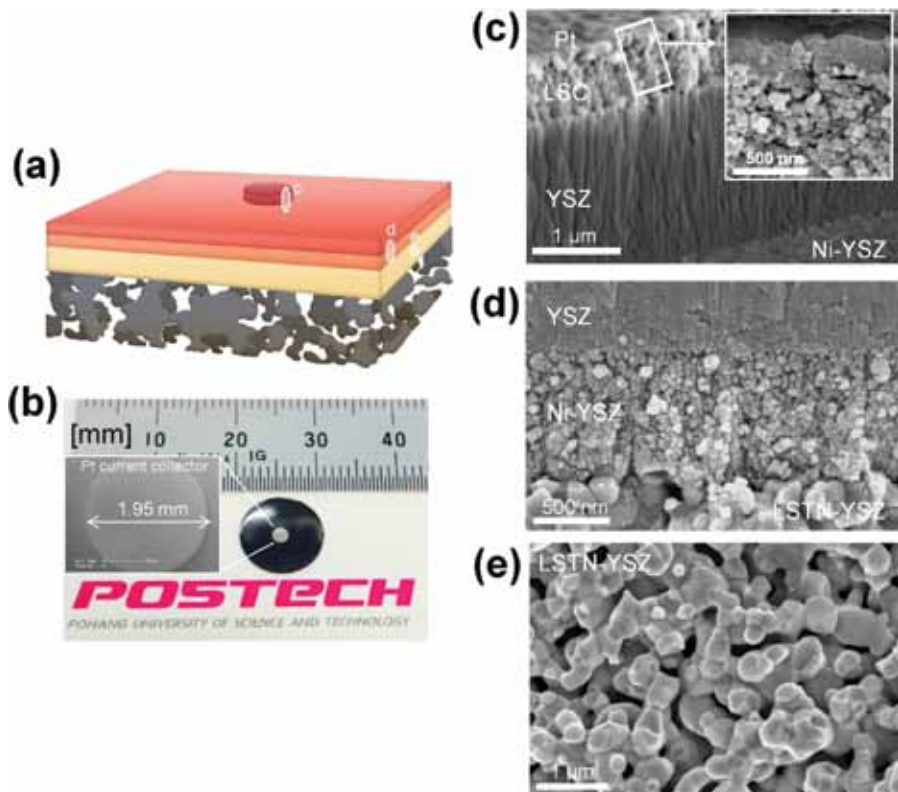
“To the best of our knowledge, this is the first demonstration of the ability of the thermal robustness of a micro-SOFC, which never has been attained in many conventional silicon-based devices,” the authors write in the open-access *Scientific Reports* paper describing their work.

The team thinks its development could help usher these bite-sized power sources into consumer electronic devices, including smartphones, laptops, drones, and more.

To build the novel micro-SOFC, the POSTECH scientists deposited a dual layer substrate on porous stainless steel. They first deposited a contact layer made of a combination of (La, Sr)(Ti, Ni)O₃ (LSTN) and yttria-stabilized zirconia (YSZ) (LSTN-YSZ) on the stainless steel. Then, on top of the contact layer, the scientists deposited a gas-tight YSZ thin-film electrolyte.

But it was not easy—according to first author Kun Joong Kim, depositing the thin-film components over steel posed the biggest challenge in developing the microcell.

Although perfecting the recipe was not simple, the team's efforts paid off.



Researchers have developed a new micro solid oxide fuel cell supported on stainless steel. (a) Schematic of the cell's thin films supported on porous stainless-steel substrate. (b) Prototype cell. Cross-sectional SEM images of (c) Pt/LSC/YSZ (inset: magnified view of Pt/LSC), (d) YSZ/Ni-YSZ/LSTN-YSZ, and (e) LSTN-YSZ contact layer.

The combination of materials allowed researchers to fabricate a cell with a total area of 78 mm² that exhibited “good mechanical stability, ease of handling, and flexibility,” the authors write in the paper.

And, because they fabricated the cells using tape casting–lamination–cofiring, which is typically used to fabricate larger SOFCs, the scientists say that commercial scale-up should be feasible. “For larger cells, we will either modify the thin-film deposition method or adopt new thick-film cell components and firing processes,” Kim says in an email.

The authors report that their experiments show that the prototype micro-SOFC performed well, exhibiting a peak power of 560 mW/cm² at 550°C.

But to really put the cell to the test, the authors subjected it to rigorous thermal cycling—between 350°C and 550°C, up

to 15°C/min, for 6 h. Even those stressful conditions failed to cause measurable degradation or appearance of cracks or delamination, the authors report.

They write in the paper, “We speculate that the porous but ductile [stainless steel] substrate may absorb the thermo-mechanical stress caused by sealants or the alumina tube during thermal cycles.”

The researchers speculate that such micro-SOFCs will be able to power small portable electronic devices that require high power density and fast thermal cycling. For example, they say the cells could fly drones for more than an hour and power smartphones for an entire week.

The open-access paper, published in *Scientific Reports*, is “Micro solid oxide fuel cell fabricated on porous stainless steel: A new strategy for enhanced thermal cycling ability” (DOI: 10.1038/srep22443). ■

Heating up rust could make large-scale solar power storage possible

Commercialized solar energy use in the United States spiked 33% in 2014, thanks to soaring solar industry expansion. Its applications are becoming more widespread as the world moves to a cleaner energy future. But, the challenge facing researchers to date has been mastering efficient capture and storage of solar power.

Researchers at Stanford University (Stanford, Calif.) recently found that ordinary metal oxides, such as rust, can be made into solar cells capable of splitting water into hydrogen and oxygen, according to a *Stanford News* report.

“Using solar cells to split H_2O by day is a way to store energy for use at night. The photons captured by the cell are converted into the electrons that provide the energy to split water,” the release explains. “Recombining hydrogen and oxygen after dark would be a way to reclaim that energy and ‘dispatch’ power back into the electrical grid—without burning fossil fuels and releasing more carbon into the atmosphere.”

Metal oxide solar cell potential is not a novel concept—but it is less efficient than a silicon solar cell when it comes to photon to electron conversion.

The Stanford team found that “as metal oxide solar cells grow hotter, they convert photons into electrons more efficiently. The exact opposite is true with silicon solar cells, which lose efficiency as they heat up,” the report explains.

“We’ve shown that inexpensive, abundant, and readily processed metal oxides could become better producers of electricity than was previously supposed,” William Chueh, assistant professor of materials science and engineering at Stanford, says in the report.

This new and surprising breakthrough could mean major changes in how we produce, store, and consume energy, with more efficiency and cost-effectiveness than we ever thought possible.

“By combining heat and light, solar water-splitting cells based on metal oxides become significantly more efficient at storing the inexhaustible power of the sun for use on demand,” says Chueh.

The study, published in *Energy and Environmental Science*, is “Significantly enhanced photocurrent for water oxidation in monolithic $Mo:BiVO_4/SnO_2/Si$ by thermally increasing the minority carrier diffusion length” (DOI: 10.1039/C6EE00036C). ■



Credit: iustatira-5; Flickr CC BY-NC-ND 2.0

Rust could be the surprising key to large-scale solar power storage, according to new research from Stanford University researchers.



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A sample from the Broburg site in Sweden. Cutaway shows molten areas of the sample; solid areas show unreacted material.

Ensuring longevity: Ancient glasses help predict durability of vitrified nuclear waste

By Jamie L. Weaver, John S. McCloy, Joseph V. Ryan, and Albert A. Kruger

Ancient glass artifacts provide a surprisingly rich source of analogues to study long-term mechanisms of glass alteration for design of new glasses for nuclear waste disposal.

How does glass alter with time? For the last hundred years this has been an important question to the fields of object conservation and archeology to ensure preservation of glass artifacts.¹ This same question is part of the development and assessment of durable glass waste forms for the immobilization of nuclear wastes. Researchers have designed experiments ranging from simple to highly sophisticated to answer this question and, as a result, have gained significant insight into the mechanisms that drive glass alteration. However, gathered data have been predominately applicable to only short-term alteration—i.e., over the course of decades.

Long-term mechanisms of glass alteration have remained elusive.² These mechanisms are of particular interest to the international nuclear waste glass community, because it strives to ensure that vitrified products will be durable for thousands

to tens of thousands of years. For the past three decades, this community has been attempting to fill this research gap by partnering with archeologists, museum curators, and geologists to identify hundred-to million-year-old glass analogues that have altered in environments (Figure 1) representative of those expected at potential nuclear waste disposal sites. Even with these partnerships, the process of identifying a waste glass relevant analogue is challenging; it requires scientists to relate data collected from short-term laboratory experiments to observations made from long-term analogues and extensive geochemical modeling.

Choosing an appropriate analogue: Initial considerations

When initially approaching the challenge of determining a glass alteration analogue, one could choose to limit the types of glasses to those that have compositions similar to nuclear waste glasses. Although it is very unlikely that an analogue will have exactly the same composition as a nuclear waste glass, it is important to study glasses that contain similar mass percentages of the baseline oxides of silica, aluminum, sodium, and, if possible, boron.

The difficulty of finding a one-to-one analogue is in part due to the complex elemental composition of nuclear waste. In general, the two most common types of vitrified

nuclear wastes are low activity waste (LAW) and high-level waste (HLW). Classification of these wastes is most often based on present radionuclides and regulations regarding how these radioactive elements are immobilized and isolated from the environment. Additionally, researchers further distinguish glasses by their relative elemental concentrations.

For U.S.-based LAW glasses, baseline components are typically ~45 mass% SiO₂ and ~20 mass% Na₂O, mixed with ~6 mass% Al₂O₃, ~9 mass% B₂O₃, and ~20 mass% of other waste-derived oxides. Alternatively, U.S.-based HLW glasses contain ~30–55 mass% SiO₂, ~15–20 mass% Na₂O, ~4–22 mass% Al₂O₃, and ~5–20 mass% B₂O₃, with other oxides comprising the balance, either added in the frit or from the waste stream. HLW glasses also may contain more iron than LAW glasses. Depending on the origin of the waste stream—i.e., industrial waste from used nuclear fuel reprocessing or legacy defense nuclear waste—these other elements can vary widely in identity, concentration, and oxidation state.

Once a waste glass of interest is identified and its compositional range defined, then it is possible to begin looking for an appropriate analogue or set of analogues (Figure 1). An ideal analogue, as stated above, should contain most, if not all, of the main elements in the waste glass in

Credit: Tamas Veres, Carolyn Pearce, and Mike Perkins (PNNL).

Capsule summary

THE CHALLENGE

Studying how glass alters over time, especially large spans of time, is a challenging research problem. However, thorough understanding of these mechanisms is critical for prediction and design of glasses that can safely store nuclear waste for long periods of time.

WHAT'S OLD IS NEW AGAIN

Ancient artifacts represent a rich source of material to study long-term glass alterations under various environmental and exposure conditions. Although artifacts often have compositions different from new glasses, they offer many potential analogues for study.

FUTURE MODELS

Better understanding of long-term glass alteration allows more accurate prediction of the performance of vitrified nuclear waste to help develop waste glasses that will be durable for many millennia.



mass% that fall within the composition range defined for the waste glass. Many previously studied analogues (see below) have two or three of the main elements of their corresponding waste glasses. In these cases, a second set of analogues that contains the relevant mass% of the other primary elements—and do not deviate more than a few mass% of the other elements—could be identified and studied. Combining both data sets could allow a researcher to develop a well rounded view of how major elements affect the long-term durability of waste glass.

Additionally, studying two or more analogues may provide insight into how a slight change in composition affects the overall durability of a sample. Understanding these interactions is important because glasses produced during vitrification often vary slightly from one melt batch to the next. These differences in composition are intentional and are designed to accommodate the special chemistry of a particular waste stream and to efficiently incorporate the waste elements into the glass.

Parallel to selecting an analogue that has a similar composition as the waste glass of interest, it also is important to choose an analogue that has been altered under relevant conditions. This is because how a glass alters can be partially determined by its altering environment.³ A candidate glass analogue should come from alteration environments similar to those that have been suggested or chosen for the disposal of nuclear waste glasses. Disposal areas are often designed to have engineered barriers, such as clay and granite, which will

Figure 1. Locations and timeline of various types of excavated vitreous materials used as analogues to study durability of modern nuclear waste glasses.

dictate chemistry of the environment in contact with the glass.

Analogues are samples with a past

In addition to the above considerations, scientists also collect information regarding the provenance, or history, of a possible glass analogue. They often mine this data from archeology, art conservation, and geology journals, or gather data through interviews with museum staff, geologists, or archaeologists who have studied the glass. Scientists can then use these findings to answer three subsequent questions about the analogue:⁴

- Is it ethical or feasible to analyze this glass?
- What ex-situ factors may have affected how this glass was altered, and what effects did they have?
- How does studying this glass help validate current glass alteration models?

Researchers should be aware of any cultural importance of a possible glass analogue and should carefully consider what physical and philosophical effects any analysis might have on an artifact's long-term preservation. Researchers should ask whether analyzing the glass will change how it may be interpreted by



future generations. It is important that researchers make these considerations with the advice of conservators, archeologists, art historians, and museum curators who specialize in the time period and geographical area associated with the artifact.

In all cases, an approved analogue should be analyzed using nondestructive methods if possible. However, if some destructive analysis is warranted to obtain critical information that otherwise could not be obtained, researchers must consider additional precautions. For instance, they should consider how destructive methods will alter the glass. They should be aware of what information they are removing from the object during analysis. They also need to consider what chemical changes the analysis technique might induce in the object, and whether this will impact its future preservation or interpretation.

Scientists studying the analogue also should consider how collected data can help validate or invalidate current glass alteration models. As stated in question

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three, a scientist should be aware of what part of the alteration model is being tested, which requires knowledge of glass corrosion science and theory.

Scientists should also consider what effect outside factors may have had on the alteration of glass analogues. Glass alteration models are developed from data collected on controlled experiments, but analogues are rarely altered under controlled conditions. Variability in alteration conditions could include annual changes of the pH of the groundwater that was in contact with the analogue, or fluctuations in microbial populations in

the soil surrounding a buried analogue. It can be difficult to know all factors that might have influenced alteration of an ancient glass during its history, and it can be challenging to separate out the effects of each factor. However, simplified experiments performed in a laboratory setting can help disentangle these factors.

Data collected in the analysis of ancient glasses also can be utilized in preservation and historical interpretation of the artifact itself or similar pieces. For example, archeologists continue to attempt to gain an understanding of the methods used to make glass recovered at the Broborg site in

Sweden (Figure 2 and sidebar). Scientists hope that further analysis of ancient glasses may uncover redox conditions used by these ancient peoples and demonstrate a connection between iron working from that time and creation of the glasses.

Previous studies

Natural glasses

Most early studies of ancient glasses for the purpose of model validation have focused on alteration of natural glasses, such as basalts, tektites, and rhyolites.⁸⁻¹⁰ Rhyolitic glasses are high-silica natural volcanic glasses also known as obsid-

Broborg hillfort case study

Archeologists define vitrified forts as stony fortifications in which a dry-wall structure has been bound by molten or calcined materials.⁵ Over the past 60 years, scientists have produced three theories of how hillforts were vitrified: *incidental*, or resulting from cooking-hearth fires, forges, or even lightning; *constructive*, meaning materials were purposefully selected and melted to fortify dry-wall structures; and *destructive*, meaning walls surrounded by organic matter, such as timber, that was set on fire either by accident or during an attack.⁶

In the case of the Broborg hillfort, located near Uppsala, Sweden, scientists believe that vitrification was intentional, and its construction was completed around 500 CE during the Migration Period (prior to the Viking age). Several excavations of the site have resulted in evidence of house foundations, suggesting that at one point it held a permanent settlement. Walking along the edge of the partially vitrified wall at the site, a casual observer will find it initially difficult to discern rock from vitrified material. However, Peter Kresten—a geologist who has spent most of his professional career investigating vitrified forts—can provide guidance that makes differences between the two materials apparent. Atop and between about one-third of the well-weathered black-and-white-speckled granite and gneiss boulders lie smooth sections of amphibolite melt. With further survey of the site, one can see that the areas where the boulders were not covered and fused with vitrified matter are more heavily eroded than their fortified counterparts. When the dust is lightly swept away from vitrified sections, one discovers a slightly cloudy material that ranges in colors from dark brown to almost clear and, in some areas, still bears marks of the charcoal used in its firing.

Amphibolite appears to have played an important role in the construction of Broborg and has



(left) Jamie Weaver at the Broborg hillfort near Uppsala, Sweden, where remains of a vitrified wall are protruding from the snow. (right) Close-up of a molten section from a hillfort, showing impressions of the charcoal used to vitrify the materials, despite ~1,000 years of weathering.

led researchers at Washington State University and Pacific Northwest National Laboratory to dig into the geology of this region of Sweden. The amphibolite used at the site is metamorphic doleritic rock formed at high water pressure and contains mainly hornblende and feldspars. Studies of the amphibolite left at the site have uncovered evidence of a wide range of localized melting and moisture evolution, from slight heating to complete liquefaction. WSU scientists have found that temperatures >1,400°C are needed to create a molten material if the melting is completed in air without

the aid of bellows. Other studies have found that amphibolite collected from the site could be melted only with the help of a forced draft and a furnace hearth covered with turf.⁷ These results suggest that to create vitrified portions of the wall, ancient people most likely had to control redox condition and water content of the melt—a technology they most likely developed based on their experiences with iron smelting. Understanding how these melts were made could inform their chemistry, which is invaluable in determination of the long-term durability of Broborg glasses. ■



(left) Melt series based on chemistry of melted amphibolite composition from the Broborg site. Melting in atmosphere and without the use of bellows required heating the material to temperatures >1,400°C, which is not likely to be reached in ancient times. (right) Final glass produced from the melting study.



Credit: John McCoy



Credit: John McCoy

Figure 2. Examples of hillfort glasses, which are manufactured glasses with compositions similar to some nuclear waste glasses. An amphibolite rock source for vitreous material, found at Broborg in Sweden, shows a molten top portion.

ian. These natural glasses range from hundreds of millions of years old (such as terrestrial, meteoritic, and lunar glasses) to only thousands of years old (some basaltic and rhyolitic glasses).

Tektites and rhyolitic glasses generally have high SiO₂ concentrations (70–75 mass%), whereas basaltic glasses have lower SiO₂ content (45–50 mass%). Unlike most nuclear waste glasses, natural glasses tend to contain low mass percentages of alkali elements. This low alkali content has made application of the alteration data collected on these natural glasses to most alkali-rich waste glasses difficult. However, even with these limitations, basaltic glasses have been used as fruitful analogues to some HLW glass formulations.¹¹

Roman glasses

An excellent example of using anthropogenic glass analogues for modeling glass alterations can be found in the recent works of Verney-Carron and the Commissariat à l'Énergie Atomique (CEA) on fractured, ~1,800-year-old glass blocks excavated in 2003 from a Roman shipwreck near the French island of Embiez.¹² Many glasses—including nuclear waste glasses—fracture on cooling, and these fractures increase the reactive surface area of the glass. Because of the slow flow of water in and out of some cracks, increased surface area results in alteration rates in the cracks different from those on the exterior glass

surfaces. The effects of these cracks on long-term alteration rates have been difficult to replicate in short-term laboratory tests because of the slow rate at which borosilicate glass alters.

In addition to showing that alteration mechanisms of archeological glass are similar to those described by a modern glass alteration model, the authors also discovered that alteration kinetics slightly varied depending on fracture location. They calculated a lower alteration rate for internal cracks that were not continually exposed to water as compared with exterior surfaces that were most likely in constant contact with water. Without renewal of fluid into cracks, the altering solution became saturated with dissolved glass elements, making it unfavorable for the glass matrix to undergo further dissolution.

In a recent follow-up to this study, Verney-Carron et al.¹³ used chemical modeling to simulate alteration of the glass in a variety of solutions, and they compared the results with reactive transport modeling of alterations in the cracks. They calculated that cracks inside the archaeological blocks had one to two orders of magnitude less alteration than the external surface because of strong coupling between rate of glass alteration and slow renewal of the internal crack altering solution. Additionally, narrow internal cracks precipitated crystals because of supersaturation of the solution in the crack with silicon, calcium, and aluminum. Formation of these crystals plugged the cracks, causing flow of water and alteration processes to halt.

On this side of the Atlantic, glass scientists at PNNL have been working with Italian archeologists to investigate the formation of alteration layers on second- to third-century CE Roman glass recovered from the *Iulia Felix* shipwreck.¹⁴ The *Iulia Felix* wreck is of particular interest, because its cargo included blue-green Roman glass fragments (Figure 3)—referred to in archeology literature as “naturally colored” glasses—and colorless and red shards, “artificially colored” by additions of reduced copper. Because ancient glassmakers usually added coloring agents in measured amounts to colorless glasses, artificially colored glass usually has a base composition similar to naturally colored Roman glasses. This slight modification makes it possible to investigate the effect of chemical composition on glass alteration within a set



Credit: Denis Strachan, Joe Ryan

Figure 3. Fragment of a Roman glass cup handle found in the *Iulia Felix* shipwreck, showing iridescent alteration layers.

environment. Further research is currently underway on these artifacts.

PNNL researchers have also focused on parts of these glasses that were encased in cemented sediments.¹⁴ The cementing material around the glass is magnesium-rich calcite, likely formed through corrosion of the glass in seawater saturated with naturally occurring dolomite and magnesium silicate precipitations. These magnesium silicate species form during alteration of some HLW glasses, and researchers identified similar species in the altering solution of nuclear waste glasses that were subjected to corrosion with a 1 mmol/kg MgCl₂ solution.^{15,16} Both cited experiments were short term, but, when coupled with results from the *Iulia Felix* glasses, the researchers determined that, in undersaturated conditions, dissolution of the glass matrix is the main cause of formation of cementing phases. Burial materials in contact with glass are important in determining alteration conditions, and, if possible, fragments should be collected with sediments in place (Figure 4).

Iron slag

Many scientists have gained insight from other ancient vitreous materials in addition to glasses. A few studies in recent years have focused on alteration of metallurgical slags—glassy materials produced when metals are smelted from ore.¹⁷ Slag is comprised of a mixture of metal oxides, typically calcium oxide, and silica. Iron blast furnace slags, for instance, are formed from silica in the iron ore and added limestone or a similar calcium source. Similar vitreous materials may form when clays from furnaces are heated to high temperatures when processing iron (Figure 5). Aged slags produced during ironworking represent an

Ensuring longevity: Ancient glasses help predict durability of vitrified nuclear waste



Credit: Denis Strachan

Figure 4. Glass collected in-situ with contacted soil at the Aquileia site in Italy. Aquileia was a major glass-producing center in Roman times.

alteration condition in which silica-rich materials are altered in the presence of metallic iron. This situation is similar to what may happen to nuclear waste glass as it ages in stainless-steel containers.

Researchers identified such altered slags from the 16th century CE and recovered from an excavated blast furnace site located in Normandy, France. Geological conditions of the burial (saturated clay) are similar to those described for disposal of some waste glasses in France. French scientists at the CEA have studied these samples using short-term wet laboratory experiments as well as analysis of long-term alteration layers on the artifact surfaces.¹⁷

Researchers designed wet laboratory experiments to closely reproduce the proposed French nuclear waste disposal settings. They covered SON68, a non-radioactive version of a French HLW glass, with an iron-steel composite and injected it with a synthetic solution at a flow rate chosen to test the glass under diffusion-controlled conditions. The researchers also used this alteration routine on synthetic archeological slags that had the same composition as excavated samples. They determined significant similarities in the increase of glass alteration in the presence of iron from comparison of the two altered samples. This result supports previous studies that have shown a deleterious effect of reduced iron, which results in an increased rate of glass dissolution, an important result that researchers can use to aid modeling of long-term durability of glasses stored in iron-based containers.¹⁸

Swedish hillfort glasses

Researchers recently have identified glasses that were produced almost 1,500

years ago on a hillfort in Sweden that have been altered by surface atmospheric conditions, which have analogies to the near-surface disposal planned for some low-level nuclear waste glasses, including those at Hanford.¹⁹ A hillfort is a building structure used as a fortified refuge, trading post, or defended settlement, where an elevated location relative to the surrounding area provides a defensive advantage.²⁰ There are many hillforts in Europe, and these fortifications usually follow the contours of a hill, consist of one or more lines of earthworks (including stockades or defensive walls), and are surrounded by external ditches. More than 100 of the European hillforts, most from the Iron Age, are vitrified, and much controversy continues to surround how this occurred (see sidebar).²⁰

A selection of glasses from the Broborg hillfort site in Sweden contain iron at concentrations between those detected in basaltic and Roman glasses. Similar to basaltic glasses, some of the Broborg glass samples contain embedded crystalline phases, such as magnetite and other spinels. The current acceptance criteria for Hanford LAW glasses allow up to a few vol% of spinel crystals. The hillfort vitrified material remains exposed today to surface weathering cycles or, in some cases, is shallowly buried by local soil.⁷ Although research on these ancient vitreous materials remains in its early stages, WSU researchers have made promising headway in an attempt to synthetically reproduce these glasses. In addition, PNNL researchers have been working with Swedish archeologists, scientists, and state officials to acquire fresh near-surface altered samples from the Broborg site.

Medieval glasses

It is sometimes helpful to analyze a glass that has been altered under two or more conditions simultaneously. In these cases, the focus of understanding is placed on whether a glass alteration model can be extrapolated to include multiple altering conditions. Alteration data from several environments can be extremely helpful when attempting to



Credit: Jamie Weaver

Figure 5. Scientific collaboration at work in Uppsala, Sweden, showing investigation of vitrified iron furnace walls. Shown (left to right) are John McCloy, glass scientist of Washington State University; Eva Hjörthner-Holder, archaeometallurgist in Arkeologerna, Geoarkeologiskt Laboratorium; and Rolf Sjöblom, geochemist with Tekedo and Luleå University of Technology.

decide what disposal setting and geology is best for a specific type of waste glass. Researchers have discovered medieval stained and nonstained glass windows to be ideal analogues for these studies, because the windows often have undergone alteration under two or more settings²¹—glass window panels facing outward are exposed to an alteration environment different from those facing inward. A few rare glass samples that have been found in architectural structures have also been excavated from groundwater-saturated soils,²¹ thus providing a third environment to compare alteration data.

Because builders, craftsmen, and church officials kept excellent records, medieval glass-manufacturing methods of most stained glass windows are well-known. Therefore, glass scientists can identify conditions under which the glasses were made. In most cases, craftsmen made medieval glasses in either soda (sodium-rich) or potash (potassium-rich) compositions through melts of a blend of washed siliceous sand and a flux.²² The flux material was either plant-based (beech or fern ash) or mineral-based (natron) and, when added to other glass-forming materials, lowered the melting temperature.

Glassmakers achieved color either by adding metals or oxides of cobalt, chromium, manganese, copper, or iron and controlling redox conditions of the melt, or by applying a thin layer of a previously colored glass to the surface of a clear glass. Minor components in these medieval

glasses are thus transition metals that are relevant to some nuclear waste glasses. Chemical durability different from the sodium-rich versus potassium-rich glasses as well as presence of multiple compositions with various transition metals provide a large dataset for studying composition-dependent alterations as well.

Scanning electron microscopy studies on medieval stained glasses from England, Italy, and France have revealed that the type of weathering has an effect on thickness and structure of the alteration layer(s). Potash glasses exposed to an exterior environment produce thick, uneven alteration layers with some pitting, along with pervasive micro-cracking.²³ In contrast, potash glasses that have been buried and exposed to groundwater have nearly even alteration layers with little to no cracking.

Potential future glasses for study

There are many less-well-studied ancient glasses from around the world that deviate significantly in composition from the glasses described above. These glasses could provide researchers with new insights into the effects of compositional and environmental differences on glass alteration. These include high-alumina glasses from India and some regions in Turkey, a special subset of Byzantine glasses that contain a moderate level of alumina plus some boron,²⁴ and certain Chinese glasses with high barium oxide content. Researchers currently are considering some of these glasses as possible analogues, although their rarity makes them challenging to acquire for study.

Researchers also need to identify high-concentration boron (~10 mass% B₂O₃) aluminosilicate glasses that have been altered in waste-disposal-relevant environments. Boron is a primary component in all waste glasses and has increased, in certain cases, the durability of silicate glasses.²⁵

Boron made its mark on the glass industry in the early 1900s, when Corning patented a series of boron-containing silicate glasses that could withstand temperature shock—Pyrex.²⁶ At this time, there were many recorded accidents related to breakage of hot glass railway lanterns during cold rainstorms. Glass breakage caused the railway signals to fail, catching innocent people unaware of oncoming trains. Replacing soda in the

glass with boron oxide solved the glasses' thermal shock problem.

Today, we find borosilicate glasses in almost every home, office space, and laboratory. In addition, commercial uses have utilized the poor durability of some boron-containing glasses. One example is Vycor, in which a phase-separated sodium borosilicate glass is selectively leached of its sodium borate phase, leaving a skeletal silicate network that is usually further consolidated to produce a low-cost nearly pure silica. Clearly, chemical durability concerns are relevant to ancient materials and to today's and likely tomorrow's commercial glass processing.

Challenges remain

The studies reviewed above are only a portion of those conducted to date on ancient glasses.^{27,28} However, their relevance to the validation of glass alteration models is significant. There are many challenges associated with analyzing ancient glasses for this purpose. However, the payoff of having a composition- and environment-dependent model of glass alteration validated by many data points, spanning days to millions of years, will be momentous. A better understanding of long-term glass alteration will allow for a more accurate prediction of the long-term performance of vitrified nuclear waste and could aid continued formulation of waste glasses that will be durable for "thousands of years".² In the process, knowledge gained on glass alteration can help create industrial glasses with engineered durability as well as help museums and historical societies in the preservation of cultural heritage objects.

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Challenges in assessing the mechanical behavior of coatings on architectural glass

Glass coatings can reduce building energy demand, but thorough understanding of the mechanical properties of these multilayer coatings is needed.

By Steve J. Bull

One of the most significant uses of glass is in architecture—building applications accounted for ~70% of worldwide glass sales in 2012. If we consider that ~40% of the world's energy demand is used to heat, cool, and light buildings, then glass coatings become a significant source of potential energy savings. Suitable glazings can reduce energy demand, helping keep buildings warm in cold climates and cool in hot climates, while allowing transmission of light. For this reason, demand for energy-efficient glazings is increasing.

Glass is mostly opaque to ultraviolet light, but transparent to visible and infrared light. For energy efficiency, glass needs to decrease infrared transmission without compromising visible light transmission. Low emissivity or energy-efficient glazings usually are based on a thin, transparent conducting layer, which may be a single layer or part of a multilayer stack with surrounding antireflection and barrier coatings. The main design factor is optical performance of the coating, but mechanical damage, particularly caused by transport or storage, also is a consideration. In most cases, a supplier produces the coatings, and the supplier delivers the coated product to a fabricator who cuts and assembles the glass into windows. Coated glass may be rejected if it is damaged in transit and if the damage affects optical performance. Therefore, an understanding of the mechanical response of multilayer coatings on glass is essential to reduce losses from damage.

Capsule summary

POTENTIAL ENERGY SAVINGS

Architectural applications account for a large percentage of worldwide glass use, making glass coatings that increase energy efficiency a rich potential source of reductions in world energy use.

THE CHALLENGE

Although demand for energy-efficient glass glazings is rising, measuring the properties of thin films on glass poses significant challenges—yet this information is critical to improve these potentially high-impact coatings.

RESOLUTION

Laboratory simulation and careful modeling approaches can replicate the properties and behavior of thin-film coated glazings and will help researchers develop more robust coatings to improve energy-efficient glass windows.

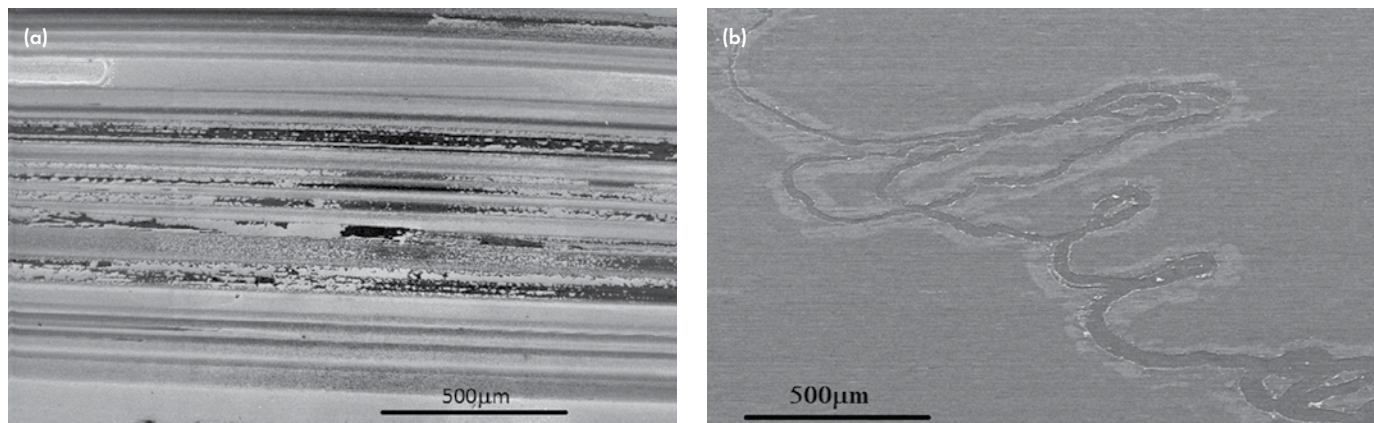


Figure 1. Transit scratches formed on coated float-glass during delivery. (a) An on-line CVD fluorine-doped tin oxide coating. (b) An off-line multilayer PVD coating based on a silver layer surrounded by ZnO antireflection and TiO_xN_y barrier coatings.

Coatings on architectural glass for energy efficiency are generally of two types:¹

- On-line coatings are coatings produced on the float-glass line, generally by chemical vapor deposition (CVD) at atmospheric pressure. Such coatings consist of a conducting oxide layer, such as fluorine-doped tin oxide, that is a few hundred nanometers thick. CVD creates a rough coating that, depending on the material selection, can be hard and difficult to damage. These coatings, therefore, can be used on the external facing panels of windows and are widely used in domestic dwellings. However, on-line coatings provide only moderate optical performance and confer limited energy savings.

- Off-line coatings are coatings generally produced by physical vapor deposition (PVD) in a separate production facility from the float-glass line. Coatings consist of multiple layers, with a total thickness of ~100 nm, deposited on standard-sized flat-glass panels (often 3 m × 2 m). Typically, the active layer is a 10-nm-thick layer of metal (silver in the U.K.), which is transparent at this thickness. Thin oxide layers, such as tin oxide and zinc oxide, surround the layer and act as antireflection coatings. Silver produces a layer of mechanical weakness in the coating stack. Therefore, off-line coatings often have an external protective layer to reduce stresses that

result from handling. These coatings also require a barrier layer to separate the antireflection coatings from the glass substrate. This layer often is based on titanium oxide or silicon nitride. Off-line coatings are not as mechanically robust as on-line coatings, even though they can be very optically efficient, but are the choice for large, substantially glass-fronted commercial buildings.

Off-line coatings often are placed on the inside of a double glazing unit. Therefore, once assembled, the propensity for damage is reduced to virtually zero. The key issue, therefore, is damage introduced during delivery. Figure 1 illustrates the main damage mechanisms that result during delivery for both coatings. A black deposit clearly is visible on the surface of on-line coatings, obscuring visibility. The off-line coating shows a well-defined but random transit scratch, where the silver layer and all subsequent layers in the coating stack are stripped. Therefore, a clear understanding of the delivery process is necessary to understand how such failures are generated and how they may be reduced.

Both types of coated glass are delivered as large panels on the back of a lorry, and cut-glass panels are loaded almost vertically onto a steel frame called a stillage (Figure 2a).

Glass handlers attempt to reduce

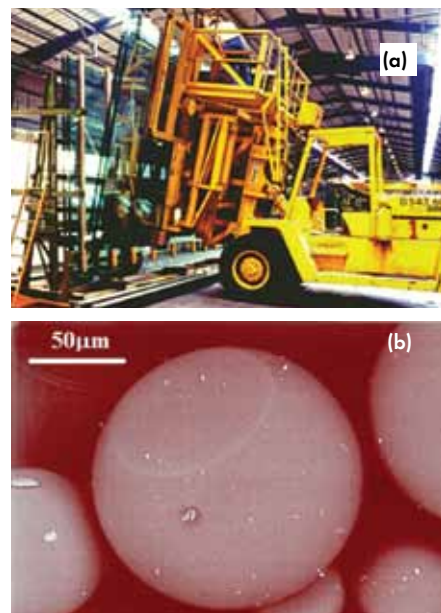


Figure 2. (a) Stillage for transport of glass. (b) Typical interlevant particles used to separate glass sheets

the amount of glass and coating debris released during cutting, but it is not always possible to completely remove debris. However, loading multiple panels together creates glass to coating contact. Therefore, to reduce contact damage, glass technicians spray a uniform layer of interlevant material on the surface to separate the glass and minimize contact stresses. Interlevant consists of poly(methyl methacrylate) (PMMA) spheres with a diameter of 70 µm

(Figure 2b), which are bigger than glass debris. In addition to interlevant, glass handlers often stack about 10 glass sheets together, separating sheets with cardboard spacers, and tightly wrap them to restrict movement. During delivery, glass plates may be agitated by acceleration and deceleration on the road—exacerbated by poor road conditions—and can move short distances with respect to each other. This movement creates transit scratches. However, often it is not clear whether such scratches are caused by interlevant particles, glass debris, or both. Therefore, laboratory simulation of the failure is needed to identify causes of such scratches.

Simulation of transit scratches

We can simulate failure using a controlled number of interlevant spheres trapped between a small flat-glass slider and a larger off-line coated glass plate.² We use estimates of interlevant density and weight and angle of repose of the glass plates in a typical stillage to determine that the load on a single interlevant sphere is ~ 7 mN, a value matched in laboratory tests. Experiments with pure interlevant or with small sharp glass debris particles (~ 10 μm in diameter) spread on the surface show that glass cullet is not necessary to form transit scratches that match those observed in commercial failures.

The mechanism of failure, therefore, depends on sliding of interlevant particles. In service these are loaded elastically on the surface, where they significantly deform. During sliding, damage occurs either in the sphere or in the coating, depending on coating properties.

- In the case of on-line coatings, the rough surface of the CVD coating critically dictates performance. Polymer spheres compress against the coating, and roughness peaks penetrate into the spheres—this is exacerbated by viscoelastic deformation of spheres such that the true area of contact increases with time until it approaches the apparent area of contact. During sliding, shear failure occurs in the polymer sphere, and a layer of polymer is transferred to the coating surface, causing the

black deposit in Figure 1(a).

- In the case of off-line coatings, the surface is much smoother and failures are generated in the coating during sliding. Lateral movement generates compressive stresses at the leading edge of the contact and tensile stresses at the rear edge because of friction. This generates through-thickness cracks at the rear of the contact, which are diverted along the weak interface between the silver layer and its underlying antireflection coating (in this case, zinc oxide). The effect is that the sphere drags the coating on top of the silver layer forward, stripping the coating from the surface as the sphere moves forward. This produces a transit scratch similar to that shown in Figure 1(b).

Adhesion failure in off-line-coated glass is a significant factor for rejection of coated glass after delivery. Therefore, it is important to determine the effect of coating designs (materials, thickness, layer stacking order, etc.) and process routes on performance. This involves modeling the stresses responsible for failure and how these are relaxed by fracture or plasticity. Stresses in coatings are a combination of residual stresses generated during deposition and stresses generated during sliding of a polymer sphere against the surface, which depends on elastic properties of the coatings and coefficient of friction between the sphere and the topmost coating layer.

Fracture behavior of coatings depends on defect distribution and fracture toughness of individual layers. If we know these properties, we can assess the likelihood of failure using finite-element modeling of the sliding contact. A major challenge for measurement is that these coatings are only ~ 10 nm thick and may exhibit size effects in deformation response.

Measurement of key properties of multilayer optical coatings

Residual stress

We can use various methods, including curvature and X-ray diffraction, to measure residual stress in a coating.³

The latter matter is well established, and the $\sin^2\psi$ method has been used widely in assessment of thin hard coatings. However, we cannot apply X-ray diffraction to most of the layers tested here, because they are not crystalline. One exception is the zinc oxide layer beneath the active silver layer, which shows some crystallinity. We measured a compressive residual stress of ~ 1 GPa of this layer at room temperature after deposition, a value that combines stresses generated during deposition at the growth temperature ($\sim 200^\circ\text{C}$) and thermal expansion mismatch stresses generated during subsequent cooling. However, the temperature of the glass substrate increases during deposition. Therefore, the contribution of thermal expansion mismatch stress to the final measured stress increases with coating thickness.⁴

We measure residual stress in amorphous coatings by change in curvature of the coating/substrate system after deposition using the Stoney equation.³ In this case, we need only coating thickness and substrate thickness and its elastic properties as well as change in radius of curvature of the sample before and after coating deposition. We measured changes in curvature with deposition of very thin coatings on thin glass-plate substrates (100- μm -thick). Figure 3 shows typical changes in residual stress as a function of coating thickness.⁴ Changes in thermal expansion mismatch stresses and relaxation of stresses by viscous processes in amorphous layers contribute to changes in residual stress measured at room temperature as coating thickness increases.

Substrate properties

We can use conventional mechanical testing approaches to measure easily bulk mechanical properties of glass, and the Young's modulus and Poisson's ratios needed for modeling are available. However, in the case of float-glass, surface properties that might be needed to model scratch failures are not necessarily the same as these bulk properties. Indeed, we often measured different properties for opposite sides of a float-glass sheet—one was in contact with molten tin during manufacture, while the other was in contact with air.⁵

It is conventional to apply coatings to the air-side of the float-glass for best adhesion. Therefore, we need to measure the properties of this side using indentation tests to generate surface specific data. We can determine hardness, elastic modulus, and fracture toughness from such indentation tests. However, properties of glass in the near-surface region can show some variation because of compositional changes, such as leaching, tin uptake, and tempering effects. Therefore, we should consider this variation to extract good data for modeling.⁵

Coating properties

Elasticity and plasticity

Conventional mechanical testing does not allow measurement of the properties of individual coating layers in an optical coating, because the materials do not exist in a form large enough to make a suitable test piece. Further, coating microstructure often is different from that of the comparable bulk material. Therefore, measured properties are different. For this reason, we often use instrumented indentation (nanoindentation) tests to measure such coatings.⁶

For thicker on-line coatings (~300 nm), we can easily measure coating properties independent of the substrate. As a rule-of-thumb, penetration depth must be less than 10% of the coating thickness, and we must produce an elastic-plastic indentation to measure coating hardness only for hard coatings on a softer substrate.⁷ Limiting depth for determination of elastic properties is much lower, and we usually determine elastic properties of the coating (contact modulus) by extrapolation of contact modulus variation with contact to zero depth, as outlined in ISO14577 ("Metallic materials—Instrumented indentation test for hardness and materials parameters," International Organization for Standardization, Geneva, Switzerland).

Practically, we cannot measure the hardness of a coating that is less than 200 nm thick, independent of its substrate, given the sharpness of commercially available nanoindenter tips. Even if we use the best possible tips, this thickness is reduced only to 50 nm, which is much larger than the great-

est thickness usually used in solar control coating designs. Therefore, with such materials, we most often measure a composite response from substrate and coating. In some cases, we may model deformation-response from the coating substrate system to extract coating properties. However, in most cases, similar to the ISO standard, we can apply an extrapolation approach if we can show it to be valid (i.e., the analysis uses sufficient indents where plastic deformation of the coating occurs).

We also are concerned that the properties of the coating may be scale sensitive and, therefore, show some variation with thickness. To assess this, we can deposit coatings to a range of thicknesses and measure their properties using nanoindentation with an appropriate modeling or extrapolation approach that accounts for the effect of the substrate. For coatings in a multilayer stack, we know that it is important to deposit the same layers underneath the coating as might be used in the intended application, so that coating microstructure remains the same—variation in coating microstructure at different thicknesses is much smaller than variation produced by substrate changes.

In general, thickness does not vary the hardness of the oxide coatings used in energy-efficient glazings, because these are predominantly amorphous and a microstructural length scale (e.g., grain size) does not affect their deformation. One exception is the crystalline zinc oxide coating, which shows a pronounced indentation size effect—hardness increases as coating thickness decreases.⁴ We need to consider this in cases where plastic deformation occurs (e.g., indentation by a sharp indenter). However, this is not an issue for model-

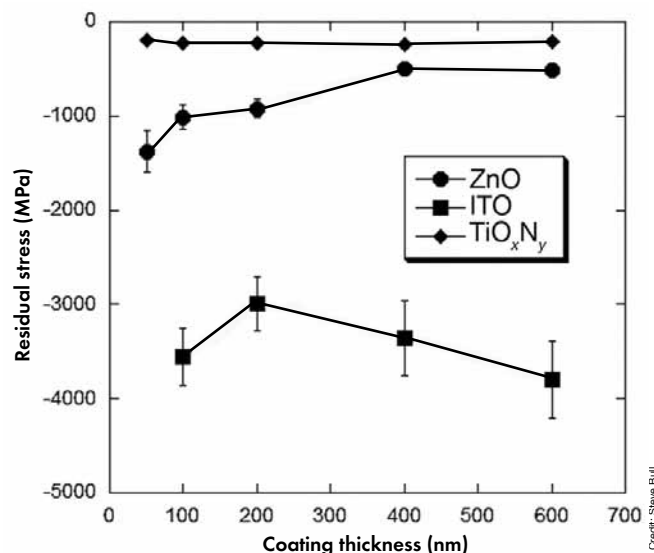


Figure 3. Variation of residual stress with coating thickness for several PVD oxides used in multilayer solar control coatings.

ing of transit scratches made by a polymer sphere.

In the case of elastic properties, there is a slight reduction in modulus as thickness is reduced in data extrapolated to zero depth, but modeling shows this represents an increasing contribution from the elastic properties of the substrate in thin-coating tests, which cannot be corrected for by extrapolation.⁴ In this case, we can use a modeling approach to show that coating elastic properties do not vary with thickness.⁸ For finite-element modeling, we can use determined elastic properties over a wide range of coating thicknesses without introducing significant errors.

Fracture toughness

All optical coatings on glass are too thin to assess by conventional mechanical tests and indentation fracture toughness tests widely used to assess bulk ceramics, because these methods require well-developed starter cracks or produce final crack sizes that are ~10 μm in length. Nanoindentation tests of thin oxide coatings on glass produce cracks, but they have a different geometry (Figure 4). With sharp indenters, or at least indenters with edges that remain sharp, the main failure mode is radial cracking, which follows indenter edges and is caused by bending of the coating around the indenter during loading. These cracks are constrained to lie

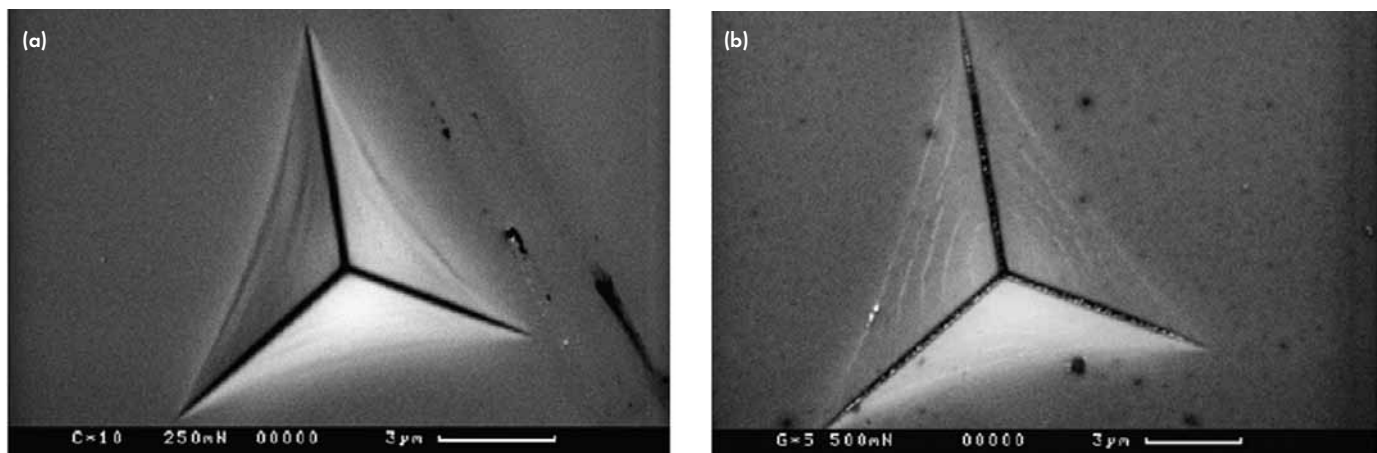


Figure 4. Cracks associated with nanoindentations in a solar control coating on glass: (a) radial cracks; and (b) radial and picture frame cracks.

within the impression, and they generally penetrate through the whole coating thickness but do not propagate into the glass substrate.

In cases where the indenter is less sharp (because of worn edges), picture frame cracking—which forms at the impression edge as the coating is bent into the dent formed in the glass—is predominant. If we want to use these cracks for toughness assessment, we must measure fracture energies from the energy dissipated during the indentation cycle.^{9–11} The area beneath the force displacement curve, or the work of indentation, represents energy dissipated in elastic, plastic, and fracture processes. When fracture occurs—either through-thickness cracking or adhesion failure—the load displacement curve can be discontinuous if the fracture event is large enough. In this case, we can estimate energy of fracture by subtracting the work of elastic plastic indentation from the total work of indentation before and after fracture.⁹

We can use other approaches when no discontinuity is observed.¹⁰ We can convert these energies to critical strain energy release rate (G_c) values using experimentally measured crack areas or to fracture toughness values using measured coating elastic moduli.⁹ There is no change in measured toughness as a function of coating thickness for any coating, which means that the fracture process is controlled by defects in the coating rather than bulk properties. Loading rate¹² and multiple loading cycles¹³ also affect the observed fracture behavior.

If we estimate failure stresses from finite-element analysis of nanoindentation tests at the onset of fracture, we find that the critical crack size responsible for failure is ~ 10 nm, which is comparable with the peak-to-valley roughness of the coating in the vicinity of the indentation. Therefore, the origin of the observed cracks probably is surface (or interface) defects that result because of surface roughness. Therefore, decreased surface roughness of the as-deposited coating increases its resistance to cracking.

Friction coefficient

We can use scratch tests to determine the friction behavior of coated glass with a range of different styli. We can conduct many tests with scratch diamonds and give low friction coefficients (<0.2), which increase slightly with load as plastic ploughing becomes more important. Coating chipping and detachment further increase friction at higher loads. However, plastic behavior of the substrate dominates generation of scratch tracks, and friction values are not representative of those that cause failure during glass sheet delivery. We have achieved experimental assessment of the coefficient of friction between a PMMA sphere and coating surface by gluing a single sphere to a nanoindenter shaft and performing a scratch at a 7-mN load to match what is expected in service.

The friction coefficient of a range of coatings against PMMA is ~ 0.6 , but it increases slightly as coating roughness increases.⁴ Thus, we need to deposit

smooth and dense coatings to decrease the chance of failure. Friction coefficient decreases with time if the coated surface is left in air for up to 24 h, after which we routinely measure values of ~ 0.3 . Thus, we suggest that it is good practice to let coated glass contaminate by air exposure before being packed for delivery.

Adhesion

We can assess adhesion of coating layers by indentation and scratch tests, but results often are semiquantitative and not useful for modeling. In some cases, drops in the load–displacement curve obtained during nanoindentation with displacement control are caused by interfacial detachment. In that case, we can use the same approach as for fracture toughness assessment to determine interfacial detachment energy and toughness.¹⁴ Alternatively, we can use wedge tests to measure detachment energies.¹⁵ In general, oxide layers adhere well to other oxide layers in a multilayer stack. Therefore, it is difficult to generate such failures.

However, in some cases, high residual stress in the coating, such as in sputtered indium tin oxide on glass, may cause spontaneous adhesion failure by buckling. In that case, we can determine adhesion energy from residual stress, coating properties, and buckle geometry.¹⁴ The poorest adhesion generally is produced when a thin metallic layer is part of the coating. In such cases, all of these methods give reasonable values for interfacial energy and are comparable to those determined from theoretical calculations.¹⁶

Modeling of coating failure

With good values for elastic and plastic properties, friction, residual stress, and adhesion energy, we can analyze the sequence of fracture around a sharp indentation or in a transit scratch and assess the effects of various coating geometries on the observed coating failures. For instance, Figure 5 shows the results of finite-element modeling of a zinc oxide/silver/zinc oxide coating stack on glass undergoing a nanoindentation test. In this model, we use cohesive zone elements on the upper and lower faces of the coating to identify failure locations.¹⁷ We parameterized these cohesive zone elements with the previously determined experimental fracture energies and fracture stresses. This shows that interfacial failure initially occurs on the upper surface of the silver layer during loading, but, on subsequent unloading, another larger interfacial crack opens up on the lower surface of the silver layer—indeed, this is what we observed in experiments.

Plastic deformation of the silver layer under the indenter causes the layer to squeeze out from the contact on loading. Accumulation of this extra material acts as a wedge to open up the crack on the top of the silver layer. On unloading, elastic stresses around the plastic zone surrounding the indenter relax and create tensile stresses at the edge of the plastically deformed zone. These open up cracks at the bottom of the silver layer, and these cracks are similar to lateral cracks observed in conventional indentation testing of bulk ceramic materials. We can develop similar models for transit scratches.

The key factor in failure of these solar control coatings is relatively poor mechanical strength of the silver layer. Increasing yield strength of the silver eventually stops adhesion failure, and the failure locus then is moved to the glass-barrier layer interface. However, generating such a high hardness metal layer requires incorporation of impurities or reduction in grain sizes, which usually compromises solar control performance of the coating by reducing conductivity of the metal layer.

Modeling approaches the answer?

Designers now widely use large-area-coated architectural glass for energy-efficiency applications. Off-line PVD coatings—which are multilayer coating stacks containing a thin metal layer surrounded by oxide or nitride antireflection and barrier layers—achieve the best performance. Mechanical weakness of the metal layer makes it a locus for failure

in these coatings. This failure tends to occur during delivery, because once the coated glass is assembled into double glazing units, coatings are on the inside and are protected from further damage.

Laboratory simulation of damage mechanisms allows development of a mechanical model of the failure mode, which can be used to optimize coating designs for mechanical resistance, provided that good material data is available for the model. To generate this has required development of several combined experimental and modeling approaches to extract the properties of a single coating layer from tests on the coating-substrate system.

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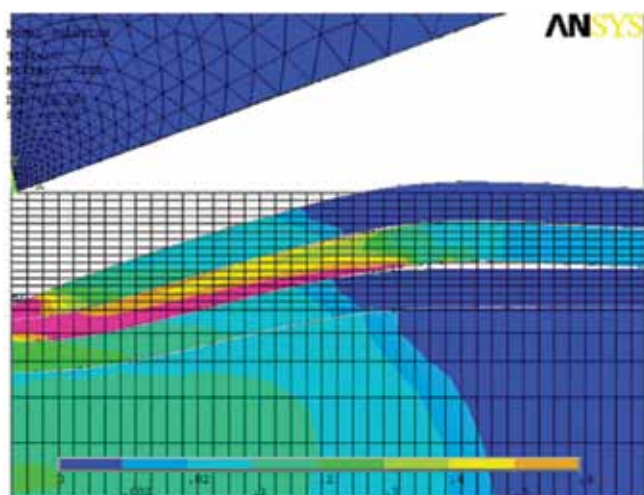


Figure 5. Finite-element model of a nanoindentation test in a ZnO/Ag/ZnO coating on glass, in which cohesive zone elements have been used to model adhesion failure.

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Appliance science:

Low-fire enamels for new preprimed steel

By Karine Sarrazy, Alain Aronica, Angelique Leseur, and Charles Baldwin

Glass-metal interfaces impact the thermal performance of household machines.

Energy reduction continues to be a key megatrend in the enamel industry as well as in the broader ceramics and consumer communities.¹ The current state-of-the-art for industrial enameling is dry electrostatic powder application in a highly automated process over cleaned-only low-carbon steel. This process uses a single coat for ground coating or a two-coat/one-fire application for cover coating. Typical firing temperatures are 810°C–850°C for 90 s at peak metal temperature to fuse the glass coating to the substrate to fully develop required properties, such as adhesion, corrosion resistance, thermomechanical resistance, and color.

The goal is to decrease firing temperature to reduce energy requirements and to allow use of thinner-gage steel. Even if a decrease of 20°C–40°C is already possible using specific preprimed steel,^{2,3} a decrease of 100°C is required for the gage reduction to maintain acceptable strength after fire, an important measure of integrity for household appliances. This technology is becoming a reality in the near future, because steel suppliers—such as ArcelorMittal (Luxembourg City, Luxembourg) and its R&D organization OCAS (Zelzate, Belgium)—are developing new steels to enable low-temperature enameling⁴ via application over a primer applied during steelmaking instead of over ground coat. Because ground coat requires the addition of ~0.5%–3% transition-metal oxide, particularly cobalt or nickel oxide, to adhere to cleaned-only steel, eliminating the need for ground coat removes this requirement and allows decreased firing temperatures.

Ferro has been collaborating with OCAS and ArcelorMittal to develop a new range of enamels for low-temperature firing at ~720°C to meet all typical industry requirements for bond and chemical and heat resistance for use in major appliances, architectural panels, or cookware. Additionally, these enamels will be more environmentally friendly, because they will not contain transition-metal oxides, such as cobalt or nickel, for bonding to steel.

We present here various types of enamels currently developed by Ferro, fired at 700°C–740°C, on precoated aluminized steels developed by OCAS. Development focuses on white cover coat for major appliances, architecture, or cookware as well as easy-to-clean (ETC) colored enamels for cavity walls of wall ovens and free-standing ranges. ETC enamels are acid-resistant ground coat enamels formulated to resist etching by baked-on acidic foodstuffs to facilitate cleanability.

Low-temperature white enamel

Titanium-opacified white enamels are usually fired at 800°C–820°C. These enamels use frits supersaturated with TiO₂, which precipitates anatase or rutile crystals during firing to provide a bright white color. Although this technology is well established, the challenge now is to develop a low-temperature alternative that fires at 700°C–740°C and can pass all tests requested by the standards that regulate architecture,⁵ appliance,^{6,7} and cookware exteriors. The enamel must be applied using a dry electrostatic process to align with the requirements of mass production.

To develop such a low-firing coating, we started with NPD787/F6, an alkali borosilicate TiO₂-based frit with a relatively low glass temperature (T_g) below 450°C. The frit was milled into powder and applied dry electrostatically over



Figure 1. White enamel NPD787/F6 applied over (left) enamel primer or (right) ground coat and fired at 700°C for 4 min.

preprimed aluminized steel supplied by OCAS. NPD787/F6 applied to enamel primer and fired at 700°C for 4 min showed no opacity because of a reaction between enamel and primer (Figure 1). However, NPD787/F6 provided a good finish and high opacity when applied over ground-coated steel and fired at 700°C for 4 min. Because of these test results, Ferro decided to continue development work over ground coat before testing over primer.

Table 1 compares test results of NPD787/F6 with a conventional high-temperature white cover coat. Measured properties include application weight (thickness), lab color, 60° gloss, and spot acid resistance. Spot acid resistance measures the enamel's resistance to staining

with a 10% aqueous solution of citric acid. The new coating formulation provided a smooth satin surface with good spot acid resistance and good hot-acid resistance with no discoloration and

no weight loss. We next attempted to make the white enamel glossier to match results obtained at 800°C.

Softening NPD787/F6 by increasing conventional fluxes (Na_2O , B_2O_3) or decreasing refractory oxides (TiO_2 , SiO_2) did not improve opacity. Additional laboratory reformulation resulted in two new glossier frits, NPD787/F18 and NPD787/F49, after firing at 700°C–740°C (Table 1). White enamel NPD787/49 also provided a smooth surface and good opacity on preprimed steel.

To confirm opacification of these new enamels after firing at 700°C, we used differential thermal analysis/thermogravimetric analysis (DTA/TGA) to characterize the crystallization process (Figure 2). Crystallization occurred at ~500°C

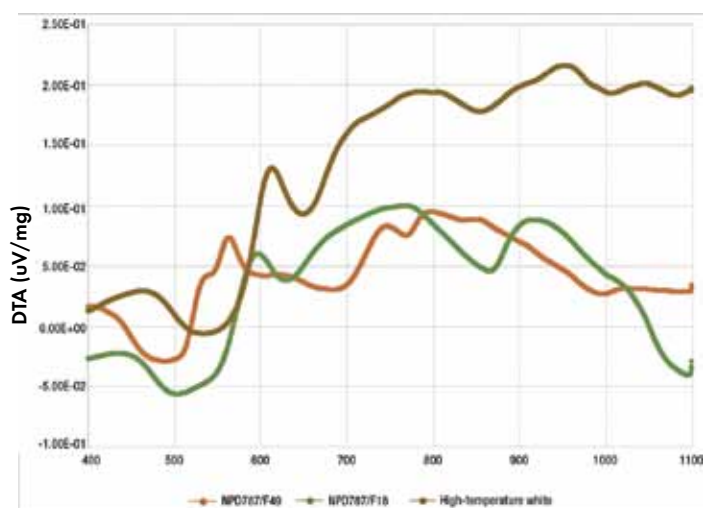


Figure 2. DTA curves of white enamel formulations.

for the new formulations—versus 550°C for conventional high-temperature white cover coat—because the new frits were softer with a lower T_g . The lower area of crystallization observed for NPD787/F49 would be expected to correspond to smaller crystals and lower opacity, because white

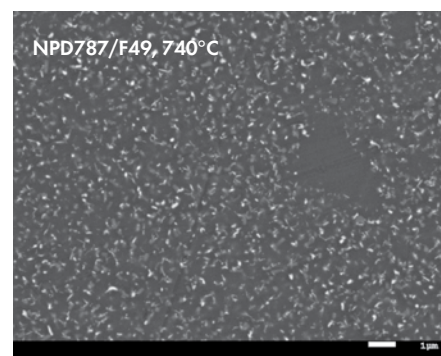
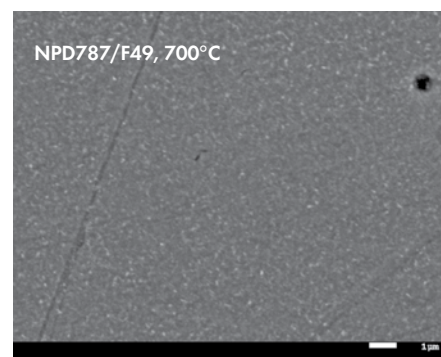
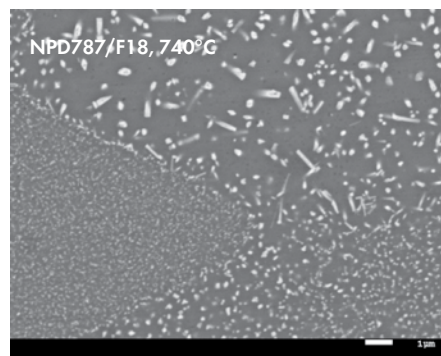
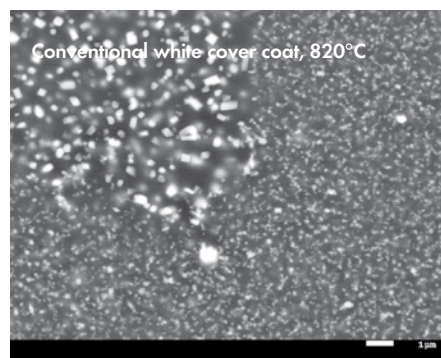


Figure 3. SEM micrographs of recrystallized titanium dioxide.

Capsule summary

POTENTIAL SAVINGS

Decreasing the firing temperature of appliance enamels represents a potential means to reduce energy use during production and permit materials savings through use of thinner-gage steel in appliances.

MATERIAL ADVANCES

Ferro is developing enamels that can be fired at lower temperatures and provide easier-to-clean surfaces, thanks to material advances in the enamel and steel industries.

PROMISING FUTURE

Initial tests of new white and easy-to-clean low-temperature appliance enamels demonstrate promising properties and, with further optimization, will lead to cost savings and improved enamel technology that is less prone to defects.

Table 1. Results with low-firing enamel NPD787/F6, NPD787/F18, and NPD787/F49

	Target 800°C	NPD787/F6			NPD787/F18		NPD787/F49	
		700°C for 4 min	700°C for 10 min	740°C for 4 min	700°C for 4 min	740°C for 4 min	700°C for 4 min	740°C for 4 min
Thickness (g/m ²)	300	220	220	220				
Color								
L (brightness/darkness)	92.8	82.4	90.8	89.7	90.2	90.0	75.5	85.2
a (green/red)	-1.1	-0.4	-1.40	-1.07	-2.3	-2.1	-3.0	-2.3
b (blue/yellow)	-2.4	-0.3	0.44	0.76	-4.9	-4.3	-12.1	-3.9
Gloss (60°)	90	50	45	62	87	104	83	70
Acid resistance [†]	AA	AA	AA	AA		AA	C	C

[†]Standard test method for acid resistance of porcelain enamels (citric acid spot test),[†] ASTM C282.

corresponds to optimal TiO₂ germination and crystal growth.

Further experiments used scanning electron microscopy (SEM) analysis to characterize low-temperature crystallization and compare it with that observed

at conventional higher temperatures. Figure 3 shows that crystallization of NPD787/F18 after firing at 740°C is similar to that observed for white cover coat fired at 820°C. Crystallization of NPD787/F49 increased from the 700°C to the 740°C firing, but crystals were smaller than for NPD787/F18. This formula also had reduced acid resistance, which could be caused by the smaller fraction of recrystallized TiO₂.

New trials are currently finalizing characterization of these three low-temperature white enamel references. Depending on chemical and physical properties, they will be defined as specific proposals for trials in architecture, appliance, or cookware markets.

test used on this family of enamels. The test consists of application and removal of three solutions: citric acid (10%), LiNO₃ (0.5 g), and ketchup (50%, mixed with water). Citric acid simulates acidic food-stuffs, such as pie filling or lemon juice. LiNO₃ is an industry test that simulates regular oven use. Ketchup consists of acetic acid and sugar, which are common components in most condiments, and is baked on at 320°C. In cleanability tests, we used a sponge and water to remove citric acid and LiNO₃, whereas we used a razor blade to remove baked-on ketchup solutions.

We first developed two frit formulations (ETC1 and ETC2) to attempt to improve enamel cleanability. Soft frit ETC1 was the starting point, from which removing metal oxides yielded frit ETC2. Although ETC2 was actually significantly harder than ETC1, ETC2 showed poor results in cleanability tests and exhibited severe staining. Adjusting the formulation of ETC2 to soften the metal oxide-free frit—such as decreasing refractories, increasing fluxes, increasing oxidizers, or increasing fluorine—did not improve cleanability.

The company further developed lab melts to evaluate the effects of added cobalt, copper, manganese, and iron oxides on hardness of the frit. Figure 5 shows the effects of these metal oxides on melt viscosity of the glass, as measured by isothermal fusion

flow tests (“Standard test methods for fusion flow of porcelain enamel frits (flow-button methods),” ASTM Designation C374. American Society for Testing and Materials, West Conshohocken, Pa.).

Figure 6 shows cleanability test results from these various frit

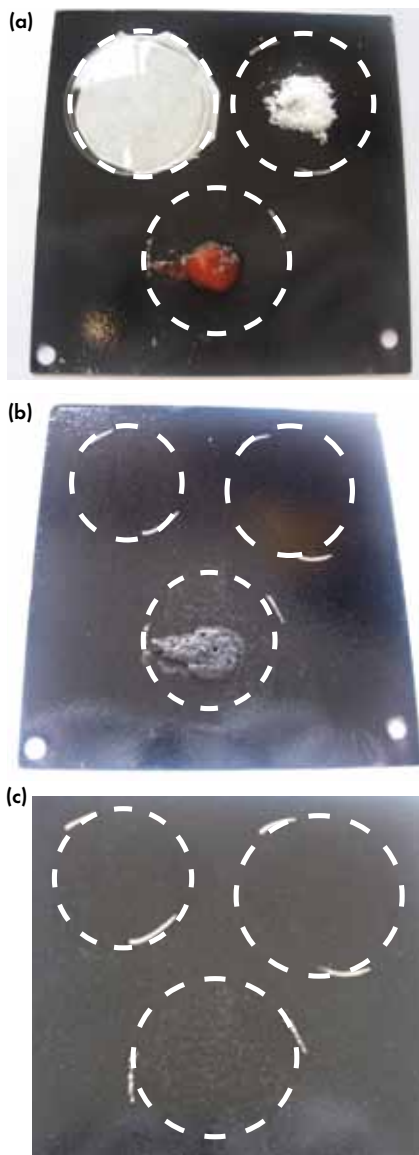


Figure 4. Typical cleanability test on ETC1. (a) The three test solutions before cooking. (b) Cooked ketchup test before cleaning. (c) Enamel surface after cleaning.

Low-temperature ETC enamel

The available color palette of ETC enamels is limited because transition-metal oxides, particularly cobalt and nickel oxides, are required to develop bonds on cleaned-only enameling grade steel. With the new approach of applying enamel over preprimed steel, it should be possible to develop uncolored base ETC enamels, which then could be colored with added pigments. This would enable a wider range of colors without changing the chemical properties of the enamel after firing at 700°C–740°C.

Figure 4 shows a typical cleanability

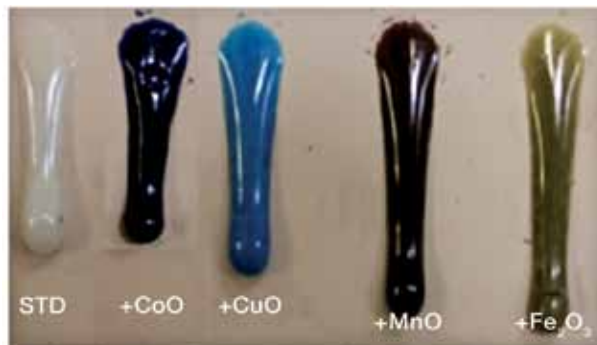


Figure 5. Fusion flow results on modified ETC frit.

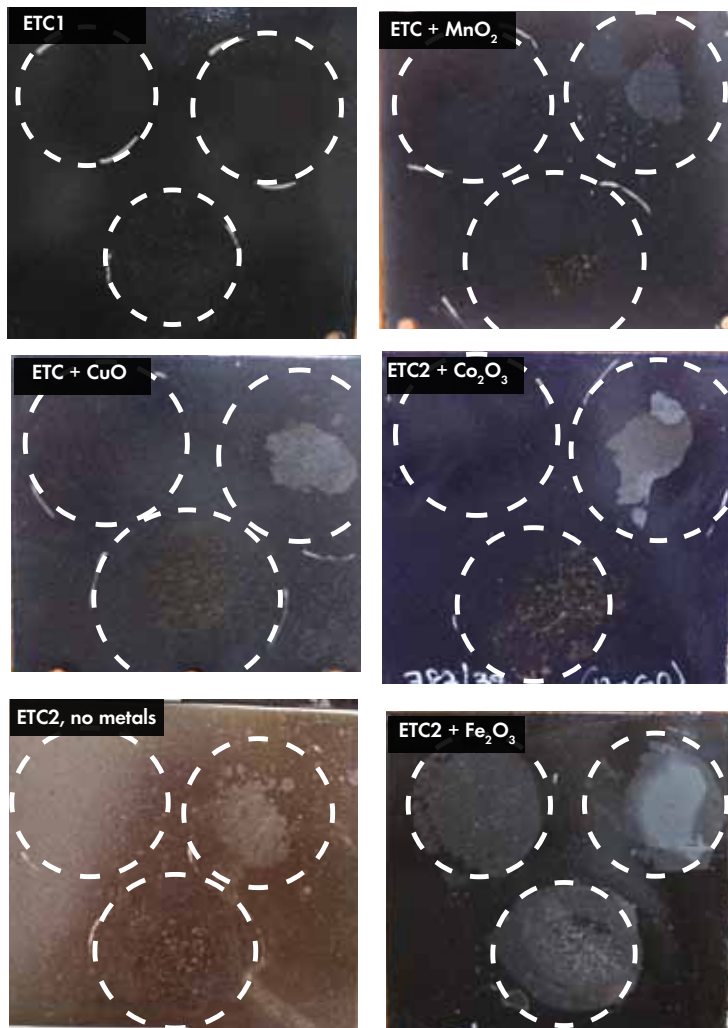


Figure 6. Cleanability test results for various ETC frit formulations.

formulations. The test reveals weak stain resistance of ETC2 compared with ETC1. Further, despite addition of Co_2O_3 , CuO , MnO_2 , and Fe_2O_3 to ETC2, only MnO_2 slightly improved surface cleanability.

We then used cross-sectional SEM to analyze the cleanability test areas exposed to LiNO_3 . Micrographs revealed that stained surface areas showed visible cracking (Figure 7). Therefore, to further improve the coating, we developed ETC3 by adding non-silicate glass formers.

ETC3 with or without metallic oxides showed good cleanability test results, with no cracks visible by SEM. ETC3 without metallic oxides is more robust terms of cleanability; however, the results also showed that in spite of good stain resistance, the coefficient of expansion of this new frit needs further optimization.

Initial steps completed

Ferro has completed initial steps to develop new white and ETC low-tem-

perature enamels with good properties developed by OCAS. This new enamelling process will lead to cost savings in the near future in terms of steel thickness, energy consumption, and enamel thickness. All these parameters also will lead to improved enamel technology that is less prone to steel- or fracture-related defects. The next step in this project will consist of optimizing enamel properties, including cleanability and hot-acid resistance, and finalizing enamel development in terms of cost and process robustness.

Acknowledgments

The authors thank Marc Leveaux at OCAS N.V. and Philippe Gousselot at ArcelorMittal for their full cooperation and continuous contribution to research and innovation.

About the authors

Karine Sarrazy is research manager

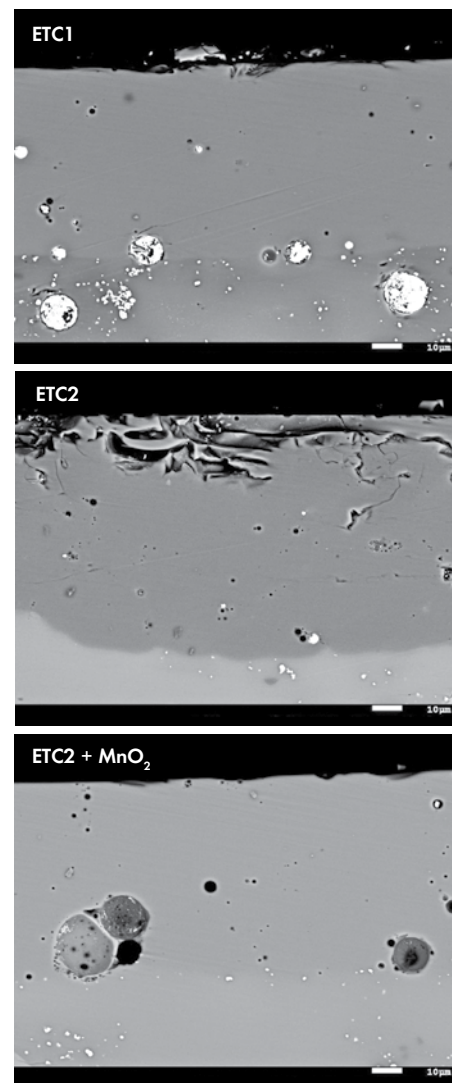


Figure 7. Cross-sectional SEM micrographs of enamel surfaces post-cleanability tests.

(Europe); Alain Aronica is R&D manager (Europe); Angélique Leseur is a technician; and Charles Baldwin is R&D manager (USA), all with Ferro Corp. (Mayfield Heights, Ohio).

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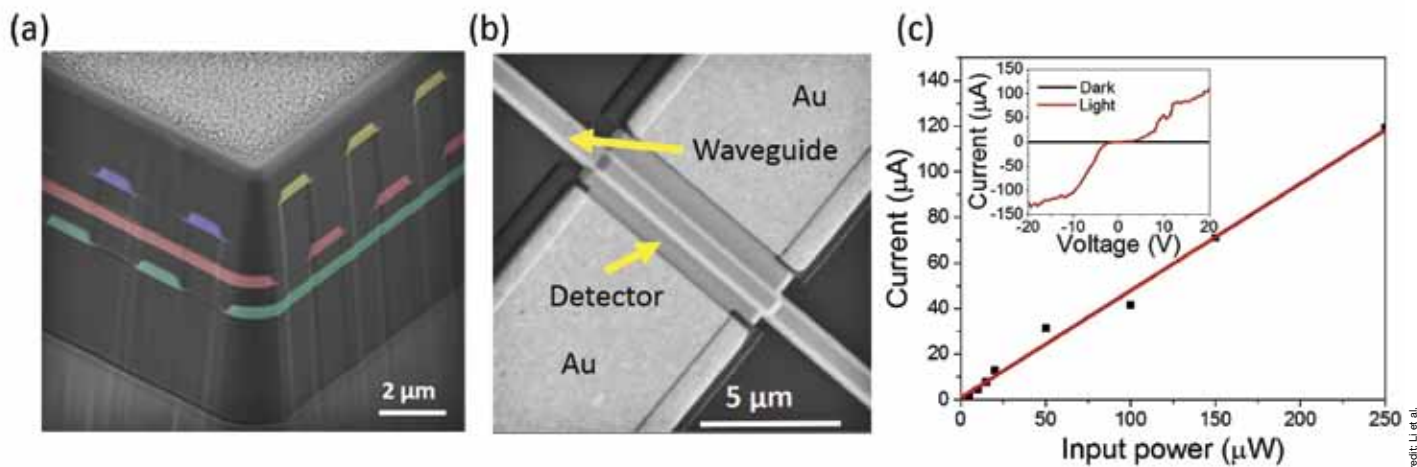


Figure 1. (a) Tilted focused-ion-beam SEM view of a multilayer woodpile photonic crystal (before delamination from the silicon handler substrate). Colors indicate various layers.² (b) SEM image of a chalcogenide glass waveguide integrated photodetector. (c) Measured photocurrent of the photodetector as a function of waveguided optical power at 1550-nm wavelength. Inset shows I - V characteristics of the detector in dark and at 250- μ W input optical power.

Amorphous thin films for mechanically flexible, multimaterial integrated photonics

By Lan Li, Hongtao Lin, Sarah Geiger, Aidan Zerdoum, Ping Zhang, Okechukwu Ogbuu, Qingyang Du, Xinqiao Jia, Spencer Novak, Charmayne Smith, Kathleen Richardson, J. David Musgraves, and Juejun Hu

Integration of amorphous chalcogenides and TiO_2 on polymers can enable photonic devices with exceptional mechanical flexibility.

Flexible integrated photonics is a new technology that started to burgeon during the past few years. It opened applications from flexible optical interconnects to conformal sensors on biological tissues. Material choice is one of the most important factors dictating performance of these flexible devices.

Organic polymers generally are compatible with flexible substrates. However, low refractive indexes of polymers (compared with semiconductors) cannot provide the strong optical confinement necessary for compact photonic integration. Besides polymers, researchers are actively pursuing semiconductor nanomembranes—thin slices of single-crystal semiconductors with submicrometer thickness—for photonic device integration on flexible substrates.

Unlike their rigid bulk counterparts, nanomembranes can bend tightly without cracking, because surface strain induced by bending linearly scales with membrane thickness. Usually, we make photonic devices from nanomembrane structures that are patterned on a rigid substrate, such as silicon. We then pick up the fabricated structures using a poly(dimethylsiloxane) rubber stamp and transfer them onto final flexible substrates. This multistep hybrid method limits processing yield and throughput.

Therefore, we turned to amorphous glasses—the material of choice for optics given their exceptionally low optical attenuation. We use these noncrystalline materials in flexible photonics because they enable monolithic fabrication and can be deposited directly onto flexible substrates without resorting to epitaxial growth. Specifically, we

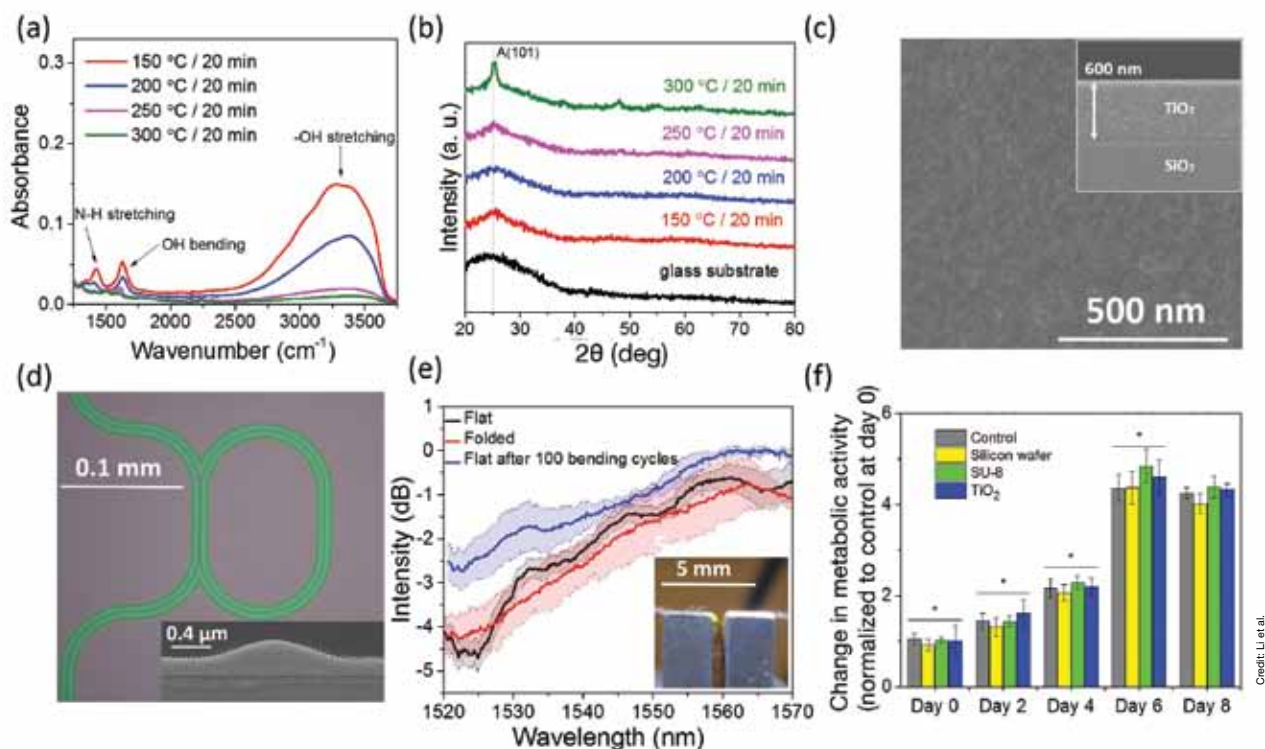


Figure 2. (a) IR spectra and (b) X-ray diffraction spectra of sol-gel TiO_2 thin films annealed at various temperatures. Arrows in (a) indicate characteristic optical absorption bands of chemical residues, and diffraction peak of the anatase phase in (b) is noted by “A(101).” (c) Top-view SEM photograph of a TiO_2 film annealed at 250°C. Inset represents a film cross-section. (d) Optical microscopy photograph of top-view of a TiO_2 racetrack micro-resonator. Inset shows cross-sectional SEM photograph of the waveguide. (e) Normalized optical transmission spectra of a flexible TiO_2 waveguide prior to and after repeated folding. Inset shows a folded TiO_2 waveguide sample under test. (f) Proliferation of human mesenchymal stem cells in indirect contact with photonic materials. *Significantly different ($p < 0.01$) from days 0–6. No significant difference was observed between days 6 and 8.¹

focused on chalcogenide glass materials and amorphous TiO_2 because they can be deposited at relatively low temperatures ($\leq 250^\circ\text{C}$) compatible with flexible substrate integration.^{1–4}

Chalcogenide glasses for 2.5-D photonic integration on flexible substrates

Chalcogenide glasses are amorphous semiconductors that contain one or multiple chalcogen elements, namely sulfur, selenium, and tellurium. Extraordinary infrared (IR) transparency makes these materials popular for optical components, such as IR windows, lenses, optical fibers, and coatings. Phase change memories, all-optical signal processing, chem-bio sensing, and on-chip light switching and modulation are other emerging applications where chalcogenide glasses are important.⁵ By incorporating a multi-neutral-axis mechanical design, we demonstrated low-loss, robust photonic devices on flexible polymer substrates capable of sustaining repeated bending down to submillimeter radii,

despite intrinsic fragility of chalcogenide glass materials.²

Besides excellent optical properties, chalcogenide glasses exhibit extreme processing versatility—they can be monolithically deposited on virtually any technically important substrate and can be shaped into functional device forms via traditional lithography or a variety of soft lithographic methods, including molding, imprinting, and ink-jet printing.⁶ Therefore, chalcogenide glasses are uniquely poised for 2.5-D photonic integration, which refers to vertical stacking of photonic devices in multiple layers.

Fink et al. showed that chalcogenide glasses readily can form planar multilayers (e.g., Bragg mirrors) via sequential thin-film deposition.⁷ We extend the process to stacking of patterned photonic devices by introducing a planarization step between film depositions. In the process, we spin-coated a polymer layer on top of a patterned chalcogenide glass film. We then thermally annealed the polymer/glass to allow the polymer to flow and planarize the surface before

cross-linking, thereby facilitating subsequent deposition and patterning. Using these techniques, we have demonstrated an array of multilayer photonic components on flexible substrates, such as vertically stacked optical resonant filters, overpass structures for waveguide crossings, and woodpile photonic crystals (Figure 1(a)).²

Recently, we showed that the approach also is applicable to integration of active optoelectronic components with passive glass photonics. Figure 1(b) shows a top-view scanning electron microscopy (SEM) photograph of a chalcogenide glass waveguide integrated with an adhesive-bonded semiconductor nanomembrane photodetector. Figure 1(c) is a plot of the measured photocurrent as a function of guided power in waveguides. Compared with traditional photodetectors, which capture only free space illumination, the much smaller optical mode volume enabled by waveguide integration underlies a much larger—and potentially much faster—optical response in these detectors. These results

open up exciting applications in which chalcogenide glasses can seamlessly integrate with other optical materials to enable unconventional functionalities.

Foldable and cytocompatible sol-gel TiO₂ photonics

We also investigated sol-gel TiO₂ as another amorphous material for flexible photonic integration. Besides sharing the same processing advantages—such as low-temperature deposition and ease of integration as chalcogenide glasses—TiO₂ is particularly attractive for biophotonic applications because it is generally considered biocompatible and has been used in dental fillers, cosmetic products, and artificial bone scaffolds.

In our work, we deposited amorphous TiO₂ films using an organic-free sol-gel process. The SEM photograph in Figure 2(c) indicates the uniformity and smooth surface of a sol-gel-coated TiO₂ thin film. Postdeposition annealing temperature is a critical parameter in determining TiO₂ film quality. Figures 2(a) and (b) show that increased annealing temperature contributed to removal of chemical residues and reduction of parasitic optical absorption. However, annealing at >250°C results in partial crystallization, which leads to optical scattering by crystalline grains. TiO₂ films annealed at 250°C feature a uniform and smooth surface (Figure 2(c)) and a relatively low optical loss of 3 dB/cm, which is suitable for photonic integration.

Using a sol-gel technique and plasma etching, we fabricated and tested TiO₂ optical waveguides and resonators monolithically integrated on flexible polymer substrates (Figure 2(d)). Similar to chalcogenide glass flexible photonics, the multi-neutral-axis design renders TiO₂ devices extremely flexible—fabricated TiO₂ waveguides can be repeatedly folded in half without introducing measurable optical degradation (Figure 2(e)). We further validated cytocompatibility of these TiO₂ devices through in-vitro cell viability tests (Figure 2(f)), which show that human mesenchymal stem cells cultured on TiO₂ devices exhibit the same level of metabolic activity as those grown on a reference cell culture

plate. Building on these results, we now focus our ongoing work on integrating TiO₂ flexible photonic sensors with biological tissue engineering platforms to enable real-time monitoring of cell growth.¹

Thin films continue to be important

Processing of amorphous thin films is far more forgiving compared with epitaxy, which is mandated for growing traditional optical crystal materials. Consequently, they can be readily mated with other functional materials to create composite structures possessing unique properties not accessible to glasses alone. Here, we demonstrated that integration of amorphous chalcogenides and TiO₂ on polymers can enable photonic devices with exceptional mechanical flexibility. On the other hand, integration of glasses with semiconductor nanomembranes enables full active-passive integration toward realizing standalone flexible “system-on-a-chip” photonic platforms. These are certainly two cases exemplifying the universal multimaterial photonic integration paradigm, in which we foresee that amorphous thin films will continue to play a pivotal role.

About the authors

Lan Li, Hongtao Lin, Okechukwu Ogbuu, Qingyang Du, and Juejun Hu are members of the Department of Materials Science and Engineering at Massachusetts Institute of Technology (Cambridge). Sarah Geiger and Xinqiao Jia are members of the Department of Materials Science and Engineering at the University of Delaware (Newark). Aidan Zerdoum and Xinqiao Jia are members of the Department of Biomedical Engineering at the University of Delaware (Newark). Ping Zhang is a member of the School of Electronic Information Engineering at Tianjin University (Tianjin, China). Spencer Novak, Charmayne Smith, and Kathleen Richardson are members of the CREOL College of Optics and Photonics in the Department of Materials Science and Engineering at the University of Central Florida (Orlando). J. David Musgraves is with IRradiance Glass Inc. (Orlando, Fla.).

Editor's note

Li will present the 2016 Kreidl Award Lecture at the Glass and Optical Materials Division Annual Meeting in Madison, Wis., on May 24, 2016.

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2016
STRUCTURAL CLAY PRODUCTS DIVISION MEETING
 in conjunction with National Brick Research Center Meeting
 May 2-4 Embassy Suites North Canton, Ohio

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The Structural Clay Products Division of The American Ceramic Society emphasizes the most efficient and economical ways to manufacture brick, pipe, red-body tile, and other structural clay products. Join us for our annual Division meeting, held in conjunction with the National Brick Research Center Meeting.

Tentative Schedule

Sunday, May 1	
Football Hall of Fame tour	(optional, on own)
Monday, May 2	
SCPD technical program	2:00 – 5:00 p.m.
Welcome reception	5:00 – 7:00 p.m.
Tuesday, May 3	
Plant tour bus departure, Embassy Suites	9:00 a.m.
Plant tour – The Belden Brick Co. (Plants 4 & 8), Sugarcreek, Ohio	10 a.m. – Noon
Lunch provided by The Belden Brick Co.	Noon – 1:00 p.m.
The Belden Brick Co. tour – continued (Plants 2 & 3)	1:00 – 3:00 p.m.
Suppliers mixer, Embassy Suites	5:30 – 7:00 p.m.
Wednesday, May 4	
NBRC management subcommittee group (by invitation only)	7:00 – 8:30 a.m.
NBRC member meeting (open to all NBRC members)	9:00 a.m. – Noon

Technical sessions

The brick MACT – What are we doing to fix it? – Susan J. Miller, Brick Industry Association, and Garth Tayler, Acme Brick

Raw-material testing for MACT and SDSs – Susan J. Miller, Brick Industry Association, and John Sanders, National Brick Research Center at Clemson University

Don't forget about silica – Susan J. Miller, Brick Industry Association, and Garth Tayler, Acme Brick

Update on recent changes to PCI specification for thin brick – Mike Walker, National Brick Research Center at Clemson University

Carbonate ceramics – A disruptive technology for the brick – Richard Riman, Rutgers University

Pigments manufacturing and flow characteristics – Don Abernathy, Huntsman Pigments–Davis Colors

Update on facade panel and thin-brick production – Don Dennison

Overview of Belden Brick – Robert F. Belden, The Belden Brick Co.

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- H9:** Component testing and evaluation of composites
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- G8:** Multifunctional coatings for sustainable energy and environmental applications

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TUESDAY, JUNE 28, 2016 | Noon – 1:15 p.m.

“Mentorship for young scientists: Developing scientific survival skills,” a special session sponsored by Saint-Gobain, will be presented by professor Federico Rosei, INRS Énergie Matériaux Télécommunications Research Centre.

PLENARY SPEAKERS

MONDAY, JUNE 27, 2016 | 8:00 – 9:00 a.m.



Shunpei Yamazaki, founder and president, Semiconductor Energy Laboratory Co. Ltd., Japan
Title: *Discovery of indium gallium zinc oxide (CAAC-IGZO) and its applications in next-generation information display devices*



A.N. Sreeram, senior vice president research & development and chief technology officer, Dow Chemical Co.
Title: *The science of materials: Impactful solutions to big global challenges*



Katherine A. Stevens, general manager, materials and process engineering, GE Aviation
Title: *SiC/SiC ceramic-matrix composites for jet engines*



Jörg Esslinger, director materials engineering, MTU Aero Engines AG, Germany
Title: *Ceramic-matrix composites (CMCs): Enabling materials for competitive aero-engines*

SCHEDULE AT A GLANCE

Sunday, June 26, 2016

Welcome reception 5:00 – 7:00 p.m.

Monday, June 27, 2016

Plenary session 8:00 – 9:00 a.m.
Concurrent sessions 9:30 a.m. – 5:30 p.m.
Lunch on own Noon – 1:20 p.m.

Tuesday, June 28, 2016

Concurrent sessions 9:30 a.m. – 5:30 p.m.
Lunch on own Noon – 1:30 p.m.
Poster session 6:30 – 8:30 p.m.

Wednesday, June 29, 2016

Concurrent sessions 9:30 a.m. – 5:30 p.m.
Lunch on own Noon – 1:30 p.m.

Thursday, June 30, 2016

Concurrent sessions 9:30 a.m. – 5:30 p.m.
Lunch on own Noon – 1:30 p.m.
Conference banquet 7:00 – 9:30 p.m.

Friday, July 1, 2016

Concurrent sessions 9:30 a.m. – Noon

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STOOKEY LECTURE OF DISCOVERY



Monday, May 23, 2016 | 8:00 – 9:00 a.m.

David L. Griscom, impactGlass research international

Title: *The life and unexpected discoveries of an intrepid glass scientist*

GEORGE W. MOREY AWARD LECTURE



Tuesday, May 24, 2016 | 8:00 – 9:00 a.m.

Hellmut Eckert, Institute of Physics in São Carlos, University of São Paulo, Brazil, and Institute of Physical Chemistry, University of Münster, Germany

Title: *Spying with spins on messy materials: 50 years of glass structure elucidation by NMR spectroscopy*

DARSHANA AND ARUN VARSHNEYA FRONTIERS OF GLASS SCIENCE LECTURE



Wednesday, May 25, 2016 | 8:00 – 9:00 a.m.

Matteo Ciccotti, Professeur de l'ESPCI, Laboratoire de Science et Ingénierie de la Matière Molle, France

Title: *Multiscale investigation of stress-corrosion crack propagation mechanisms in oxide glasses*

DARSHANA AND ARUN VARSHNEYA FRONTIERS OF GLASS TECHNOLOGY LECTURE



Thursday, May 26, 2016 | 8:00 – 9:00 a.m.

Matthew J. Dejneka, research fellow, Corning Glass Research Group

Title: *Chemically strengthened glasses and glass-ceramics*

NORBERT J. KREIDL AWARD FOR YOUNG SCHOLARS



Tuesday, May 24, 2016 | Noon – 1:00 p.m.

Lan Li, Massachusetts Institute of Technology

Title: *Materials and devices for mechanically flexible integrated photonics*

TECHNICAL PROGRAM

S1: Fundamentals of the glassy state

Session 1: Glass formation and structural relaxation

Session 2: Fundamentals and applications of glass-crystallization

Session 3: Structural characterization of glasses

Session 4: Computational and theoretical studies of glass

Session 5: Mechanical properties of glasses

Session 6: Non-oxide and metallic glasses

Session 7: Glass under extreme conditions

S2: Larry L. Hench memorial symposium on bioactive glasses

S3: Optical and electronic materials and devices—Fundamentals and applications

Session 1: Amorphous ionic and electronic conductors: Materials and devices

Session 2: Optical fibers

Session 3: Optical materials for components and devices

Session 4: Laser interactions with glass

Session 5: Glass-ceramics and optical ceramics

S4: Glass technology and cross-cutting topics

Session 1: Glass surfaces and functional coatings

Session 2: Liquid synthesis and sol-gel-derived materials

Session 3: Challenges in glass manufacturing

Session 4: Waste immobilization—Waste form development: Processing and performance

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PRACTICAL TIPS FOR GETTING YOUR RESEARCH PUBLISHED

Wednesday, May 25, Noon – 1:15 p.m. | Madison Room

All student and young professional attendees are invited. Professor Mario Affatigato will provide advice for students on the wild world of scientific publishing. Specifically, he will discuss:

- Content suitable for publication;
- Desire for cutting-edge work;
- Reviews and topical issues;
- Vexing problem of plagiarism, naive and intentional;
- Technical issues with manuscripts, including image resolution and English language editing;
- Getting involved as a journal reviewer;
- Writing clarity and the expected audience;
- Some of the ethical principles behind research publishing; and
- Brief mention of the current environment in the scientific journal world, with an emphasis on trends, such as open access publishing and discussions on impact.

Expect a friendly, educational presentation with ample time for questions.

Mario Affatigato is coeditor of the *International Journal of Applied Glass Science*. He holds the Fran Allison and Francis Halpin Professorship in Physics at Coe College, where he has developed a research effort primarily investigating the relationship between the optical properties and structure of glassy materials. He is a Fellow of The American Ceramic Society and the U.K. Society of Glass Technology, and, in 2015, became a Research Corporation Cottrell Scholar.

Lunch will be provided on a first come, first served basis.

STUDENT, POSTDOC, AND YOUNG PROFESSIONAL CAREER DISCUSSION ROUNDTABLES

Wednesday, May 25, 5:45 – 6:45 p.m. | Capitol B

Students, postdocs, and young professionals are invited to an informal group discussion with nine panelists representing industry, national labs, and academia. This is an opportunity to ask questions of professionals in a casual environment on a number of diverse topics (work-life balance, career opportunities, etc.). The career professionals will rotate every 15 minutes, so attendees will get a chance to have candid discussions with several professionals during this session. Light refreshments will be served.

PANELISTS:

Academia:

Juejun Hu, Massachusetts Institute of Technology
Mathieu Bauchy, University of California, Los Angeles
Liping Huang, Rensselaer Polytechnic Institute

National lab:

Tayyab Suratwala, Lawrence Livermore National Laboratory
Todd Alam, Sandia National Laboratories
Joseph Ryan, Pacific Northwest National Laboratory

Industry:

Mathieu Hubert, CelSian Glass & Solar, The Netherlands
John Mauro, Corning Incorporated
Clara Rivero-Baleine, Lockheed Martin Corporation

GOMD STUDENT POSTER CONTEST INFORMATION

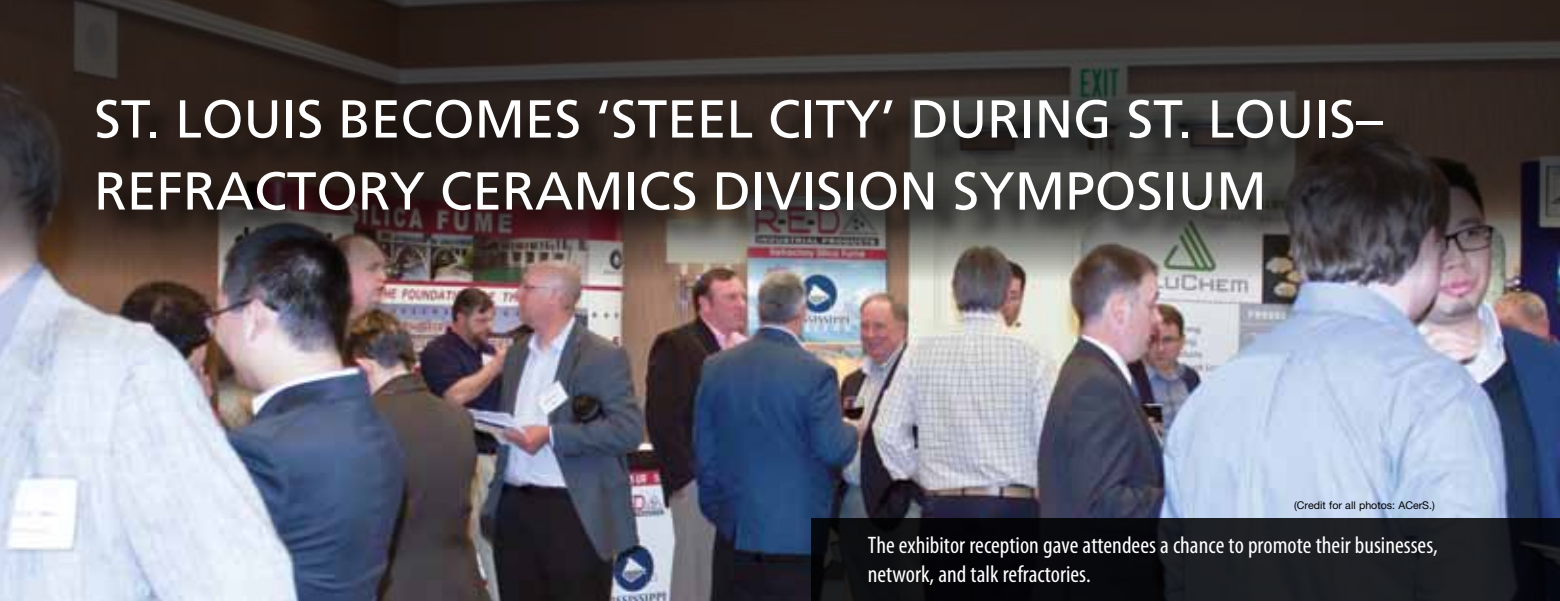
Sponsored by **CORNING**

The GOMD student poster contest, sponsored by Corning Incorporated, will take place on Monday evening as part of the regular poster session, 6:30 - 8:30 p.m. in Senate A/B. This year's contest is organized by Mathieu Bauchy of UCLA.

- Set up posters 3:20 – 5:00 p.m. Pins will be provided.
- Students are expected to remain with their poster for judging.
- All posters must be removed from the boards at 8:30 p.m.
- Winners announced at the conference dinner on Tuesday, 7:00 – 10:00 p.m. Winning posters will remain on display.

Good luck to all students, and thanks to Corning for their generous sponsorship!

ST. LOUIS BECOMES 'STEEL CITY' DURING ST. LOUIS-REFRACTORY CERAMICS DIVISION SYMPOSIUM



(Credit for all photos: ACerS.)

The exhibitor reception gave attendees a chance to promote their businesses, network, and talk refractories.



Abel Carriquiry from Peru takes the opportunity to network during a break at the St. Louis Section–Refractory Ceramics Division symposium.



The assembled group of T.J. Planje award winners.



Andus Buhr (left) from Almatris in Germany fields a question while symposium co-organizer Simon Leiderman listens.

It has been said that if you are in the refractory business, you are in the steel business. So, this year's theme for the 52nd Annual Symposium on Refractories was especially fitting—Refractories for the Ferrous Industry: A Historical Perspective, Present, and Future Directions.

The joint effort, produced by the St. Louis Section and the Refractory Ceramics Division, took place March 30–31 in St. Louis, Mo. The symposium featured 17 presentations for about 210 attendees. The international scope of the steelmaking industry was reflected in the presentations, with speakers from Norway, Germany, Canada, China, Brazil, Austria, and the United States. The audience, too, included about 25 attendees from abroad. ■



Bjørn Myhre (left) from Elkem Silicon Materials (Norway) accepts the St. Louis Section's T.J. Planje award from Jeff Smith (Missouri S&T).



ACerS president Mrityunjay Singh presents Patty Smith with an ACerS Global Ambassador certificate recognizing her work organizing the St. Louis Section and the Refractories Symposium.



Paul Ormond accepts an ACerS Global Ambassador certificate from President Singh in recognition of contributions to the refractory community and participation in the MS&T Materials Camps.



Keith Bowman
Keith Bowman

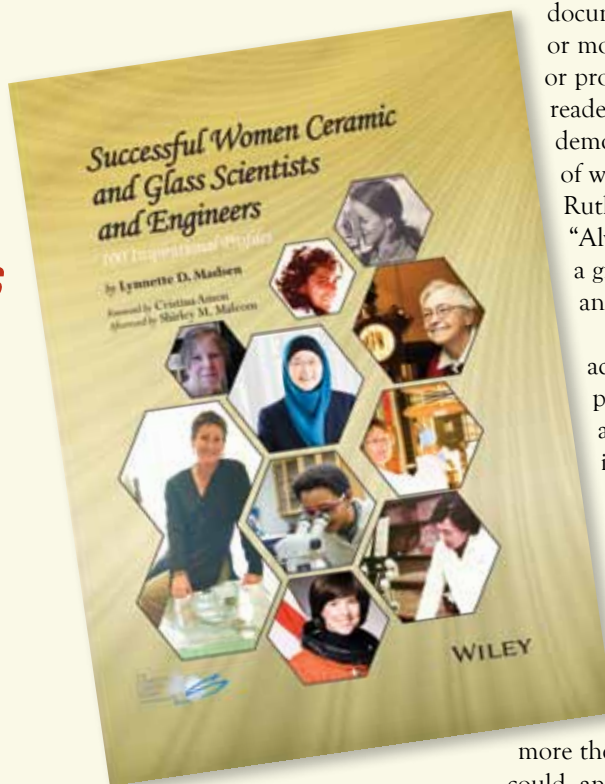
Successful Women Ceramic and Glass Scientists and Engineers: 100 Inspirational Profiles

by **Lynnette Madsen**

As I began reading *Successful Women Ceramic and Glass Scientists and Engineers*, I recognized how much I have been honored to have many of the profiled women as colleagues across my professional career. Many subjects of the one hundred profiles could dot a timeline mapping my experiences as a member of the ceramics community, and I relished the opportunity to learn, learn more, or relearn about all the women included in the book.

But I initially had not considered that the book topic—women in ceramics and glass—would also inspire me to think of all of the inspirational women I have known across other science and engineering fields as well. For me, that list includes many of the superb women who serve or have served The American Ceramic Society. Like many of the women in Madsen’s new book, they are often the first people I think of when I hear only their first names. I believe other readers will similarly recognize that we are blessed to have so many inspiring women in the ceramics and glass community, and, as is evident in much broader contexts as well, that celebrations of women and their contributions have been too rare. The words of wisdom shared by Doreen Edwards—“We need to make our own definitions of success”—apply to all.

As she notes in the introduction, Madsen overcame challenges inherent



in establishing any collection of profiles from a large group of individuals. But she also was challenged to choose just one hundred women to profile and determine how to organize the profiles and how to include appropriate representation from other areas of diversity, including race, ethnicity, and nationality. The book provides a listing of women based on various categories, including regions, career categories, and ethnicity. But the compilation also provokes thought about the (in)visibility of other aspects of diversity, including disability and sexuality, for many other colleagues—either from lack of representation, disclosure, or both.

Many women profiled in the book share private moments and information about themselves that might give some pause, but the personal moments add richness to the professional achievements. Photos shared with the profiles

document important achievements or moments in the women’s personal or professional lives that enrich the reader experience. The book clearly demonstrates that hearing the voices of women in our field is important. Ruth H.G.A. Kiminami states, “Always fight for your dreams with a great dose of optimism, perseverance, and kindness.”

The mixture of professional achievements, personal accomplishments, words of wisdom, and sobering commentaries is much richer than might be apparent to a reader paging through the book for the first time. Stories describing intentional and unintentional discrimination and slow progress toward truly inclusive workplaces likely will make the reader appreciate even

more the successes of colleagues. Or it could, and should, also make the reader frustrated that progress has been so slow. But supportive spouses, clever approaches for overcoming inflexible workplace policies, and exceptional mentors and advocates are key elements in many of the stories.

When reading profiles of women I know, I found it important to ask myself if I knew all the achievements listed and if I had ever heard the women describe challenges they have overcome. In several cases, I felt like I was hearing the real voices of these successful women for the first time. As Marina Pascucci advises, “Try not to listen to naysayers—success is the best revenge.”

Keith Bowman is dean of the College of Science & Engineering at San Francisco State University (San Francisco, Calif.). ■

Calendar of events

May 2016

2–4 Structural Clay Products Division Meeting – Embassy Suites, North Canton, Ohio; www.ceramics.org/clay2016

2–4 Missouri Concrete Conference – Rolla, Mo.; www.dce.mst.edu

8–11 ➤ ICCPS-13: 13th Int'l Conference on Ceramic Processing Science – Nara, Japan; unit.aist.go.jp/ifmri/tl-int/iccps13

10–12 ➤ 78th Annual PEI Technical Forum – Louisville, Ky.; www.porcelainenamel.com

18–22 ➤ WBC2016: 10th World Biomaterials Congress – Montreal, Canada; www.wbc2016.org

22–26 GOMD 2016: Glass and Optical Materials Division Meeting 2016 – The Madison Concourse Hotel and Governor's Club, Madison, Wis.; www.ceramics.org/gomd2016

23–25 27th AeroMat Conference and Exposition – Meydenbauer Center, Bellevue, Wash.; www.asminternational.org/web/aeromat-2016

June 2016

8–10 ACerS Southwest Section meeting – Hilton Birmingham Perimeter Park, Birmingham, Ala.; www.ceramics.org/sections/southwest-section

26–30 ➤ HTCMC 9 and GFMT: 9th Int'l Conference on High-Temperature Ceramic-Matrix Composites and Global Forum on Advanced Materials and Technologies for Sustainable Development 2016 – Toronto Marriott Downtown Eaton Centre Hotel, Toronto, Canada; www.ceramics.org/htcmc9_gfmat2016

27–29 ➤ Electroceramics XV – Limoges, France; www.electroceramics15.com

July 2016

3–6 ➤ Microwave Materials and Their Applications – Seoul, South Korea; www.mma2016.com

5–8 12th European SOFC and SOE Form: 20th Conference in Series with Exhibition – Kulture- und Kongresszentrum Lucerne, Switzerland; www.EFCF.com

10–13 3rd Int'l Congress on 3D Materials Science 2016 – Pheasant Run Resort, St. Charles, Ill.; www.tms.org/meetings/2016/3DMS2016

11–13 Cements 2016: 7th Advances in Cement-Based Materials – Northwestern University, Evanston, Ill.; www.ceramics.org/cements2016

17–21 6th Int'l Conference on Recrystallization and Grain Growth – Omni William Penn Hotel, Pittsburgh, Pa.; www.tms.org/meetings/2016/ReXGG2016

25–26 Diversity in the Minerals, Metals, and Materials Professions – Northwestern University, Evanston, Ill.; www.tms.org/meetings/2016/diversity2016

28–31 Innovations in Biomedical Materials and Technologies – Rosemont Hyatt, Chicago, Ill.; www.ceramics.org/biomed2016

31–Aug. 5 ➤ Gordon Research Conference on Ceramics and Solid State Studies – Mount Holyoke College, South Hadley, Mass.; www.grc.org/programs

August 2016

21–23 ➤ ICC6: Int'l Congress on Ceramics – Dresden, Germany; www.icc-6.com

September 2016

4–8 ESG 2016/SGT100: Society of Glass Technology Conference – Sheffield, U.K.; www.sgt.org

28–29 59th Int'l Colloquium on Refractories 2016 – Aachen, Germany; www.ecref.eu

October 2016

1–6 ➤ 6th Int'l Conference on Electrophoretic Deposition – Gyeongju, South Korea; www.engconf.org/conferences

23–27 ➤ MS&T16, combined with ACerS 118th Annual Meeting – Salt Lake City, Utah; www.ceramics.org; www.matscitech.org

January 2017

18–20 EMA 2017: ACerS Electronic Materials and Applications – DoubleTree by Hilton Orlando Sea World, Orlando, Fla.; www.ceramics.org

22–27 ICACC'17: 41st Int'l Conference and Expo on Advanced Ceramics and Composites – Hilton Daytona Beach Resort/Ocean Walk Village, Daytona Beach, Fla.; www.ceramics.org

Ceramic Tech Today blog

www.ceramics.org/ceramictechtoday

Dates in **RED** denote new entry in this issue.

Entries in **BLUE** denote ACerS events.

➤ denotes meetings that ACerS cosponsors, endorses, or otherwise cooperates in organizing.

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Skulls, mummies, and nuclear fuels? Diversity in materials science

Re-entering academia as a more seasoned, nontraditional student with a background in finance, I knew that I wanted to change course and pursue a scientific career—but materials science and engineering was not on my radar. As a female student, I struggled to decide if engineering was the right path for me. But after visiting an engineering professor at Boise State University (BSU) and discovering what a materials science and engineering degree offers in terms of interdisciplinary opportunities, I decided to take a fork in the road—I enthusiastically embraced materials science and engineering as my career choice.

During my first few years back to school, personal circumstances allowed me to pursue my education only part-time. Fortunately, many materials science and engineering faculty at BSU involve undergraduates in their research. I was thrilled when the Advanced Materials Laboratory at BSU and the Center for Advanced Energy Studies in Idaho Falls, led by Darryl Butt, hired me as an undergraduate researcher in early 2014. Our group participates in wide-ranging research programs that include projects on materials processing, materials in extreme environments (including nuclear applications), and materials associated with cultural heritage and art conservation.

I started in Butt's group working on two cultural heritage projects aimed at characterizing pigments from a painted, pre-Columbian skull and a 2,000-year-old Fayum mummy portrait. I was delighted to discover how materials science could unearth new discoveries on ancient artifacts—some days it was hard to believe I was working on artifacts with such a rich history.

Jennifer Watkins processes oxygen sensitive pyrophoric nitride ceramics in an argon back-filled glovebox.



Credit: Jennifer Watkins

But, as I immersed myself in materials science research and studies, I became more and more fascinated with nuclear energy. Fortunately, my pigment research led to an opportunity to contribute to additional research to develop advanced nuclear fuels. I jumped in, seeking out supplementary education through an online minor in nuclear engineering at Kansas State University. Through these new opportunities, I learned about novel synthesis and fabrication methods for advanced fuels and developed my skills with various characterization tools.

I am fortunate to work in such a diverse field—how many people can say that on any random day they could handle archaeological remains in the morning and manufacture a uranium pellet in the afternoon? My experience also helped me secure a spot in the Department of Homeland Security's Nuclear Forensics Undergraduate School during the summer of 2015. The school provided me with hands-on experience in radiochemistry and further advanced my knowledge of nuclear materials and applications.

I am often asked why I want to work in the nuclear field. Even though I have gained substantial understanding of nuclear science, I continue to be fascinat-

ed by how much energy can be generated from something so small. In fact, when I decided to re-enter academia, I did not plan to pursue an advanced degree. But my experiences thus far and the opportunities for collaboration with leading researchers and scientists have adjusted my plans—a few weeks ago, I accepted an offer from BSU to pursue my Ph.D.

The opportunity to pursue something I am passionate about has negated any initial uncertainties about my success as a nontraditional student and researcher. And, like most scientists, I am hopeful that my work ultimately improves our world. I feel privileged to know that in my role as a nuclear research scientist, I can impact technological innovations that advance our nation's energy security and climate change goals.

Jennifer Watkins is a senior undergraduate at Boise State University. She is a member of the Materials Science Club and Society of Women Engineers and serves as a peer ambassador for the Boise State College of Engineering. In her free time, Watkins enjoys reading, wine tasting, and enjoying the beautiful Idaho outdoors. ■



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19 K 39.0983 Potassium	20 Ca 40.078 Calcium	21 Sc 44.955912 Scandium	22 Ti 47.867 Titanium	23 V 50.9415 Vanadium	24 Cr 51.9961 Chromium	25 Mn 54.938045 Manganese	26 Fe 55.845 Iron	27 Co 58.933195 Cobalt	28 Ni 58.6934 Nickel	29 Cu 63.546 Copper	30 Zn 65.38 Zinc	31 Ga 69.723 Gallium	32 Ge 72.64 Germanium	33 As 74.9216 Arsenic	34 Se 78.96 Selenium	35 Br 79.904 Bromine	36 Kr 83.798 Krypton
37 Rb 85.4678 Rubidium	38 Sr 87.62 Strontium	39 Y 88.90585 Yttrium	40 Zr 91.224 Zirconium	41 Nb 92.90638 Niobium	42 Mo 95.96 Molybdenum	43 Tc (98.0) Technetium	44 Ru 101.07 Ruthenium	45 Rh 102.9055 Rhodium	46 Pd 106.42 Palladium	47 Ag 107.8682 Silver	48 Cd 112.411 Cadmium	49 In 114.818 Indium	50 Sn 118.71 Tin	51 Sb 121.76 Antimony	52 Te 127.6 Tellurium	53 I 126.90447 Iodine	54 Xe 131.293 Xenon
55 Cs 132.9054 Cesium	56 Ba 137.327 Barium	57 La 138.90547 Lanthanum	58 Ce 140.116 Cerium	59 Pr 140.90765 Praseodymium	60 Nd 144.242 Neodymium	61 Pm (145) Promethium	62 Sm 150.36 Samarium	63 Eu 151.964 Europium	64 Gd 157.25 Gadolinium	65 Tb 158.92535 Terbium	66 Dy 162.5 Dysprosium	67 Ho 164.93032 Holmium	68 Er 167.259 Erbium	69 Tm 168.93421 Thulium	70 Yb 173.054 Ytterbium	71 Lu 174.968 Lutetium	
87 Fr [223] Francium	88 Ra [226] Radium	89 Ac (227) Actinium	90 Th 232.0377 Thorium	91 Pa 231.03688 Protactinium	92 U 238.02891 Uranium	93 Np (237) Neptunium	94 Pu (244) Plutonium	95 Am (243) Americium	96 Cm (247) Curium	97 Bk (247) Berkelium	98 Cf (251) Californium	99 Es (252) Einsteinium	100 Fm (257) Fermium	101 Md (258) Mendelevium	102 No (259) Nobelium	103 Lr (262) Lawrencium	

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