

AMERICAN CERAMIC SOCIETY

bulletin

emerging ceramics & glass technology

APRIL 2020

Smog begone!
**How development of
ceramic automotive
catalytic substrates
and filters helped
reduce air pollution**



New issue inside
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MANUFACTURING

Silicon nitride for surgical implants | Transparent ceramics: Emerging opportunities

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by Douglas M. Beall and Willard A. Cutler



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Breaking in: A guide to working with big business

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Employee of Corning Inc. in a manufacturing facility.
Credit: Corning Inc.

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As seen on Ceramic Tech Today...



Credit: Zhang et al., *International Journal of Nanomedicine*
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Films fix flaws: MXenes for guided bone regeneration

A lot of research focuses on environmental and energy applications of MXenes, but there are plenty of potential biomedical applications as well. Three researchers at Sichuan University in China investigate using MXene films as a barrier membrane in guided bone regeneration.

Read more at www.ceramics.org/mxenefilms

Also see our ACerS journals...

Changes in fluoride removal ability of chicken bone char with changes in calcination time

By M. Kikuchi, Y. Arioka, M. Tafu, and M. Irie

International Journal of Ceramic Engineering & Science

Black ZrO₂ synthesized by molten lithium reduction strategy for photocatalytic hydrogen generation

By D. Zu, H. Wang, T. Yang, et al.

Journal of the American Ceramic Society

Permeability behavior of silicon carbide based membrane and performance study for oily wastewater treatment

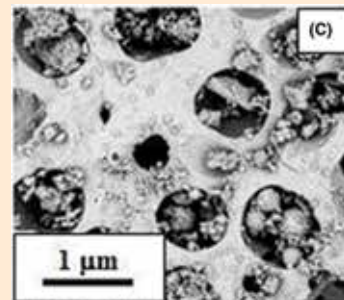
By D. Das, N. Kayal, G. A. Marsola, et al.

International Journal of Applied Ceramic Technology

Prospects of antibacterial bioactive glass nanofibers for wound healing: An in vitro study

By S. Saha, A. Bhattacharjee, S. H. Rahaman, et al.

International Journal of Applied Glass Science



Read more at www.ceramics.org/journals

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POSTMASTER: Please send address changes to American Ceramic Society Bulletin, 550 Polaris Parkway, Suite 510, Westerville, OH 43082-7045. Periodical postage paid at Westerville, Ohio, and additional mailing offices. Allow six weeks for address changes.

ACSBA7, Vol. 99, No. 3, pp 1 - 64. All feature articles are covered in Current Contents.

Integrated energy management looks to graduate smart homes to the next level

The annual Consumer Electronics Show, held in Las Vegas every January, introduces scores of new innovations in electronics, tech, gadgets, and more. The show debuts everything from the mundane to the innovative, profound, weird, wacky, and even completely unnecessary—and it is the perfect place to track trending technologies and electronics evolutions.

There are many gadget highlights from the most recent CES 2020, including speakers with curved display screens, watches with optical blood oxygen sensors, AI-powered prosthetics, and foldable phones. But a prevalent theme at

CES in recent years is further upgrades, new iterations, and continued integrations of smart tech for the home.

According to McKinsey & Company, the United States market for connected homes grew at a rather rapid compound annual growth rate of 31% since 2015.

Homes now can have light switches controlled by voice commands, video doorbells that allow homeowners to interact with someone on their doorstep even when they are not home, and surprising devices—from washers and dryers to toilets and trash cans—that are Bluetooth connected and wifi-enabled.

One of the most successful penetrations of these smart home technologies to date relates to energy management. After all, residential and commercial buildings account for 40% of total energy use in the U.S.

And as CES this year indicates, smart home energy management may soon gain the ability to graduate to advanced classes. For instance, Schneider Energy debuted its solution to makeover power management in buildings at CES 2020 with its Energy Center control panel, a modern, smart, and integrated upgrade of the fuse box.



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According to a *TechCrunch* article, “The new product is part of a broader range of Square D home energy management devices that Schneider is aiming at homeowners. The company provides a broad suite of energy management services and technologies to commercial, industrial and residential customers, but is making a more concerted effort into the U.S. residential market beginning in 2020.”

Although the available details are vague, the idea is to give consumers a better way to manage and control their electricity use. Data from Statista shows that energy management is a particular segment of the market that is increasing its penetration into smart homes, predicted to almost double from 23.5 million homes in 2020 to 45.1 million homes in 2024.

Schneider is not the first company to target energy management—Leviton



Credit: Nigel Howe, Flickr (CC BY-NC 2.0)

Advanced ceramics and high-tech glass are not limited to smartphones—a similar rapid technological evolution is occurring in the smart homes market as well

already offers smart circuit breakers to provide granular energy usage data. And startup Span previously released its plan to upgrade the residential fuse box with solutions to better integrate and manage

alternative energy sources in residential homes, such as electricity generated from rooftop-installed solar panels, which represent a growing sector of the residential energy market.

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However, such technologies are the forefront, so most solutions to integrate smart and connected home tech cannot yet do so with these energy management solutions, “likely due to the fact that none of the major smart home services are designed to handle anything this complex,” according to an article on *The Verge*.

Offering a way to better integrate and manage more diverse energy sources seems to be the next chapter in smart homes, and a step toward not just integration but true home automation—allowing buildings to work even more efficiently without requiring human commands and interventions.

This step is an important point and the next chapter for all types of “smart” devices in people’s lives and on display at CES 2020—much of what people think of as “smart” is not really smart but rather connected.

These devices are equipped with Bluetooth and have the capability to connect to the internet, but most still require considerable user input for their functionality. To be smart, devices must be more insightful, “with technology automatically learning patterns within a home to find and suggest ways to control devices and ultimately save energy,” according to a Smart Electric Power Alliance article.

The Smart Electric Power Alliance article continues: “Imagine a future where home energy management is fully automated and optimized—taking care of everything from adjusting the load of a house amid fluctuating temperatures and peak demand prices, to preventing a pipe from bursting and causing severe water damage as temperatures fall below freezing. Autonomous home energy management could fundamentally alter the way utilities manage the grid by creating a coordinated network between the smart grid and smart home.”

That is an exciting possibility for residential consumers, but it also undoubtedly means opportunity for ceramic and glass materials as well—because just like the smartphone market, the residential housing market is inexorably linked to ceramic and glass materials.

“As the complexity of these systems increases, so does the methodology, the science, the innovation—all that is able to come into these systems more and more,” Eastman Chemical Co.’s Julia Schimmelpenninckh says in the December 2019 *ACerS Bulletin* feature

article about homes. “So the opportunities for innovation increase with the complexity.”

And luckily for the ceramic and glass industries, innovation is something these materials enable quite well. ■



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Glass comes to foldable phone displays

On February 12, Samsung debuted its latest device in a slate of foldable phones currently on the market—although the electronics giant’s new model, the Samsung Galaxy Z Flip, is the first to feature a foldable glass display.

Foldable devices have captured much attention in the smartphone world for the past several years despite challenges to making foldable smartphones. Namely, one of the biggest challenges with a smartphone that bends in half is its screen—users want a large, high-resolution display, but designing one with the durability to repeatedly bend across 180° is not easy. That is why existing foldable phones—including ones introduced by Huawei, Motorola, and Samsung—have all donned plastic screens thus far.

The new Galaxy Z Flip uses Samsung’s proprietary material, called Ultra Thin Glass (UTG), for the display. UTG is seemingly manufactured by South Korean glass company Dowoo. Samsung became a major stakeholder in the company in late 2019 “as part of efforts to secure stable supply of key materials for the second generation of the Galaxy Fold,” according to an article on Korean news website MK. And Samsung around the same time filed for the trademark “Samsung Ultra Thin Glass” with the European Union Intellectual Property Office, according to *Forbes*.

“Dowoo can currently produce around 500,000 UTG units per month—which should be enough to support Samsung’s foldable OLED needs in 2020,” states an article on OLED-info.com. “With the new investment [that of Samsung], Dowoo plans to increase its production capacity in the future. Dowoo’s display glass is less than 100 μm thick—and can be made thinner up to 30 μm.”

The details of those production processes and capabilities are hard to come by or verify currently, but it stands to reason that there is a reasonably large market for thin, bendable glass that could be incorporated into smartphones and other electronics devices.

Yet a significant question is how well the UTG display holds up over time, as the thin glass is bound to be stressed each time the phone flips open and closed. And what about bumps, bumbles, and drops—does the thin glass easily snap in the face of daily device abuse?

Recent reports seem to indicate that the durability of the new Galaxy Z Flip is not that great. In fact, some testing calls into question whether the devices even feature glass displays at all. According to an article on *The Verge*, tech reviewer Zack Nelson tested the new Samsung device on his YouTube channel JerryRigEverything and found the Z Flip’s display starts showing permanent marks and scratches far earlier than actual glass would.

“If you’ve watched Zack’s videos before, you’ve likely heard that modern smartphones have ‘scratches starting at a [Mohs hardness] level 6, with deeper grooves at a level 7,’” *The Verge*



Credit: Samsung

The new Samsung Galaxy Z Flip is the first flip phone to feature a foldable glass display.

article reports. “The Z Flip starts picking up damage at level 2 and more significantly at 3, which is on par with the plastic screens of the Galaxy Fold and more recent Motorola Razr.”

Samsung admits that its screen has a “protective layer” on top of the glass, but Nelson speculates that the Galaxy Z Flip may instead actually use a hybrid plastic polymer with glass mixed in rather than a thin sheet of glass.

So the picture is not clear for the Samsung Galaxy Z Flip. Nonetheless, there does seem to be significant potential for foldable glass displays, as Dowoo is not the only company working toward this goal—Corning also is working on developing bendable glass.

“We do have an active effort underway for bendable glass, and we believe it will be the ultimate solution in this space,” a Corning spokesperson says in an email. “While we can’t put a specific timeframe on it right now since the glass is still in development, we believe that our glass solution will be ready in the next 12–18 months.” ■

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Transparent ceramics: Emerging opportunities

By Jason Chen

Sales of transparent ceramic materials worldwide will grow at a compound annual growth rate (CAGR) of 19.0% in the next five years, reaching \$765.9 million by 2024.

Transparent ceramics are defined as inorganic, nonmetallic polycrystalline materials that transmit light with wavelengths in the visible electromagnetic spectrum. A ceramic is considered transparent when its real in-line transmission is 60% or higher at wavelengths between 300 nm and 800 nm in samples that have a minimum thickness of 0.8 mm. In recent years, the definition has expanded to include materials that also allow light with wavelengths in the ultraviolet and infrared regions of the spectrum to pass through.

Several major segments by application for transparent ceramics include

- **Optics and optoelectronics**, used in lasers, bulbs, optical fibers, display panels, and more,
- **Aerospace and defense**, used in reconnaissance and sensor windows, infrared heat-seeking devices, aircraft blast shields, and more,
- **Security and protection**, used in armors for military vehicles, bulletproof sheets for civil automobiles, burglary-safe panels for construction projects, and more,
- **Sensors and instrumentation**, used in scintillators, infrared temperature sensors, medical equipment sensors, thermoluminescent dosimetry, and more,
- **Healthcare**, used in prostheses, skull implants, dental products, biological labeling, and more,
- **Consumer products**, used in home appliances, wearable devices, and more,

- **Other applications**, used in cutting tools, high-temperature parts, solar cells, and more.

The optics and optoelectronics sector currently represents the most important field of application for transparent ceramics, projected to grow at a CAGR of 19.5% to \$714.5 million by 2024.

Transparent ceramic materials for this market are segmented into six segments: oxides, fluorides, selenides, sulfides, nitrides, and mixed systems.

The aerospace and defense sector is characterized by relatively slow growth, but the demand could increase a little in the next few years as governments in the United States, China, and other countries allocate more resources to fight new threats and upgrade their defense systems.

The security and protection sector is projected to grow at the largest CAGR of 30% over the next five years to \$8.9 million by 2024. However, compared to glass and crystal, transparent ceramics will still account for only a little more than 0.1% of the security and protection market, which indicates potential strong growth beyond 2024 for magnesium aluminate spinel and aluminum oxynitride (the two most promising transparent ceramics for this sector).

The most popular application for transparent ceramics in the sensors and instrumentation sector is for the manufacture of scintillators. Scintillators are used to fabricate gamma-ray spectrometric and high-energy radiographic instruments, which are employed in healthcare applications, such as computed tomography and stationary digital imaging, and in oil and gas drilling.

The use of transparent ceramics in health products will expand with the help of advanced processing technologies. Ceramics are biocompatible, hard, and shatter-resistant, making them ideal

Table 1. Global market for transparent ceramics, by application, through 2024 (\$ millions)

Application	2018	2019	2024	CAGR% 2019–2024
Optics and optoelectronics	245.4	293.2	714.5	19.5
Aerospace and defense	18.9	20.6	31.8	9.1
Security and protection	2.1	2.4	8.9	30.0
Sensors and instrumentation	2.0	2.1	4.9	18.5
Healthcare	0.7	0.7	1.9	22.1
Consumer	0.4	0.4	1.1	22.4
Other	1.5	1.6	2.8	11.8
Total	271.0	321.0	765.9	19.0

for biomedical implants and protective casings for electronics.

Within the consumer sector, transparent ceramics are being developed for producing heat-resistant plates, shields, and windows for home appliances, such as cooktops, ovens, and barbecue grills. Transparent ceramics are also used in the fabrication of protective covers for wearable devices, such as wristwatches.

The category of other applications for transparent ceramics comprises uses in energy, mechanical, chemical, and other industrial sectors. Currently, the main application within this sector is represented by the fabrication of cutting tools and bearings. Other growing applications are in the production of windows for high-temperature furnaces, chemical processing equipment, and industrial blasting equipment, and for the fabrication of full-spectrum solar cells.

About the author

Jason Chen is a research analyst for BCC Research. Contact Chen analysts@bccresearch.com.

Resource

J. Chen, “Transparent ceramics: Emerging opportunities” BCC Research Report CHM149A, February 2020. www.bccresearch.com. ■

SOCIETY, DIVISION, SECTION, AND CHAPTER NEWS

Your voice counts

This summer ACerS will conduct an in-depth membership study designed to inform ACerS' strategic priorities and enhance member value. The survey will address interests related to membership, professional challenges, and current and emerging trends that impact the industry.

This is your chance to have a voice in shaping future priorities for the Society. All members are encouraged to share your feedback with us when you receive the survey. After all, ACerS is your society! ■

Volunteer Spotlight



ACerS Volunteer Spotlight profiles a member who demonstrates outstanding service to the Society.

Kristin Breder is a senior principal scientist and group leader at Saint-

Gobain Research North America. She has been with Saint-Gobain for 20 years. Her work includes mechanical characterization and failure analysis of ceramics, abrasives, and polymers as well as fundamental studies on abrasives, abrasive grains, and their relationship to grinding process.

Breder holds four patents related to abrasives and has contributed two book chapters on ceramics testing. She has 32 publications on ceramics and materials in peer-reviewed journals, 47 publications in conference proceedings, and has authored three ASTM standards on ceramics.

Breder has been a member (and then the chair) of the Membership Services Committee of ACerS since 2014. She has been a member of the awards selection committees for the John Jeppson and the Du-Co Young Professional awards. She is also a current member of the ACerS Strategic Planning Committee. From 2004 to 2006, she was an associate editor of the *International Journal of Applied Ceramic Technology (IJACT)*. She continues to be a reviewer for *IJACT* and the *Journal of the American Ceramic Society*.

We extend our deep appreciation to Breder for her service to our Society! ■

FACULTY SOUGHT FOR CENTRAL OHIO TECHNICAL COLLEGE TWO-YEAR CERAMIC ENGINEERING DEGREE PROGRAM

Responding to a critical need of the ceramic manufacturing industry, Central Ohio Technical College (Newark, Ohio), in partnership with the Edward Orton Jr. Ceramic Foundation and The America Ceramic Society, plans to establish a two-year associate's degree in ceramic materials engineering technology. COTC expects to welcome its first student cohort in fall 2020 pending approval from the Higher Learning Commission accrediting body.

COTC has opened a faculty search to lead the academic side of the program. "This is a great opportunity for someone who understands the needs of the industrial community and has a passion for working with young people," says Mark Mecklenborg, ACerS executive director.

The position will begin as a part-time appointment and is expected to increase to full-time as the program extends beyond the first cohort.

The part-time faculty member is responsible for providing professional, quality work as a teaching faculty member. Responsibilities include facilitating student learning by conducting and teaching lecture and lab courses at the undergraduate level, using active learning methods and a variety of instructional strategies designed to assist the learner in meeting the objectives of the program, assessing student learning, and providing academic assistance to students as needed.

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Society, Division, Section, and Chapter news (cont)

Names in the news



Ravichandran

Jayakanth Ravichandran, assistant professor of chemical engineering and materials science at the University of Southern California, has been recognized with a 2020 Young Leaders Professional Development Award from the Functional Materials Division of The Minerals, Metals and Materials Society (TMS). ■

Members—Would you like to be included in the Bulletin's Names in the News? Please send a current head shot along with the link to the article to mmartin@ceramics.org. The deadline is the 30th of each month. ■

ACerS has experts

We invite you to share your knowledge by participating as a presenter in the 2020 ACerS Webinar Series.

Some requested topics include: learning from failures in research; women in science: trials and tribulations; and the decision to pursue a Ph.D.

If you are interested in presenting on a topic mentioned above or you have levelled up to “expert” on a ceramic/glass topic, reach out to Yolanda Natividad at ynatividad@ceramics.org to be a potential webinar presenter in 2020. We ask that your proposed topic is educational and not promotional in nature.

Check out the webinars at ceramics.org/webinars that have been presented this year. ACerS members also may view recordings of past webinars as a benefit of your membership. ■

In memoriam

John (J.D.) Mackenzie
John T. Jones
John Clayton

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

www.ceramics.org/ceramictechtoday

AWARDS AND DEADLINES

Nominations close May 15 for three awards

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.

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 *Bulletin* during the previous calendar year on a subject related to electronic ceramics.

Electronics: Lewis C. Hoffman Scholarship

This scholarship recognizes academic interest and excellence among undergraduate students in the area of ceramics/materials science and engineering.

Award criteria and nomination forms can be found at ceramics.org/members/awards. Contact Erica Zimmerman at ezimmerman@ceramics.org if you have questions. ■

Nominations open for ECD awards

The **Engineering Ceramics Division** invites nominations for the 2021 James I. Mueller, Bridge Building, Global Young Investigator, and Jubilee Global Diversity awards. The deadline for submitting nominations for all four awards is **July 1, 2020**.

The Mueller Award recognizes the contributions of James I. Mueller to the Engineering Ceramics Division and to the field of engineering ceramics and the accomplishments of individuals who have made similar contributions. The award consists of a memorial plaque, certificate, and an honorarium of \$1,000. If you have questions, contact Surojit Gupta at gsurojit1@gmail.com.

The Bridge Building Award recognizes individuals outside of the United States who have made outstanding contributions to engineering ceramics. The award consists of a glass piece, certificate, and an honorarium of \$1,000. If you have questions, contact Valerie Wiesner at valerie.l.wiesner@nasa.gov.

The Global Young Investigator Award recognizes an outstanding scientist who is conducting research in academia, in industry, or at a government-funded laboratory. Candidates

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Awards and deadlines (cont)

must be ACerS members and 35 years of age or younger. The award consists of \$1,000, a glass piece, and certificate. If you have questions, contact Hisayuki Suematsu at suematsu@etigo.nagaokaut.ac.jp.

The Jubilee Global Diversity Award recognizes exceptional early- to mid-career professionals who are women and/or under-represented minorities (i.e., based on race, ethnicity, nationality, and/or geographic location) in the area of ceramic science and engineering. Three awards are given annually and consist of a certificate, complimentary registration, and \$500 honorarium to be presented during the plenary session of ICACC. The awardees present invited talks at ICACC. If you have questions, contact Michael Halbig at michael.c.halbig@nasa.gov.

For full criteria and nomination forms, visit <https://ceramics.org/acers-spotlight/nominations-open-for-ecd-mueller-bridge-building-and-global-young-investigator-awards-3>. ■

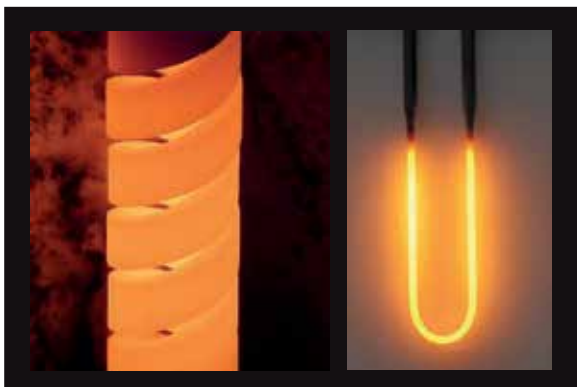
Announcing: The European Ceramic Society—American Ceramic Society Joint Award

The European Ceramic Society—American Ceramic Society Joint Award recognizes individuals who foster international cooperation between The American Ceramic Society and The European Ceramic Society, in demonstration of both organizations' commitment to work together to better serve the international ceramics community.

The award shall be presented in alternate years at the ACerS Annual Meeting (with Materials Science & Technology Conference) in even years and the European Ceramic Society Biennial Meeting in odd years. The award consists of \$1500 honorarium, registration for the event for one person, a certificate(s), and a piece of ceramic or glass artwork from the host society. The 2020 nomination deadline is **March 31, 2020**; in subsequent years it will be **January 15** annually.

For more information, please visit <https://ceramics.org/awards/the-european-ceramic-society-american-ceramic-society-joint-award>. ■

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Students and outreach (cont)

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MS&T announces discount rates for Material Advantage undergraduate students

MS&T has long been the home of Material Advantage students looking to present their research, compete for prizes, and network with other students and mentors.

The 2020 registration rate for undergraduate students is \$25 for Material Advantage members and \$55 for nonmembers. Graduate student registration rates are \$110 for members and \$140 for nonmembers. In addition, there will be several options for students to apply for a travel grant, including Material Advantage Chapter travel grants and more.

To view the technical program and more, go to www.matscitech.org/MST20. ■



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Students and outreach (cont)

ACerS GGRN for young researchers

Put yourself on the path toward post-graduate success with ACerS Global Graduate Researcher Network. GGRN addresses the professional and career development needs of graduate-level research students who have a primary interest in ceramics and glass.

GGRN aims to help graduate students

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- Build a network of peers and contacts within the ceramic and glass community, and
- Access professional development tools.

Are you a current graduate student who could benefit from additional networking within the ceramic and glass community? Visit www.ceramics.org/ggrn or contact Yolanda Natividad, ACerS membership engagement manager, at ynatividad@ceramics.org. ■

CERAMIC AND GLASS INDUSTRY FOUNDATION

ACerS and CGIF present Engineered Concrete design challenge to students

ACerS and The Ceramic and Glass Industry Foundation were sponsors and participants of STEMfest!, held at The Works in Newark, Ohio, on Saturday, Feb. 29 and Sunday, March 1, 2020.

STEMFest! is an annual STEM event that engages middle and high school students in design-based problem-solving challenges from a variety of disciplines that relate to what scientists and engineers encounter every day in their careers.

Students are encouraged to form a team, or work individually, to solve a real-world STEM problem developed by area businesses and organizations. Winners receive awards as well as potential scholarships and internships. The teams select one of three or four "Problem Solving Challenges" and present their designs and solutions to a panel of judges during the STEMfest! competition.

Students, their parents, and members of the public also had the chance to learn about STEM careers during the hands-on career

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displays and discussions portion of the event. CGIF staff members were on hand to perform some demonstrations from the Materials Science Classroom Kit, as well as discuss career pathways in the area of ceramics and glass science and engineering.

The 2020 Problem Solving Challenges were:

- **Materials Engineering—Engineered Concrete:** Sponsored by The American Ceramic Society and Central Ohio Technical College
- **Engineering Design—Bridge Architecture:** Presented by Newark City Engineers and Central Ohio Technical College (middle school only)
- **Environmental Impact Challenge—Pollution & Water Quality:** Presented by Denison University Department of Chemistry and Biochemistry
- **Thermal Energy Challenge—Home Insulation Efficiency:** Presented by Owens Corning Science and Technology Center

The Engineered Concrete design challenge, presented by ACerS and the CGIF was adapted from the lesson of the same name included in the Materials Science Classroom Kit that was originally developed by members of the PCSA. Participating design challenge



Students enjoying a Materials Science Classroom Kit demonstration.

Credit all images: ACerS

students received instructions and the materials for the competition in November 2019 and completed research and concrete design, mixing, and testing of multiple iterations of their products over the course of several months as preparation for the STEMfest! event.

ACerS and the CGIF wish to thank Milind Pawar, ACerS member and graduate student at The Ohio State University, for serving as a judge for the Engineered Concrete design challenge.

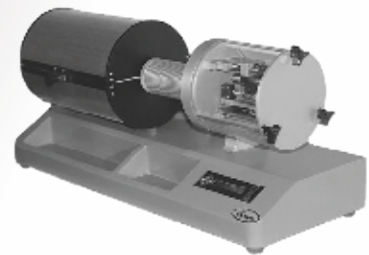
The STEMfest! design challenge program can be viewed at https://attheworks.org/wp-content/uploads/2019/12/STEMfest2020_HighSchool.pdf. ■

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advances in nanomaterials

Nanomechanical materials testing at over 2,000°C

Researchers at the University of Illinois at Urbana-Champaign (UIUC), Sandia National Laboratories, and the University of California, Davis, created a new method for conducting nanomechanical materials testing at ultrahigh temperatures.

Incorporating ultrahigh-temperature (UHT) materials in jet turbines is a main area of focus for aerospace engineers. And while progress has been made—engines containing UHT ceramics are now commercially available—a big challenge researchers face in bringing more UHT materials to commercialization is properly testing potential materials.

“A decade ago, advancements in aeronautical materials involved testing large, expensive models and years of development,” a UIUC press release explains. “Scientists and engineers now use microscale experimentation to help create new materials and understand the chemical and physical properties that lead to material failure.”

However, there is a hitch to microscale materials testing—researchers struggle to conduct these tests at the extreme temperatures experienced during flight.

“Unfortunately, it’s really difficult to perform experiments with new materials or combinations of existing materials at ultrahigh temperatures above 1,000°C because you run into the problem of destroying the testing mechanisms themselves,” Shen Dillon, professor of materials science and engineering at UIUC, says in the UIUC press release.

To overcome this problem, Dillon and his colleagues created a new ultrahigh-temperature testing method by combining targeted laser heating and transmission electron microscopy (TEM).

In an email, Dillon explains what gave them the idea to try combining these two common techniques.

“We have worked with Khalid Hattar’s group through CINT (Center for Integrated Nanomaterials) for a number of years. They installed the laser as part of a pump-probe system that they were planning to use for other experiments. He mentioned this to me during their install process because he knew that we were really interested in doing high temperature mechanical experiments,” Dillon says. “We started using it to test metallic samples at much lower temperatures soon after it was installed ... [but] we imagined that using samples or sample substrates with lower thermal conductivity, i.e. ceramics, would allow us to access much higher temperatures.”

Unlike other methods for heating materials, lasers are very good at heating a local area to extremely high temperatures while maintaining low temperatures in the neighboring regions. Additionally, by monitoring the experiments using TEM, the researchers could simultaneously observe the deformation mechanisms and determine the temperature at which the experiment took place.

Dillon says they were initially concerned about the high temperatures affecting the nanomechanical tester (a Bruker PI-95) even though the heating was localized, but some simple thermal modelling suggested that everything should work, so they went ahead with the experiment.

The researchers decided to test zirconium dioxide, a material often used in fuel cells and thermal barrier coatings, because Dillon's group was already collaborating with professor Ricardo Castro's group at UC Davis to characterize deformation of nanograin zirconia.

Testing was performed between 25°C and 2,050°C (77°F and 3,722°F), "a temperature well above anything that you could do previously," Dillon says in the press release. Overall, the experiments demonstrated how the laser-TEM technique "enables testing over a broad temperature range extended to the ultrahigh-temperature regime," the researchers write.

Dillon says they are just finishing another paper related to studying bicrystal Coble creep and sintering in zirconia using similar experimental methodologies. They also have an NSF CER-supported project that extends this approach to systematically investigate the effects of applied electric field on interfacial transport kinetics, thermodynamics, and mechanisms, and they have performed some preliminary experiments characterizing diffusion at heterophase boundaries in ceramic-ceramic composites.

"We envision this methodology impacting a range of applications, varying from the effects of irradiation on interfacial transport to the effects of interfacial creep in the development of metal-oxide scales," Dillon says.



Credit: Shen Dillon

University of Illinois at Urbana-Champaign professor Shen Dillon, left, and Sandia National Lab researcher Christopher Barr, right, with the new ultrahigh-temperature testing setup.

The paper, published in *Nano Letters*, is "In situ transmission electron microscopy for ultrahigh temperature mechanical testing of ZrO₂" (DOI: 10.1021/acs.nanolett.9b04205). ■

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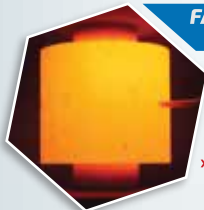
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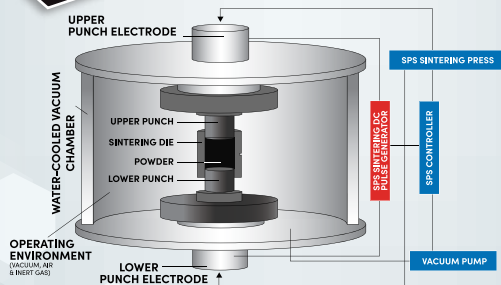
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research briefs

Atomic layer deposition offers advantages for preserving lumber

Researchers at the Georgia Institute of Technology found they could manage moisture content, mold growth, and thermal conductivity of lumber by treating it using atomic layer deposition (ALD).

ALD is a vapor phase technique for depositing a thin film onto a surface through self-limiting chemical reactions. "For wood-based products, a single-cycle ALD process is in fact very similar to current pressure treatments that are carried out in high pressure or vacuum chambers," the Georgia Tech researchers write in the paper on their research.

Georgia Tech assistant professor of materials science and engineering Mark Losego explains in an email that it is "pretty well known" that ALD of metal oxides on cellulosic materials leads to hydrophobicity, i.e., the property of being water repellent. So they were pretty sure the ALD process would make bulk wood lumber hydrophobic as well, "at least to some extent."

The researchers used a single-cycle ALD process (1cy-ALD) to infuse blocks of pine, cedar, or poplar with subnanometer layers of three fairly benign metal oxides: aluminum oxide, zinc oxide,



By treating wood using atomic layer deposition, Georgia Tech researchers found they could manage moisture content, mold growth, and thermal conductivity.

Credit: Georgia Tech, YouTube

Research News

New state-of-the-MOF materials

Researchers from Kyoto University and National Institute of Advanced Industrial Science and Technology in Japan reviewed the latest advancements and perspectives in the field of metal-organic frameworks (MOFs). Tens of thousands of MOFs have been synthesized since they were first discovered in the late 1990s. So far, researchers have reported about 10 MOFs that can be turned into a glass state. Some MOFs are transformed into glass by cooling their liquid state. Others require a mechanical grinding-like treatment for glass to form. These liquid and glass MOFs could provide a new state of material that demonstrates porosity, ion conductivity, and optical properties. For more information, visit <https://www.eurekaalert.org>. ■

and titanium oxide. Only one type of metal oxide was deposited on each block, so the efficacies could be tested separately.

To evaluate hydrophobicity, the researchers collected water contact angle measurements on the three types of wood species. Despite initial differences in hydrophilicity (attraction to water), “all lumber varieties show an increase in hydrophobicity after 1cy-ALD treatments,” the researchers write.

However, water contact angle measurements only really test the blocks’ surface properties. To more directly evaluate water repellency in the bulk of the wood, the researchers fully submerged the pine blocks in water to gravimetrically measure water uptake over time.

Of the three metal oxides, only titanium oxide prevented bulk uptake of water. In a Georgia Tech press release, graduate student and lead author Shawn Gregory explains why the titanium oxide provided the best water resistance. “We hypothesize that this is likely because of how the precursor chemicals for titanium dioxide react less readily with the pore surfaces and therefore have an easier time penetrating deep within the pores of the wood,” he says.

Losego says they were also surprised to find the TiO₂-coated pine demonstrated rot resistance. They realized this fact after leaving the blocks sitting in a humid environment for several months and witnessed no evidence of mold growth.

In the press release, Gregory says, “We suspect that this has something to do with its hydrophobic nature, although there could be other chemical effects associated with the new treatment process that could also be responsible. That’s something we would want to investigate in future research.”

There was one more important finding from the study—the treated wood blocks showed decreased thermal conductivity.

Perhaps surprisingly, Losego says thermal conductivity was the initial reason they were driven to investigate ALD of bulk wood. “We knew the thermal conductivity is affected by moisture content, so we thought that the ALD could help reduce thermal bridging in wood studs,” he says.

In the press release, Shannon Yee, Georgia Tech associate professor in mechanical engineering and study co-author, says

Unique material could unlock new functionality in semiconductors

Rensselaer Polytechnic Institute researchers synthesized an organic-inorganic hybrid crystal made up of carbon, iodine, and lead, and then they demonstrated it was capable of two material properties previously unseen in a single material. It exhibited spontaneous electric polarization that can be reversed when exposed to an electric field (i.e., ferroelectricity). It simultaneously displayed a type of asymmetry known as chirality. The researchers say this unique combination of ferroelectricity and chirality, when combined with the material’s conductivity, can enable other electrical, magnetic, or optical properties. For more information, visit <https://news.rpi.edu>. ■

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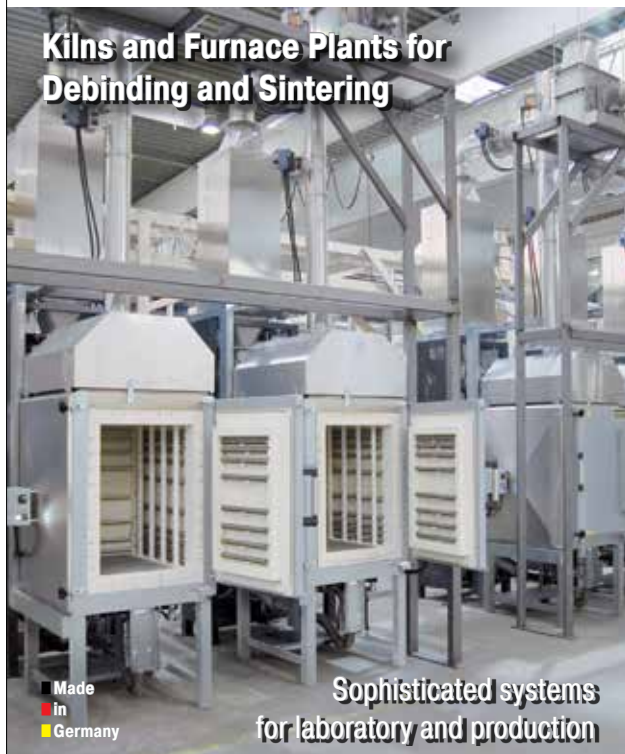
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“Lumber treated with this new process [ALD] can be up to 30 percent less conductive, which could translate to a savings of as much as 2 million BTUs of energy per dwelling per year.”

The paper, published in *Langmuir*, is “Single-cycle atomic layer deposition on bulk wood lumber for managing moisture content, mold growth, and thermal conductivity” (DOI: 10.1021/acs.langmuir.9b03273). ■

The origin of self-affine roughness

In a recent study led by researchers from the University of Freiburg (Germany), they look to understand one aspect of roughness—its self-affinity.

Self-affinity describes a fractal whose pieces are scaled by different amounts depending on the axis. In the paper, the researchers note that rough surfaces are often self-affine fractals, something that has been observed from the atomic to the tectonic scale. This observation is interesting because it means rough surfaces have patterns in their bumpy contours rather than random jagged ups and downs.

There currently is no unifying explanation for the origins of this self-affinity. But “[the] fact that scale-invariant roughness is observed from microscopic to geological scales hints that a common mechanism is active across vastly different length scales,” the researchers write.

For their study, the researchers investigated self-affinity by looking at roughness on a small scale. Macroscale surface changes, “whether natural or engineered, involve mechanical deformation at the smallest scales,” so understanding small-scale roughness will shed light on macroscale roughness.

The researchers used molecular dynamics calculations to simulate simple biaxial compression for three benchmark material systems: single-crystal gold, the model high-entropy alloy $\text{Ni}_{36.67}\text{Co}_{30}\text{Fe}_{16.67}\text{Ti}_{16.67}$, and amorphous $\text{Cu}_{50}\text{Zr}_{50}$.

Research News

Tickling an atom to investigate atomic impurities in nanomaterials

Researchers led by the University of Leeds (U.K.), in collaboration with colleagues at the Sorbonne University in Paris, France, showed it is possible to develop a diagnostic technique for single atoms that is loosely related to the idea of a tuning fork. It involves firing a beam of electrons at a single atom in a solid, which causes the atom and the atoms that surround it to vibrate. This movement creates a unique vibrational energy fingerprint, akin to the fixed tone from a tuning fork, which can be recorded by an electron microscope. But if a single atom impurity is present, such as another chemical element, the vibrational energy fingerprint of that impurity will change, i.e., the material will “sound” different at this precise location. For more information, visit <https://scitechdaily.com>. ■



Credit: Ivan Radic, Flickr (CC BY 2.0)

Roughness plays a big role in determining friction and adhesion between materials, which greatly affects processes in both scientific and industrial fields. So understanding roughness can help control these factors.

“Each material represents a unique limit of structural order: a homogeneous crystal, a crystal with stoichiometric disorder, and a glass with no long-range order,” they explain. “They are known to exhibit a different micromechanical or molecular mechanism of deformation ... [but] despite their differences in structure and material properties, all three systems develop rough surfaces with a self-affine surface topography when compressed.”

Based on their results, the researchers conclude that the emergence of self-affine roughness at small scales is not due to a specific deformation mechanism. Instead, it is likely due to the type of deformation taking place.

“[The] statistical nature of plasticity appears to be the principal reason that surfaces develop self-affine roughness during deformation,” they write. That is, plastic flow deformation occurs through intermittent strain bursts. When the researchers carried out continuum mechanical calculations on systems that deformed via smooth laminar flow, self-affinity did not emerge.

In the conclusion, the researchers speculate that similar results may hold for deformation processes occurring at much larger scales, as long as deformation occurs in a discrete manner rather than as a smooth, continuous flow.

“Our results pave the way for a thorough understanding and control of surface roughness created in a variety of processes, such as machining or wear,” they conclude.

The paper, published in *Science Advances*, is “The emergence of small-scale self-affine surface roughness from deformation” (DOI: 10.1126/sciadv.aax0847). ■



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Coronavirus shapes the global ceramic tile industry

From ghost flights to quarantined cruise ships, the effects of the rapidly-spreading novel coronavirus on the travel industry are overwhelmingly apparent to anyone watching the news. But the coronavirus's effects on other aspects of society are just as significant, affecting everything from science conferences and sports festivals to global financial markets.

There are countless ways the coronavirus affects the global ceramic and glass communities. But one ceramic market in particular is experiencing a noticeable shift due to the virus—the global ceramic tile industry.

China is the world leader in manufacturing ceramic tile. In 2018, the country produced roughly 5.7 billion square meters of ceramic tile, according to a Statista article. Compare that to the 13.1 billion square meters produced globally, as stated in a recent ACIMAC Research Department report, and it is evident China produced more than 40% of all ceramic tiles manufactured that year.

India comes in a distant second to China, as the Statista article says the



Workers shape a ceramic tile at a manufacturing plant in Morbi, Gujarat, India.

country produces only about 1.15 billion square meters in 2018. But the coronavirus presents India with a chance to close that gap.

Morbi, a region in the Indian state of Gujarat, alone accounts for about 90% of the total production of ceramic products in India, as stated in a Messe München India and EAC International Consulting report. Last year saw difficulties for tile makers in Morbi, including more than 400 of the 550 wall tile plants

closing due to a coal gasifier ban and a drop in production due to reduced domestic demand caused by a sluggish real estate market and poor retail sales.

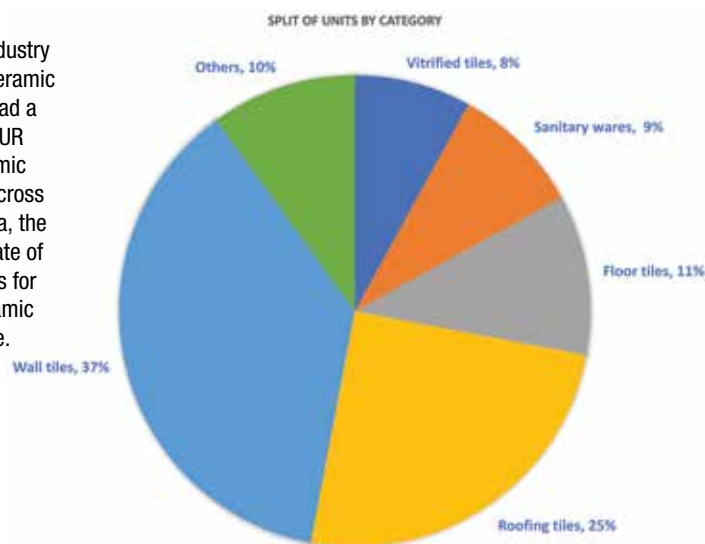
Over the past couple months, however, exports from China stopped as the country focused on tackling the coronavirus—which gave tile makers in Morbi an opening.

“We are expecting a 10% increase in exports from Morbi as result of current crisis in China. Some of the tile makers have already started exporting to Europe and African countries,” says Nilesh Jetpariya, president of Morbi Ceramic Association, in a *Financial Express* article.

If the coronavirus continues to affect Chinese exports for a prolonged time, however, India may face shortages in raw materials, in particular the abrasives needed for polishing vitrified tiles. If that happens, “ceramic units will have to import from Spain, which would be costlier,” says Dinesh Sadsania, a leading exporter of ceramic products from Morbi, in the *Financial Express* article. ■

Ceramics in Morbi

The Indian ceramic industry is dominated by the ceramic tiles industry, which had a market of 4.9 billion EUR in 2017. Though ceramic clusters are present across different states in India, the Morbi region in the state of Gujarat alone accounts for about 90% of the ceramic products market share.



Credit: Data from Messe München India and EAC International Consulting report “Status quo and outlook 2022: Indian ceramics industry.” Accessed 11 March 2020 from https://www.indian-ceramics.com/wp-content/uploads/2018/10/Ceramics_Industry_Report.pdf

ceramics in energy

Perovskite films easily healed with moderate compression or heat

Brown University researchers found cracks in perovskite films are easily healed by applying compression or moderate heat.

“In materials science, things that are easy to make also tend to be easy to break,” says Nitin Padture, the Otis E. Randall University Professor and Director of the Institute for Molecular and Nanoscale Innovation at Brown University, in a Brown press release. “That’s certainly true of perovskites, which are quite brittle.”

However, “that means you can also fix them easily!” he adds in an email.

Padture and colleagues looked to heal cracks in organic-inorganic halide perovskites (OIHPs) using two simple techniques—moderate compression or heat.

They used two typical OIHPs for the study: methylammonium lead triiodide ($\text{CH}_3\text{NH}_3\text{PbI}_3$ or MAPbI_3) and formamidinium lead triiodide ($\alpha\text{-HC}(\text{NH}_2)_2\text{PbI}_3$ or $\alpha\text{-FAPbI}_3$).

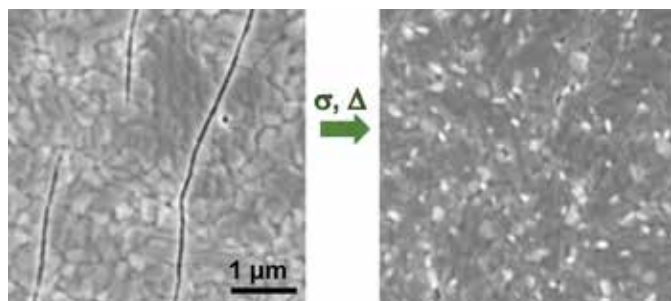
To test the viability of mechanically healing perovskite thin films, the researchers wrapped OIHP thin films on substrates around a glass mandrel. The applied stress was either tensile (causing the film to crack) or compressive (causing, hopefully, the film to heal) depending on how they wrapped the film around the mandrel.

The researchers also tested the viability of thermal healing by heat-treating MAPbI_3 and $\alpha\text{-FAPbI}_3$ thin films at 100°C for 5 minutes and 140°C for 10 minutes, respectively.

Using X-ray diffraction, the researchers confirmed that compressive stress at room temperature or heat treatment at moderate temperatures healed cracks in the thin films on a time-dependent basis.

Padture says this proof-of-concept study lays the groundwork to move beyond thin films and investigate healing perovskite solar cells with these techniques. In addition, Padture says what they learned about cracks in this study will help them investigate fracture at the interface between the films and substrate.

The paper, published in *Acta Materialia*, is “Facile healing of cracks in organic-inorganic halide perovskite thin films” (DOI: 10.1016/j.actamat.2020.01.040). ■



With just some compression (σ) or a little heat, a cracked perovskite film (left) can be fully healed (right).

Credit: Padture Lab, Brown University

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Corning DuraTrap® GC gasoline particulate filter (foreground) and a Corning FLORA® low-mass substrate for catalytic converters (background).

Smog begone! How development of ceramic automotive catalytic substrates and filters helped reduce air pollution

Credit: Corning Incorporated.

By Douglas M. Beall and Willard A. Cutler

Ceramic-based mobile emissions control products have prevented billions of tons of hydrocarbons, nitrogen oxides, carbon monoxide, and particulates from entering the atmosphere—and researchers continue to innovate to make these products even better.

Deep in the heart of your car's exhaust system—withstanding temperatures of more than 1,800 degrees Fahrenheit and staying tough over hundreds of thousands of miles of bumpy roads—a highly engineered ceramic material is an important part of helping to prevent harmful emissions from escaping into the air you breathe.

By keeping the air cleaner, these same ceramic products help save as many as 160,000 lives each year and help to prevent just as many cases of heart disease and asthma.¹

From 1975 through today, light and heavy-duty vehicle emissions dropped by an astounding 99%. Ceramic-based mobile emissions control products prevented more than 4 billion tons of hydrocarbons, 4 billion tons of nitrogen oxides (NO_x), and 40 billion tons of carbon monoxide from entering the atmosphere.

The clean-air movement brought economic benefits as well. Emissions-related technologies represent about \$37 billion in annual economic activity, with a significant portion of the industry involving ceramic-based components.²

Capsule summary

A DIRTY PROBLEM

By the mid-1900s, health hazards associated with poor air quality had come into stark view. Governments set new federal air pollution regulations that presented both a technological challenge and business opportunity for industry manufacturers.

CERAMIC SOLUTION

In the 1970s, Corning scientists invented a substrate for catalytic converters based on cordierite and also a ceramic-based wall-flow particulate filter for diesel engines. Nowadays, ceramic substrates and filters are the keystone of mobile pollution control ecosystems.

INNOVATION AHEAD

Most emissions in a typical drive cycle are produced in the first minutes of operation, so the remaining frontier of a zero-emission internal combustion vehicle is tackling this first minute of operation.

Innovations in ceramics will continue to be needed for at least the next couple of decades. Consumers throughout the world, not only in areas with untreated mobile pollution sources, are demanding better air quality. Areas of focus that continue to drive ceramic innovations include

(1) Ensuring that real-world emissions are as low as emissions on the certification test;

(2) Gaseous emission limits reducing, particularly NO_x emissions, while supporting the reduction of greenhouse gases; and

(3) Reducing the last remaining bursts of gaseous and particulate emissions from internal combustion engines, the emissions that occur during the first minute or two of vehicle operation—an event that can happen several times during a drive with hybrid vehicles.

A growing dirty problem

By the late-1940s, industrial activity in the United States had grown for nearly a century. Little attention was paid to the consequences of smoke and pollutants from coal-burning factories. Suburbs were growing. More and more families could afford cars. With city traffic crowding new highways, unprecedented levels of harmful chemicals flowed into the air, especially in major population centers—both in the U.S. and in other developed countries.

The health hazards associated with poor air quality came into stark global view in London in late 1952, when airborne pollutants, mostly arising from the use of coal, mixed with fog to form a thick blanket of smog over the city. Historical accounts vary on the details, but most agree between 4,000 and 6,000 people died over the course of five days from the choking effects of the Great Smog.^{3,4}

The following year, New York City was covered with a toxic mix of sulfur dioxide and carbon monoxide that blanketed the city. During one week, according to reports, as many as 260 deaths were attributed to the smog. More deadly smog crises would hit both New York and London again over the following decades.⁵

With the public health effects of air pollution now beyond dispute, the U.S.

began its first attempt to legislate a solution with the Air Pollution Control Act of 1955, which focused on research and information, leaving the states to devise ways to deal with polluters. A few states, notably California, had a plan to combat the issue.

The U.S. government made several more attempts to regulate sources of air pollution. However, the Clean Air Act of 1963, the Motor Vehicle Air Pollution

Control Act of 1965, and the Air Quality Act of 1967 all lacked the teeth to make a difference in the deadly problem.

But in 1970, sweeping new federal regulations required clean-air compliance from every segment of industry. The new Environmental Protection Agency (EPA) was formed with the mandate to enforce the regulations.

The Clean Air Act of 1970 established air-quality standards that strictly

Clean Air Act of 1970

When it comes to clean air in the United States, one of the most important pieces of legislation that made it possible is the Clean Air Act (CAA) of 1970.

Prior to CAA 1970, there were a few federal acts involving air pollution.¹ The first was the Air Pollution Control Act of 1955, which provided funds for federal research on air pollution. This was followed by CAA 1963, which established a federal program within the U.S. Public Health Service and authorized research into techniques for monitoring and controlling air pollution. In 1967, the Air Quality Act was enacted to expand federal government activities, including conducting the first-ever extensive ambient monitoring studies and stationary source inspections.

However, CAA 1970 marked a major shift in the federal government's role in air pollution control by substantially expanding the government's enforcement authority.¹ It authorized development of comprehensive federal and state regulations to limit emissions from both stationary (industrial) sources and mobile sources. In particular, it established four major regulatory programs affecting stationary sources:

- National Ambient Air Quality Standards (NAAQS)
- State Implementation Plans (SIPs)
- New Source Performance Standards (NSPS)
- National Emission Standards for Hazardous Air Pollutants (NESHAPs)

In 1977 and 1990, two sets of major amendments were added to CAA 1970.¹ The 1977 amendments focused on ensuring attainment and maintenance of NAAQS by establishing major permit review requirements; the 1990 amendments substantially increased the federal government's authority and responsibility.

To date, several in-depth benefit-cost analyses of the CAA Amendments performed by the Environmental Protection Agency all show

extreme benefits thanks to this legislation. For example, a 2011 EPA study² estimated the central benefits exceed costs by a factor of more than 30 to one, largely due to reductions in premature mortality associated with reductions in ambient particulate matter.

However, the gains in clean air made thanks to CAA 1970 and its amendments may be challenged in the future. The Trump administration has pursued the rollback of almost 100 environmental rules, 58 of which are now completed. Of these completed rollbacks, 16 involve air pollution and emissions rules.³

Recent studies also show air quality improvement is leveling off. A 2018 report⁴ by the U.S. PIRG Education Fund and Environment America Research & Policy Center found 2018 had more days of pollution than each of the previous five years, a finding supported by studies by the American Lung Association⁵ and Carnegie Mellon University.⁶

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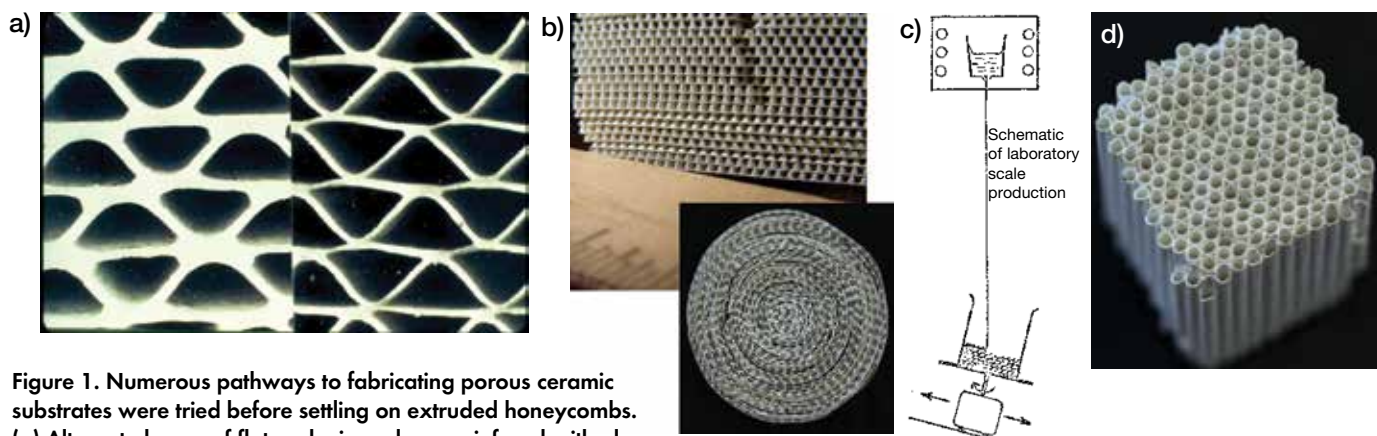


Figure 1. Numerous pathways to fabricating porous ceramic substrates were tried before settling on extruded honeycombs. (a) Alternate layers of flat and crimped paper infused with glass-ceramic (CERCOR material); (b) layers of cordierite glass-ceramic sheets with small “nubbins” to increase the surface area and permit exhaust flow; (c) open, layered structure similar to ribbon candy formed by buckling a hot glass stream and later ceraming the part; and (d) glass tubing fused together, then ceramed, looking like a packet of hollow cigarettes.

limited levels of six pollutants that threatened public health: sulfur dioxide, nitrogen dioxide, particulate matter, carbon monoxide, ozone, and lead.⁶ (See “Clean Air Act of 1970”)

For automakers, the government’s marching orders were clear: design vehicles that could run on unleaded gasoline and incorporate a new device—the catalytic converter—to reduce carbon monoxide and hydrocarbons by 90% from car exhaust. By 1975, all American-made vehicles were required to meet the new emissions requirements—no exceptions. European countries implemented similar regulations in 1992.

A technical challenge, a business opportunity

Around the same time in 1970, Corning was bringing its materials science expertise to General Motors with the idea of a lightweight, chemically strengthened glass windshield made on its new fusion-draw process. GM had little interest in Corning’s windshield glass, as the recently developed Pilkington’s float process was more economical. While at GM, Corning president Tom MacAvoy showed GM president Ed Cole a sample of a unique glass-ceramic material called CERCOR®. Light and highly resistant to heat, Corning envisioned marketing CERCOR material as a heat exchanger for gas turbine engines. Cole told MacAvoy that the industry was moving away from turbine engines, but

he liked Corning’s ingenuity. He urged the company to investigate the substrate opportunity for catalytic converters.

GM and other automakers had settled on internal combustion engines and the catalytic converter, first patented by French-born mechanical engineer Eugene Houdry in 1955, as the solution for reducing harmful engine emissions. The converter required unleaded gasoline and a durable substrate with low resistance to flow that also provided a lot of surface area, allowing the exhaust to pass over the platinum group catalysts supported on high-surface-area gamma-alumina.

Many capable people were trying to solve the problem, including those at GM, 3M, W.R. Grace, Engelhard, Johnson Matthey, and more. But as of 1970, no clear winning substrate technology emerged. Corning chief technology officer Bill Armistead responded to MacAvoy’s challenge by launching an internal emissions control (EMCON) project and directed significant funding into its R&D efforts.

Advanced glass was Corning’s best-known specialty, but the company’s skill in ceramics dated back to the late 19th century, when engineers designed and created durable ceramic crucibles for melting glass. In 1920, the company formed a ceramics research group and soon began making ceramic refractory bricks with extreme chemical durability. The bricks were ideal for lining the con-

tinuously operating melting tanks used to mass-produce light bulbs as well as one of the company’s newest products: PYREX kitchen ware.

Corning approached the emissions-control project by researching a wide variety of designs and materials. Under consideration, for example, were (Figure 1)

- Alternate layers of flat and crimped paper infused with CERCOR glass-ceramic material;
- Layers of cordierite glass-ceramic sheets with small “nubbins” to increase the surface area and permit exhaust flow;
- An open, layered structure, not unlike ribbon candy, formed by buckling a hot glass stream and later ceraming (i.e., heat treating to induce crystallization of the glass) the part; and
- Glass tubing fused together, then ceramed, looking like a packet of hollow cigarettes.

Competitors were testing potential solutions just as wide-ranging.

• American Lava Corporation (a subsidiary of 3M) developed a way to alternate layers of flat and corrugated ceramic-impregnated paper, which was then slowly fired. The design used zircon-mullite and cordierite-mullite compositions.

• W.R. Grace devised a ceramic powder-filled plasticized polyolefin sheet with ribs. The sheet was rolled and heat-sealed, providing parallel airflow paths. The polymers were burned off to form the final product.



Figure 2. Results from the first extrusion of cellular ceramic (left), and drawings from the patented idea filed in November 1971. First tested in July and first scaled in October 1971.

- General Motors' in-house team used a packed bed of catalyst-coated ceramic beads rather than a structured ceramic.

Technically, several of the designs worked. But the question of manufacturing—specifically, how to produce millions of substrates per year, at a low cost—was most challenging.

A breakthrough solution

The tide turned in July of 1971 when two young Corning scientists—Ed Bush and Rod Bagley—were in a meeting with a colleague at the company's research campus, Sullivan Park. Bagley described an idea he had mulled over—using extrusion to make a cellular substrate structure.

He sketched his idea on blackboard. The original design had offset slots in both sides, but it was not clear to the others how it would work. To clarify the concept, he ran to the mason's shop and grabbed a soft refractory brick and used a diamond saw to make a 3D model, further demonstrating the concept.

The first practical embodiment was a custom-made brass prototype die about 25 mm in diameter, which produced parts with 50 cells per square inch and cellular walls roughly 0.5 mm thick (Figure 2). Bagley extruded alumina through the brass die to test the idea.

Meanwhile, scientist Irwin Lachman was developing a cordierite-mullite composition with a remarkably low coefficient of thermal expansion (CTE)—a material that could remain stable and functional despite the repeated extreme

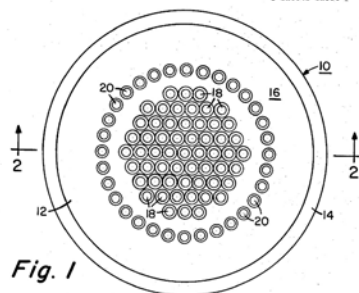


Fig. 1

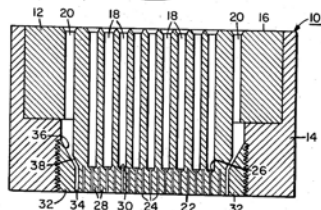


Fig. 2

INVENTOR
Rodney D. Bagley

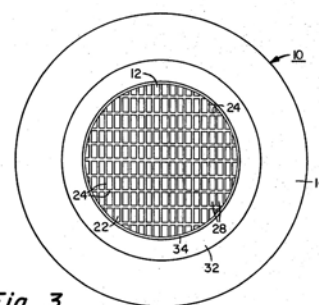


Fig. 3

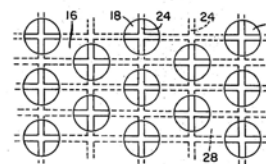


Fig. 4

INVENTOR
Rodney D. Bagley

Credit: Beal and Cutler

heating and cooling experienced within a car's powertrain system. Lachman worked with Ron Lewis to improve the material. The resulting synthetic cordierite not only had very high temperature capabilities ($T_m > 1,400^\circ\text{C}$) but also great thermal shock resistance.

The first successful 4.66-inch (118 mm) diameter extrusion of cordierite was in 1971 (this diameter is still a common diameter for automotive exhaust components). Corning branded the new ceramic substrate as Celcor[®] and received its first order—from Ford Motor Company—by the end of 1971.

It worked so well that Corning—even as it was financially challenged in the increasingly global color TV glass market—shelved other potential substrate solutions and quickly invested \$25 million into a new environmental factory in Erwin, N.Y. This Corning factory depreciated over five years, as car companies claimed they would improve their engines so that catalytic converters would not be needed in the future. Instead, the market continued to grow, and the factory is still operational today.

What's special about cordierite?

Naturally occurring cordierite is a mineral compound containing magnesium, iron, aluminum, and silicon. It is found, among other places, near veins of tin in the mines of Southern England. It draws its name from French geologist Louis Cordier, who included the mineral in a much-celebrated geological gallery at the National Museum of Natural

History in Paris in 1813.

The synthetic version of cordierite that Corning scientists created included no iron and contained magnesium, aluminum, and silicon ($\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$), creating a new material for emissions-control products.

Irwin Lachman was working with cordierite for other applications at the time of Bagley's invention of the die. Lachman considered cordierite an attractive choice of material for several reasons.

First, the application required a very high level of thermal shock resistance, and cordierite was known to have a very low CTE. Second, cordierite had good high-temperature stability and therefore could survive even the highest temperatures that would be encountered in the application, and it also had good chemical stability in the environment that would be encountered in the vehicle exhaust. Third, cordierite could be synthesized from relatively inexpensive and commonly available batch materials such as talc ($\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$), kaolinite clay ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), and gibbsite ($\text{Al}(\text{OH})_3$) or corundum (Al_2O_3).

Lachman combined these raw materials along with a methyl cellulose binder and water to produce a plastic mass with a putty-like consistency, which could be pushed easily through Bagley's extrusion die to produce the honeycomb structure. The extruded honeycomb parts were then dried to remove the water and fired to a high temperature, allowing the raw materials to react together to form the cordierite phase.

Smog begone! How development of ceramic automotive catalytic substrates and . . .

Lachman found that, after firing, the raw materials converted to over 95% cordierite phase. The fired ceramic honeycomb was porous, containing about 30 vol% of porosity in the walls. The presence of the porosity proved to be advantageous compared to a dense ceramic because the pores in the ceramic walls allowed the washcoat containing the catalyst to be slip casted onto the walls. The porosity also fortuitously decreased the heat capacity of the honeycomb relative to a dense ceramic, allowing it to heat up faster in use to the temperature where the catalyst became active.

Furthermore, the surface pores served as anchor points for the high surface area washcoat and catalyst, increasing the adhesion capability of the catalyst in the harsh environment of thermal cycling and mechanical vibrations that would be encountered when in the vehicle.

One of the most interesting findings was that dilatometric measurements of the synthetic cordierite honeycomb structure showed the bulk CTE was *lower* than expected based on what they knew about the crystal structure of mineral cordierite (also known as iolite) from X-ray diffraction data describing the lattice expansion.

The thermal expansion of cordierite is anisotropic with a negative thermal expansion in the *c*-axis of the crystal and positive expansion in the *a* and *b* axes of the cyclosilicate. The average

of the expansion coefficients in the three directions is $1.8 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ (from 25°C – 800°C), which is a very low value relative to most ceramic materials. However, measurements of the thermal expansion of the cordierite honeycombs consistently showed expansion coefficients of half that value or even lower.

Researchers also found their choice of raw materials and firing cycle could change the amount of suppression of the thermal expansion. Analysis of the microstructure of the ceramic walls of the substrates revealed the reasons for the suppression of the bulk thermal expansion. Ronald Lewis discovered the cordierite crystallites had a preferred orientation, with a majority of the crystallites oriented with the negative expansion *c*-axis lying within the plane of the ceramic walls. Therefore, the thermal expansion coefficient within the plane of the ceramic walls was depressed, relative to the average lattice expansion value.

Lewis and Lachman determined the preferred orientation resulted from the position of the platy silicate raw materials during the extrusion process as the materials passed through the thin slots in the extrusion die. This discovery led to the granting of a U.S. patent to Lachman and Lewis for an anisotropic cordierite monolith with designed preferred orientation and low bulk thermal expansion coefficient.⁷

Ed Bush discovered the CTE hysteresis sometimes observed in this material was due to microcracking, which could be intentionally induced to become engineered expansion joints (serving a similar function to expansion joints on a bridge), further lowering the CTE of the structure. Control over the size and density of stable engineered expansion joints in the matrix is important. The combined impacts were found to be capable of reducing the thermal expansion coefficient of the cordierite honeycomb by up to an order of magnitude or more (compared to the average crystallographic value), which is important to creating the severe thermal shock resistance required for this application.

Ceramic flow-through substrates

The first commercial ceramic substrates were low cell density (about 200 cells/in²) with thicker walls (about 12 mil or 0.012" or 0.3 mm) with a substrate volume about four times that of engine displacement (i.e., cylinder volume of the engine). As material and processing technology progressed, higher cell densities, thinner walls, and higher porosities became possible.

For historical reasons, substrates are commonly referred to by their cell density/wall thickness moniker. For example, a 400/4 substrate is one in which the "400" defines the cell density in cells/in² (or cpsi) and the "4" defines the nominal

wall thickness in 0.001" increments (or mils). In the average U.S. gasoline engine sedan, there are two or three substrates at work to meet the rigorous U.S. gaseous emissions standards. Right off the engine, close-coupled substrate(s) with high cell density (750/2 or 900/2) provide a lot of geometric surface area to allow the catalyst to do the initial gaseous conversions. In the underfloor position there is generally a lower cell density substrate like 400/4, to help clean up final emissions. Table 1 describes common macro

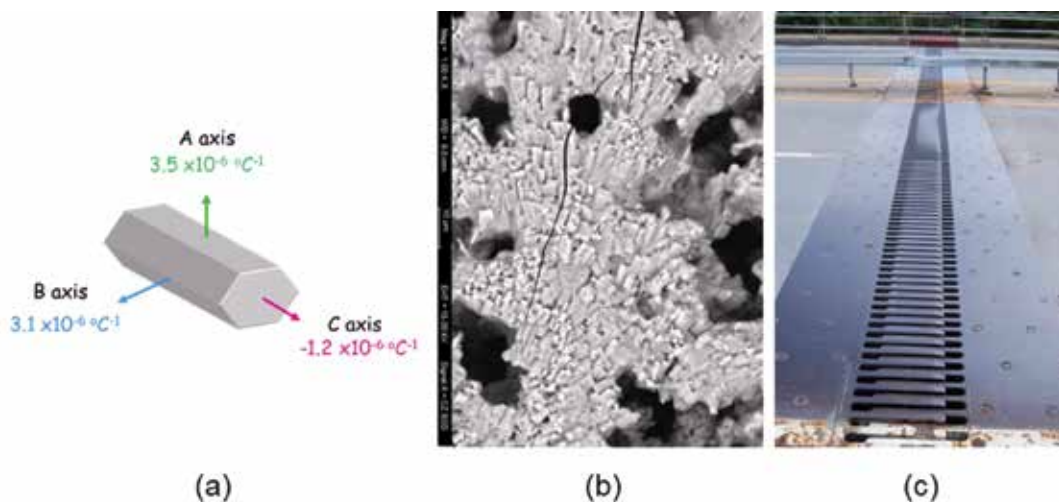


Figure 3. (a) Schematic of orthorhombic cordierite crystallite showing anisotropy in thermal expansion. (b) SEM micrograph of the web surface of a cordierite honeycomb showing orientation of individual cordierite crystallites and microcracks that act as engineered expansion joints. Dark areas are pores. Width of view is approximately 80 μm . (c) Expansion joint in a bridge, which allows for thermal expansion of the bridge components, but the length of the bridge itself remains constant.

and micro properties of ceramic substrates and filters.

Development of particulate filters

The flow-through honeycomb substrate was the ideal platform for supporting catalysts that eliminate harmful gaseous air pollutants but did little to remove harmful particulates from exhaust. Particulates in exhaust are often the result of incompletely combusted fuel and influenced by varied factors including, but not limited to, ambient temperature, altitude, fuel quality, vehicle power-to-weight ratio, drive cycle, and engine hardware and software. The World Health Organization (WHO) cautions that microscopic carbon particles, when inhaled, “can penetrate the lung barrier and enter the blood system. Chronic exposure to particles contributes to the risk of developing cardiovascular and respiratory diseases, as well as of lung cancer.”⁸

In the late 1970s, an aluminum manufacturer asked Corning if they had a product that could be used to filter impurities from molten aluminum. Corning scientist Rod Frost, who had led the development of the process for producing the ceramic honeycomb substrate, conceived of the wall-flow particulate filter as a possible solution for this application.

As sometimes happens in R&D, the concept did not work for this particular application. However, months later, diesel engine manufacturers came to Corning looking for a con-

cept to filter soot particles from diesel exhaust for diesel engines running in confined spaces, like mining vehicles. Rod thought his ceramic-based wall-flow concept might work better for this application. He had some prototypes made and testing proved his design worked very well.

Corning DuraTrap filters are still produced with the Frost design, as are almost all other particulate filters. These filters have a cellular honeycomb ceramic with engineered wall porosity to capture fine particles. Individual channels are open and plugged at alternating ends, like a checkerboard. Exhaust gases enter the open (inlet) channels, flow down the channel, and escape only through the engineered porosity of the cellular walls. The walls offer little flow resistance and particles become trapped in the porosity and collect on the filter walls instead of being released into the atmosphere. The cleaned gas exits the filter through the adjoining (outlet) channels. Filters can be used in their bare state, or in conjunction with catalysts to assist in gaseous-emissions reduction, or to aid soot burning.

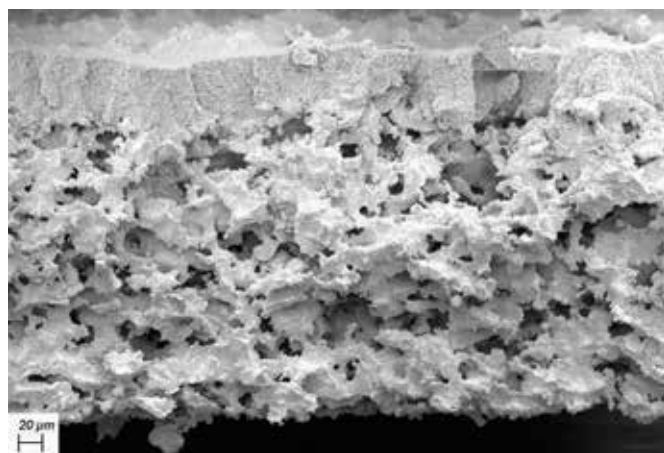


Figure 4. Pictured is a cross-section of a cell wall of a diesel particulate filter, showing an accumulated soot layer at 200x on top of the porous ceramic wall.

Diesel engines often produce a lot of soot particles, from both a particle number and a mass perspective. This particle output, combined with low engine-out temperatures, often results in a soot cake on the inlet wall surfaces of the wall-flow diesel particulate filter (DPF). The porous deposit of nano to submicron soot particles on the filter wall can increase the native filtration efficiency (Figure 4). The same mechanism operates in gasoline particulate filter (GPF) applications, where the number of particles in the exhaust can be high but the particles have less mass. Collecting a soot cake in gasoline applications is more difficult and typically does less to aid filtration.

In both diesel and gasoline applications, the captured particles remain in the filter until exhaust conditions are appropriate to burn the particles to “clean” or “regenerate” the filter. Regenerating wall-flow filters can take place passively (as a by-product of the time/temperature/atmosphere) or actively (triggered by additional sensors and software). For example, in the diesel case, active regeneration increases the filter inlet temperature in the presence of the appropriate oxygen-containing species to burn the combustible particles, returning the filter to its nearly clean state. Filters generally last the life of the vehicle.

The selection of filter materials depends on the filtration efficiency requirements (microstructure) and the heat capacity requirements (material choice, porosity, cell density/wall thickness). For gasoline

Table 1. Flow-through ceramic substrates for automobiles through heavy duty vehicles

	Common	Possible	Comments
Macro properties			
Cell density (cpsi)	200-900	100-3000	>900 cpsi - very high pressure drop (Δp)
Wall thickness (mil)	2-8	2-17	< 2 mil carries significant cost and isostatic strength implications
Cell shape	square hexagonal	asymmetric triangular rectangular round	Cell shape is often balanced with cost, isostatic strength, geometric surface area and Δp
Isostatic strength (bar)	7-10.5	5-15	Usually important for canning strength
Diameter (mm)	~101-330 (4-13")	~50-610 (2-24")	Larger diameters can be available as assemblies of smaller blocks
Length (mm)	~76-300 (3-12")	~50-600 (2-24")	Length can be dictated by Δp and sintering dimensional control implications
CTE ($\times 10^{-6}/^{\circ}\text{C}$, RT to 800 $^{\circ}\text{C}$)	0.05-1.0	0-50	Very low coefficient of thermal expansion (CTE), enables thermal shock resistant structures
Wall porosity (%)	25-55	5-70	More porosity leads to lower mass, but also potentially lower strength
Mean pore size (μm)	2-20	0.5-30	Engineered porosity is more important for filters than flow through substrates

Smog begone! How development of ceramic automotive catalytic substrates and . . .

vehicles and many heavy-duty diesel vehicles, where passive regeneration dominates, cordierite is the material of choice due to its low cost, low heat capacity, high-melting-point, low CTE, and low-thermal-conductivity with macrostructural and microstructural flexibility.

In some diesel applications, particularly in the diesel passenger car segment, the mass of the soot collected is high, resulting in the possibility of higher exotherms when the filter is regenerated. The heat generated only has two paths to dissipate. It can be carried away in the exhaust gas or is adsorbed by the filter. In regeneration conditions with little exhaust flow, high-heat-capacity alternatives to cordierite, such as silicon carbide or aluminum titanate, are used to adsorb the exothermic reaction to prevent filter damage.

How these products are used in the system

Ceramic substrates and filters are the keystone of mobile pollution control ecosystems. The vehicle emission system is composed of ceramic substrates and filters and ceramic-based catalysts held in place by a ceramic-based mat material. The ceramic substrates and filters provide a thermal-mechanically-stable base that can withstand extremes in temperature and vibration and last for the life of the vehicle. In the case of filters, they also provide the engineered microstructure that allows for the particulate filtration function. All flow-through

substrates and, in many cases, wall-flow filters are sent to catalyzers to apply a washcoat and catalyst. These catalysts can be platinum-group metal-based catalysts supported on high surface area alumina, ceria, zirconia, zeolite, or can be other catalysts or sorbents.

After coating, the composite product is sent to a canner and wrapped in a ceramic-based mat material (Figure 5). The ceramic-based component must have sufficient isostatic strength to withstand the canning process, where the material is squeezed (stuffing process) or compressed (tourniquet process) into the can. The ceramic-based mat provides heat insulation and a holding force to maintain the ceramic in the can while it accommodates the differential shrinkage between the ceramic (low expansion) and the metallic can (high expansion). The canned system is then welded into the complete exhaust system and integrated with sensors, so that computers on the vehicle assure the emissions are compliant under all conditions.

Innovation continues

The growth of pure electric vehicles—which have no tailpipe emissions issues (although issues exist at the electricity source and from the braking system)—are changing the baseline and societal expectations. However, even with aggressive battery electric vehicle penetration, the combination of geographic expansion of regulations and regulation tightening in existing regions is likely to increase ceramic substrate and filter volume over the next 10 to 15 years. To stay competitive, some internal combustion engine-based vehicles are going well beyond regulatory requirements—to nearly emissions free—to meet consumer expectations.

Because of advancements in engine, vehicle, and emissions control, the capacity exists to make vehicles achieve “negative emissions” after the first couple of minutes of vehicle operation in many cities, meaning that the air coming out of the tailpipe is cleaner than that going in.

Most of the gaseous and particulate emissions in a typical drive cycle are produced in the

first minute or two of operation. Once catalysts are hot, they are extremely effective. Therefore, the remaining frontier of a zero-emission internal combustion vehicle is tackling this first minute of operation, including vehicles with multiple engine starts like start/stop vehicles and hybrid vehicles, which may switch between electric and conventional engines several times during the drive cycle. Due to low engine outlet temperatures (increased engine efficiency or frequent starts and stops), active devices may also play a role.

Current designs and materials for ceramic-based substrates, filters, catalysts, and mats will continue to play an important role in meeting future requirements. However, new ceramic processes, materials, microstructures, and designs will be needed to enable products that heat-up faster and have higher pollution removal efficiencies. Thinner walls, higher porosity levels, tighter pore size control, and additional durability are all on the list.

Cellular ceramics proved to be an effective way of packing a lot of geometric surface area into a small volume, and the cellular form factor is an important success factor for mobile emissions remediation (Figure 6). There are likely benefits for cellular ceramics beyond mobile emissions as similar benefits may extend to catalysis, sorption, and filtration in other industries such as petrochemicals, fine chemical, clean water, and more. For these reasons, ceramic filters and substrates are likely to remain a key factor in the improvement of the environment around the world.

Regulations, impact, and societal benefits

Over the past nearly 50 years, ceramic substrates and filters brought dramatic benefits to society. Since the U.S. adopted the Clean Air Act of 1970, the nation’s economy grew fourfold. At the same time, ambient air pollution dropped more than 70%. The greatest contributor to this improvement in air quality—reduced vehicle emissions—is largely due to the three-way catalytic converter, so called because it mitigates NO_x , CO , and hydrocarbon. With the addition of the wall-flow filter, in some locales the air coming out of the engine can be cleaner than air going into the engine.

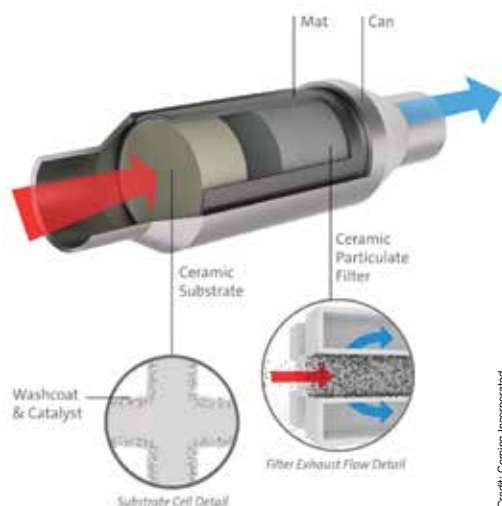


Figure 5. Ceramic substrates and filters and ceramic-based catalysts held in place within a metal can using a ceramic-based mat material.

Credit: Corning Incorporated.

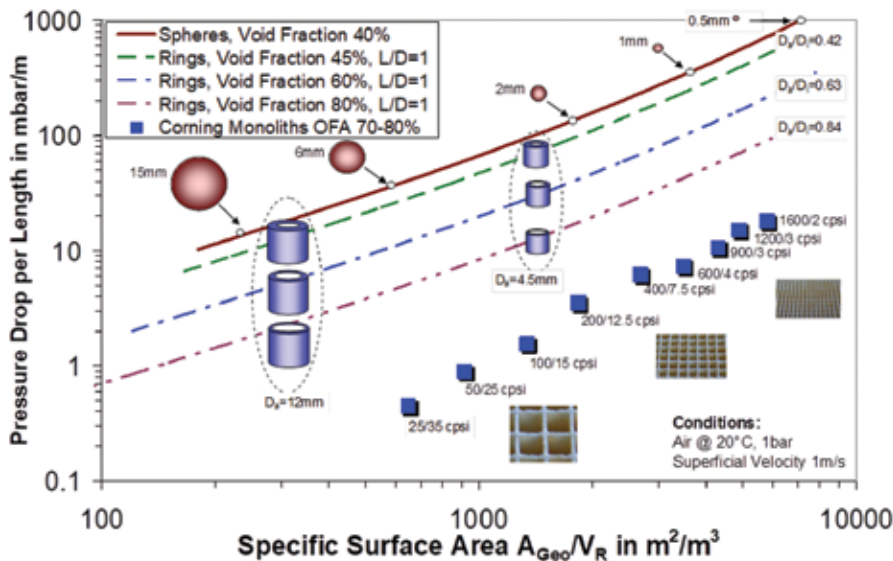


Figure 6. Pressure drop as a function of specific surface area for various form factors of media, where L is ring length, D is ring diameter, and OFA is open frontal area.

The success of Clean Air Act standards in its early years gave the EPA impetus to add even more-stringent amendments in 1990. These standards, according to government studies, are saving an additional 160,000 lives per year. The 1990 law also prevents an estimated 13 million lost workdays, 1.7 million cases of exacerbated asthma, and roughly 130,000 cases of heart disease each year.¹ Beyond these benefits, the emissions-control systems turned out to be a bargain for automakers as well. Catalyst systems represent less than 5% of the sticker price of most vehicles, and the societal benefit—even as regulations became more stringent over the years—is still about \$10 for every \$1 spent.

Regulations for heavy-duty vehicles and nonroad machineries were also adopted, expanding ceramic substrates and filters to larger vehicles and increasing component size up to 20 times larger than light-duty vehicles, requiring new manufacturing developments to make ceramic structures and catalyze the large-frontal area substrates and filters.

The honeycomb ceramic substrates and filters had such a profound impact on air quality that in 2002, the National Inventors Hall of Fame inducted the three Corning scientists who developed the innovation: Dr. Irwin Lachman, Dr. Rod Bagley, and Mr. Ron Lewis. They were recognized for creating the extrusion method for forming the thin-walled structures. And in 2005, those same three scientists won the National Medal of Technology.

What's ahead?

Despite the short-term fluctuations of governments, the overall global direction is toward tightening standards for vehicular emissions. This attention to air quality intersects the continuing increase in vehicle usage. Many forecasts indicate vehicle growth of up to 50% over the next 20 years. Virtually all this growth will be in heavily populated and developing countries.

Following this trend, expect to see the eventual expansion of regulations in the Asean and Africa regions, as well as the tightening of regulations in emerging economies. China, India, and South America now are implementing vehicle tailpipe regulations on par with those in Europe but still with looser gaseous emissions than in North America. Because the technology is already available to make the air even cleaner and easily deployed, for example, from the U.S. to Europe, further regulatory tightening in all regions is quite likely.

Though vastly improved over the past 50 years, air quality in the U.S.—specifically, in large cities and traffic-heavy California—still has room for significant improvement. Reductions in real world driving emissions for NO_x and particulates are still needed. Some in California are exploring new standards that would enable the use of gasoline particulate filters, matching the low particulate output of European and Chinese gasoline vehicles.

Improvement also continues in long-term emissions standards for heavy-duty

vehicles. Some California lawmakers are working to tighten NO_x emissions by more than 90% by 2027 to address urban ozone issues. They are also considering doubling or tripling the regulatory full-useful life and emissions warranty for trucks and other heavy-duty equipment. Similar initiatives are in the works in Europe and China.

Acknowledgements

The authors would like to thank Tim Johnson, Anne Kenlon, Kerstin Stobbe, and Agatha Lutoborski for their help with this article.

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Application note

"If you would seek health, look first to the spine."

—Socrates



Silicon nitride—A ceramic surgical implant material

By Don Bray and Bryan McEntire

Silicon nitride is used in many industries. For the healthcare industry, it is a relatively new adoption—but one with a lot of potential.



Credit: Don Bray, SINTX

Little did materials scientist Ashok Khandkar and orthopedic surgeon Aaron Hoffman realize the impact that a ceramic material—silicon nitride—would have on the quality of life for many people with spine disease. Today, SINTX Technologies, the company they helped establish, is making silicon nitride spinal fusion implants and exploring many new applications for the material.

Background

Silicon nitride is an inorganic and nonmetallic material made of silicon and nitrogen, two elements that are essential in biologic systems. It is made by mixing highly refined raw powders that are formed into desired shapes. The final product is finished in furnaces under high pressure and heat. Dense silicon nitride is a very hard, abrasion- and corrosion-resistant solid. Unlike familiar ceramics such as porcelain or glass, silicon nitride has very high strength with the highest fracture resistance of any advanced ceramic.

Silicon nitride was first synthesized in 1857 and was commercialized in the 1950s. Later, research funded by the United States, European Union, and Japanese governments helped further development and reduced manufacturing



costs. Because of its advantages, silicon nitride was soon adopted in many industries, particularly ones in which extreme conditions precluded the use of other metal, plastic, or composite materials.

Khandkar and Hoffman initially worked to develop a silicon nitride ball bearing for artificial hips. At the time, the news was filled with reports of some patients reacting to toxic metal wear particles due to higher wear rates of metal bearings in hips, thus leading to the search for new bearing materials. The company also submitted an FDA 510K approval for a product for spinal fusion based on animal data showing rapid healing of silicon nitride to bone.

The company received approval for the spinal fusion device and deferred a clinical trial of the hip bearing. Starting in 2015, the company invested heavily in the basic research and development related to silicon nitride and discovered additional properties, such as surface resistance to bacterial colonization. SINTX Technologies is now a materials technology company focused on developing new products based on its silicon nitride platform.

Advantages of silicon nitride

An ideal biomaterial

Existing biomaterials have limitations—metal implants fret and corrode, plastics oxidize, and allograft bone never fully heals. Toxic metal wear led to a

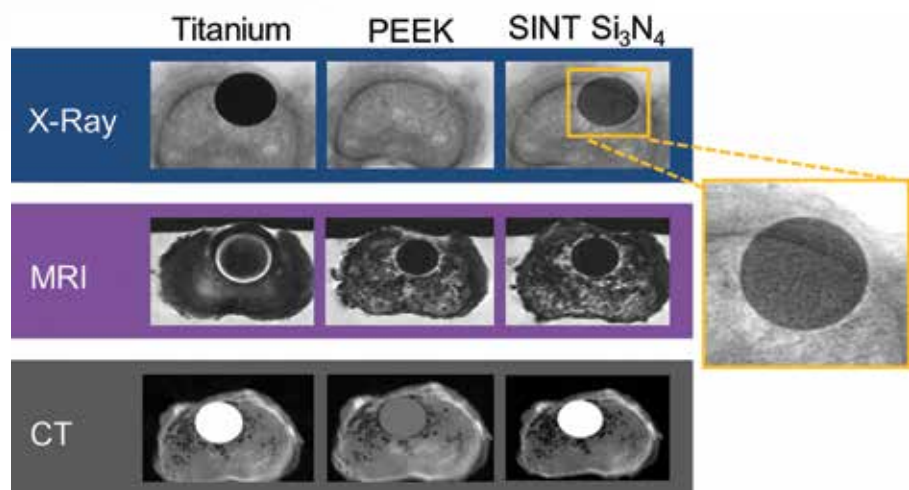


Figure 1. Comparison of implant visibility during medical imaging.

recall of all-metal hip bearings, while fretting corrosion is a new concern in artificial hips. Metal allergies to total knee implants remain an unsolved clinical problem.

Silicon nitride has none of these concerns. Its wear rate is extremely low, and the wear particles are soluble and can be cleared from the body. Silicon nitride is chemically resistant, and it has a high dielectric constant, which confers resistance to fretting corrosion. Clinical data proves its efficacy—with more than 35,000 human spine implantations over 10 years and fewer than 30 FDA-reported adverse events, silicon nitride has an exceptional safety record.

In addition to spinal fusion implants, silicon nitride can be polished to a smooth and wear-resistant surface for

hip and knee replacement bearings.^{1,2} Because of its inherent resistance to bacterial adhesion, silicon nitride is also suitable as a dental implant material, an application SINTX is actively pursuing.

Favorable imaging

On X-ray images, plastic implants are invisible while metals obscure the visibility of bone. CT scans and MRI images are also distorted by metal implants. Here again, silicon nitride shows its advantages. Implants made of silicon nitride are visible on X-ray images without obscuring the underlying bone details (Figure 1). Also, silicon nitride implants allow for distortion- and artifact-free MRI and CT images, thus giving a clear assessment of the implant-tissue interface and visualization of adjacent anatomy.³

Credit: Don Bray, SINTX

Silicon nitride—A ceramic destined to change the world

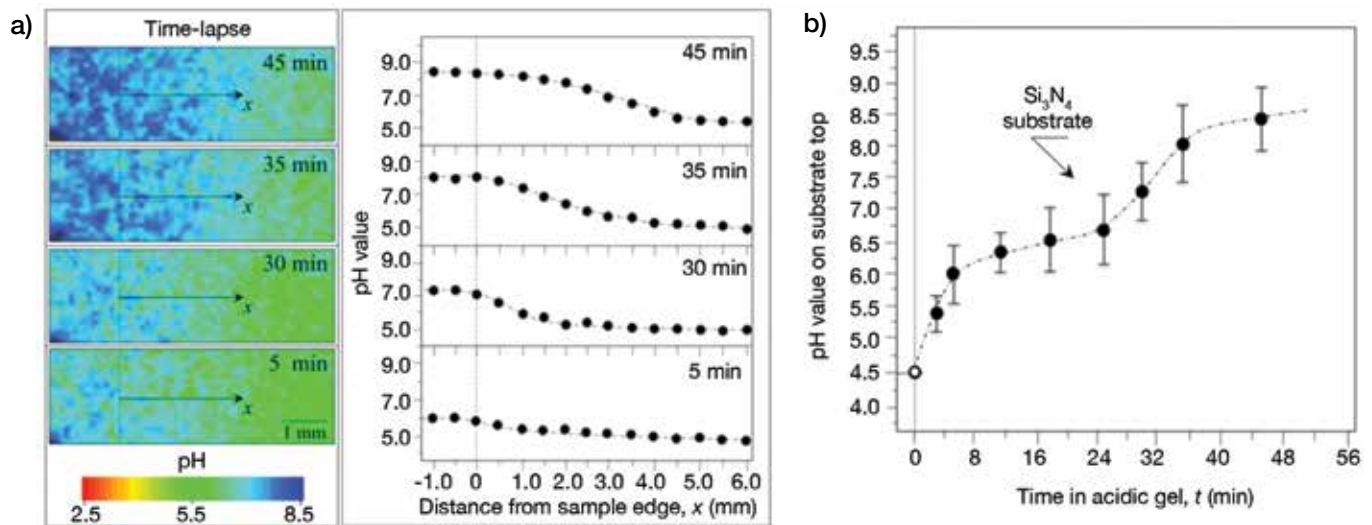


Figure 2. a) Evolution of pH near a silicon nitride surface when placed in an acidic gel; b) average surface pH over time for same experiment. Republished with permission from The Royal Society of Chemistry, from Boschetto et al.⁵; permission conveyed through Copyright Clearance Center, Inc.

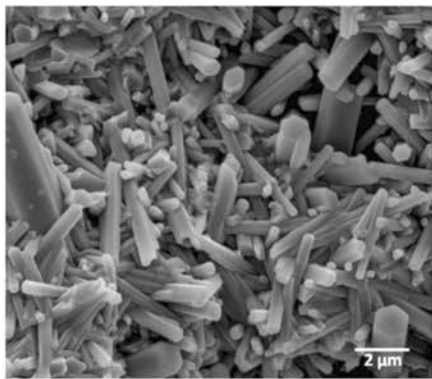


Figure 3. Surface microstructure of bioactive Si₃N₄.

Versatile surface chemistry

Surface chemistry is a major factor in the success of any implantable device. Compared to the polymer polyetheretherketone (PEEK) or titanium, silicon nitride is hydrophilic, i.e., it attracts body fluids containing proteins and bone-forming cells that are critical to bone healing. Simple manufacturing variations, such as glazing or heating in a nitrogen or oxidizing atmosphere, can modify implant surface chemistry, which allows tailoring of implant chemistry to specific biomedical applications.⁴

At the surface level, silicon nitride hydrolyzes, resulting in local, microscopic release of silicic acid and ammonia, according to the reaction shown below. Silicic acid enhances osteogenic processes near the material surface, and the ammonia creates an environment that discourages bacterial growth. This dual effect is highly desirable in any bone fusion implant.

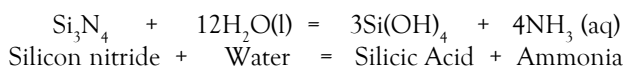


Figure 2a shows the evolution of pH near a silicon nitride surface when placed in an acidic gel. Dissolution of ammonia causes a local increase of near-surface pH over

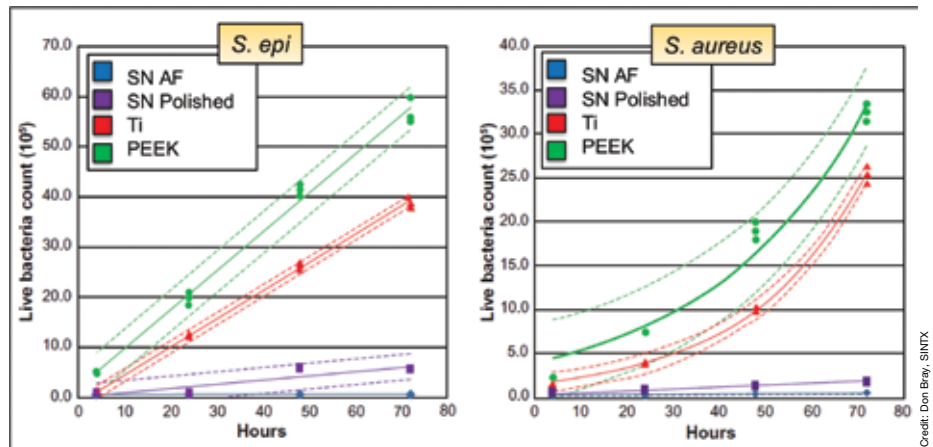


Figure 4. Counts of living bacteria (y -axis) versus incubation time (x -axis) for the bacteria *S. Epidermidis* and *S. Aureus*.

time. Figure 2b shows the average surface pH over time from the same experiment.⁵

Silicon nitride's surface topography is equally supportive of bone healing. The surface of as-fired silicon nitride consists of anisotropic grains that are typically 1 μm up to 10 μm with individual features (e.g., asperities, sharp corners, points, pits, pockets, and grain intersections) that can range in size from less than 100 nm to 1 μm. While this structure is morphologically different from surface functionalized titanium, it has some common features (e.g., sharp corners, points, and pockets). Research shows that this type of surface microstructure is important in resisting bacterial attachment while concurrently promoting mammalian cell adhesion and proliferation (Figure 3).

Antibacterial properties

Bacterial infection of any biomaterial implant is a serious clinical problem. Silicon nitride offers a potential easy solution—it is inherently resistant to bacterial colonization and biofilm formation. In addition, a recent study showed a direct bactericidal effect against an oral pathogen, *P. Gingivalis*. This property is probably multifactorial, reflect-

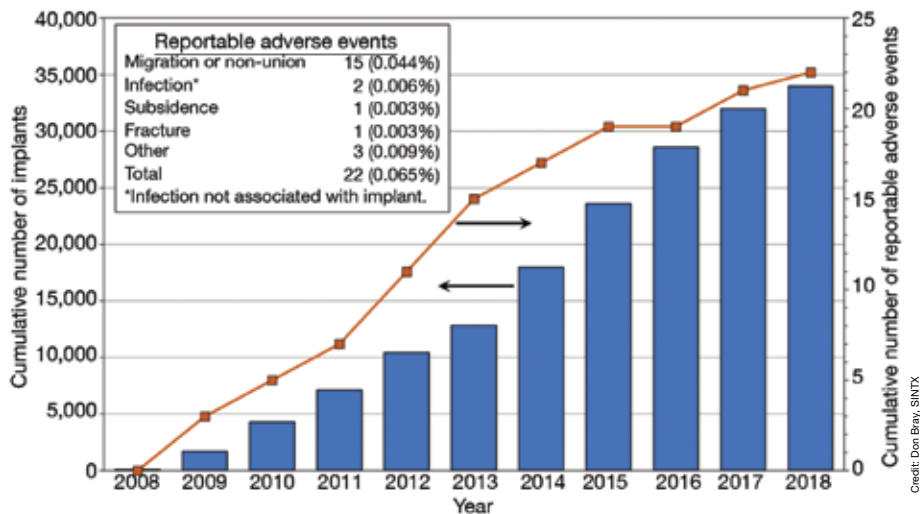


Figure 5. Silicon nitride in spinal applications: +33,000 implants in nine years.

ing the combined effects of surface chemistry, pH, texture, and charge. The ability to vary these surface properties for specific implants is an advantage of the material. In several studies, silicon nitride demonstrates significantly lower bacterial biofilm formation compared to polymers or metals. Independent studies performed outside SINTX corroborated these findings.

The graphs in Figure 4 are representative of in vitro tests done with several bacterial species. For simplicity, the graphs show results with *S. Epidermidis* and *S. Aureus*.⁶ Both common nosocomial pathogens are common causes of implant-associated infection. The graphs show counts of living bacteria (y-axis) versus incubation time (x-axis). In all cases, two forms of silicon nitride (as fired and polished) showed lower bacterial counts than either polymer or metal.

Promote bone growth

Silicon nitride stimulates osteoblasts (bone-forming cells) to form bone while suppressing osteoclasts (bone resorbing cells). A manufacturing change called “nitrogen-annealing” results in a near-200% increase in bone formation by cells exposed to silicon nitride. This finding has excellent implications for accelerating bone healing, bone fusion, and implant integration into the skeleton. Other data shows living cells adhere preferentially to silicon nitride over polymer or metal surfaces.

Cell adhesion promotes tissue development and enhances the bioactivity of materials. Cell adhesion to silicon nitride is a function of pH, chemical, and ionic changes at the material’s surface. The surface chemistry and nanostructure topography of silicon nitride provide an optimal environment for the stimulation of bone growth. Silicon nitride implants demonstrate greater new bone formation at 3, 7, 14, and 90 days compared to polymer or metal implants.⁷ The amount of regenerated bone associated with silicon nitride implants is 2–3 times greater than polymer or metal implants three months after surgery.

Clinical studies

The first use of silicon nitride in spinal fusion was in a small Australian clinical trial in the mid-1980s.⁸ The implants used were anterior lumbar interbody fusion (ALIF) devices fashioned from a reaction-bonded silicon nitride. A 31-year follow-up of seven surviving patients was recently published, showing sustained implant stability, no subsidence, no migration, and excellent bone integration, even three decades after implantation. This study is the longest reported clinical history for a synthetic biomaterial used in spine. Cumulative silicon nitride implantations through 2018 total about 35,000. Of these, fewer than 30 FDA-reportable adverse events manifested, with no implant-related

infections relative to an industry standard of 3–10% (Figure 5).

Future

With an expanding, ageing, and more active population, biomaterial innovations will lead to improved biomedical implant safety, higher-performance, and lifetime durability. Already well-proven in diverse industrial applications and currently used as intervertebral spinal fusion cages, silicon nitride has the foundational evidence to be applied likewise across a range of biomedical applications.

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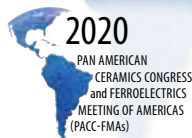
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SCHEDULE OF EVENTS

Sunday, July 19, 2020

- Conference registration 3:30 – 7 p.m.
- Welcome reception 5:30 – 7 p.m.

Monday, July 20, 2020

- Conference registration 7 – 5 p.m.
- Opening ceremony and plenary session 8:30 – 11:30 a.m.
- Lunch/Technology fair 11:30 a.m. – 1 p.m.
- Concurrent technical sessions 1 – 5:00 p.m.
- Coffee break 3 – 3:20 p.m.
- Technology fair and poster session, including reception 5:30 – 7 p.m.

Tuesday, July 21, 2020

- Conference registration 7 a.m. – 5 p.m.
- Concurrent technical sessions 8:30 – 11:30 a.m.

- Lunch/Technology fair 11:30 a.m. – 1 p.m.
- Concurrent technical sessions 1 – 5 p.m.
- Coffee break 3 – 3:20 p.m.

Wednesday, July 22, 2020

- Conference registration 7:30 a.m. – Noon
- Concurrent technical sessions 8:30 a.m. – Noon
- Technology fair 8:30 a.m. – Noon
- Afternoon on own/ ACerS-organized Panama Canal tour available for an additional fee Noon – 5 p.m.
- Conference dinner 7 – 9 p.m.

Thursday, July 23, 2020

- Conference registration 8 a.m. – Noon
- Concurrent technical sessions 8:30 a.m. – Noon

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Hotel Monteleone is located right in the French Quarter of New Orleans, among a variety of specialty shops selling art and antiquities from around the world, and restaurants serving authentic New Orleans Cajun cuisine. Tourist attractions are located just steps from the hotel, including Jackson Square, Bourbon Street, the French Market, and the Riverwalk. New Orleans itself is steeped in European traditions and Caribbean influences. The Big Easy offers visitors sweet sounds and savory aromas fueled by three hundred years of history.

The GOMD Executive Committee, program chairs, and volunteer organizers sincerely hope you will join them in New Orleans for GOMD 2020 to find new collaborative opportunities and to exchange ideas in the international glass community.

We look forward to seeing you in New Orleans!

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SCHEDULE OF EVENTS

Sunday, May 17, 2020	
Registration	4 – 7 p.m.
Welcome reception	6 – 8 p.m.
Monday, May 18, 2020	
Registration	7 a.m. – 5:30 p.m.
Stookey Lecture of Discovery	8 – 9 a.m.
Concurrent sessions	9:20 a.m. – 5:40 p.m.
Otto Schott Award luncheon sponsored by Schott AG	Noon – 1:30 p.m.
GOMD general business meeting	5:45 – 6:30 p.m.
Poster session and student poster competition	6:30 – 8:30 p.m.
Tuesday, May 19, 2020	
Registration	7:30 a.m. – 5:30 p.m.
George W. Morey Award lecture	8 – 9 a.m.
Concurrent sessions	9:20 a.m. – 6 p.m.
The Norbert J. Kreidl Award for Young Scholars	Noon – 1 p.m.
Lunch on own	Noon – 1:30 p.m.
Conference banquet	7 – 10 p.m.
Wednesday, May 20, 2020	
Registration	7:30 a.m. – 5 p.m.
Varshneya Frontiers of Glass Science lecture	8 – 9 a.m.
Concurrent sessions	9:20 a.m. – 5:40 p.m.
Lunch on own	Noon – 1:30 p.m.
Thursday, May 21, 2020	
Registration	7:30 a.m. – Noon
Varshneya Frontiers of Glass Technology lecture	8 – 9 a.m.
Concurrent sessions	9:20 a.m. – Noon



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May 17–21, 2020
Hotel Monteleone
New Orleans, Louisiana

TECHNICAL PROGRAM

S1: FUNDAMENTALS OF THE GLASSY STATE

- Glass Formation and Structural Relaxation
- Glass Crystallization and Glass Ceramics
- Structural Characterizations of Glasses
- Topology and Rigidity
- Atomistic Simulation and Predictive Modeling of Glasses
- Data-based Modeling and Machine Learning for Glass Science
- Mechanical Properties of Glasses
- Non-Oxide Glasses
- Glass Under Extreme Conditions

S2: GLASS AND WATER: DEGRADATION OF AMORPHOUS MATERIALS

- Glass-water Interfacial Reactions and Dynamics During Initial Dissolution
- Soluble Glasses and Glasses as Ion Release Devices
- Glass-Water Interactions for Long-Term Durability

S3: OPTICAL AND ELECTRONIC MATERIALS AND DEVICES —FUNDAMENTALS AND APPLICATIONS

- Laser Interactions with Glasses
- Charge and Energy Transport in Disordered Materials
- Optical Fibers and Waveguides
- Glass-based Optical Devices
- Optical Ceramics and Glass-Ceramics
- Glasses and Glass-Ceramics in Detector Applications
- Rare-earth and Transition Metal-doped Glasses and Ceramics for Photonic Applications

S4: GLASS TECHNOLOGY AND CROSS-CUTTING TOPICS

- Glass Surfaces, Interfaces, and Coatings
- Sol-gel Processing of Glasses and Ceramic Materials
- Challenges in Glass Manufacturing
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POSTER SESSION/RECEPTION & STUDENT POSTER COMPETITION

Organizers

Joy Banerjee, Corning Inc., U.S.A.

Mostafa Ahmadzadeh, Washington State University, U.S.A.

GOMD offers great opportunities for students to learn, network, and win prizes!

Calendar of events

April 2020

13–17 2020 MRS Spring Meeting & Exhibit – Phoenix, Ariz.; www.mrs.org/spring2020

May 2020

5–6 6th Ceramics Expo – I-X Center, Cleveland, Ohio.; <https://ceramics.org/event/6th-ceramics-expo>

6–7 Ceramic Manufacturing Solutions Conference – I-X Center, Cleveland, Ohio; <https://ceramics.org/event/ceramic-manufacturing-solutions-conference>

17–21 2020 Glass and Optical Materials Division Annual Meeting – Hotel Monteleone, New Orleans, La.; www.ceramics.org/gomd2020

27–29 MagForum 2020: Magnesium Minerals and Markets Conference – Grand Hotel Huis ter Duin, Noordwijk, Amsterdam; <http://imformed.com/get-imformed/forums/magforum-2020>

June 2020

7–10 ➔ Ultra-high Temperature Ceramics: Materials for Extreme Environment Applications V – The Lodge at Snowbird, Snowbird, Utah; <http://bit.ly/5thUHTC>

July 2020

19–23 Pan American Ceramics Congress and Ferroelectrics Meeting of the Americas (PACC-FMAs 2020) – Hilton Panama, Balboa Avenida Aquilino de la Guardia, Panama City, Panama; www.ceramics.org/PACCFMAs

August 2020

2–7 ➔ Solid State Studies in Ceramics, Gordon Research Conference – Mount Holyoke College, South Hadley, Mass.; <https://www.grc.org/solid-state-studies-in-ceramics-conference/2020>

16–21 Materials Challenges in Alternative & Renewable Energy 2020 (MCARE2020) combined with the 4th Annual Energy Harvesting Society Meeting (AEHSM 2020) – Hyatt Regency, Bellevue, Wash.; www.ceramics.org/mcare2020

23–27 ➔ International Congress on Ceramics (ICC8) – Bexco, Busan, Korea; www.iccs.org

30–Sept.27 ➔ 2nd Global Forum on Smart Additive Manufacturing, Design and Evaluation (SmartMADE) – Osaka University, Nakanoshima Center, Japan; <http://jwri.osaka-u.ac.jp/nconf/Smart-MADE-2020>

September 2020

21–23 China Refractory Minerals Forum 2020 – InterContinental Dalian, Liaoning, China; <http://imformed.com/get-imformed/forums/china-refractory-minerals-forum-2020>

October 2020

4–8 ACerS 122nd Annual Meeting with Materials Science & Technology 2020 – David L. Lawrence Convention Center, Pittsburgh, Pa.; www.matscitech.org

November 2020

8–13 7th Int. Conference on Electrophoretic Deposition (EPD 2020) – Santa Fe, New Mexico; <http://www.engconf.org/conferences/materials-science-including-nanotechnology/electrophoretic-deposition-vii-fundamental-and-applications>

29–Dec 3 2020 MRS Fall Meeting & Exhibit – Boston, Mass.; www.mrs.org/fall2020

January 2021

20–22 Electronic Materials and Applications (EMA2021) – DoubleTree by Hilton Orlando at Sea World Conference Hotel, Orlando, Fla.; www.ceramics.org

24–29 45th International Conference and Expo on Advanced Ceramics and Composites (ICACC2021) – Hilton Daytona Beach Oceanfront Resort, Daytona Beach, Fla.; www.ceramics.org

March 2021

27–31 ➔ The Int'l Conference on Sintering 2021 – Nagaragwa Convention Center, Gifu, Japan; <https://www.sintering2021.org>

23–28 14th Pacific Rim Conference on Ceramic and Glass Technology (PACRIM 14) – Hyatt Regency Vancouver, Vancouver, British Columbia, Canada; www.ceramics.org

August 2021

29–Sept 2 ➔ 17th European Ceramic Society Conference – Dresden, Germany; www.ecers2021.org

September 2021

14–17 20th Biennial Worldwide Congress Unified International Technical Conference on Refractories – Hilton Chicago, Chicago, Ill.; www.ceramics.org

October 2021

17–21 ACerS 123rd Annual Meeting with Materials Science & Technology 2021 – Greater Columbus Convention Center, Columbus, Ohio; www.ceramics.org

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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Ceramic & Glass

APRIL 2020 • VOLUME 1 • ISSUE 2

MANUFACTURING

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BREAKING IN: A GUIDE TO WORKING WITH BIG BUSINESS

HOW TO BREAK IN: SMALL COMPANIES CAN USE THEIR STRENGTHS AND FLEXIBILITY

ONE STARTUPS' SECRETS TO BIG COMPANY SUCCESS



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Ceramic & Glass Manufacturing is published four times per year by The American Ceramic Society. The American Ceramic Society is not responsible for the accuracy of information in the editorial, articles, and advertising sections of this publication. Publication of articles does not comprise endorsement, acceptance, or approval of the data, opinions, or conclusions of the authors on the part of the Society or its editors. Readers should independently evaluate the accuracy of any statement in the editorial, articles, and advertising sections of this publications. Vol. 1, No. 2, pp 1-16.

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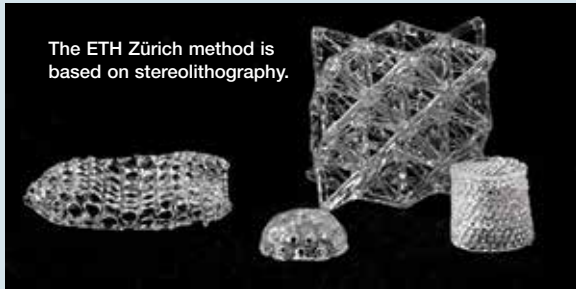
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INDUSTRY NEWS



The ETH Zürich method is based on stereolithography.

ETH ZÜRICH RESEARCHERS DEVELOP 3D GLASS PROCESS

Researchers from ETH Zürich developed a technique to produce complex glass objects with 3D printing. The method is based on stereolithography, one of the first 3D-printing techniques developed during the 1980s. The researchers developed a special resin containing a plastic and organic molecules to which glass precursors are bonded. Wherever light strikes the resin, it hardens. The plastic monomers combine to form a labyrinth-like structure, creating the polymer. The ceramic-bearing molecules fill the interstices of this labyrinth.

AGC GLASS USES AI IN NEXT-GEN RESEARCH

AGC Glass Europe, a European leader in flat glass, and Citrine Informatics are collaborating to use artificial intelligence to accelerate the development of next-generation glass. Citrine Informatics is a technology platform that uses AI to bring new materials to market faster and capture materials-enabled product value. The companies said the collaboration will work on ways to meet increasing global demand for strong scratch- and abrasion-resistant glass in the automotive and communication industries.



The partners aim to meet global demand for scratch- and abrasion-resistant glass.

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ACeRS – NIST PHASE EQUILIBRIA DIAGRAMS

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NIST NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

NEW PRESIDENT OF WORLD REFRACTORY ASSOCIATION



The World Refractory Association elected Carol Jackson, chairman and CEO of HarbisonWalker International, as incoming president of WRA for a two-year term beginning January 2020.

She succeeds Stefan Borgas, CEO of RHI Magnesita, who has led the organization since January 2018. Formed in 2014, WRA has grown from 12 to 22 members in the last two years and has itself become a member of the World Steel Association.



The partnership aims to cut the time for designing and validating 3D printed components.

GE, OAK RIDGE, PALO ALTO PARTNERSHIP AWARDED 3D FUNDING

GE, Oak Ridge National Laboratory, and the Palo Alto Research Center a Xerox company, were awarded an estimated \$1.3 million to accelerate the development of 3D printed turbomachinery parts. The funds were granted by the U.S. Advanced Research Projects Agency-Energy (ARPA-E) Design Intelligence Fostering Formidable Energy Reduction and Enabling Novel Totally Impactful Advanced Technology Enhancements (DIFFERENTIATE) program. Within the program, the partners aim to reduce the timeline for designing and validating 3D-printed components by as much as 65 percent.

CENTER FOR GLASS SCIENCE AND TECHNOLOGY CREATED

By combining materials science expertise with large-scale medical research, Missouri S&T researchers hope to meet clinical demands for glass-related solutions through a new Center for Glass Science and Technology. The new center will build on Missouri S&T's previous glass research, which includes the development of bioactive glass to treat open wounds and cancers. The center was formed with funding from the University of Missouri System, and the grant will provide equipment and dedicated lab space to support research related to the university's NextGen Precision Health Initiative.



Recent research at Missouri S&T targeted bioactive glasses and bioceramics to repair bone, heal soft tissue wounds, and eradicate infection. Credit: Sam O'Keefe/Missouri S&T



Fuyao Group has opened five plants in the U.S.

FUYAO GROUP EXPANDS ITS DAYTON, OHIO, PLANT

China's auto glass manufacturer Fuyao Group said it will invest \$46 million in U.S. dollars in new equipment at its Dayton, Ohio, plant, which will bring 100 new jobs. In 2014, Fuyao bought a former General Motors facility closed in 2008 and turned the abandoned plant into a two-million-square-foot factory. Fuyao Group has opened five factories in the United States. The Dayton factory now has 2,300 employees.



Allied Glass makes containers for Tanqueray gin and many other brands.

ACQUISITION OF ALLIED GLASS COMPLETED

An affiliate of London-based private investment firm Sun European Partners completed the acquisition of Allied Glass for an undisclosed sum. Allied is headquartered in Leeds, England, and is one of the largest U.K.-based manufacturers of glass packaging containers for the premium spirits and food and drinks markets. Sun said the business has doubled its customer base, which includes craft manufacturers and big blue-chip manufacturers, and delivered sales growth of 13 percent per annum.

MORE INDUSTRY NEWS

RHI MAGNESITA ACQUISITION STRENGTHENS ITS NORTH AMERICAN MARKET



RHI Magnesita employs more than 14,000 people.

RHI Magnesita, a global supplier of refractory products, systems and solutions, acquired Missouri Refractories Co., Inc. (MORCO). The acquisition fits into Austria-based RHI Magnesita's strategy to strengthen its position in the North American refractory market, the company said. RHI Magnesita produces more than 400 monolithic mixes, which serve industries including steel, cement, lime, and glass. The company also provides refractory material for the petrochemical industry. The company employs more than 14,000 people in 35 main production sites and more than 70 sales offices around the world.

SUMITOMO INVESTS IN COLORADO-BASED MATERIALS FIRM

Investment and trading firm Sumitomo Corp. of Americas said it would invest in additive manufacturing research and development firm Elementum 3D Inc. Erie, Colo.-based Elementum 3D develops advanced metals, composites, and ceramics for additive manufacturing applications, including a patented metal powder blended with ceramics. Sumitomo said its investment will help the materials firm to expand the sales and marketing of its powder. The powder product will be used in several of Sumitomo's current industries, such as steel, mineral resources, aerospace, and tubular. Sumitomo also recently invested in the additive manufacturing companies Sintavia, AREVO, and Shapeways.



Sumitomo hopes to expand the sales of Elementum's powder product.



Carnegie Mellon researchers will establish an AI Center of Excellence

AIR FORCE FUNDS AI MATERIALS RESEARCH AT CARNEGIE MELLON

The Air Force Research Laboratory's Materials and Manufacturing Directorate awarded a multimillion-dollar cooperative agreement to Carnegie Mellon University to fund cutting-edge research and develop Ph.D. students in an emerging area of materials science: using artificial intelligence and machine learning to discover, analyze, design and develop both existing and new high-tech materials. A kickoff meeting for a Center of Excellence was held in December at Carnegie Mellon.

Medallion Resources' proposed rare-earth plant has a smaller footprint than traditional plants.



MEDALLION RESOURCES TAKES STEP TOWARD RARE EARTHS PRODUCTION

Medallion Resources has engaged international engineering group Stantec to evaluate sites in the U.S. for its planned rare earth element extraction plant. The plant, which will use feedstock sourced from the southeast U.S., will use Medallion's hydrometallurgical process to extract a rare earth element concentrate from byproduct monazite sand. After years of test work and development, Medallion said it recently completed the design of this process. The proposed plant has a small footprint and capital costs that are a fraction of traditional rare-earth mining and processing facilities, according to Medallion.



O-I separated its asbestos liabilities from its glass-making operations.

O-I SUBSIDIARY FILES FOR CH. 11 PROTECTION

Paddock Enterprises, a wholly-owned subsidiary of O-I Glass, filed for Chapter 11 bankruptcy protection under the weight of thousands of asbestos injury claims. In December 2019, O-I created a new holding company structure where O-I Glass became the new parent entity with Owens-Illinois Group and Paddock as direct, wholly-owned subsidiaries. O-I's legacy asbestos-related liabilities are isolated within Paddock, structurally separating them from the company's glass-making operations. O-I Glass and O-I Group were not included in the Chapter 11 filing.

DEPARTMENT OF ENERGY SELECTS EMC FOR SOLAR MODULE DEVELOPMENT

The U.S. Department of Energy Solar Energy Technologies Office selected Energy Materials Corp. to advance perovskite photovoltaic module research and development. The selection supports EMC's scale-up of high-efficiency and stable perovskite solar modules. The project will demonstrate high-speed printing of entire perovskite devices on paper-thin flexible glass, including the transparent conductor layers that are traditionally done by costly vacuum deposition techniques. EMC said the process can greatly lower the cost of building solar panel factories. EMC was selected as a part of the Solar Energy Technologies Office Fiscal Year 2019 funding program.



EMC plans to fabricate the panels on its high-speed, roll-to-roll manufacturing lines.



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TRADE SHOWS

ACERS NEW MANUFACTURING CONFERENCE JOINS CERAMIC EXPO

(IN CONJUNCTION WITH CERAMICS EXPO)

May 6–7, 2020 | Cleveland, Ohio, USA

ACerS' inaugural Ceramic Manufacturing Solutions Conference will feature practical programming covering solutions to problems faced by ceramic manufacturers. The program will feature sessions on testing, quality, health and safety, ceramic processing, and raw materials. Registration includes 1.5 days of technical programming, a networking reception on Wednesday evening, and a networking lunch on Thursday. All events occur in the ballroom at the I-X Center in Cleveland, Ohio.

<https://ceramics.org/event/ceramic-manufacturing-solutions-conference>

CERAMIC MANUFACTURING SOLUTIONS CONFERENCE

HOUSTON HOSTS FORUM ON OILFIELD MARKETS AND MINERALS

June 8–10, 2020 | Houston, Texas, USA

Oilfield Minerals & Markets Forum 2020 will be held June 8–10 in Houston at the Hilton Houston Post Oak. Expert speakers will cover key issues and the supply and demand of industrial minerals used in the oilfield market. The forum brings together major players across the global supply chain, from mineral producers, processors, and traders to logisticians, financiers, and end use consumers.

www.imformed.com



MAGNESIUM MINERALS THE FOCUS OF MAGFORUM 2020

May 27–29, 2020 | Noordwijk, Amsterdam

MagForum is for those in the development, supply, processing, logistics, and market application of magnesium minerals. Delegates will have an opportunity to visit the magnesia operations of Nedmag at Veendam on Wednesday, May 27.

www.imformed.com

CHINA'S CHANGING MARKET FOR REFRACTORY MINERALS KEY TOPIC FOR SEPTEMBER EVENT

Sept. 21–23, 2020
Dalian, China

The China Refractory Minerals Forum 2020 will be held September 21–23 at the InterContinental, Dalian. The forum is a networking and knowledge acquisition opportunity on the issues, trends, developments, and outlook for China's

refractory minerals and their market demand domestically and worldwide. There will also be an opportunity to visit China's primary refractory magnesia producing center on Thursday, Sept. 24, in Haicheng, Liaoning. www.imformed.com





81ST CONFERENCE ON GLASS PROBLEMS COMING IN OCTOBER

Oct. 26–29, 2020 | Columbus, Ohio, USA

The Conference on Glass Problems is the largest glass manufacturing conference in North America, attracting manufacturers and suppliers worldwide. The 81st conference will be held October 26–29 at the Greater Columbus (Ohio) Convention Center and is organized by the Glass Manufacturing Industry Council in partnership with Alfred University. The conference focuses on technical issues facing professionals responsible for the operations of glass manufacturing companies.

<http://glassproblemsconference.org>

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HOW TO BREAK IN: SMALL COMPANIES ARE FAST AND FLEXIBLE

By David Holthaus

More than 20 years ago, Joe Pegna and a couple of Ph.D. students at Polytechnique Montreal began working on an advanced manufacturing process to produce high-performance fibers that could be deployed in stressful environments in defense, automotive, energy, and other applications.

By 2006, they had advanced and refined the process enough to start a company called Free Form Fibers.

Today, from the home base in Saratoga Springs, N.Y., Free Form Fibers markets its low-cost, high-performance ceramic fibers to a variety of high-tech customers, touting its status as the only business in the world with the technical capability to produce such materials in a cost-effective way.



Free Form Fibers senior engineer Ram Goduguchinta. Credit: FFF

After starting from scratch in 2006, Free Form Fibers today is a company with seven full-time employees and a list of big clients that buy its products.

"We're the little engine that could," said CEO and co-founder John Schneider. "We've been able to get things done that big guys have spent 35 years working on."

That creative drive to innovate is one of the keys to the company's success. Schneider and others agree it's an important component of growth for any small, high-tech business with an eye on expanding.



John Schneider

As small companies look to break into big markets and grow, they can use the advantages they already have to gain work from their much larger counterparts as suppliers and partners.

Industry experts say capitalizing on the speed and flexibility that small companies enjoy is a way to break into working with the big companies. They can exploit their capacity to quickly conduct research, develop products that fill a void, and scale up production.

"The advantage of small company is flexibility," said Doug Freitag, technical director of the U.S. Advanced Ceramics Association, a trade group that lobbies for the advanced ceramics industry.

That's especially true as big multinational companies tend to look for ways to contain their research and development costs and seek to outsource some of that work.



Exothermics takes delivery of a large plasma deposition system.
Credit: Exothermics

“Little companies can act as almost an outsourced R&D department where the really high-risk stuff can take place,” Schneiter said.

ONE COMPANY’S BIG BREAK

That’s essentially what happened at Exothermics, an Amherst, N.H.-based company that has developed and commercialized nonoxide ceramics, refractory materials, and specialty thin films for use in aerospace, defense and semiconductor markets.

Founded in 1996, the company’s growth trajectory took off in 2008 when Lockheed Martin approached it with a problem related to the stealth materials in its F-22 fighter jet.

“We ended up developing something for them that basically solved their issues,” said Steve DiPietro, founder and CEO of Exothermics. “That put us on the map.”

Exothermics’ technology, and its previous relationships with people at Lockheed and elsewhere, got it noticed when the big defense contractor needed help quickly.

It’s how productive relationships between large and small companies are often initiated, cultivated, and expanded.

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HOW KYOCERA WORKS WITH SMALLER COMPANIES

Like many other big companies, Kyocera started as a small one. Born in 1959 as the Kyoto Ceramic Co. with 28 employees, Kyocera is now a global giant with more than 75,000 employees and sales of more than \$14 billion.

Some of its growth over the years has come through partnerships with small companies, as well as acquisitions, and company executives say they are always looking over the horizon for new technologies and applications.

"We are reading articles, we go to trade shows and conferences, we read patents," said Mark Wolf, vice president of Kyocera's Fine Ceramics Group. "Anything that small companies do that's public,



Mark Wolf

advertising what they have, we're generally going to pay attention to."

Like all big tech companies, Kyocera has a substantial research and development budget, but the company can't do everything on its own, so it looks to small firms to supplement its own work.

"We're interested in new materials, new methods and processes, new equipment," Wolf said.

The company's R&D team explores emerging technologies in new markets and attempts to solve challenges that arise from relying on conventional materials, said Jay Scovie, deputy general manager for corporate communications at Kyocera.



Some of Kyocera Fine Ceramics' products. Credit: Kyocera

A champion for large and small manufacturers

Large and small ceramics manufacturers alike have a friend in Washington in the U.S. Advanced Ceramics Association.

Based in D.C., USACA is an organization that champions the business interests of advanced ceramic producers and their industry customers.

The association was formed in 1985 to facilitate the commercialization of the United States' advanced ceramics industry and has become a leading voice of the industry before the U.S. Congress and federal agencies.

USACA and its member companies work to identify new commercial market opportunities for advanced ceramics and promote their use in new high-efficiency and high-performance products for transportation, aerospace, defense, energy, and industrial applications.

The organization functions through working groups led by USACA members. It establishes working groups on an as-needed basis and currently staffs groups in ceramic fiber and ceramics matrix composite manufacturing, nuclear ceramics, transparent armor, and workforce development.

USACA will hold its 45th annual conference on January 25–28, 2021.

More information about USACA is available at advancedceramics.org.

For example, in mid-2019, Kyocera announced an expanded partnership with Cambridge, Mass.-based 24M to validate that company's manufacturing platform to mass produce a semi-solid lithium ion battery system.

Then in January, the two companies announced the launch of a residential energy storage system using 24M's new manufacturing process.

"24M and Kyocera working together will do something that neither company could do on their own," Scovie said.

The two companies have been long-time partners and Kyocera has been an investor in 24M for some time.

It's the type of business relationship that evolves and grows over time and is typical of how Kyocera and other big companies interact with small firms.

Kyocera will fund research and capital investment at small firms in order to be at the head of the line when it comes to commercializing the company's technology.

"We really like to work with companies first to get to know them before we do anything else," Wolf said.

Sometimes, Kyocera will simply fund good, small-company ideas. In other cases, especially where intellectual property issues may arise, it will enter into joint development contracts, Wolf said.

"I've paid companies millions of dollars to direct their R&D in a direction that Kyocera wants," Wolf said.

Sometimes these arrangements result in acquisitions, as Kyocera staff has opportunities to see what the culture of the small company is like, how its staff works, and what their capabilities are.

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HOW TO MINIMIZE PERCEIVED RISKS

While small companies can be fast and flexible, they can present risks to big corporations, Freitag said. And small-business owners should take steps to minimize those risks.

Small companies should anticipate thorough reviews before the big companies agree to do business with them, he said. This review often takes the form of an audit of the small firms' manufacturing systems, employee capabilities, finances, and other measurements.

"The big companies are very risk averse," Freitag said. "The last thing they want to do is spec your ceramic material into a system then have that company fail."

Free Form Fibers and other companies are compliant with the guidance and recommendations of the Defense Contract Audit Agency, the agency responsible for auditing government defense contracts. Compliance means documented policies and procedures are in place and rigorously followed to meet the government's requirements.

"Good, strong bookkeeping is as important as excellent technical work for small companies and startups, just as it is for larger organizations," Free Form's Schneiter said.

There are programs in place to assist small companies in working with the bigs. One is the Department of Defense Mentor-Protégé program, under which small businesses are partnered with larger companies. It's designed to help small businesses expand their footprints in defense industry work.

"It helps them put into place everything they need to be a good supplier to a big company," Freitag said.

The DOD said the program has helped more than 190 businesses become part of the military's supply chain.

The DOD also maintains its Title III program, which provides funding to ensure domestic industrial defense capabilities, commercialize research and development, and scale up emerging technologies. The program has been in existence since the 1950s and has helped many small businesses transfer technology to prime contractors, Freitag said.

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Defense department officials regularly issue requests for information that could lead to opportunities for small companies. For example, in November 2019, the department issued a request for information on producing ultrahigh and high-temperature composites for hypersonic and strategic systems, and in December it issued a call for proposals to strengthen the industrial base for the production of light and heavy rare earth elements.

One CEO's secrets to success

Steve DiPietro, founder and CEO of Exothermics, offers these tips on working with big companies:

1. Building relationships with a larger company is all about building trust and credibility. This can take some time if you are coming in as an outsider.
2. It's good to find a difficult problem to work on.
3. You must believe in your proposed solution to the problem and be willing to spend painful amounts of your own money to get there.
4. Over the long haul, it is good to have one or more champions for your cause on the inside.
5. You must have a long-term horizon that is not exclusively focused on revenue or profits. You need to have a Pope-like perspective on helping your customer. The profits will come later.
6. Become part of the larger company's long-term development strategy and planning. This integration gives you insight into future areas of interest for collaborative product development and revenue generation.
7. Be willing to accept some of the administrative and bureaucratic requirements that are imposed by essentially all large DOD/aerospace and even commercial concerns (e.g., quality management systems such as ISO, AS9100, SAP systems, liability insurance). These requirements are just a carrying cost for working with large firms.
8. One of the main challenges is to become like a junior version of a large company while not loading up with the administrative overhead that slows things down. In the eyes of the large company, the value of working with a small company is that they have the opportunity to work with someone that can move rapidly with the minimum level of administrative overhead. You need to engage in a balancing act that preserves the character of your entrepreneurial vision while offering an acceptable mechanism for the large company to work with you.

Small companies can use their speed and flexibility to respond quickly to such opportunities, Freitag said.

Once a small company gets connected to a big one, maintaining that relationship and cultivating trust among people on the inside will help create long-term opportunities, DiPietro said. "That's what's of enduring value—when you have champions on the inside," he said.

But he cautions small companies to maintain the traits that led to their success in the first place, even as they work with multinationals that will make many demands on them.

"You're being faced with a set of bureaucratic requirements that you have to manage," he said. "You have to actively push back and not allow your company to be a clone of a Boeing or Lockheed or a Ford or a GM. Preserve those things in your corporate culture that contribute to your creative spirit and lack of bureaucracy." ▽



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A SHORT LIST OF RESOURCES AVAILABLE TO SMALL MANUFACTURERS

There are many resources and programs available to small businesses through the U.S. Small Business Administration, the U.S. Department of Commerce and others. These programs have been cited as especially relevant to small, high-tech manufacturers:

SMALL BUSINESS INNOVATION RESEARCH (SBIR) PROGRAM

A competitive program that encourages domestic small businesses to engage in federal research and R&D with the potential for commercialization.

<https://www.sbir.gov>

SMALL BUSINESS TECHNOLOGY TRANSFER (STTR)

A federal program that requires small businesses to collaborate with research institutions to bridge the gap between basic science and commercialization of resulting innovations.

<https://www.sbir.gov/about/about-sttr>

PROCUREMENT TECHNICAL ASSISTANCE PROGRAMS

Established to expand the number of businesses participating in government contracts. Administered by the Defense Logistics Agency's Office of Small Business in cooperation with states, local governments and nonprofit organizations.

<https://www.dla.mil/SmallBusiness/PTAP>

DEPARTMENT OF DEFENSE RAPID INNOVATION FUND

A vehicle for small businesses to provide the department with technologies that can be rapidly inserted into acquisition programs that meet specific defense needs. Administered by the Office of the Secretary of Defense, Assistant Secretary of Defense for Research and Engineering and Office of Small Business Programs.

<https://defenseinnovationmarketplace.dtic.mil/business-opportunities/rapid-innovation-fund/>

DEPARTMENT OF DEFENSE MENTOR-PROTÉGÉ PROGRAM

The oldest continuously operating federal mentor-protégé program in existence. Helps eligible small businesses expand their footprint in the defense industrial base by partnering with larger companies.

<https://business.defense.gov/Programs/mentor-protege-program>

SMALL BUSINESS ADMINISTRATION'S ALL SMALL MENTOR-PROTÉGÉ PROGRAM

Mentors and protégés in the All Small program can form joint ventures. These joint ventures would qualify for set-aside contracts that the small business is eligible for.

<https://www.sba.gov/federal-contracting/contracting-assistance-programs/all-small-mentor-protege-program>



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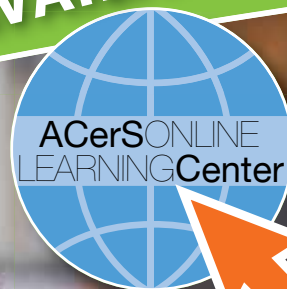
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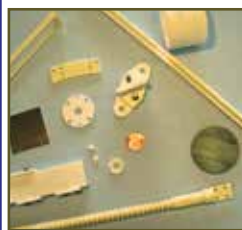


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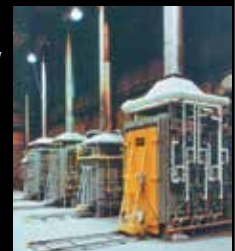
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Traditional ceramics: A mechanical strength perspective

Stability in harsh environments due to excellent electrical, mechanical, and thermal properties makes ceramics the materials of choice for numerous applications despite the emergence of newer materials like plastics and composites.¹ However, ceramic materials do face disadvantages, particularly brittleness.

Brittle fracture in ceramics is often attributed to the strong covalent and ionic bonding within a ceramic body. There is still much to learn about brittle fracture on a detailed level, though, and so ceramic brittleness and its catastrophic failure is a frequently researched subject, particularly in loading applications.

One ceramic material used frequently in loading applications, such as veneers, is porcelain. Porcelain is a type of traditional ceramic. Traditional ceramics are primarily made from three basic components: clay, which provides plasticity; silica, which maintains the shape and stability of a ceramic body at high temperature; and feldspar, which influences vitrification.

In traditional ceramics, quartz inversion during a cooling process may cause development of microcracks, which act as stress concentration centers.^{2,3} Consequently, the subcritical crack growth due to quartz inversion is reported to cause unpredictable catastrophic failure of the working ceramic bodies in loading applications.

However, in porcelains, the mullite hypothesis says that the strength of porcelain can be greatly increased by feltlike interlocking of mullite needles in the ceramic body. Thus, a realistic assessment of the role of quartz and mullite on the strength of porcelains is needed to create stronger and less brittle porcelains.

Currently, the lack of reported elastic moduli in the literature has resulted in erroneous conclusions pertaining to the mechanical behavior of porcelains. Therefore, my current project uses the modulus of elasticity approach to investigate the role of quartz and mullite on the mechanical strength of different porcelains, in addition to particle size reduction, flexural strength, and fractographic techniques.

Formulation is achieved by varying the compositions of kaolin, quartz, feldspar, and bauxite in percentage weight (wt %) with a particle size of 44 μm . The flexural strength of porcelain samples is determined by dynamic four-point-bend testing while modulus of elasticity is obtained by using two methods, namely dispersive and acoustic emission. The pulse-echo technique is used to determine Poisson's ratio (ν). Then, Poisson's ratio, the material's apparent bulk density (ρ), and the longitudinal velocity will be used to determine the elastic modulus (E).

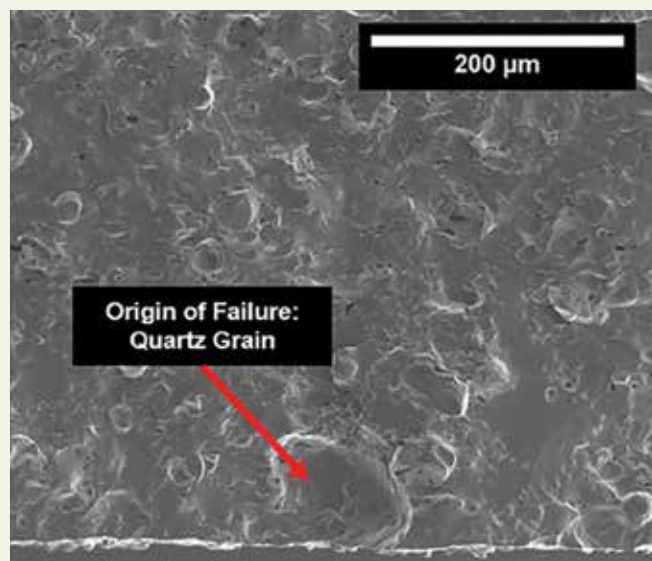


Figure 1. The fractographic technique is used to observe failure points, as shown in this SEM-micrograph of a large quartz grain at tensile surface.

Though I have not yet started doing laboratory work, I feel excited to work on this project as it addresses two major questions surrounding the strength of porcelains and the unpredictable catastrophic failure of working porcelains.

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