

*Celebrating 100 years*

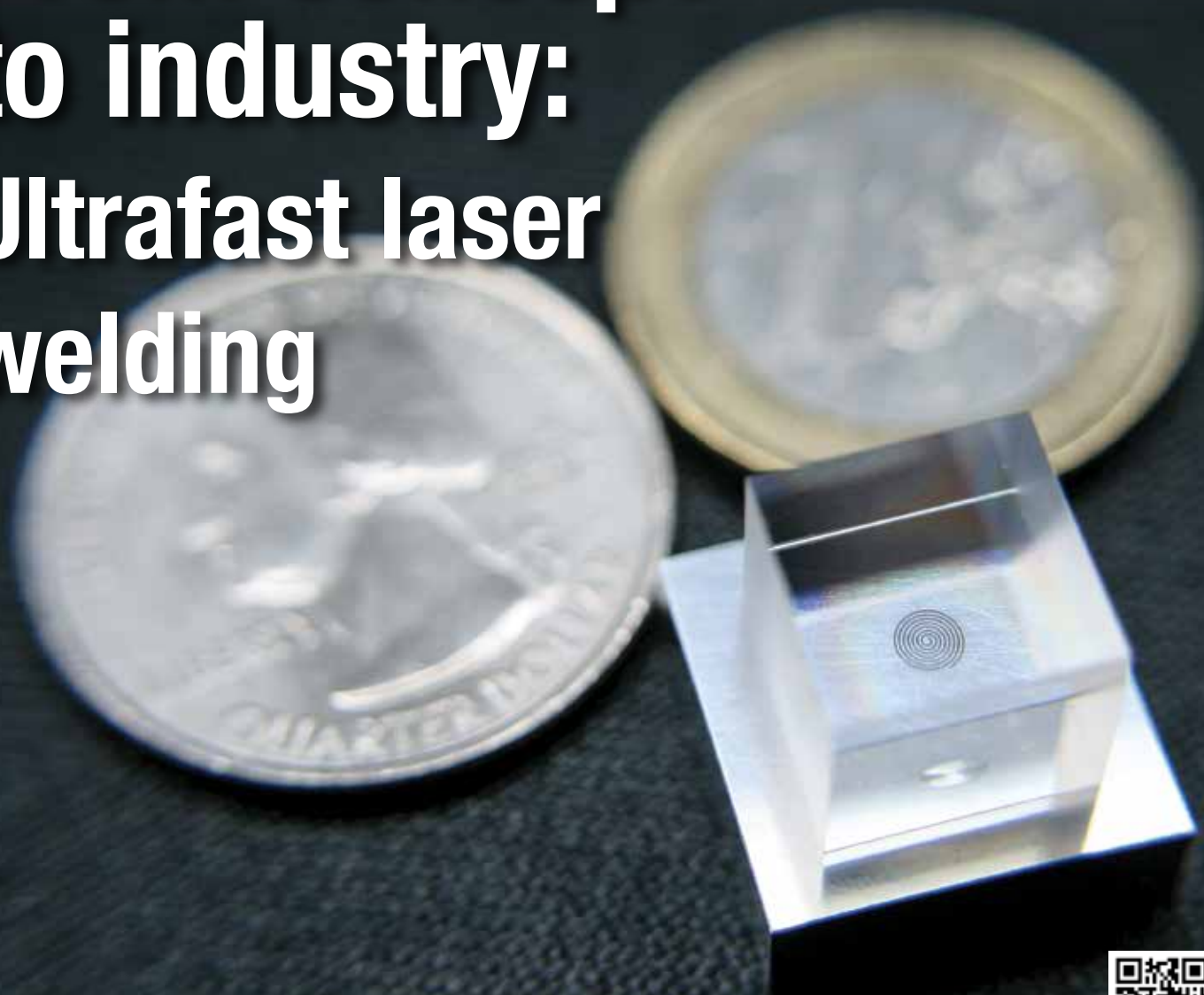
AMERICAN CERAMIC SOCIETY

# bulletin

emerging ceramics & glass technology

MAY 2021

## From concept to industry: Ultrafast laser welding





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## NEXT GENERATION GLASS RESEARCH for Sustainable Manufacturing

Thanks to a \$1.7 million grant through the New York State Department of Environmental Conservation focused on glass recycling and de-carbonization of the glass manufacturing process, Alfred University is excited to announce the launch of the **Center for Glass Innovation (CGI)**.

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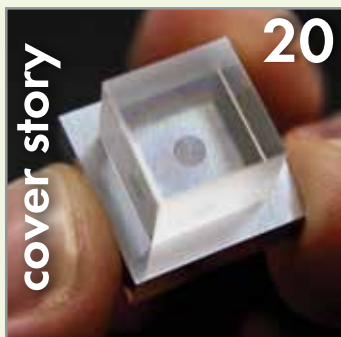
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INNOVATION

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# contents

May 2021 • Vol. 100 No. 4

## feature articles

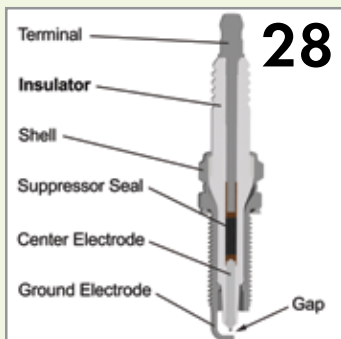


20

### From concept to industry: Ultrafast laser welding

*Ultrashort pulse laser welding has the potential to transform optomechanical component manufacturing—and research around the world is helping to move this technique from concept to industry.*

by Richard M. Carter

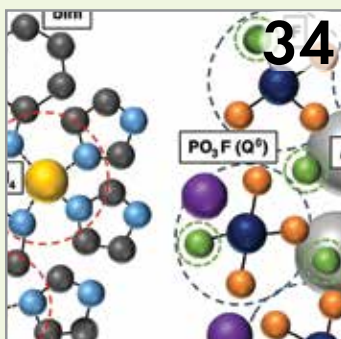


28

### Making sense of failure modes and effects analysis: Case study of a spark plug insulator

*Failure modes and effects analysis (FMEA) is a systematic approach to reduce technical risks of a product or process by identifying all potential failure modes and their causes.*

by William J. Walker, Jr.



34

### Hybrid composites made from metal-organic framework and inorganic glasses

*Elaborating on the recent discovery of glass formation at the phosphate-imidazolate join, we fabricate amorphous organic-inorganic composites using metal-organic frameworks and inorganic glass.*

by Courtney Calahoo, Louis Longley, Thomas D. Bennett, and Lothar Wondraczek



36

### Building bridges in the time of Corona: Virtual Student Exchange Program

*In August 2020, a group of Ph.D. students participated in a two-day Virtual Student Exchange Program, which offered them a way to replicate some of the in-person experiences of meetings that are currently unavailable due to the coronavirus pandemic.*

by Iva Milisavljevic, Jenna Metera, Kana Tomita, and Yiquan Wu

## department

|                           |    |
|---------------------------|----|
| News & Trends             | 3  |
| Spotlight                 | 8  |
| Research Briefs           | 14 |
| Advances in Nanomaterials | 16 |

## columns

|   |    |
|---|----|
| Business and Market View                              | 5  |
| <i>Welding equipment and supplies: Global markets</i> |    |
| by BCC Publishing Staff                               |    |
| Into the Bulletin Archives—1950s                      | 6  |
| by Lisa McDonald                                      |    |
| Deciphering the Discipline                            | 48 |
| <i>Joining of similar and dissimilar materials</i>    |    |
| by Alessandro De Zanet                                |    |

## meetings

|  |    |
|--|----|
| MCARE 2021 combined with the 4 <sup>th</sup> Annual Energy Harvesting Society Meeting (EHS 2021)   | 38 |
| Materials Science and Technology (MS&T21)  | 40 |
| PACRIM 14 including Glass & Optical Materials Division 2021 Annual Meeting (GOMD 2021)             | 42 |
| Recap of St. Louis Section/Refractory Ceramics Division 56 <sup>th</sup> Symposium on Refractories | 43 |

## resources

|                        |    |
|------------------------|----|
| Calendar               | 44 |
| Classified Advertising | 45 |
| Display Ad Index       | 47 |



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May 2021 • Vol. 100 No. 4



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## As seen on Ceramic Tech Today...



Credit: Dorothea OLDANI, Unsplash

## Mantis shrimp inspire tough composites and sophisticated optical sensors

Artists and scientists alike find inspiration in nature. But two recent scientific studies found inspiration in the same creature: the mantis shrimp. The creature's incredibly tough materials and complex eyes inspired innovations that could lead to fracture-resistant biocomposites and highly advanced optical sensors.

Read more at [www.ceramics.org/mantis-shrimp](http://www.ceramics.org/mantis-shrimp)

## Also see our ACerS journals...

### Electric field assisted solid state interfacial joining of TaC-HfC ceramics without filler

By A. Nisar, T. Dolmetsch, T. Paul, et al.

*Journal of the American Ceramic Society*

### Laser-induced modification and external pressureless joining $\text{Na}_2\text{FeP}_2\text{O}_7$ on solid electrolyte

By M. Hiratsuka, T. Honma, and T. Komatsu

*International Journal of Ceramic Engineering & Science*

### Effect of the thickness of nickel film interlayer on direct-bonded aluminum/alumina as substrate in high-power devices

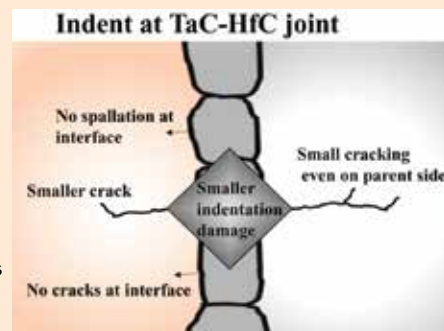
By W. Chao, C. Lin, and K. Lin

*International Journal of Applied Ceramic Technology*

### Investigation of alumina doped 45S5 glass as a bioactive filler for experimental dental composites

By Y. B. Elalmis, B. K. Ikizler, S. K. Depren, et al.

*International Journal of Applied Glass Science*



Read more at [www.ceramics.org/journals](http://www.ceramics.org/journals)

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ACSBA7, Vol. 100, No. 4, pp 1–48. All feature articles are covered in Current Contents.

# news & trends

## Endless Frontier Act: Will applied research become a main focus for the National Science Foundation?

If you conduct basic science research in the United States, chances are high that you have received funding through the National Science Foundation (NSF). Created by Congress in 1950 “to develop and encourage the pursuit of a national policy for the promotion of basic research and education in the sciences,” NSF is now the funding source for approximately 27% of the total federal budget for basic research conducted at U.S. colleges and universities.

However, in recent years, a push to make NSF a more applied research agency has gained steam in Congress due largely to increased investments made by China.

“From 2000 to 2017, R&D spending in the United States grew at an average of 4.3% per year, ... But spending in China grew by more than 17% per year during the same period,” a *Nature* article overviewing the recent “State of U.S. Science and Engineering 2020” NSF report explains. “The United States accounted for 25% of the US\$2.2 trillion spent on R&D worldwide in 2017, and China made up 23%.”

These statistics have concerned U.S. government officials, and the past year only heightened the concerns. “The coronavirus pandemic has shown the science and technology gap between the United States and the rest of the world

is closing fast and that threatens our long-term health, economic competitiveness, and national security,” writes Senate Majority Leader Chuck Schumer (D-NY) in a statement. “America cannot afford to continue our decades-long underinvestment and expect to lead the world in advanced scientific and technological research.”

These concerns are a major reason why, in November 2019, Schumer first spoke publicly about a proposal to have the federal government increase support for applied research during a conference organized by the National Security Commission on Artificial Intelligence. He said the goal of the proposal was to

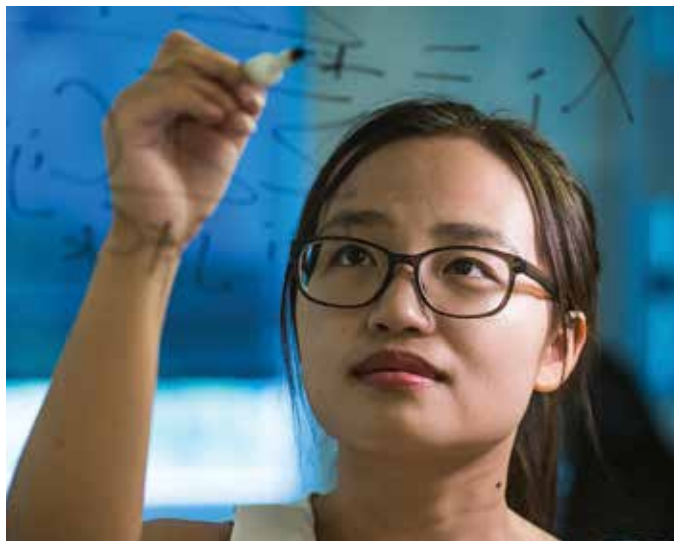
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Credit: Jeff Fallow, Rice University

**Rice University graduate student Wendy Hu, pictured, is leading development of a new technique to help clinicians obtain meaningful patient groupings. Research like Hu's that uses big data falls under the key technology areas promoted by the Endless Frontier Act.**

create a new research-funding entity provisionally called the "National Science and Technology Foundation" that would focus on emerging technologies such as artificial intelligence, quantum computing, robotics, and 5G telecommunications.

At that time, Schumer did not indicate whether the entity would be permanent or if it would expire at the end of a suggested five-year funding period. However, it became clear that the initiative was meant to be a permanent change when Schumer and three bipartisan colleagues introduced the Endless Frontier Act in May 2020.

The bill, which is named in reference to the seminal "Science—The Endless Frontier" report by Vannevar Bush, would implement two major changes to NSF. One, the bill would establish a new Directorate for Technology within NSF, bringing the total numbers of directorates to eight. Two, NSF would be renamed the National Science and Technology Foundation (NSTF) to reflect the addition of the technology directorate.

"The Directorate for Technology would fund an assortment of university centers, testbeds, fellowships, and technology consortiums, with a total recommended budget rising to \$35 billion within four years, dwarfing the agency's current \$8.3 billion topline. The bill also proposes to create a multi-billion dollar technology hub program within the Commerce Department focused on catalyzing R&D partnerships in areas that are not already leading centers of innovation," an FYI article on the bill elaborates.

While the bill's sponsors argue this reorganization of NSF is necessary to remain competitive on a global level, some leaders in the science community expressed concerns that it would dilute NSF's purpose.

"I believe it would be a mistake for a technology directorate at NSF to serve as an offset to private funding for commercial innovation and entrepreneurship," says Arden Bement, who led NSF from 2004 to 2010, in a *Science* article. "Federal funding for applied technology research and development should be need-based and channeled through mission agencies."

The Endless Frontier Act did not receive a vote before the end of the last Congress. But this February, Schumer announced that he planned to use the Endless Frontier Act as the "centerpiece" of a legislative package aimed at bolstering U.S. competitiveness with China in critical technology sectors.

Several former NSF directors and former chairs of the agency's governing body signed a letter in support of the bill (including Bement, who previously expressed reservations.)

"As former NSF directors and National Science Board Chairs with decades of government experience, we are supportive of the spirit of this legislation," they write. "We note that basic research is increasingly coupled to applications that respond to national needs. ... The Endless Frontier Act would position NSF to take up that challenge."

Besides the Endless Frontier Act, there are several other proposals for increasing applied research funding that do not involve drastically reorganizing NSF. For example, Representative Ro Khanna (D-CA) introduced separate legislation in 2020 to establish a "Federal Institute of Technology" that would fund dozens of R&D hubs across the country, while Sen. Mark Warner (D-VA) proposed creating a new division in NSF that would fund partnerships between universities and local workforce organizations.

In addition, the recently released report by the National Security Commission on Artificial Intelligence recommends establishing a new agency called the National Technology Foundation that "would complement successful existing organizations, such as the NSF and DARPA, by providing the means to more aggressively move science into engineering."

On March 26, the House Science Committee introduced a bipartisan bill that offers an alternative vision for the National Science Foundation than that of the Endless Frontier Act. Learn more about their bill at <https://bit.ly/3cQtGwj>, which proposes to double the National Science Foundation budget over five years and add a directorate to the agency focused on "societal challenges." 100

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## Welding equipment and supplies: Global markets

By BCC Publishing Staff

The total market for welding-related products is expected to reach \$37.2 billion by 2025, with a compound annual growth rate (CAGR) of 5.1% from 2021 to 2025. The growth in the safety and protective equipment segment is expected to be the highest, with a CAGR of 7.6% during the forecast period.

Welding is a fabrication process that joins material like metals and thermoplastics by applying some pressure and either with or without the presence of filler material. Advantages of welding as a joining method include considerable freedom in design, a joined piece that is as strong as the base metal, and general equipment is not costly. Disadvantages of welding include possible residual stresses and distortion of the workpieces, edge preparation of the workpiece, and potential metallurgical changes.

Welding processes can be classified by the method used in producing the welding heat and by the way the filler material is fed into the weld. Welding processes are typically classified as:

- **Arc welding:** Arc welding is a group of welding processes wherein coalescence is produced by heating with an electric

arc or arcs, mostly without the application of pressure and with or without the use of filler metal, depending upon the base plate thickness. Examples: carbon arc welding, metal inert gas welding, plasma arc welding, stud arc welding.

- **Resistance welding:** Resistance welding is a welding process wherein coalescence is produced by heat obtained from the resistance of work to the flow of electric current in a circuit of which the work is a part and by the applications of pressure. No filler metal is needed. Examples: spot welding, seam welding, projection welding, percussion welding.

- **Solid-state welding:** A solid-state welding process produces coalescence at temperatures essentially below the melting point of the base materials being joined without the addition of a filler metal. Pressure is always applied. Examples: cold welding, diffusion welding, ultrasonic welding, hot pressure welding.

- **Thermochemical welding:** The thermochemical welding process is a fusion welding process in which no outside heat is required to melt the workpieces to be joined. These welding processes involve exothermic reactions. Examples: gas welding, plastic welding, thermit welding, atomic hydrogen welding.

- **Radiant energy welding:** Radiant energy processes focus an energy beam on the workpiece. Heat is generated only when the energy beam strikes the workpiece. Examples: electron beam welding, laser beam welding, hybrid laser welding.

- **Other processes:** There are many other welding processes, some of which are used on a selective basis. Others are in the development stage. Examples: braze or bronze welding, induction welding, magnetic pulse welding.

The predicted CAGR of 5% for the arc welding technology segment is the highest among all the types of welding technologies. The CAGR of 2.3% for thermochemical welding technology is the lowest, as this includes gas welding, which is slowly becoming obsolete in many developed countries.

Various types of electrodes, filler rods, wires, fluxes, and gases are used in addition to the basic welding equipment. These are called “consumables” because they are consumed during welding. The consumables market is expected to continue increasing in value and grow at a faster rate than the equipment market because the market for consumables includes older equipment as well as new equipment.

The Asia-Pacific region is expected to increase at the highest CAGR of 5.4% due to the number of developed countries moving their industries to this region. Most of the leading welding technology companies are from the Europe and North America regions.

### About the author

BCC Publishing Staff provides comprehensive analyses of global market sizing, forecasting, and industry intelligence, covering markets where advances in science and technology are improving the quality, standard, and sustainability of businesses, economies, and lives. Contact the staff at [info@bccresearch.com](mailto:info@bccresearch.com).

### Resource

BCC Publishing Staff, “Welding Equipment and Supplies: The Global Market” BCC Research Report AVM040F, September 2020. [www.bccresearch.com](http://www.bccresearch.com). 

Table 1. Global market for welding-related products, by type of equipment, through 2025 (\$ millions)

| Type of equipment                             | 2019     | 2020     | 2021     | 2025     | CAGR%<br>(2021–2025) |
|---|----------|----------|----------|----------|----------------------|
| Welding equipment and consumables             | 18,254.2 | 17,822.2 | 19,584.6 | 23,275.1 | 4.4%                 |
| Various gases for welding                     | 6,418.8  | 6,010.8  | 6,178.1  | 7,693.7  | 5.6%                 |
| Automated and robotic welding and accessories | 3,009.5  | 2,865.9  | 3,656.9  | 4,768.6  | 6.9%                 |
| Safety and protective equipment               | 1,063.8  | 1,054.6  | 1,106.3  | 1,484.7  | 7.6%                 |
| Total   | 28,746.3 | 27,753.5 | 30,525.9 | 37,222.0 | 5.1%                 |



# Into the Bulletin Archives—A look back at our 100 years in print

Since May 1922, the *ACerS Bulletin* has served the ACerS community, providing them updates on member news, Division meetings, and the latest research in ceramics and glass.

In celebration of Volume 100 this year, the *Bulletin* editorial team is running a special column in each issue of the 2021 *Bulletin* that looks at the history of the *Bulletin* by decade. This issue highlights the 1950s.

We hope you enjoy following the journey of the *Bulletin* from its early years to today. As an ACerS member, you have access to all 100 years of the *Bulletin* on the *Bulletin Archive Online* at <https://bulletin-archive.ceramics.org>. <sup>100</sup>

## Into the Bulletin Archives—1950s

From the very first issue of this decade, the 1950s *Bulletins* took a significant turn in terms of cover art—instead of real-life images, simple hand-drawn illustration were used. While the artwork generally featured machinery and parts, December issues were holiday themed.

For the Society, the 1950s is a significant decade because, for the first time since its founding, the Society raised enough money to build its own headquarters building in Columbus, Ohio. The building, which was located near the Park of Roses, was dedicated on

December 4, 1954. The January 1955 issue reported on the event, including a quote from ACerS general secretary Charles S. Pearce on how the building came to be.

“...it was back in 1938 that G. G. Hanson of Consolidated Feldspar insisted that The Society should have an office building of its own, a ceramic structure of which it could be justly proud, and presented his check for \$10 to start the building fund. This first contribution was carried on the books for 12 years before it had company and the action that culminated in the new building got under way.”

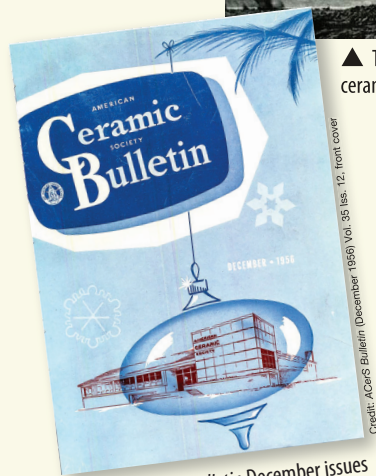
—ACerS *Bulletin*, Vol. 34.,  
Iss. 1., January 1955

The Society added two Divisions during the 1950s. Basic Science was announced in the March 1951 issue as being “composed of those members who are interested in the fundamental facts, principles, and theories that underlie the various manufacturing processes of the ceramic industry.” Electronics was announced in January 1958, and in the following February issue, a couple of corporate members expressed their approval of the new Division, such as A. J. “Jo” Gitter of Whittaker, Clark & Daniels, Inc.



Credit: ACerS *Bulletin* (January 1955) Vol. 34 Iss. 1, p. 19

▲ The first Society-owned headquarters building was considered “a true ceramic show place,” being made from brick, terra cotta, glazed tiles, and more.



Credit: ACerS *Bulletin* (December 1956) Vol. 35 Iss. 12, front cover

▲ The cover of *Bulletin* December issues in the 1950s were holiday themed, like this one from 1956.



Credit: ACerS *Bulletin* (July 1957) Vol. 36 Iss. 7, p. 283

▲ ACerS held its first ceramographic exhibit in 1957 during the 59<sup>th</sup> Annual Meeting in Dallas, Texas. Opinion surveys on the event unanimously recommended that it be continued annually. Pictured is one of the entries: an electron micrograph showing the nature of the surface structure development of a barium titanate dielectric material.



## DIVISIONS OF THE SOCIETY

During the 1950s, the Society had nine Divisions.

- **Basic Science (new)**
- Design
- **Electronics (new)**
- Enamel
- Glass
- Materials and Equipment
- Refractories
- Structural Clay Products
- White Wares

"In my opinion, this is a forward moving step. In the past several years I have had many, many contacts with the electronic industry and increasingly I felt that somewhere it would have to 'hone' for certain types of components with which the electrical engineering field are not really familiar."

—ACerS Bulletin, Vol. 37., Iss. 2.,  
February 1958

In contrast to the approval surrounding the new Electronics Division, another member felt quite differently about the existing Design Division. "After 25 years closely associated with the art movement in ceramics, ... I am still thoroughly convinced that the weakest link in American ceramics today is Design," William Manker wrote in a December 1951 letter. "What we are lacking and that which we are primarily interested in—the Design Division—should and must continue to be an integrated part of The American Ceramic Society."

Manker offered several suggestions that he believed would help the Design Division flourish, and in January 1953, some of his suggestions were fulfilled when members Spencer Davis and Louis G. Farber launched a new magazine called *Ceramics Monthly*.

"The idea for a magazine devoted exclusively to the total ceramic art and craft field did not originate entirely in the minds of the publishers. The actions and comments of the thousands of active but much neglected ceramists stimulated the idea. ... Here then is the line of communication so obviously needed by individuals from all walks of ceramic life."

—*Ceramics Monthly*, Vol. 1,  
Iss. 1., January 1953

Over 40 years later, in 1996, the Society would acquire *Ceramics Monthly*. Learn more

about *Ceramics Monthly* and explore recent issues at <http://bit.ly/CeramicsMonthly>.

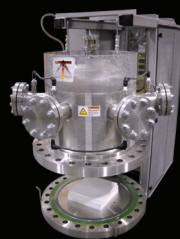


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# SOCIETY DIVISION SECTION CHAPTER NEWS



## Welcome new ACerS Corporate Partners

ACerS is pleased to welcome its newest Corporate Partner:  
– Uncountable



To learn about the benefits of ACerS corporate partnership, contact Kevin Thompson, membership director, at (614) 794-5894 or [kthompson@ceramics.org](mailto:kthompson@ceramics.org). <sup>100</sup>



## Find the materials and supplies you need in *ceramicSOURCE*

With more than 80 pages of listings, ACerS' annual buyer's guide *ceramicSOURCE* 2021 is certain to help you find the perfect materials, equipment, consultants, providers, supplies, and more. Whether you are looking for additives, refractories, a new kiln, laboratory services, dinnerware, or glass products—and everything in between—*SOURCE* has you covered.

Be a part of ACerS online resource directory and printed buyer's guide by adding your company's free listing at <https://ceramicsource.org>. ACerS Corporate Partners may enhance their listing by adding logos, white papers, PDFs, and more. <sup>100</sup>

## Ceramic Tech Chat: Yuichi Ikuhara

Hosted by ACerS Bulletin editors, *Ceramic Tech Chat* talks with ACerS members to learn about their unique and personal stories of how they found their way to careers in ceramics. New episodes publish the second Wednesday of each month.

In the March episode of *Ceramic Tech Chat*, Yuichi Ikuhara, professor of engineering innovation at the University of Tokyo, discusses his work using scanning transmission electron microscopy to characterize grain boundaries in ceramics, the recent advances that have greatly improved this technique's resolution capabilities, and where he sees TEM headed in the future.

Check out a preview from his episode, which features Ikuhara discussing the big advances in TEM he has seen over the course of his career.

"Previously, we are studying conventional transmission electron microscopy. So, we can observe the microstructure, but not so high resolution. But in this century, we have a very big evolution, that is spherical aberration corrected scanning transmission electron microscopy. This is very powerful technique, and the resolution of Cs-STEM is now less than 1 angstrom. Actually, we have a very high spatial resolution less than 0.5 angstrom. This means that you can even observe the hydrogen columns or lithium columns in materials."

Listen to Ikuhara's whole interview—and all of our other *Ceramic Tech Chat* episodes—at <http://ceramictechchat.ceramics.org/974767>. <sup>100</sup>



**Scanning TEM unlocks grain boundary secrets:  
Yuichi Ikuhara**



## Volunteer spotlight

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



Xiong

**Hui (Claire) Xiong** is associate professor in the Micron School of Materials Science and Engineering at Boise State University. Xiong received her Ph.D. in electroanalytical chemistry from the University of Pittsburgh. She conducted postdoctoral work at Harvard University and Argonne National Laboratory. Xiong received the NSF CAREER Award in 2015. She also is a CAES fellow and a Scialog Fellow.

Xiong is an active member of ACerS and serves as an officer of the Electronics Division. She was one of the lead organizers for the Electronic Materials and Applications conferences in 2020 and 2021.

We extend our deep appreciation to Xiong for her service to our Society! 100

## ECD seeks secretary nominations

The Engineering Ceramics Division invites nominations for the incoming 2021–2022 Division secretary. Nominations and a short description of the candidate's qualifications should be submitted to Surojit Gupta at [surojit.gupta@und.edu](mailto:surojit.gupta@und.edu) by **July 21, 2021**. 100

## IN MEMORIAM

Mark Davis

John May

Lynn Owen

Don Steffen

Some detailed obituaries can also be found on the ACerS website, [www.ceramics.org/in-memoriam](http://www.ceramics.org/in-memoriam).



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Title: *Digitalization and the Future of Formulating in Modern R&D Labs*

Cost: Free

**MAY 11**  
**11 AM – 12 PM EST**

ACerS Corporate Partner Uncountable is sponsoring the complimentary webinar “Digitalization and the future of formulating in modern R&D labs” on **Tuesday, May 11, 2021**.

The biggest lever that R&D leaders have today is to implement better data tools that make the learning process faster and easier. Will Tashman, co-founder of Uncountable, will discuss the benefits of implementing a true data infrastructure and the challenges that organizations will face along this journey.

For more information and to register, visit <http://bit.ly/Uncountable>. 100



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# AWARDS AND DEADLINES



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[ceramics.org/members/awards](http://ceramics.org/members/awards)



| Society Awards      | Nomination Deadline | Contacts  | Description   |
|---------------------|---------------------|---|---|
| <b>ACerS Fellow</b> | Sept. 2, 2021       | Erica Zimmerman<br><a href="mailto:ezimmerman@ceramics.org">ezimmerman@ceramics.org</a> | Elevation to ACerS Fellow recognizes outstanding contributions to the ceramic arts or sciences through broad and productive scholarship in ceramic science and technology, by conspicuous achievement in ceramic industry, or by outstanding service to the Society. Visit <a href="http://bit.ly/SocietyFellows">http://bit.ly/SocietyFellows</a> to download the nomination form. |

| Division                    | Award                                      | Nomination Deadline |
|-----------------------------|--|---------------------|
| <b>GOMD</b>                 | <b>Alfred R. Cooper Scholars</b>           | May 15              |
| <b>Electronics</b>          | <b>Edward C. Henry</b>                     | May 15              |
| <b>Electronics</b>          | <b>Lewis C. Hoffman Scholarship</b>        | May 15              |
| <b>Bioceramics</b>          | <b>Young Scholar</b>                       | July 1              |
| <b>Bioceramics</b>          | <b>Global Young Bioceramicist</b>          | July 1              |
| <b>Bioceramics</b>          | <b>Larry L. Hench Lifetime Achievement</b> | July 1              |
| <b>Bioceramics</b>          | <b>Tadashi Kokubo</b>                      | July 1              |
| <b>Engineering Ceramics</b> | <b>Jubilee Global Diversity</b>            | July 1              |



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Michael Halbig  
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### Description

The award recognizes undergraduate students who have demonstrated excellence in research, engineering, and/or study in glass science or technology.

The 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.

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

The award recognizes excellence in research among current degree-seeking graduate students and postdoctoral research associates.

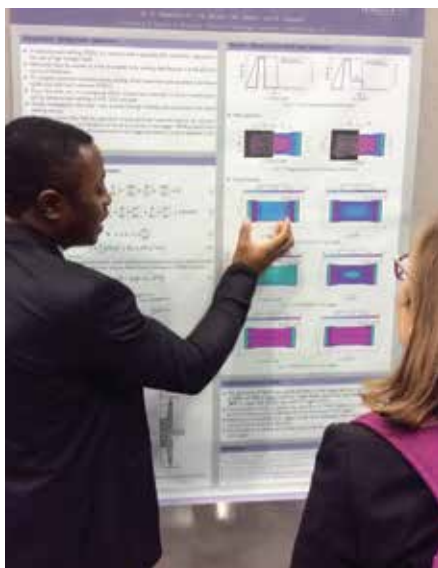
The award recognizes the outstanding young ceramic engineer or material scientist who has made significant contributions to the area of bioceramics for human healthcare around the globe.

The award is presented to a deserving individual(s) in recognition of lifetime dedication, vision, and accomplishments in advancing the field of bioceramics, particularly toward innovation in the field and contribution of that innovation to translation of technology toward clinical use.

The award is presented in recognition of outstanding achievements in the field of bioceramics research and development.

The award recognizes exceptional early- to mid-career professionals who are women and/or underrepresented minorities (i.e., based on race, ethnicity, nationality, and/or geographic location) in the area of ceramic science and engineering.

# STUDENTS AND OUTREACH



FOR MORE  
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**Yolanda Natividad**  
**ynatividad@ceramics.org**

## MS&T21 speakers: You may be eligible for the GEMS award!

The Basic Science Division organizes the annual Graduate Excellence in Materials Science (GEMS) awards to recognize the outstanding achievements of graduate students in materials science and engineering. The award is open to all graduate students who are making an oral presentation in any symposium or session at the Materials Science & Technology (MS&T) meeting.

In addition to their abstract submission, students also must submit a nomination packet to the chair of the GEMS award selection committee, John Blendell, by **Sunday, Aug. 15, 2021**. For further details, go to [www.ceramics.org/GEMS](http://www.ceramics.org/GEMS). <sup>100</sup>

## An abundance of student opportunities available at MS&T21

There are many opportunities available at this year's MS&T. Make sure to sign up for the following student contests:

- Undergraduate student poster contest
- Undergraduate student speaking contest
- Graduate student poster contest
- Ceramic mug drop contest
- Ceramic disc golf contest
- Humanitarian pitch competition
- ...and MORE!

For more information on any of the contests or student activities at MS&T, visit [www.matscitech.org/students](http://www.matscitech.org/students), or contact Yolanda Natividad, member engagement manager. <sup>100</sup>

## ACerS GGRN for young ceramic and glass researchers

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

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- Engage with The American Ceramic Society,
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## ACerS Associate Membership and Young Professionals Network

The American Ceramic Society offers one year of Associate Membership at no charge for recent graduates who have completed their terminal degree. Join today to receive the benefits of membership in the world's premier membership organization for ceramics and glass professionals. Start your free year-long membership by visiting [www.ceramics.org/associate](http://www.ceramics.org/associate) or contact Yolanda Natividad, member engagement manager.

Also, consider joining ACerS Young Professionals Network (YPN) once you have become an ACerS member. YPN is designed for members who have completed their degree and are 25 to 40 years of age. YPN gives young ceramic and glass scientists access to invaluable connections and opportunities. For more information, visit [www.ceramics.org/ypn](http://www.ceramics.org/ypn). You may join this growing community by logging in to your ACerS Personal Snapshot and marking "Young Professionals Network (YPN)" under the Interest Groups and Communities tab. <sup>100</sup>



# CERAMIC AND GLASS INDUSTRY FOUNDATION

## 2020 GRATITUDE REPORT



*We thank our generous donors for their support of our programs to attract, inspire, and train the next generation of ceramic and glass professionals. Despite the difficulties encountered due to the pandemic, we are pleased to present to you some highlights of 2020—examples of how your gifts have impacted and motivated students.*

### CGIF presents 2020 Gratitude Report

The Ceramic and Glass Industry Foundation is pleased to share its 2020 Gratitude Report—an overview of the year's highlights presented in appreciation for all of those who have donated to support our endeavors. We know that the growth of our outreach programs would not be possible without our donors, thus we offer our Gratitude Report to show what YOU have accomplished with your gifts of support.

Our mission of inspiring and training the next generation of ceramic and glass professionals is being fulfilled every day because of our donors. Despite the pandemic, your gifts enabled us to launch a new materials science teaching resource, our Mini Materials Demo Kit, a collection of seven simple demonstrations for use by parents, teachers, and students who are utilizing online and at-home teaching resources.

We invite you to read more about what your donations have done for students who are impacted through the CGIF at <http://foundation.ceramics.org/2020Report>. As always, we truly appreciate you and your gifts of support.

If you would like to provide a gift to the CGIF to support our work, please contact CGIF development director Marcus Fish at [mfish@ceramics.org](mailto:mfish@ceramics.org) or 614-794-5863. You may also donate online at <https://myacers.ceramics.org/donate>. <sup>100</sup>

### VIRTUAL MEETING

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[ceramics.org/cements2021](https://ceramics.org/cements2021) » June 23–25, 2021



## Researchers confirm existence of unique nonlinear optical response in nanocrystal-in-glass composites

Researchers in China discovered nanocrystal-in-glass composites exhibit a unique nonlinear optical response called transverse second-harmonic generation that would allow for the creation of light sources with expanded capabilities.

Nonlinear optics refers to the study of a wide range of interesting physical phenomena based on nonlinear relationships. “Nonlinear” means there is not a direct or straight-line relationship between variables. In other words, nonlinear materials and processes exhibit responses that are disproportionate compared to the external force(s) triggering it.

In the case of nonlinear optics, the nonlinear optical process called second-harmonic generation is highly studied because of its ability for high-resolution imaging. In this process, which is also known as frequency doubling, photons that interact with a nonlinear material will “combine” to form new photons with twice the frequency of the initial photons. This phenomenon allows for the creation of low-power and compact light sources that operate in wavelengths typically requiring more power and larger devices.

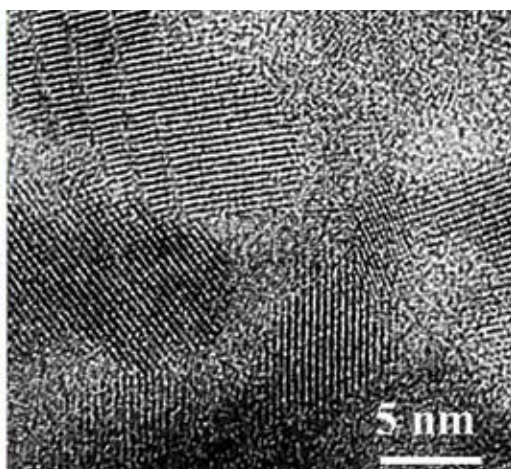
Since second-harmonic generation was identified in 1961, there now are numerous commercial devices based on this phenomenon

available for purchase. These devices mainly use various crystals as the nonlinear material. However, researchers have started exploring the potential of using nanocrystal-in-glass composites instead.

Nanocrystal-in-glass composites are composed of tiny crystals embedded in a glassy matrix. These composites have emerged as an alternative to glasses and crystals in display applications because of some superior optical properties. For example, nanocrystal-in-glass composites containing high-quartz nanocrystals present zero thermal expansion over a wide temperature range of 0–50°C, which makes them ideal for use as optical mirror blanks in ultrastable space telescopes.

Some nanocrystal-in-glass composites, such as those embedded with  $\text{LiNbO}_3$  and  $\text{LiNb}_{0.5}\text{Ta}_{0.5}\text{O}_3$ , are nonlinear materials and so have potential for use in second-harmonic generation devices. However, the recent study led by professors Shifeng Zhou and Huakang Yu from South China University of Technology reveals these composites may have additional potential not previously realized.

Originally, their goal was to study the nonlinear optical response of nanocrystal-in-glass composites with different



Optical microscopy (left) and transmission electron microscopy (right) images of a glass composite embedded with  $\text{LiNbO}_3$ . Researchers in China discovered this composite exhibits a unique nonlinear optical response that is rarely seen in bulk materials.

Credit: Shifeng Zhou

## Research News

### Concrete on the double

A team of researchers at Oak Ridge National Laboratory and the University of Tennessee developed a concrete mix that demonstrates high early strength within six hours of mixing, potentially doubling the production capacity for the precast industry. Quick performing concrete shortens manufacturing time for prefabricated assemblies, but these early-strength mixes have short setting times and require specific curing methods. The researchers evaluated commercially available components including steel, glass, and carbon fibers. The result was a self-compacting mix that not only showed early strength but also maintained its workability for 30 minutes. For more information, visit <https://www.ornl.gov/news>. <sup>100</sup>

### Scientists obtain high-entropy carbide using electric arc plasma

Scientists at Tomsk Polytechnic University in Russia synthesized a high-entropy carbide consisting of five various metals using a vacuum-free electric arc method. High-entropy carbides are a new class of materials simultaneously consisting of four or more various metals and carbon. Their main feature lies in the capability to endure high temperatures and energy flux densities. The high-entropy carbide created by the scientists consists of titanium, zirconium, niobium, hafnium, tantalum, and carbon. They plan to explore synthesizing high-entropy carbides of other chemical compositions using this method as well. For more information, visit <https://news.tpu.ru/en/news>. <sup>100</sup>

domain structures in a counter-propagating laser scheme, i.e., a scheme where two laser beams enter the composite from opposite sides and interact.

While the nonlinear optical response in certain regions of the LiNbO<sub>3</sub>-embedded composites was as expected, the researchers surprisingly observed a local and brighter signal in an extreme region of the microstructure map—a signal that disappeared when either of the two laser beams were blocked.

“It is strange that this brighter signal would vanish while the background second-harmonic generation still existed when either individual pulse is blocked,” Zhou explains in an email.

They realized that the brighter signal must be due to a special nonlinear response called transverse second-harmonic generation. Transverse second-harmonic generation differs from standard second-harmonic generation in two significant ways.

**1. Phase matching mode:** Standard second-harmonic photons are generated from the same pulse, whereas transverse second-harmonic photons are generated from oppositely propagating pulses.

**2. Generation condition:** The only requirement for achieving standard second-harmonic generation is to fulfill the phase matching condition, i.e., minimize mismatch between interacting waves along the propagation direction. Transverse second-harmonic generation requires that, in addition to the phase matching condition, overlap of the oppositely propagating pulses in time and space must occur as well.

This difference in origin means that standard second-harmonic generation and transverse second-harmonic generation manifest differently as well. While the signal from standard second-harmonic generation can easily be observed at any position where the fundamental pulse passes, the signal from transverse second-harmonic generation can only be observed at the position where oppositely propagating pulses overlap.

The discovery of transverse second-harmonic generation in nanocrystal-in-glass composites is surprising because the phenomenon is typically observed in low-dimension nonlinear

materials—seeing it in bulk nonlinear materials is rare. However, the fact it was discovered for the first time in a microstructured nonlinear photonic glass is exciting because of what it offers in terms of application.

“Standard second-harmonic generation is usually used for laser frequency conversion,” Zhou says. In contrast, because the transverse second-harmonic generation signal reflects the time domain information of the fundamental pulses, “the signal can be used for pulse group velocity and pulse width measurement of ultrashort optical pulses with femtosecond time scales.”

While this study focused on nanocrystal-in-glass composites embedded with LiNbO<sub>3</sub>, “It should be noted that the strategy should be universal to a wide category of nanoparticle-in-glass composites,” Zhou says. He adds that the researchers are now working on developing a novel fabrication strategy to further expand the nanocrystal-in-glass composites to more extreme regions, and they are exploring the composites’ potential for other unique properties and new practical applications.

The paper, published in *Advanced Materials*, is “Manipulating nonlinear optical response via domain control in nanocrystal-in-glass composites” (DOI: 10.1002/adma.202006482). <sup>100</sup>

### Fast *p*-type oxide semiconductor rolled into life with liquid metal

An RMIT-led research team has introduced ultrathin beta-tellurite to the 2D semiconducting material family, providing an answer to the decades-long search for a high mobility *p*-type oxide. There are two types of semiconducting materials: “*n*-type,” which have abundant negatively-charged electrons; and “*p*-type,” which possess plenty of positively-charged holes. A barrier to oxide devices has been that while many high-performance *n*-type oxides are known, there is a significant lack of high-quality *p*-type oxides. The new beta-tellurite is a *p*-type, and the researchers isolated it using a specifically developed synthesis technique that relies on liquid metal chemistry. For more information, visit <http://www.fleet.org.au/news>. <sup>100</sup>

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## Researchers demonstrate potential of MXenes as additives in ultrahigh-temperature ceramics

Researchers at Indiana University–Purdue University Indianapolis investigated the phase stability and transformation of titanium carbide MXenes at high temperatures to better understand its potential as an additive in ultrahigh-temperature ceramics.

Titanium carbide ( $\text{Ti}_3\text{C}_2\text{T}_x$ ) is the most studied layered carbide from the MXene family. It is known to have impressive material properties, including high in-plane mechanical stiffness ( $330 \pm 30$  GPa), high electrical conductivity ( $10,000$ – $20,000$   $\text{S} \cdot \text{cm}^{-1}$ ), and high temperature phase stability (up to  $800^\circ\text{C}$  under inert environments).

Recently, researchers have investigated using titanium carbide MXenes as additives in metal and ceramic matrix composites to endow the composites with improved in-matrix mechanical and electrical properties. However, this application faces a slight obstacle—these composites require processing at relatively high temperatures, and titanium carbide MXenes exhibit variable rates of oxidation and degradation when exposed to high temperatures in the presence of oxygen.

Technically, “all the ultrahigh-temperature ceramics (carbides, nitrides, and borides) get oxidized in air. But the oxide shell is too thin (at relatively low temperatures) that we don’t discuss them,” explains Babak Anasori, assistant professor at Indiana University–Purdue University Indianapolis, in an email. “But if we make UHTCs at 1-nm size [as is the case with MXenes], then oxidation becomes a major issue.”

Processing in low-oxygen environments offers a partial solution, but researchers still need to consider the possible effect of high temperatures on the MXene’s electrical, catalytic, and mechanical properties. In particular, early studies have established that titanium carbide MXenes undergo a three-stage

phase transformation process from a 2D flake to a 3D crystal-line structure when annealed at high temperatures:

1. Low-temperature annealing up to  $200^\circ\text{C}$  results in loss of water molecules trapped between the MXene layers.

2. Medium-temperature annealing causes desorption of functional groups. Specifically, temperatures of  $300$ – $500^\circ\text{C}$  result in removal of  $-\text{OH}$  groups,  $-\text{F}$  groups desorb at  $500$ – $750^\circ\text{C}$ , and complete desorption of all  $-\text{F}$  and  $-\text{OH}$  groups occurs above  $800^\circ\text{C}$ .

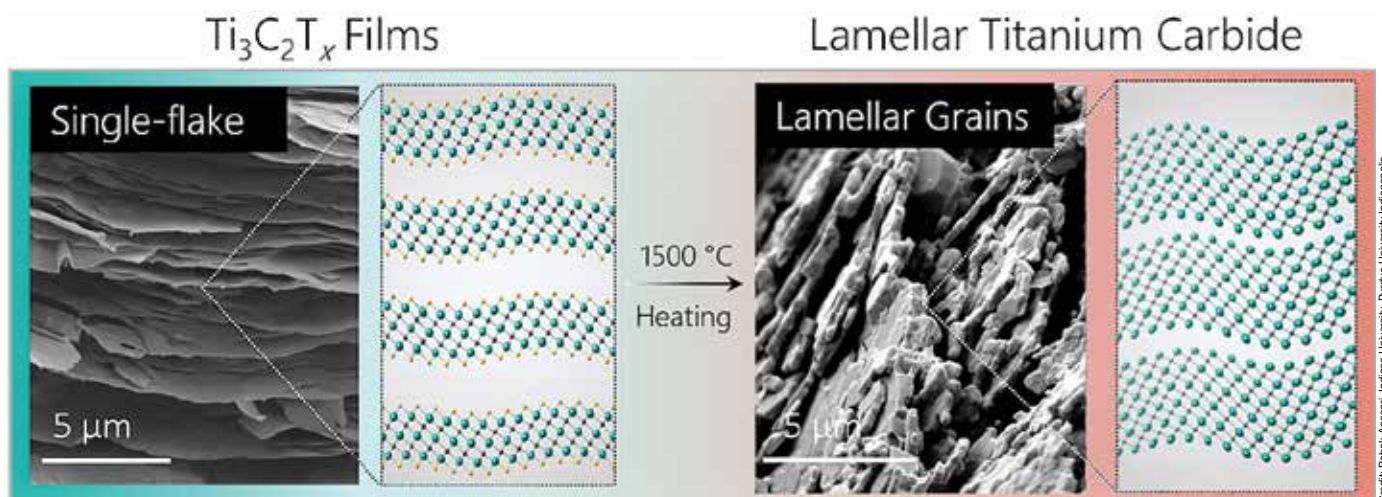
3. High-temperature annealing above  $800^\circ\text{C}$  causes partial phase transformation to cubic  $\text{TiC}$ , while full transformation occurs at temperatures above  $1,200^\circ\text{C}$ .

Processing at temperatures above  $800^\circ\text{C}$  is required for many metal and ceramic matrix composites, yet detailed characterization of the phase transformations at these temperatures is not complete. Fortunately, the new study led by Anasori looks to fill this gap in knowledge.

The researchers explain that titanium carbide MXenes offer a unique advantage compared to other 2D materials when it comes to investigating the material properties. “Fundamentally,  $\text{Ti}_3\text{C}_2\text{T}_x$  is a nano-lamellar titanium carbide, which means that  $\text{Ti}_3\text{C}_2\text{T}_x$  has compositional similarity to the bulk crystalline three-dimensional (3D) nano-lamellar transition metal carbides that have been studied for the past  $>50$  years,” they write. As such, “We can use previously established literature on the bulk carbides as a guide on the potential phase transformation behavior of  $\text{Ti}_3\text{C}_2\text{T}_x$  during high-temperature annealing.”

Based on what is known about bulk carbides, the researchers hypothesized that titanium carbide MXenes would have two main ranges of phase transition:

1. Low-temperature phase transition to a mix of 3D crystalline  $\text{Ti}_2\text{C}$  +  $\text{TiC}$  at temperatures of  $700$ – $1,000^\circ\text{C}$ .



Researchers at Indiana University–Purdue University Indianapolis showed titanium carbide MXene flakes undergo a phase transformation upon sintering to become a stable, bulk 3D crystal.

2. High-temperature phase transition to purely  $\text{TiC}_y$  at temperatures above  $1,000^\circ\text{C}$ .


The researchers used wet chemical selective etching to synthesize two different forms of titanium carbide MXene films for their experiments: nondelaminated multilayer stacks (clay) and delaminated single-to-few layers of flakes (single-flake). Then, they investigated the phase transformations using in-situ heated X-ray diffraction paired with a 2D detector (up to  $1,000^\circ\text{C}$ ) and ex-situ methods using tube furnace and spark plasma sintering for high-temperature annealing (up to  $1,500^\circ\text{C}$ ).

The results confirmed their original hypothesis. In particular, they found that the titanium carbide MXene keeps its 2D layered structure up to  $600^\circ\text{C}$ , after which stable 3D crystalline phases of  $\text{Ti}_2\text{C}$  and  $\text{TiC}_y$  are formed. These phases remain stable up to annealing temperatures of  $1,000^\circ\text{C}$  in both single-flake films and multi-layer clay films, after which there is a subsequent transition to cubic  $\text{TiC}_y$  above  $1,000^\circ\text{C}$ .

In an email, Ph.D. student Brian Wyatt explains why confirmation of this transition from a 2D to 3D crystalline phase is an important finding. Generally, the layers of 2D materials are held together by weak van der Waals forces. For 2D materials such as graphene or hexagonal boron nitride, the van der Waals bonds can become weak spots in the composites.

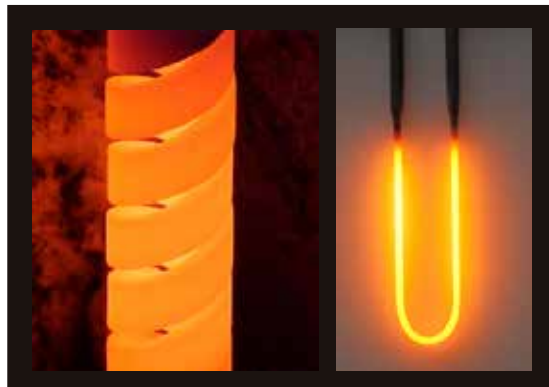
"In contrast, we can turn MXenes' inter-flake van der Waals bonds to primary bonds through high-temperature sintering," Wyatt says. This transformation will allow the MXene to "behave as a type of 'glue' within UHTCs to improve the bonding between ceramic grains, and thus form fracture and thermal shock resistance UHTCs with improved oxidation properties."

Anasori's group is already working on forming MXene UHTC composites using different ceramic materials and exploring their mechanical and oxidation properties. "We believe MXenes for the extreme environment will become a new and fast-growing research area," Ph.D. student S. Kartik Nemani says in an email.

The paper, published in *Journal of Physics: Condensed Matter*, is "High-temperature stability and phase transformations of titanium carbide ( $\text{Ti}_3\text{C}_2\text{T}_x$ ) MXene" (DOI: 10.1088/1361-648X/abe793). 

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## Hydrogenation stabilizes borophene for practical use

Researchers led by Northwestern University researchers, along with colleagues from the University of Florida and Argonne National Laboratory, experimentally investigated the hydrogenation of borophene to see how well it stabilizes the material for practical use.

Borophene, a 2D sheet of boron atoms, was first theorized in the 1990s and experimentally confirmed in 2015. Much like graphene, borophene is flexible, strong, and lightweight. It also forms in several different crystal structures (polymorphs), so structural manipulation of borophene is easier than it is for graphene. For these reasons, some feel borophene may serve certain applications such as wearable device applications better than graphene.

However, borophene rapidly oxidizes in air, which means integrating borophene into practical devices is difficult. To date, experimental characterization of borophene has required ultrahigh-vacuum conditions to avoid unwanted reactions with air.

Fortunately, passivation may address borophene's volatility. Passivation refers to processes used to reduce the chemical reactivity of a material.

Hydrogenation, or reaction with hydrogen, is one type of passivation process that first-principles calculations suggest could be used to stabilize borophene and suppress ambient oxidation.

While theoretical explorations of hydrogenated borophene, or "borophane," have taken place, "atomically well-defined synthesis and characterization of borophane polymorphs have not yet been achieved," the researchers explain in the paper. So, their aim was to investigate this synthesis.

To create borophane, they exposed borophene to atomic hydrogen in ultrahigh-vacuum conditions. Then, they extensively studied the bonding structure and properties by combining scanning tunneling microscopy and spectroscopy, inelastic electron tunneling spectroscopy, and density functional theory.

Similar to the high degree of polymorphism in borophene, the researchers observed eight different borophane polymorphs. All of the polymorphs demonstrated markedly improved chemical and morphological stability in ambient conditions. In particular, degradation in borophane was only detected after 1 week of ambient exposure.

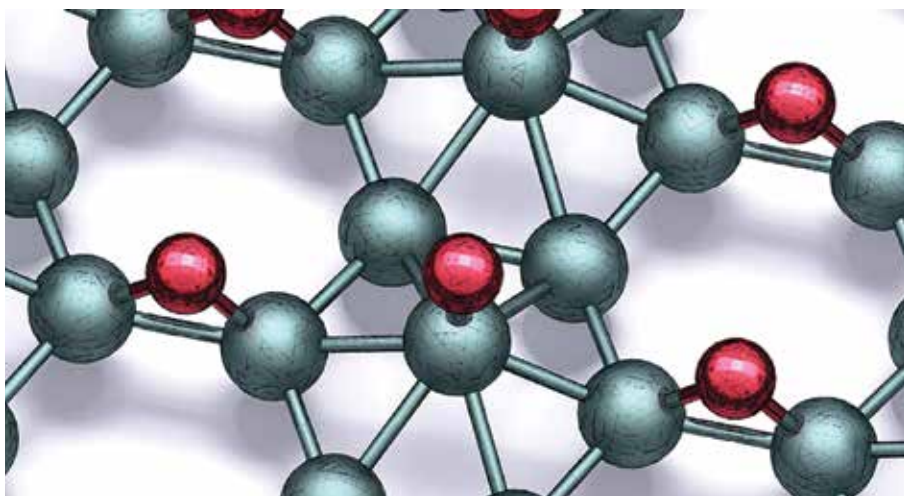
"This oxidation peak is clearly smaller than that of the borophene sample after 1 hour of ambient exposure, indicating that the oxidation rate is reduced by more than two orders of magnitude," they write. These findings show "that diverse hydrogen-bonding motifs on borophene impart ambient stability."

In addition to providing ambient stability on the order of days, which is a sufficient time window for most ambient characterization and processing methods, the researchers also found that the hydrogenation of borophene was reversible using thermal annealing.

"Specifically, borophane samples could be recovered to pristine borophene without apparent degradation after annealing the sample to ~300°C," they write. This finding is significant because it means pristine borophene can be regained once ambient processing is complete or robust encapsulation layers are applied.

In a Northwestern University press release, senior author and Walter P. Murphy Professor of Materials Science and Engineering Mark C. Hersam says their findings will allow researchers to more rapidly explore borophane's properties and its potential applications.

The paper, published in *Science*, is "Synthesis of borophane polymorphs through hydrogenation of borophene" (DOI: 10.1126/science.abg1874). <sup>100</sup>



Hydrogenating the 2D material borophene offers a way to stabilize it against ambient oxidation. In this image, grayish teal balls represent boron and red balls represent hydrogen.

### LIVE VIRTUAL CONFERENCE

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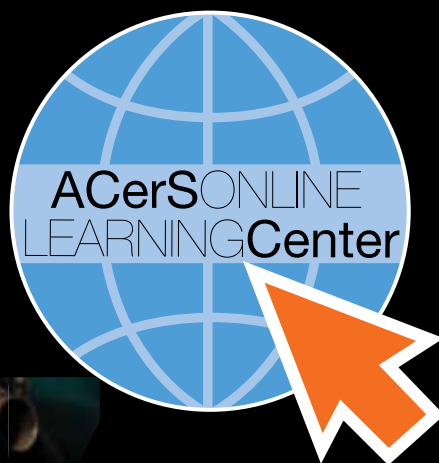
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# From concept to industry: Ultrafast laser welding

By Richard M. Carter

Ultrashort pulse laser welding has the potential to transform optomechanical component manufacturing—and research around the world is helping to move this technique from concept to industry.

When manufacturing optomechanical components, one of the more problematic steps is bonding the optical components to a structural body. Such optical components can include a whole variety of glass and crystal materials, but these materials are normally chosen for their optical properties rather than their structural or mechanical performance. Because it usually is inadvisable to manufacture the structural chassis from similar (i.e., glass) materials, this decision inevitably leads to a significant mismatch between the properties of the optic and its mount, most significantly in their thermal properties.

There are essentially two options when joining dissimilar materials. The first option involves using some form of clamp or mechanical fixing. In this case, the mismatch in thermal properties is less of an issue; however, such an approach requires additional design engineering and several manufacturing steps. Thus cost, mass, volume, and scope for misalignment can all increase, which can significantly impact applications.

The other option is to use some form of bonding interlayer. This interlayer can take the form of an adhesive, frit, or solder, for example, depending on the precise materials involved. However, creating a reproducible manufacturing process with these materials is rarely as simple as one might wish. For example, issues in the use of adhesives for high-precision optics are well known and can be attested to by

## Capsule summary

### A SHORT HISTORY

Since researchers at Osaka University (Japan) published a paper in 2005 showing that ultrashort laser pulses could weld transparent materials together, a substantial amount of research worldwide has demonstrated new material combinations as well as generated knowledge on fundamental laser–material interactions.

anyone who has attempted to develop a reproducible high-precision adhesive process. Therefore, in general, considerable time and money usually are needed to develop sufficient art for a reliable, high-precision process.

Furthermore, these bonding processes fundamentally rely on the introduction of an interlayer in some form, which often generates its own problems. A common manifestation is creep, or part movement during curing. But often more problematic is material aging performance, with outgassing a particularly significant issue for systems where strict atmospheric control needs to be maintained to avoid reducing system lifetimes and/or performances (e.g., vacuum systems and some lasers).

Because of these limitations, an alternative, direct joining technique would be preferred. The obvious solution therefore would be to directly weld the two components together.

Be it a torch, arc, friction, or laser, welding relies on a source of thermal energy to locally melt material over a scale of millimeters to centimeters. Depending on the technique, the welding process may also involve adding a “filler” material to bridge a gap between the parts. The materials then will mix in a liquid state, potentially combine chemically, and cool to form a welded bond.

When dealing with broadly similar materials, this process generally works extremely well, with the welded material often stronger than the bulk. However, this process breaks down when considering dissimilar material welding, largely due to differences in thermal expansion—when a material begins to solidify, thermal stresses accumulate and result in an almost immediate failure of the weld. Thermal stresses can be problematic when considering two different metals, but the issue is even more extreme for

### BROAD POTENTIAL

The applicability of the technique is very broad, from the types of materials welded together to the wavelength of laser used, thus giving laser manufacturers the ability to offer a suitable system provided they have sufficient knowhow of the focusing and material handling requirements.

optical and mechanical components as these materials can differ in expansion rates by an order of magnitude or more.

Because joining of dissimilar materials is still fundamentally desired, the trick would seem to be to minimize the volume of material that needs to be heated, thus reducing the total amount of thermal stress introduced. Minimizing the volume of heated material can be best achieved by limiting the total thermal energy introduced around the weld zone, i.e., to use a very focused source of heat. The laser is an ideal tool for localized heating because the energy can be deposited exactly where it is required by focusing and scanning the beam across the material interface.

Of course, the use of lasers for welding is not at all new. But there is a significant difference in capabilities depending on what type of laser is used. In general, lasers can be divided into two broad categories. The first is “continuous wave lasers.” As the name suggests, these lasers provide a continuous beam of light. If directed onto a material, it provides a continuous thermal input very much like a welding torch and so is not terribly well suited to preventing thermal expansion issues during welding.

The alternative is to pulse the output of the laser because it is then possible to separate the peak intensity in the pulse from the average power delivered by temporally concentrating the energy. The pulse duration and the repetition rate are thus both key factors in how the laser–material interaction will work—and thus key to controlling the thermal profile during welding. For most industrial laser processes, pulse durations are in the order of milli- to nanoseconds ( $10^{-3}$  to  $10^{-9}$  s). While short, this duration does not provide a sufficiently high localization of thermal energy to allow for dissimilar material welding. Instead, an

### BUDDING INDUSTRY

A few companies have adopted ultrashort pulse laser welding as an industrial process because, despite the high cost, this technique offers the key advantages of a limited thermally affected zone, the ability to join a range of materials, and the ability to do so without introducing an interlayer.

“ultrashort” pulsed laser is required, one which produces laser pulses in the order of pico- to femtoseconds ( $10^{-12}$  to  $10^{-15}$  s).

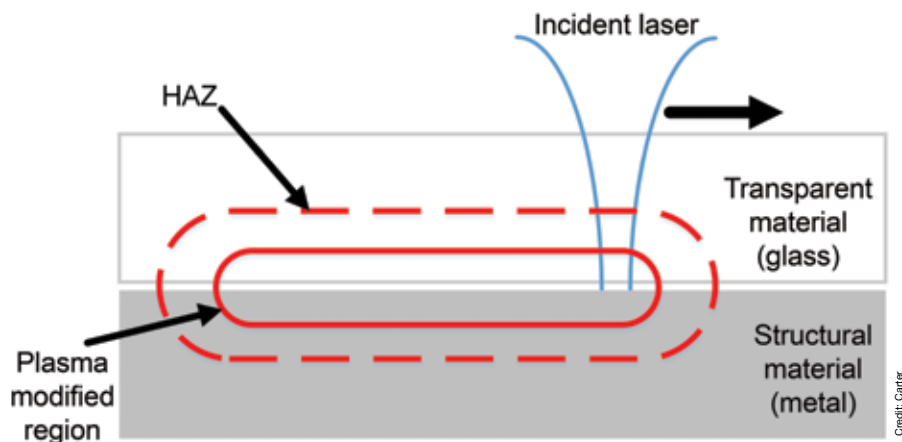
Over these extremely short timescales, the laser–material interactions take on some features that can be beneficially exploited, which can be further enhanced by focusing the laser into a very small area. First, the duration of any individual pulse is sufficiently short so there is no time for heat to dissipate through thermal conduction. Hence, the thermal energy will, for a short time, be just as concentrated as the laser energy. Second, while the pulses contain a low amount of energy (10–15  $\mu$ J is typical), the combination of temporal and spatial concentration—typically the pulses are focused into spots of 1–4  $\mu$ m diameter for this type of welding—results in truly enormous energy densities, typically in the order of MW of peak power and  $\text{GWcm}^{-2}$  in intensity. This amount of energy is more than enough to not only melt but also vaporize material.

The combined effect is that there is a highly concentrated thermal gradient. The area of the weld zone is molten—as is required for welding—but the bulk of the material experiences almost no temperature rise. Furthermore, the thermally affected zone, defined here as the zone which has seen sufficient temperature to melt, is strictly limited in size to only a few 100  $\mu$ m around the focus. It is this concentration of the temperature gradient that allows for welding of dissimilar materials without excess thermal stress because the total volume of heated material is strictly limited.

However, the extreme thermal gradient presents a new issue to the welding process—material at the focus of the laser beam will be not only melted but also vaporized (in fact, a plasma will be formed). This area essentially becomes a small pocket of high-pressure gas and,



# From concept to industry: Ultrafast laser welding



**Figure 1. Schematic illustration of ultrashort pulse laser welding. The incident laser is focused through the glass onto the metal surface. By translating the laser along the interface, a weld is formed from an inner plasma affected region and an outer heat-affected zone (HAZ).**

depending on the precise dynamics of the laser used, may be surrounded by a region of melted material. Thus, if a weld is attempted on the edge of the parts, this gas will escape as a jet, carrying off much of the melt volume with it and resulting in ablation rather than welding. This phenomenon limits the technique to a lap weld, where the laser is focused through one of the two materials (Figure 1). In this way, the gas is confined until it can cool and form a bond.

There clearly is a limit to the materials that can be welded with this technique as at least one must be transparent to the laser. Typically, these types of lasers operate at a wavelength of around 1,000 nm (in the near infrared part of the spectrum), although there are a range of other wavelengths commonly available. At this wavelength, most optical glasses and crystals will be transparent but all metals and most semiconductor materials will not. Ceramics pose an interesting issue as while several ceramics are nominally transparent at this wavelength, they often are highly scattering due to the fine structure and thus may not be suitable for focusing a laser beam through.

The final aspect of laser-material interactions using ultrashort pulses is the most intriguing: the ability to trigger what is referred to as nonlinear absorption. To understand this process requires some understanding of how light behaves and how it is absorbed by

atoms. Light is composed of individual photons, where each photon has a specific amount of energy that is related to the wavelength. At 1,000 nm, glass is transparent because the photons do not have the correct energy to interact with the atoms within the glass. However, if several photons arrive at a single atom at essentially the same time, their combined energy will be enough to trigger absorption.

Under normal circumstances, the probability of photon absorption happening is essentially zero, but a focused ultrashort laser pulse has such extreme photon density that the probability becomes not only nonzero but quite likely. The net result is that it is possible to trigger absorption in otherwise transparent materials but only at the focus of an ultrashort laser pulse—nowhere else is the photon density sufficient to permit this phenomenon. In other words, it is possible to trigger absorption inside the volume of a transparent material.

This ability is extremely powerful because it allows us to control the energy deposition within an otherwise transparent material in three dimensions by carefully positioning the focus using microstages or some form of optical scanner. This effect has been known for some time and has been exploited in a range of applications to alter the glass properties (e.g., to change its refractive index and make waveguides; or to change the reaction of the glass to etchant

chemicals, enabling selective etching). However, its potential to applications of laser welding was not appreciated until relatively recently.

## A brief history of ultrashort pulse laser welding

The story of ultrashort pulse laser welding starts in 2005 at Osaka University (Japan) with a paper demonstrating that it was possible to not only modify transparent material using an ultrashort laser pulse but also possible to weld them together.<sup>1</sup> In this paper, they focused a femtosecond laser on the interface region between two glass plates in intimate contact, fusing them into a weld. The importance of this result was clearly well appreciated by the team at Osaka because they were careful to obtain a patent within Japan before publishing.

Further publications from Osaka—including joint publications with RWTH-Aachen University (Germany) and IMRA American Inc. (a Michigan-based U.S. laser company)—expanded the understanding of the process and the range of glasses demonstrated, and also further reinforced the copywrite protection with additional patents in Germany, Japan, the U.S., and worldwide. However, the process they demonstrated relies on the formation of a fusion filament, a feature particular to femtosecond lasers, and it was only applied to transparent (i.e., broadly similar) materials.

This publication set off a bewildering array of research worldwide, with many publications demonstrating new material combinations as well as in-depth studies on the fundamental laser-material interactions, including development of sophisticated theoretical modeling systems. Readers of the *Bulletin* may be interested to note that this work has included welding of transparent ceramics,<sup>2</sup> but for the purpose of this article we will concentrate on developments toward dissimilar material welding.

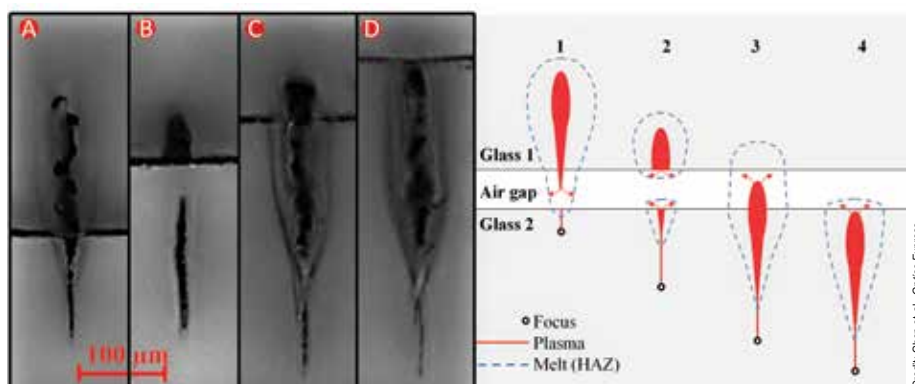
It would not be till 2007 that Osaka published the first paper in the area of dissimilar material welding, when they demonstrated glass-silicon welding.<sup>3</sup> This achievement was significant as

it showed that welding was possible between a transparent and opaque material. However, the chemical compatibility of silicon and glass (essentially silicon dioxide) still left open the question of whether the process could be applied more universally to dissimilar materials. Shortly thereafter, the University of Kassel (Germany) published their own demonstration of silicon–glass welding in early 2008,<sup>4</sup> indicating international interest in this development.

Osaka continued to lead in this area, providing the next milestone in 2008 with a publication demonstrating the welding of copper and glass using a femtosecond laser.<sup>5</sup> This study was a major breakthrough as copper, silicon, and oxygen have limited chemical interactivity and thus limited scope to form a purely chemical bond. Nevertheless, a bond was indeed demonstrated, and it could now be said that truly dissimilar material welding was possible.

The following years would see surprisingly few publications in this area, as the majority of research seemingly aimed at similar material welding. It was not until 2011 that the next novel material demonstration was made: The University Laval (Canada) with a paper exhibiting welding of fused silica to both copper and tungsten.<sup>6,7</sup>

It is worth noting that up to this point all dissimilar material welding was carried out using a femtosecond laser. Although pico- and femtosecond lasers are both referred to as ultrashort lasers, there are significant differences in the way the laser–material interactions occur and thus their capabilities. As previously mentioned, femtosecond lasers are able to generate high aspect ratio filaments of plasma due to a complex self-focusing effect in material, while almost entirely avoiding the generation of molten material when using a low repetition rate (typically 1–10 kHz). This situation results in an extremely high aspect ratio weld structure but one which is generally quite intolerant to any gap between the two materials because even a micrometer level gap will allow the gas to escape. As such, submicrometer contact (often referred to as optical contact) is required



**Figure 2. Illustration of the use of a correctly positioned focal point to create a melt volume to bridge a gap, or local roughness, between two materials. Taken from Chen et al.<sup>8</sup>**

between the two materials to prevent plasma from escaping—in effect holding the material together using van der Waal's force until it can be reinforced into a weld.

Higher repetition rate and picosecond laser systems, in comparison, produce a teardrop-shaped weld feature that typically exhibits a small but significant melt volume. This melt has been demonstrated to be useful in assisting confinement of the plasma (Figure 2) and thus in reducing the requirement for surface contact during the bonding process. However, it has the disadvantage that more care needs to be taken to ensure that the laser focus is accurately positioned and that increased gaps between materials will result in decreased weld strengths.<sup>8</sup>

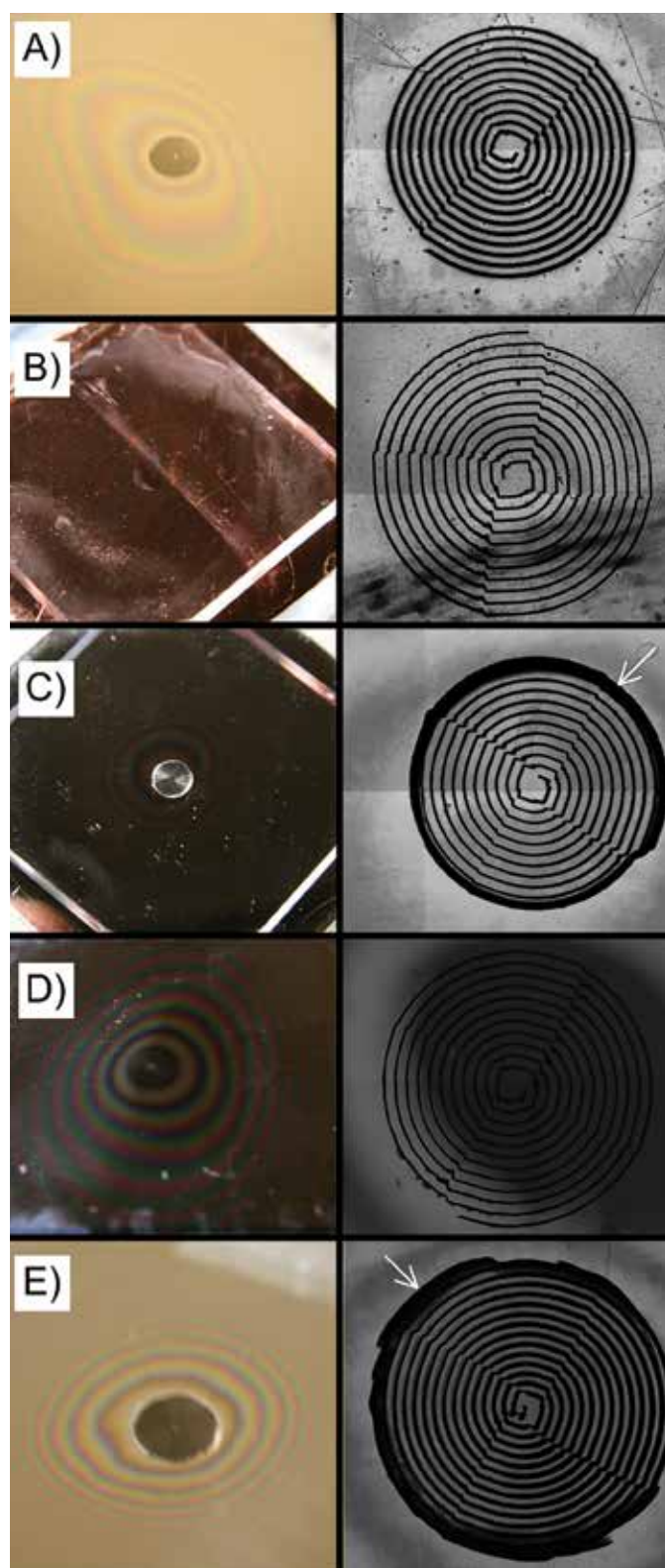
In my own research group at Heriot-Watt University (Edinburgh, Scotland), we demonstrated welding using a picosecond laser with a paper in 2014.<sup>9</sup> In this case, additional proof-of-principle demonstrations expanded the range of materials to include new metals and a crystal: aluminum, stainless steel, silicon, and copper welded to glass and sapphire (Figure 3). Since 2014, several other universities have published works demonstrating welding of various combinations of these materials, indicating a widespread interest in this novel capability, including the universities of Tampere (Finland),<sup>10</sup> Okayama (Japan),<sup>11</sup> Nara (Japan),<sup>12</sup> Kyiv (Ukraine),<sup>13</sup> Guangdong (China),<sup>14</sup> and the Key State Labs in Xian (China).<sup>15</sup> However, it is only very recently that there were published demonstrations of further material com-

binations. In 2020, NASA (U.S.) demonstrated the bonding of several glasses and crystals to metals, including invar, titanium, calcium fluoride, and Zerodur (a specialist low thermal expansion glass from Schott).<sup>16</sup> More recently, Jena (Germany) made a significant contribution by demonstrating silicon–copper welding, where the silicon is the transparent optical material.<sup>17</sup>

## Current capabilities and potential

The range of materials with published demonstrations include optical glasses, crystals, metals, ceramics, and semiconductors. Thus, the applicability of the technique is very broad. While some material combinations appear to be simpler to weld than others (usually demonstrated by a wider, more forgiving process parameter space), there is no clear link between material properties and the capability or ease of welding.

From my own experience, it is possible to weld almost any two materials provided they can be prepared properly. All that is required is time, effort, and sufficient samples to carry out a study of the welding parameter space. Indeed, the only material we have failed to weld entirely was not a single material but rather a mix of silicon carbide and aluminum with individual grains of each at multimicrometer sizes. Because these grains are larger than the laser spot size used for welding, the process is not really applied to a single material and hence unsurprising that no single set of welding parameters could be obtained for the combination.



**Figure 3.** Our first of proof-of-principle welds in (A) Al-SiO<sub>2</sub> (B) Cu-SiO<sub>2</sub> (C) Stainless steel-BK7 optical glass (D) Si-SiO<sub>2</sub> (E) Stainless steel-Sapphire. These 2.5-mm diameter spirals are typical of a “spot weld arrangement.” Note the backlash in our stage control—we have come quite a way since these samples! Taken from Carter et al.<sup>9</sup>

In terms of suitable optical sources, most work was carried out with lasers operating around 1,000 nm. However, there are examples in the literature from 515–1,558 nm, demonstrating that the precise wavelength (within reason) of the laser is not critical. Pulse durations also seem to widely vary, albeit always within the ultrashort pulse range, with demonstrations from 50–2,000 femtoseconds, which is likely more indicative of available lasers within disparate research labs than any fundamental requirement on pulse duration. Similarly, the repetition rates used vary from 1 kHz to several MHz, although here there seems to be a clear difference between femtosecond filament-based welding at low repetition rates and pico-/femto- processes at repetition rates exceeding 100 kHz, which rely on the formation of a melt volume and are more tolerant to surface roughness.

The overall picture, therefore, is that although the welding process requires optimization for each material combination and laser system, the required technical specifications for that laser are quite broad, allowing a wide range of laser manufacturers to be able to offer a suitable system provided they have sufficient knowhow of the focusing and material handling requirements.

The quality of bonds is more difficult to assess because the majority of these tests are in a proof-of-principle stage with limited statistical information regarding the strength and yield, among other properties. To the best of my knowledge, the only in-depth analysis looking to optimize a dissimilar welding process was carried out at Heriot-Watt,<sup>18</sup> although readers will readily appreciate that this kind of detailed research does not always make it into published papers—particularly where there is a potential for industrial application and new product development.

Where the quality of these welds was investigated, they tend to rely on some form of mechanical strength for quantitative evaluation. Typical arrangements involve a pull, shear, or some form of wedge/knife inserted in the gap between the components after welding, with the force required to break the bond being logged. It is prudent to be cautious in directly comparing the absolute values of weld strength between published techniques because the techniques usually are not directly comparable to one another and there is as yet no clear ISO standard for evaluating these types of bonds. Nevertheless, such results are suitable for identifying an optimum for a specific welding process and will, at the least, provide a reasonable estimate of expected strength, as can be seen in Figure 4.

One of the issues that becomes quickly apparent is in how the welds fail. This failure is almost always in the glass—often with a volume of glass left attached to the structural component after failure—because the welding process creates a defect in the glass. At the boundary of the glass melt zone, there is an abrupt change to the fictive temperature, which manifests as a visual change in refractive index as well as local stress. Studies indicate it is this line that most often fails (Figure 5).<sup>9</sup> The glass-metal weld, despite exhibiting complex chemistry, microcracks,<sup>19</sup> which in standard welding would all be causes for concern but is not normally the source of failure.

Thus, in almost every case it is the optical component that fails first. Because the optical component usually is a brittle material, the difficulty then is that the “weld” failure is brittle

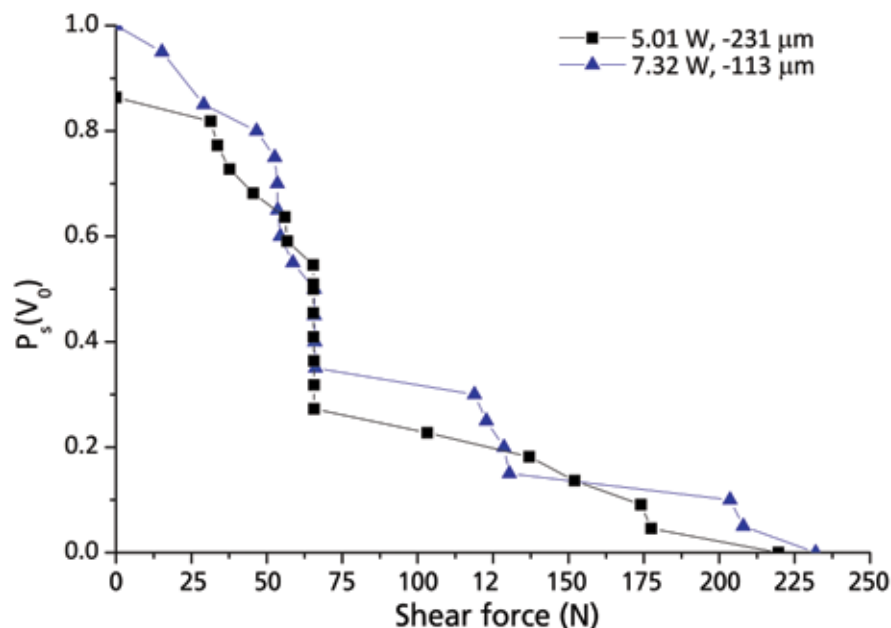
Credit: Carter et al., Applied Optics



as well—with all the accompanying statistical variation this failure implies. To obtain reliable data on the strength of the bonds, it is thus not only necessary to carefully design a measurement technique but also to measure a large number of components to build up a statistically meaningful set of data. Nevertheless, results from across the published record suggest bonds that are at least as strong as equivalent adhesives and, in some cases, more than ten times stronger—certainly sufficient for current applications.

It is crucial to note that these studies reveal that both the strength and the reliability (yield) is tightly intertwined with material preparation. As mentioned, to obtain a strong, reliable bond, the materials should ideally be placed in intimate, optical contact before welding. This placement is clearly more readily achieved with materials that are extremely flat, smooth, and clean. While easily achieved in optical components (as these are already prepared to high specifications with sub micrometer or even 10s of nanometer roughness and flatness), preparing structural materials to the same specification is a nontrivial problem that scales with the required contact area.

The common option at present is to polish the material to an essentially mirror finish via lapping, thus providing both the required roughness and flatness. This process is both slow and expensive, but with the right equipment and expertise, it is reliable for most materials (aluminum being a key exception). Unfortunately, lapping requires the target surface to be both flat and



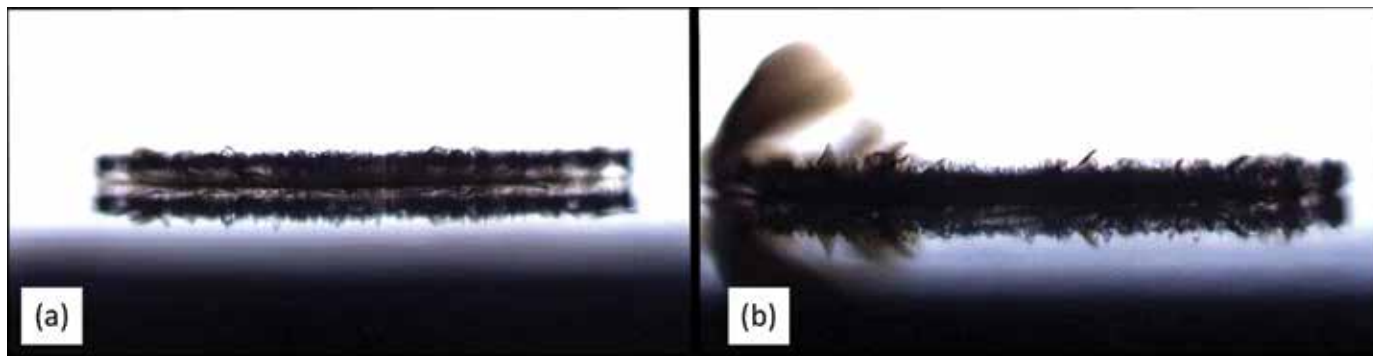
**Figure 4.** Example of weld strength statistics for Al-BK7 bonding. In this case, 20 samples for each of two processing parameters (average power and focal position) were tested. The bonds here are single 2.5-mm diameter spot welds and are required to sustain only 7 N of shear force for this application. Taken from Carter et al.<sup>9</sup>

exposed as curved or recessed surfaces (both often desired for optical mounts) are out of the question. While other polishing techniques may be applicable (e.g., chemical, laser) it has yet to be demonstrated that both the required level of flatness and roughness can be achieved in a reliable fashion.

The alternative technique, which Heriot-Watt has developed extensively, is to use the melt generated in a 100s kHz process to fill the gap between the two materials, thus relaxing the requirement to polish the metal surface. To date we have successfully welded surfaces with SA roughness in the order of approximately 1  $\mu\text{m}$ , which is consistent with a

high-quality machined or ground surface. However, care still is required so that the surface preparation method does not detrimentally affect the welding because yields varying between 80–97% have been obtained for different preparations methods of what is nominally the same surface finish.

Thus strengths, yields, and material combinations are appropriate for a wide range of applications where a mechanical bond is required. But what of applications where more than a purely mechanical attachment is required? Hermeticity has been demonstrated in glass–glass and glass–silicon welding, but it remains to be



**Figure 5.** Illustration of the weld failure mechanism for a glass–metal bond. Both images are taken side on with a reflection of the weld and the metal surface visible toward the bottom of the images. Image (a) is an untested example. Image (b) was taken part-way through the welding process, and a crack in the glass has developed. Adapted from Carter et al.<sup>9</sup>

## From concept to industry: Ultrafast laser welding



**Figure 6.** (a) Photograph of an Oxford Lasers C-Series laser micromachining station. Images of example laser welded parts: (b) One-inch, 4-mm thick quartz waveplate welded to stainless steel, (c) Fused silica plate welded to the end of a 40-mm diameter aluminum tube, (d) BK7 prism (18-mm height) welded to a mounting plate. Images courtesy of Oxford Lasers. [www.oxfordlasers.com](http://www.oxfordlasers.com).

demonstrated if this is the case more generally—although it would certainly be extremely surprising given the weld cross-sections studied thus far to find that it is not. However, a point to consider is that thermal expansion is still an issue for the assembled component. Using an ultrashort laser pulse allows the materials to be bonded but does not change the fundamental fact that two dissimilar materials will expand at differing rates. A purely mechanical bond can be achieved using a series

of small spot welds, thus limiting the issue, but for a hermetic seal a continuous perimeter weld will be required which, by necessity, will cover a longer line and larger area, thus giving larger expansion stresses.

### Industrial availability and applications

An early adopter of an industrial process was IMRA American Inc. (from at least 2006). Although they currently hold some of the key patents publicly

available, information suggests they offer only similar material (glass–glass) welding as a process. A second early entry came in the form of a Finnish company, Primoceler Oy. This company appears to have been a spin out from the collaborative work between Osaka and Tampere around 2013.<sup>10,11</sup> Initially aimed at glass–glass welding, the company also offered glass–silicon welding as part of an all-glass or silicon–glass packaging capability. In 2018, Primoceler was acquired by Schott AG (who also holds an Osaka patent), who now offers glass–glass and glass–silicon welding.

Trumpf (a German laser systems company) also is known to be active in this area, with joint publications with the Universities of Jena and Aachen.<sup>20,21</sup> For the most part, this research seems to be in the area of glass–glass welding (including as part of its own production process for optical fiber end caps), although in the last few years Trumpf showed glass–metal optical mounts with welds at exhibitions, which seems to be as far as the company has taken such applications.

The most recent entry into the field is Oxford Lasers (U.K.). They, in collaboration with Heriot-Watt University, developed a prototype laser welding system for glass–metal welding, including demonstrations of aluminum–BK7 and quartz–stainless steel. Importantly, this prototype is a fully integrated platform based on a standard turnkey laser micromachining platform (Figure 6), the general form of which will be familiar to industrial laser uses. Therefore, it includes the expected capacity to, among other things, generate user-defined laser beam toolpaths and correct for working distances and aberrations in optics, but also an adjustable laser amplifier allowing for both a pico- and femtosecond welding on demand.

The key advantages in ultrashort laser welding are the limited thermally affected zone, the ability to join a range of materials, and the ability to do so without introducing an interlayer. However, the cost of an ultrashort laser system is currently in the order of more than \$100k, suggesting applications either require economy of scale or high-value

products. From the current industrial players, this suggestion would seem to bear out because, although there are few public examples to draw on. Schott seems to have taken aim at semiconductor device packaging while Trumpf and Oxford Lasers appear to be well set up to cater to high-value optical, optomechanical, and aerospace device fabrication.

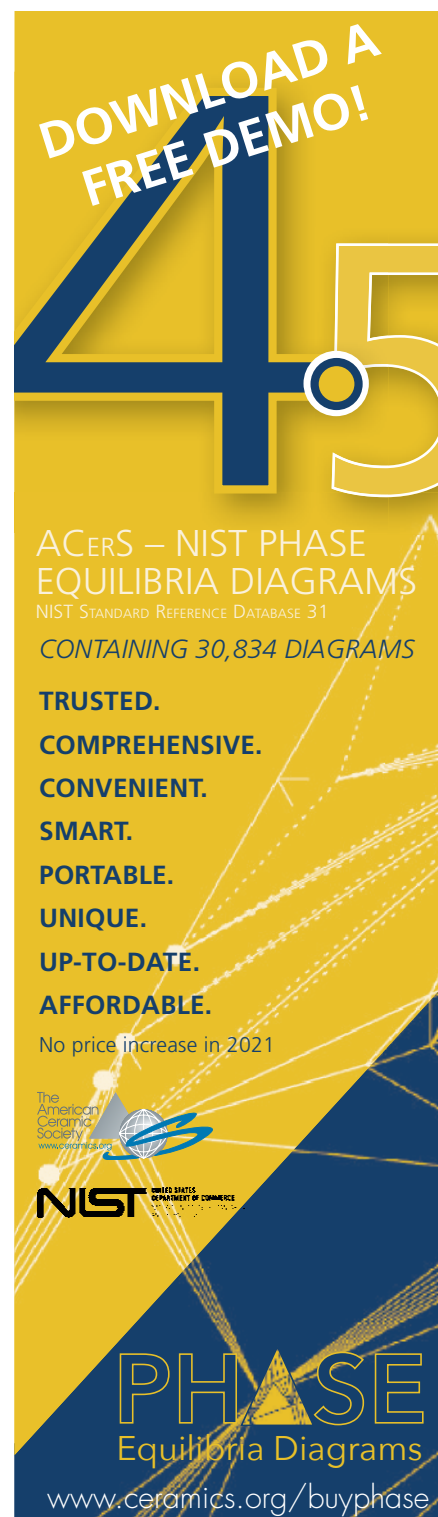
In truth, while high-value manufacturing has led the way, the range of potential applications is truly staggering because dissimilar material bonding is an issue in almost all areas of manufacturing, from microdevices up to building-scale applications. We have every expectation that this technique will rise to its potential and prove to be a significant new capability.

### About the author

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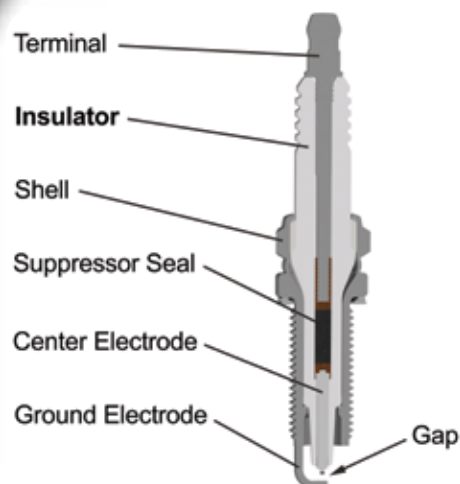
# Making sense of failure modes and effects analysis:

## Case study of a spark plug insulator

By William J. Walker, Jr.



Figure 1. Cross section of a spark plug showing the major components. Spark plug insulators are typically made from 95–96% alumina.



Credit: Tenneco

**F**ailure modes and effects analysis (FMEA) is a systematic approach to reduce technical risks of a product or process by identifying all potential failure modes and their causes. Effective FMEA can anticipate and prevent problems, reduce costs, shorten product development times, and result in safer products and processes with high reliability.

The right time to start a FMEA is during the concept phase of the product life cycle. It could be a new design, a new technology, or a new process, but it also could be a new application of an existing design or process. A FMEA is a living document. When changes are made to an existing design or process because of new technology, new requirements, or new knowledge about the product, including experience gained during the product life cycle, the FMEA needs to be updated.

The two most commonly used FMEAs are Design FMEAs and Process FMEAs. As the names suggest, Design FMEA applies to the design of a product, and Process FMEA identifies potential failure modes from manufacturing, assembly, and logistical processes to ensure that the final product meets the requirements of the design. Design FMEA and Process FMEA are closely linked, as the former provides the inputs for the latter.

The focus of this article is on Design FMEA for a ceramic material used to make spark plug insulators. A spark plug, shown in Figure 1, can be seen as an electrical feed-through that creates a high-energy spark to initiate combustion in an internal combustion engine. The ceramic insulator is generally cylindrical with a bore along the axis to support the components that transport electrical current from the ignition lead to the gap where the spark is created. The external surface is contoured to interlock with the shell that threads into the cylinder head of the engine. The shell is electrically grounded and is attached to a ground

electrode, which defines the sparking gap. The spark plug also has an internal resistor that suppresses electromagnetic interference.

A critical function of the insulator is to isolate the high voltage. When the dielectric breakdown strength of the material is exceeded, a pinhole or puncture through the ceramic is formed and that puncture becomes the preferred path for the electric current, which is to say that it sparks through the puncture instead of firing across the gap, and the engine misfires.

Today's engines require as much as 45 kV to create a spark, an increase from 36 kV not too many years ago. Dielectric breakdown strength of the ceramic is a critical property to withstand the high voltage without damage. Improvements of existing ceramic materials and development of new ceramic materials with higher dielectric breakdown strength are ongoing efforts.

The Design FMEA for the insulators will be considered on two different levels: the spark plug, and the ceramic material. The methodology for preparing a Design FMEA for a material is not documented in the standard FMEA handbooks and thus presents a challenge. This article describes the approach taken by Tenneco Inc.

## A seven-step approach

The new FMEA handbook defines a seven-step approach to FMEA (Table I).<sup>1</sup> Step 1 is *planning and preparation*, an important part of which is to define the scope of the FMEA—what it is that is being analyzed, and what is outside the scope of the investigation. The scope is limited to the area of design responsibility. Outside that limit, it is someone else's design responsibility and excluded from the FMEA. Another part of Step 1 is putting together the team, which needs to be multidisciplinary to get the perspectives of all stakeholders.

Step 2 is *structure analysis*, which identifies the different system elements that go into the product and how these components are interrelated (for example, the mating of parts and the interfaces where matter or energy is transferred between system elements). System elements include assemblies, subassemblies, components, and characteristics. By breaking the system up into separate system elements, each of the elements can be treated as individual focus elements that are analyzed in terms of each one's function and failure. One way to perform structure analysis is using a structure tree, which shows how the system elements go together to form the overall system.

Step 3 is *function analysis*, which assigns functions to the system elements and links the functions based on cause and effect. (Functions should be described in terms of what the system element does in relation to the next higher level on the structure analysis.) With a good function analysis, Step 4, *failure analysis*, can be done in a very systematic manner, which helps to ensure thoroughness of the process.

**Table I. Seven-step process for conducting failure modes and effects analysis.**

|                                      |   |
|--------------------------------------|---|
| System analysis                      | Step 1. Planning and preparation<br>- Product identification and boundaries<br>- Assemble a cross-functional team |
|                                      | Step 2. Structure analysis  |
|                                      | Step 3. Function analysis   |
| Failure analysis and risk mitigation | Step 4. Failure analysis<br>Step 5. Risk analysis   |
| Risk communication                   | Step 6. Optimization<br>Step 7. Results documentation   |

## History of FMEA in the automotive industry

The FMEA methodology was first developed in the 1940s by the United States military as "Failure Mode, Effect and Criticality Analysis."<sup>a</sup> NASA adopted it in the early 1960s for the Apollo program. In the late 1970s, the automotive industry began to use FMEA following the highly publicized safety issues with the Ford Pinto.

Following the release of the ISO 9000 international quality management standards in 1988, the U.S. automotive industry developed QS 9000, which included Design and Process FMEAs as part of advanced product quality planning. FMEA is now used by a wide range of industries, including software, semiconductors, food service, and healthcare.

The automotive industry follows FMEA standards developed by the Automotive Industry Action Group (a U.S. industry group) and Verband der Automobilindustrie (a European industry group). The two organizations recently released a joint handbook that reconciles the differences that previously existed in order to have a common standard for the global automotive industry.<sup>b</sup> This project was completed before the new handbook was released and relies heavily on the earlier Verband der Automobilindustrie guidelines<sup>c</sup> to meet the requirements of a customer in Europe. <sup>100</sup>

a. United States Military Procedure MIL-P-1629, *Procedure for performing a failure mode, effects and criticality analysis*, Nov. 9, 1949, since updated to MIL-STD-1629.

b. *Failure modes and effects analysis—FMEA Handbook*, Automotive Industry Action Group and Verband der Automobilindustrie, First Edition, 2019.

c. Verband der Automobilindustrie, "Product and Process FMEA," *Quality Management in the Automotive Industry* Volume 4, Second Edition, 2012.

In Step 4, *failure analysis*, all possible failure modes are listed for each system element. The failure modes may be the loss of function, which can occur suddenly, intermittently, or as a degradation over time. A failure mode also may be performance outside of the specified range. Once failures are identified, they are linked based on cause and effect. The failure effect is the consequence of the failure mode and occurs at the next level up. The failure cause is the reason (in the next level down) why the failure mode occurred. The way the failures are linked usually follows the function tree.

Step 5, *risk analysis*, assigns risk to each identified failure mode based on severity, occurrence, and detection. The FMEA handbook has established guidelines for rating these risks (Table II),<sup>1</sup> which run on a scale of 1 to 10.

Step 6, *optimization*, addresses the areas of highest risk by identifying actions necessary to reduce the risk and evaluating the effectiveness of those actions.

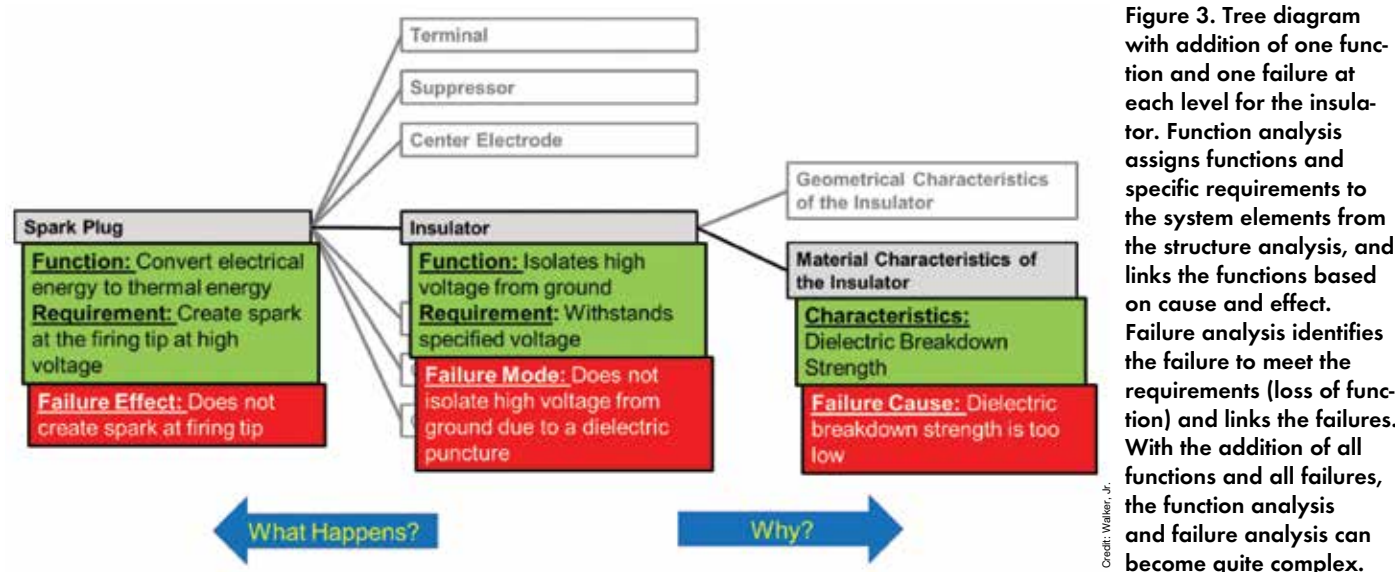
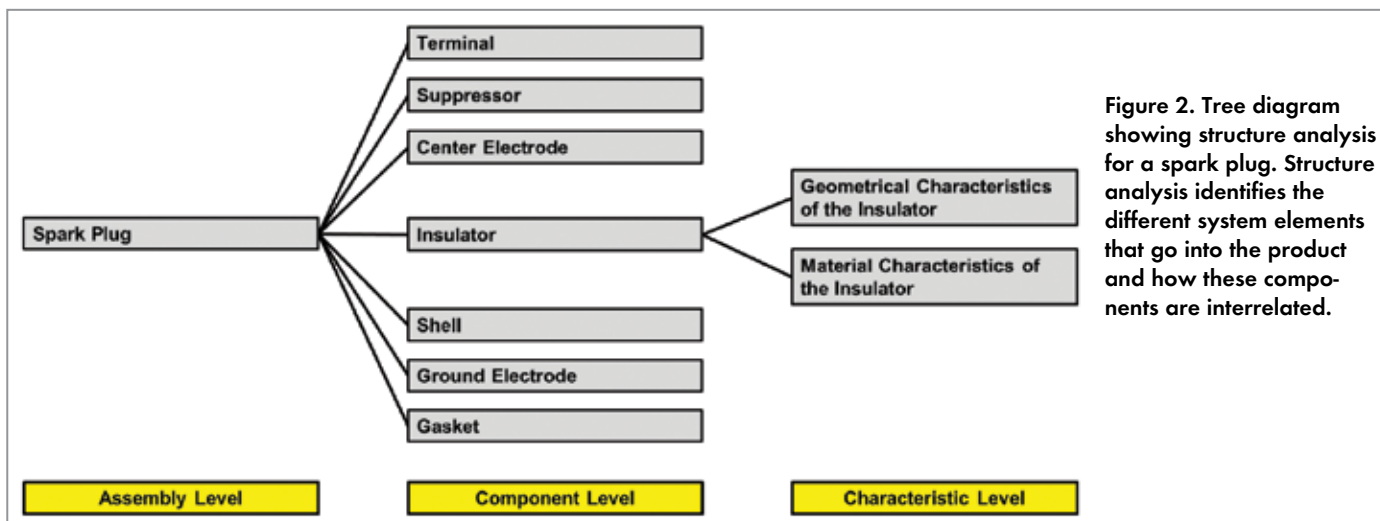
Once these steps are all completed, Step 7 is *results documentation*, which covers documenting and communicating the results. The complete FMEA form can be quite extensive, and includes the function analysis, failure analysis, risk analysis, and optimization in a single document that is organized according to the structure analysis. Historically, the completed FMEA was a paper document in the form of an FMEA table. However, it has become common for the FMEA to be documented in electronic format using FMEA software, which allows the data to be presented in multiple formats, including the FMEA form, the structure tree, and the function and failure nets. The software also simpli-

**Table II. Criteria for severity, occurrence, and detection ratings.\***

| Rating | Effect    | Severity                                  | Occurrence                             | Detection                   |
|--------|-----------|---|--|-----------------------------|
| 10     | Very High | Safety, regulatory                        | New technology                         | Test method to be developed |
| 9      |           |   |  |                             |
| 8      | High      | Loss or degradation of primary function   | New design based on similar technology | Unproven test method        |
| 7      |           |   |  |                             |
| 6      |           |   |  |                             |
| 5      | Moderate  | Loss or degradation of secondary function | Detail changes to previous design      |                             |
| 4      |           |   |  |                             |
| 3      | Low       | Objectionable effect on function          | Mature technology                      | Proven test method          |
| 2      |           |   |  |                             |
| 1      | Very Low  | No discernable effect                     | Failure not possible                   | Failure always detected     |

\*Adapted from the AIAG & VDA FMEA Handbook.<sup>1</sup>

# Making sense of failure modes and effects analysis: Case study of a spark plug



fies integration of multiple FMEAs to describe an entire product or system.

The results of the Design FMEA are the basis for product drawings and design specifications and are used as the inputs to the Process FMEA. The Design FMEA also will identify certain special characteristics, which can be directly responsible for the failure of a product and require special attention for process control.

For a complex system such as an automobile, FMEAs will be developed on several different levels; together, they provide a complete description of the system. Within a single organization, multiple FMEAs may be integrated into a larger FMEA, but between companies they typically remain separate. In either case, the outputs of an (internal or external) customer's FMEA become the

inputs to an (internal or external) supplier's FMEA in the form of specifications and drawings.

## Applying FMEA to a spark plug

The structure analysis for a spark plug (Figure 2) shows that the spark plug is made up of a number of different components. Each of these components is made of some material and has some particular geometry, so at the lower level, there are geometrical characteristics of the component and the material characteristics of the component. The scope of the FMEA is limited to the area of design responsibility. The spark plug designer specifies a geometry and a material for each of the components. The details of why the material has the properties that it does are not necessary.

The next step is function analysis. In the simplified example shown in Figure 3, only one function for the spark plug and one function for the insulator are considered, but in fact most system elements will have a number of functions.

In addition to the functions, there are specific requirements, such as to form a spark at the firing tip. This requirement comes from the design of the engine. It is outside of the design responsibility of the spark plug designer; it is specified by the customer. It is one of the inputs into the Design FMEA. Looking at the insulator, its function is to isolate the high voltage so the current can be delivered to the firing tip with a requirement to withstand the voltage needed to form the spark.

The next level down from the insulator is the system element "Material



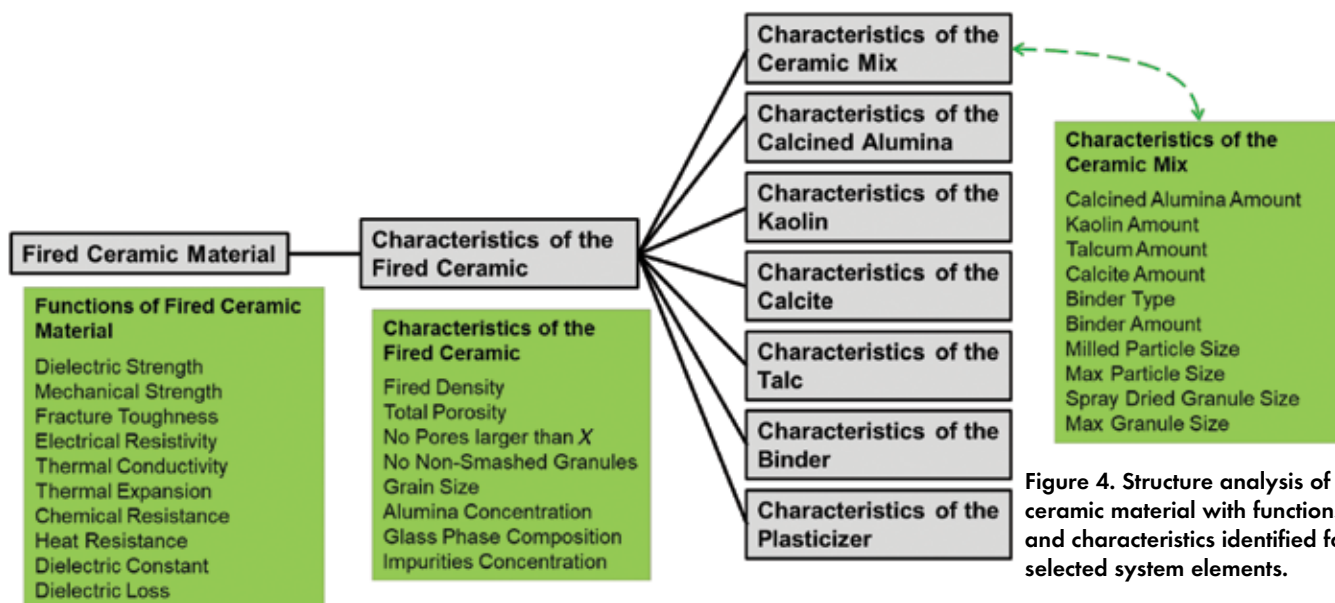


Figure 4. Structure analysis of the ceramic material with functions and characteristics identified for selected system elements.

characteristics of the insulator,” which is the set of properties that are needed to meet the performance requirements of the insulator. To withstand the voltage requirement, the key property is dielectric breakdown strength.

In Step 4, failure analysis, all possible failures are listed for each system element. In the Figure 3 example, the failure mode is that the insulator does not isolate the high voltage because of a dielectric failure. The failure effect, on the spark plug level, is a spark is not created at the firing tip. The failure cause is the dielectric breakdown strength of the ceramic is too low. Because this is a Design FMEA, it might be better to say the ceramic material was incorrectly specified with insufficient dielectric breakdown strength.

### Applying FMEA to a ceramic material

As we develop a Design FMEA for the ceramic material—which on the spark plug FMEA is the box marked as “Material characteristics of the insulator”—we need to think about what the system elements are. In other words, we need to consider the components of the material that give us the set of properties that are handed to us in the form of a materials specification from the spark plug designer.

The well-known relationships between performance, properties, characteristics (or structure), and processing that are the foundation of materials science were the

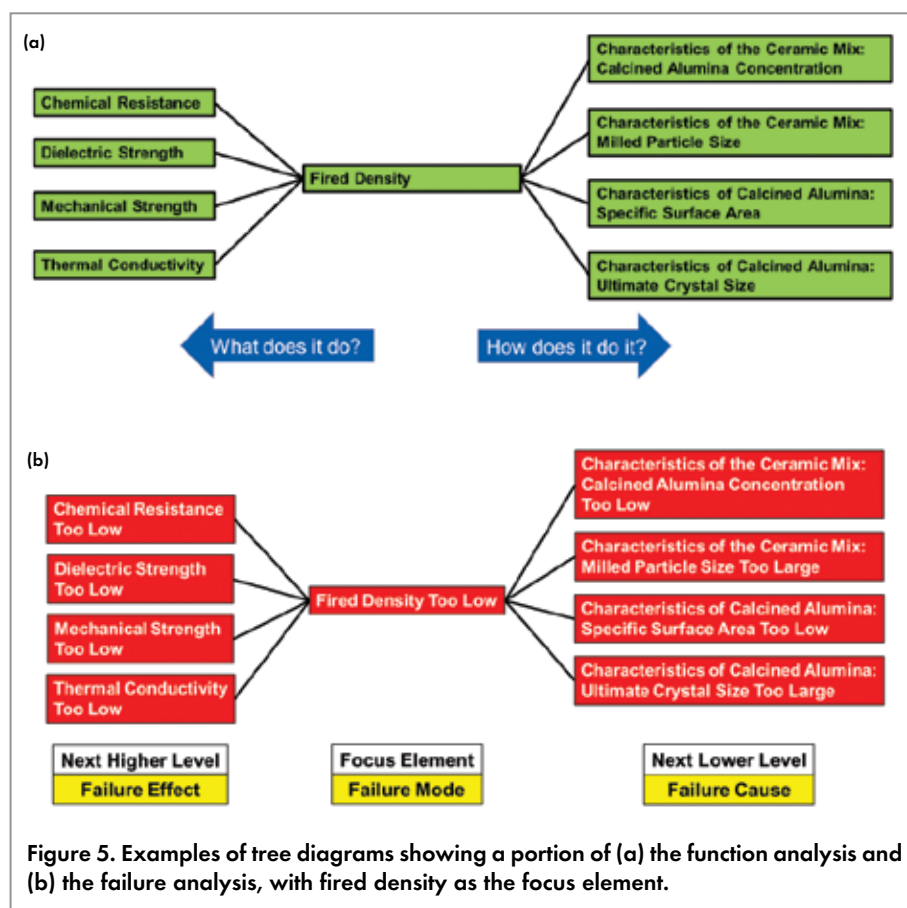


Figure 5. Examples of tree diagrams showing a portion of (a) the function analysis and (b) the failure analysis, with fired density as the focus element.

basis for developing the Design FMEA for the ceramic material.

Performance of a material is its response to a complex set of conditions. A material property is the response to a single well-defined energy field. Examples include mechanical strength, dielectric breakdown strength, and thermal conductivity.

Material characteristics are physical and chemical aspects that fully describe the material—the structure and the composition. The National Academy of Sciences Materials Advisory Board defined characteristics as “those features of the composition and structure (including defects) of a material that

# Making sense of failure modes and effects analysis: Case study of a spark plug

are significant for a particular preparation, study of properties, or use, and suffice for reproduction of the material.<sup>2,3</sup> Characteristics can be considered as intrinsic properties—they are not the response to an external stimulus. Examples include density, grain size, pores size, and defect structure.

Processing is the path taken to obtain the desired characteristics. From an engineering perspective, the performance of the material depends on its properties, the properties depend on the characteristics, and the characteristics depend on the processing. A scientific understanding of cause and effect goes in the other direction. The processing defines the characteristics, the characteristics control the properties, and the properties work together to determine the performance.

In FMEA language, the term characteristics is used to broadly describe the requirements of the material. In materials language, these requirements are better described as properties. For the ceramic material FMEA, properties and characteristics were separated into different system elements on different levels.

In the structure analysis for the ceramic material (Figure 4), the highest level is the fired ceramic. The ceramic is the product that is being designed. The requirements of the fired ceramic material are the properties from the spark plug Design FMEA. The next level is the characteristics of the fired ceramic, and one more level down includes the characteristics of the raw materials that go into the ceramic and also the characteristics of the ceramic mix, which is the characteristics of the feed-stock used to make the ceramic.

Functions of selected system elements are included in Figure 4. The functions for the fired ceramic are the properties from the spark plug Design FMEA, such as dielectric breakdown strength and mechanical strength. The characteristics of the fired ceramic are those features of the composition and structure (including defects) that fully describe this material: the fired density, the amount of porosity, the size of the pores, the size of the grains, the size of any defects, and the chemical and phase compositions. The characteristics of the ceramic mix are the amounts of each

raw material that go into the formulation, the particle size after milling, the spray dried granule size, and so on. These characteristics are inputs into the Process FMEA. The characteristics of the raw materials are the specifications that are given to the suppliers. For calcined alumina, the specifications are the ultimate crystal size, the specific surface area, and chemical composition.

Once all of the functions and characteristics are assigned, the next step is to link functions based on cause and effect. The function net for fired density is shown in Figure 5a. Dielectric strength is dependent on fired density, and fired density is dependent of several factors at the characteristic level. But dielectric breakdown strength is not the only property that is influenced by fired density. Others include mechanical strength, chemical resistance, and thermal conductivity. Because each characteristic of the fired ceramic can influence several properties, and because each characteristic of the fired ceramic can depend on several characteristics of the raw materials and of the ceramic mix, the function

**Table III. Completed FMEA**

## System Element: Characteristics of the fired insulator

### Function: Fired density

#### Requirement: Density specification

| Potential Failure Effect       | Severity | Potential Failure Mode   | Potential Failure Cause                             | Current Preventive Control | Occurrence | Current Detection Controls                    | Detection | Risk Priority Number | Action Priority | Recommend- ed Actions                            | Responsibility and Target Completion Date | Action Results |            |           |                      |                 |
|--------------------------------|----------|--------------------------|---|----------------------------|------------|---|-----------|----------------------|-----------------|--|---|----------------|------------|-----------|----------------------|-----------------|
|                                |          |                          |   |                            |            |   |           |                      |                 |  |   | Severity       | Occurrence | Detection | Risk Priority Number | Action Priority |
| Dielectric strength is too low | 8        | Fired Density is too low | Milled Particle Size is too large                   | DOE on particle size       | 2          | Laser Scattering Method<br>Prototype Testing  | 2         | 32                   | Low             | None – all current design controls are adequate. |   |                |            |           |                      |                 |
|                                |          |                          | Calcined alumina concentration is too low           | DOE on ceramic composition | 2          | Chemical Analysis by XRF<br>Prototype Testing | 2         | 32                   | Low             | None – all current design controls are adequate. |   |                |            |           |                      |                 |
|                                |          |                          | Calcined alumina specific surface area is too low   | DOE on raw materials       | 2          | BET method<br>Prototype Testing               | 2         | 32                   | Low             | None – all current design controls are adequate. |   |                |            |           |                      |                 |
|                                |          |                          | Calcined alumina ultimate crystal size is too large | DOE on raw materials       | 2          | Laser Scattering Method<br>Prototype Testing  | 2         | 32                   | Low             | None – all current design controls are adequate. |   |                |            |           |                      |                 |

analysis becomes a complex network of causes and effects. FMEA software, such as APIS<sup>4</sup> or Q-SYS,<sup>5</sup> provide a tool for navigating these complex networks of interactions. Once the function analysis is completed, the FMEA software will show any system element and all other system elements that are linked to it.

The next step is failure analysis. Figure 5b shows the failure net corresponding to Figure 5a. One failure mode is that the fired density is too low, or more specifically, the specification for fired density is too low. One effect of this failure mode is that the dielectric strength is too low, which would lead to an increased likelihood of dielectric failure. Low fired density also results in degradation of other properties: mechanical strength, chemical resistance, and thermal conductivity. The failure causes for low density are not enough calcined alumina in the mix, milled particle size too large, and the raw material being improperly specified.

The tree structure is valuable for visualizing all of the relationships between the different elements. However, for risk analysis, optimization, and results documentation, visualization is easier with a table. The table is considered to be the standard format for documenting the completed FMEA, with groupings of columns containing the structure analysis, function analysis, failure analysis, risk analysis, and optimization for each focus element. With FMEA software, it is easy to shift between the tree structure and the tabular format. In fact, the software is necessary to develop the complete FMEA and to integrate it with the FMEAs for all of the other systems and subsystems that make up a complex product, such as an automobile.

In Step 5, risk analysis, dielectric failure in a spark plug is very severe, so it has a rating of 8, corresponding to loss of primary function. For prevention of this failure mode (specifying the density too low), there is a long history of working with similar materials and experimental work conducted as a “design of experiments” in the lab, which sets forth confidence in the specification. Therefore, occurrence will be low, with a rating of 2. A lot of

prototype testing has been conducted, including building spark plugs and putting them in engines. Therefore, a problem with the design would be detected, and detection gets a rating of 2.

The severity, occurrence, and detection ratings are combined to determine the areas of highest risk—the weaknesses of the design. Earlier FMEA methodology by the Automotive Industry Action Group, a U.S. industry group, used a risk priority number (RPN), which was calculated by multiplying the three ratings, and the areas with highest RPN were identified as needing improvement. Some companies would set a threshold value for RPN (such as >100 or >150) where improvements would be required. For this example, the  $RPN = 8 \times 2 \times 2 = 32$ , which is well below the more stringent threshold of 100, hence no further action is required. A weakness with the RPN is that it assumes equal weighting of severity, occurrence, and detection.

The current joint methodology by the Automotive Industry Action Group and Verband der Automobilindustrie (see sidebar: “History of FMEA...”) uses an action priority, which is a more complex function of severity, occurrence, and detection, that places the highest weight on severity and the least weight on detection. In the example in Table III, the action priority (determined from the FMEA Handbook) is low, so no further action is required. If, however, the action priority was high, countermeasures would be needed to lower the action priority, which is Step 6, optimization. In the automotive industry, the severity is inherited from the customer requirements, and therefore cannot be changed. The emphasis for optimization should first be on reducing the occurrence of the failure, and second on improving the detection. Reducing the occurrence is preferred because it involves the process of engineering the problem out of the product, while detection merely contains the problem.

## Summary

FMEA is a systematic method to identify weaknesses with a design or a process. When done properly, an FMEA

can anticipate and prevent problems, save money, and provide a guide to a safer, more reliable product or process.


The steps to performing Design FMEA are first to define the boundaries of the system and then to develop the structure analysis, which shows how the different components of the system interact. Next, functions are assigned to each element of the system and the functions are linked based on cause and effect. Failures are then assigned to each function and the failures are linked, again based on cause and effect. Risk is assigned to each failure mode and the areas of highest risk are identified in order to optimize the product and reduce the overall risk. Finally, the results are documented and communicated.

The FMEA is a living document and needs to be updated over the life cycle of the product as more information becomes available or as changes are implemented. For a ceramic material, the well-known relationships between performance, properties, characteristics (structure and chemistry), and processing of materials were used to establish the cause and effects relationships of the Design FMEA.

## About the authors

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- <sup>2</sup>National Academy of Sciences Materials Advisory Board MAB-229-M, March 1967.
- <sup>3</sup>J.W. McCauley, “Materials characteristics: Definition, philosophy and overview of conference,” pp. 1–11 in *Materials Characterization for Systems Performance and Reliability*, Edited by J.W. McCauley and V. Weiss, Plenum Press, New York (1986).
- <sup>4</sup>APIS IQ-Software, APIS Informationstechnologies GmbH.
- <sup>5</sup>QSys FMEA, ib seteq GmbH. 



# Hybrid composites made from metal-organic framework and inorganic glasses

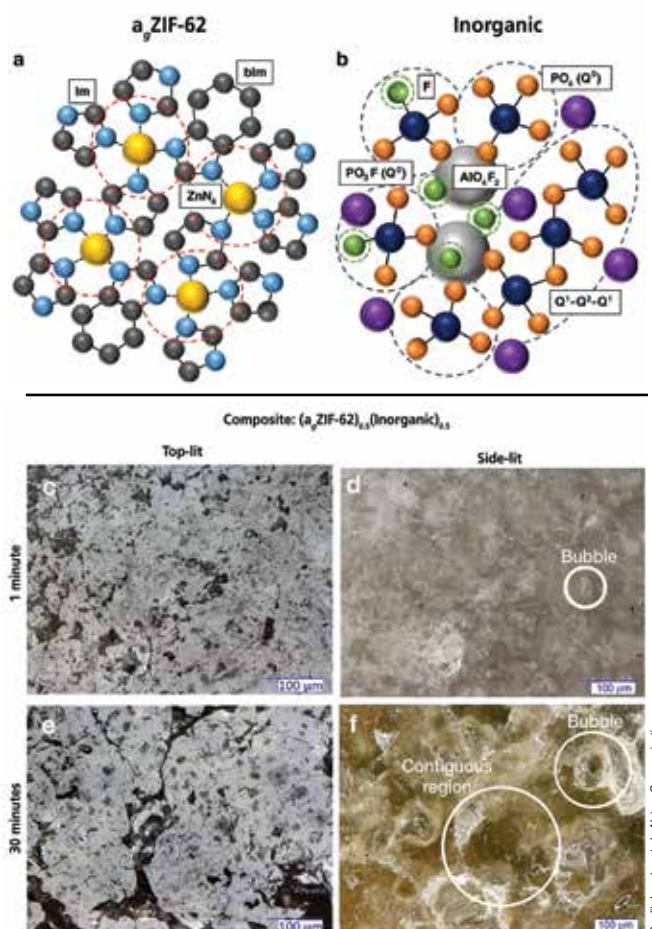
By Courtney Calahoo, Louis Longley, Thomas D. Bennett, and Lothar Wondraczek

Inorganic glasses are a long-studied class of materials, usually obtained through quenching from a liquid phase at sufficient speeds to avoid recrystallisation. The variety of glasses is vast; it comprises many subfamilies with very distinct optical, electronic, and mechanical properties.

In contrast, metal-organic frameworks (MOFs) are a relatively new class of materials. They are comprised of inorganic nodes joined by organic linkers, and in their crystalline state, these materials typically display high porosity. Despite their comparative novelty, over 80,000 distinct crystal structures are recorded in the literature. A handful of these MOFs are shown to melt when heated in inert atmospheres, and they form glasses upon cooling.<sup>1</sup> Albeit still small in variety, researchers have demonstrated already that this new class of MOF-derived glasses exhibit unique properties, such as the persistence of porosity in the liquid and quenched states.<sup>2</sup>

Recent work shows that similar hybrid glasses also can be formed through a combination of phosphate chains and imidazolate linkers.<sup>3</sup> Elaborating on this discovery of glass formation at the phosphate-imidazolate join, we fabricated amorphous organic-inorganic composites.<sup>4</sup> These composites were prepared by combining crystalline ZIF-62 ( $\text{Zn}(\text{Im})_{1.75}(\text{bIm})_{0.25}$ ) (Figure 1a), which is a zinc imidazolate framework, with a variety of sodium fluoroaluminophosphate glass powders,  $(1-x)[\text{Na}_2\text{O}]_x[\text{P}_2\text{O}_5]_{1-x}[\text{AlO}_3]_y[\text{AlF}_3]_{1-y}$  (Figure 1b), in equal mass amounts and then heating both components into the liquid state. Isothermal treatment was conducted to investigate the impact of liquid phase mixing on composite properties.

Thermogravimetric analysis,  $^1\text{H}$  nuclear magnetic resonance (NMR) spectroscopy, and Fourier transform infrared spectroscopy confirmed that the integrity of the organic linkers in the MOF remains unchanged on composite formation; this finding is in line with results observed on single phase MOF glasses.<sup>1</sup> PXRD results also confirmed that the composites are mainly amorphous, though a small degree of recrystallization of a dense ZIF-zn ( $\text{Zn}(\text{Im})_2$ ) phase was observed, the extent of which depended both on treatment time and inorganic glass composition. Differential scanning calorimetry revealed two separate



**Figure 1. Structural chemistries.** (a) Representative atomistic structure of ZIF-62, showing  $\text{ZnN}_4$  tetrahedra connected by imidazolate (Im) and benzimidazolate (bIm) organic ligands. (b) Local structure of  $(1-x)[(\text{Na}_2\text{O})_{1.6}(\text{P}_2\text{O}_5)]-x[(\text{Al}_2\text{O}_3)[\text{AlF}_3]]$  glass series. Key: N – light blue, Zn – purple, C – dark grey, P – blue, O – red, Al – light grey, F – orange, Na – green, H – omitted for clarity. **Glass flow.** Top-lit and side-lit z-scan digital microscopy images of the  $(a_9\text{ZIF-62})_{0.5}(\text{inorganic glass})_{0.5}$  composites heat treated for 1 minute (c–d) and 30 minutes (e–f). Reproduced with adaptations from Ref. 4 under CC-BY license.

glass transitions, identified as due to the inorganic and ZIF glass components, in each of the composite samples.

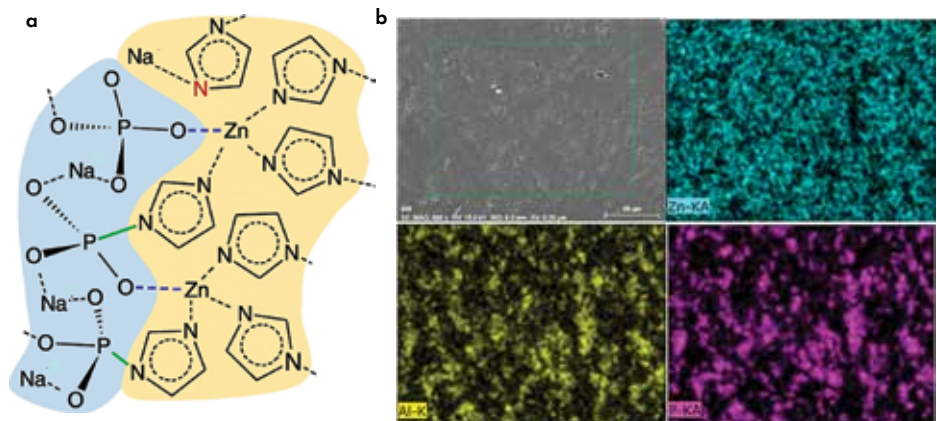
Electron, optical, and laser scanning microscopy revealed that longer isothermal heat treatments and lower inorganic glass transition temperatures produced more homogenous consolidated microstructures with fewer remnant particles. This finding is attributed to the increased degree of liquid flow allowed by less viscous (lower glass transition temperature) melts and longer heat treatment times. In line with this observation, the densities of the composites fell between the values of the two end-members and longer heat times results in higher densities. Clear evidence of flow can be observed

in the side-lit images (Figure 1c–f), with bubbles, islands, and surface droplets being found in the composites.

Structural studies via Raman spectroscopy and solid state  $^{31}\text{P}$  NMR shed light on generation of interfacial bonds between the two phases (Figure 2a). Specifically, Raman spectroscopy revealed new Na–N bonding, while solid-state nuclear magnetic resonance found evidence of organic protons in close proximity to phosphorus atoms, likely due to P–N bonding. Pair distribution function studies confirmed that the short-range order of both glassy phases remains intact on composite formation. A differential pair distribution function treatment also was used to investigate the atomic scale structure of the composite samples;<sup>5</sup> however, this investigation did not reveal any unambiguous evidence of new bonding in the bulk of the composites. Energy-dispersive X-ray spectroscopy shows clear domains originating from the inorganic glass (phosphorus, aluminum) and those originating from the MOF glass (zinc) (Figure 2b).

Nanoindentation mapping revealed clear separate regions of low hardness and stiffness (a ZIF-62) and high hardness and stiffness (inorganic), while much of the composite material had mechanical properties between the endpoint materials. Longer heat treatment of 30 minutes resulted in smaller inorganic regions, perhaps indicating a finer grain composite and larger interfacial volume. Scratch testing also confirmed the fabrication of a true composite material with chemically bonded components, with the scratch resistance, i.e., indenter displacement and work of deformation, of the composite material being between the organic and inorganic component. The variable temperature  $\text{Na}^+$  ionic conductivity of some of the composite samples was investigated as well. The ionic conductivity and activation energy of the composites were found between the values measured for inorganic glass and values measured for other similar MOFs.<sup>6</sup>

Taken together, these results indicate the formation of a class of composite materials that are formed of distinct glass domains originating from the inorganic and ZIF glasses, which are bonded at the interface. The nanoindentation and conductivity measurements indicate that the functional properties of this class of



**Figure 2. Interface and microstructure. (a) Representation of the MOF glass–inorganic glass interface derived from spectroscopic investigations. (b) Domain structure of the composite as measured by energy-dispersive X-ray spectroscopy. Reproduced with adaptations from Ref. 4 under CC-BY license.**

materials depend on the degree of mixing in the liquid phase and therefore the degree of consolidation observed in the material formed on cooling. Additionally, the observation of a small degree of ZIF-zni recrystallisation, an effect not previously observed in ZIF-62 liquids,<sup>1</sup> indicates broader interactivity of the MOF and inorganic glass components at elevated temperatures.

As previously stated, composite formation has the ability to produce new classes of materials with advantageous properties above and beyond those of the end-members. However, creating a strong bond between materials with substantially different chemical, thermal, and mechanical properties presents a challenge. Nature in her inventive parsimony often extracts the benefits of composite formation in spite of these issues, through combining materials with very different properties, i.e., small inorganic crystallites in an organic polymeric protein matrix in constructions, such as nacre, eggshell, or bones. Beyond the benefits of a soft matrix, proteins often provide necessary scaffolding to promote the ideal crystal growth and habit. Clearly, emulation of this practice in man-made materials such as the composites produced in this work can lead to the discovery of useful materials.


Much work is yet to be done in overcoming the issues with composite formation in man-made materials with drastically different end-members. As indicated by Umeyama et al.,<sup>3</sup> more systematic exploration of the design space, e.g., through creation of phase diagrams, are needed for inorganic–organic hybrid materials in order to optimize process-

ability and formability of functional materials. However, the results presented in this work indicate there also is great potential for new materials discovery.

## About the authors

Courtney Calahoo and Lothar Wondraczek are post-doctoral fellow and professor, respectively, at the Otto Schott Institute of Materials Research at the University of Jena, Germany. Louis Longley and Thomas D. Bennett are Ph.D. student and Royal Society University Research Fellow, respectively, in the Department of Materials Science and Metallurgy at the University of Cambridge, U.K. Contact Wondraczek at lothar.wondraczek@uni-jena.de.

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**Figure 1. Participants in the Virtual Student Exchange Program. Top from left to right:** Iva Milisavljevic (Alfred Univ., USA); Jenna Metera (UC San Diego, USA); Kana Tomita (Tokyo Inst of Tech, Japan); Amanda Bellafatto (Colorado School of Mines, USA)

**Middle from left to right:** Rohit Malik (Univ of Seoul, Korea); Haoming Luo (Shanghai Inst Ceramics, China); Gillian Micale (MIT, USA); Yuuki Kitagawa (Kyoto Univ, Japan)

**Bottom from left to right:** Tun Wang (Shanghai Univ, China); Tea-Hyung Kim (Seoul National Univ, Korea); Meir Sharchar (UC San Diego, USA); Chao Wang (Tsinghua Univ, China)

# Building bridges in the time of Corona: Virtual Student Exchange Program

By Iva Milisavljevic, Jenna Metera, Kana Tomita, and Yiquan Wu

Student exchange programs represent a unique opportunity for students to gain valuable research and cultural experiences, knowledge, and connections within the scientific community. Overall, these programs make a positive impact on the professional and personal development of every young researcher.

Unfortunately, most of the funded projects and limited school funding that support graduate studies do not include or are insufficient to provide opportunities for student exchange. Moreover, amid COVID-19 and all the restrictions and travel bans that exist, engaging in exchange programs has become even more challenging. Fortunately, virtual platforms offer a way to replicate some of the in-person experiences of meetings that students currently lack.

In August 2020, a group of Ph.D. students participated in a two-day virtual event called the Virtual Student Exchange Program. Alfred University professor Yiquan Wu organized this student networking event as a part of his National Science Foundation project on Collaborative Exchange Research and Materials In Ceramic Sciences (CERAMICS) program. The CERAMICS program was established by Wu with his NSF CAREER award, which is funded by the NSF Division of Materials Research Ceramics Program. Goals of the CERAMICS program include drawing more students to study ceramic



materials in the pursuit of advanced degrees, better preparing them for their future careers, and training them to be capable of performing in an international research environment at the forefront of science and engineering.

During the Virtual Student Exchange Program, 12 Ph.D. students from four countries gave talks (Figure 1). These student speakers came from the United States (5), China (3), Japan (2), and South Korea (2). They presented to the attendees some interesting facts about their graduate research institutions, university culture, research environment, as well as some of their research interests and past and current projects. For example, Iva Milisavljevic from Alfred University presented her research on the potential of a novel solid-state single crystal growth technique developed in her lab to fabricate high-quality single crystals (Figure 2).

Participation in the Virtual Student Exchange Program was a worthwhile experience for all of the students and, in a way, their first link in a network of new connections to share common ideas that could support the long-term partnership between other researchers and their institutions.

Even more importantly, the Virtual Student Exchange Program enabled them to share not only the exciting results of their research but also a piece of their lives outside the research environment. As Jenna Metera, one of the speakers, says, “I feel that researchers and especially grad students, in general, get lost in their work and truly identify with it. However, the work is not our whole life, and I genuinely enjoyed learning about the other aspects of the other students’ lives at their universities.” Learning about other cultures and lifestyles of other people is one of the most rewarding aspects of exchange programs, which enrich students’ lives and support their personal growth.

After the Virtual Student Exchange Program ended, Metera and another speaker Kana Tomita shared some

thoughts on their experiences with student exchange programs, their opinion on the topic, and the future of student exchange.

“Overall, student exchange allows the students to learn to think in different ways, which is the thing that really aids in better research skills,” says Metera, who is currently a Ph.D. student at the University of California, San Diego. Tomita from Japan had a similar experience, saying, “In my opinion, constant lab student exchange is preferable. In that way, mutual understanding of both the culture and research topic gets much deeper.”

Concerning the current status and availability of student exchange programs, the situation varies from institution to institution. According to Metera’s experience, most collaborations are organized between principal investigators at the university and with other institutions; students can foster relationships as well, but they do so more easily within the university. Spending some time abroad, on the other hand, is highly encouraged at Tomita’s university. However, many graduate students do not have access to travel funds in their institutions, which lowers their chances of landing a student exchange opportunity or means they may have to look for such opportunities elsewhere. In that case, a suggestion from Metera may be the answer to the existing situation, especially nowadays when we are all highly engaged in different social media platforms. Metera suggests establishing a centralized area or message board

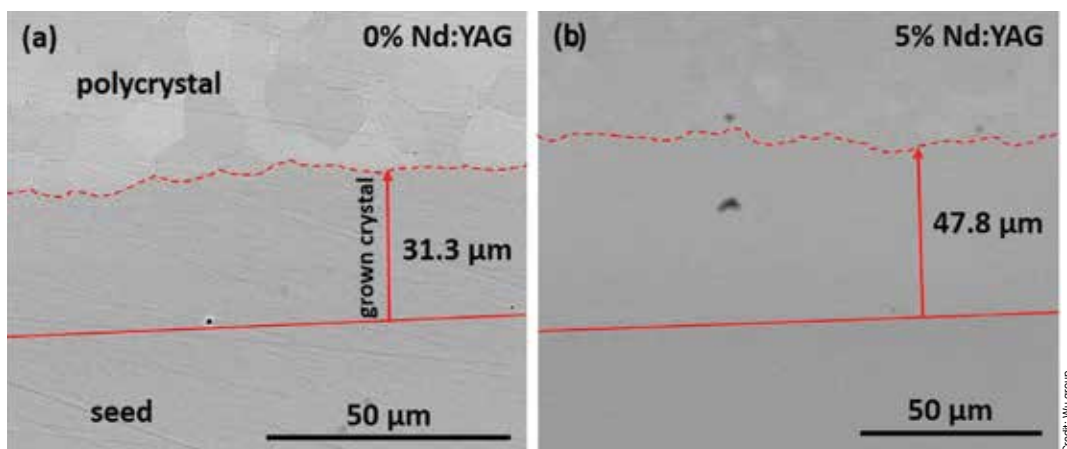
dedicated to finding other people and beginning collaborations for all future collaborations and student exchange opportunities. “I guess that could be a cross between LinkedIn and ResearchGate,” explains Metera.

If there is something to be learned from this whole pandemic situation, it is that we need to reimagine our ways of communicating and value the potential of online networking more. Moreover, for us to collaborate and, therefore, grow on a personal and professional level, we need to develop a positive attitude toward the growth itself and open our minds to new ideas and life-long learning.

With that being said, we conclude with a comment from one of the speakers, Gillian Micale from the Massachusetts Institute of Technology: “After this exchange program, I now believe the great barrier to collaboration is the sheer wealth of research which exists today. For collaboration to occur, we must overcome unknown unknowns and find one another in a vast world of information. This is one of the most beautiful aspects of science: by working together, we inspire growth in countless ways.”

## About the authors

Iva Milisavljevic is a Ph.D. student at Alfred University. Jenna Metera is a Ph.D. student at the University of California, San Diego. Kana Tomita is a Ph.D. student at the Tokyo Institute of Technology, Japan. Yiquan Wu is professor of ceramic engineering at Alfred University. Contact Wu at [wuy@alfred.edu](mailto:wuy@alfred.edu). 



**Figure 2. SEM images of (a) undoped and (b) 5 at% Nd<sup>3+</sup> doped YAG single crystals grown using solid-state single crystal growth (SSCG) method. The SSCG method enables the growth of a single crystal by converting polycrystalline ceramics.**

Credit: Wu group

# LIVE VIRTUAL CONFERENCE

## MATERIALS CHALLENGES IN ALTERNATIVE AND RENEWABLE ENERGY 2021 (MCARE 2021)

## 4<sup>TH</sup> ANNUAL ENERGY HARVESTING SOCIETY MEETING (EHS 2021)

### MATERIALS CHALLENGES IN ALTERNATIVE AND RENEWABLE ENERGY (MCARE 2021)

Organized by The American Ceramic Society and its new Energy Materials and Systems Division, MCARE 2021 is a premier forum to address opportunities of emerging materials technologies that support sustainability of a global society. This conference brings together leading global experts from universities, industry, research and development laboratories, and government agencies to collaboratively interact and communicate materials technologies that address development of affordable, sustainable, environmentally friendly, and renewable energy conversion technologies. If your research seeks sustainable energy solutions on a global scale, you should attend MCARE 2021.

This cutting-edge international conference features plenary and invited talks, thematically focused technical sessions, and poster presentations, enabling participants to network and exchange ideas with professional peers and acclaimed experts. The conference atmosphere engages and promotes the participation of scientists and engineers of all ages to include students and early-stage researchers.

### MCARE 2021 ORGANIZING CO-CHAIRS



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### MCARE 2021 TECHNICAL PROGRAM

**S1:** Materials for Solar Fuel Production and Applications

**S2:** Advanced Materials for Energy Storage

**S3:** Joint with **EHS Symposium 4:** Challenges in Thermal-to-Electrical Energy Conversion Technology for Innovative Novel Applications

**S4:** Advanced Materials for Perovskite and Next Generation Solar Cells

**S5:** Spectral Conversion Materials for Energy Applications

**S6:** Joint with **EHS Symposium 1:** Materials, Components and Devices for Self-powered Electronics

**S7:** Advanced Materials & Nanodevices for Sustainable and Eco-Friendly Applications

**S8:** Advanced Materials for Fuel Cells and High Temperature Electrolysis

**S9:** Critical Materials for Energy Applications

**S10:** Lifecycle Impacts of Clean Energy Materials

**S11:** Materials for Super Ultra-Low Energy and Emission Vehicles

**S13:** Theory and Experiment Meeting in Energy Materials Research

**S14:** Chemical and Biological Sensors: Materials, Devices and Systems

**S15:** Young Scientists Forum on Future Energy Materials and Devices

### HONORARY SYMPOSIUM

**Frontiers of Solar Energy Harvesting and Functional Nanomaterials: New Materials for Photovoltaics, Solar Fuels and Multifunctional Optoelectronic Devices**

(Special symposium in honor of Prof. Yoon-Bong Hahn, Jeonbuk National University)



# SAVE THE DATE July 19–22, 2021

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Hosted and organized by:  
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### 4<sup>TH</sup> ANNUAL ENERGY HARVESTING SOCIETY MEETING (EHS 2021)

Since its inception, the EHS workshop has been highly successful in bringing the academic community from around the world together to openly discuss and exchange ideas about energy harvesting. Those researching energy harvesting know it has become the key to the future of wireless sensor and actuator networks for a variety of applications, including monitoring of temperature, humidity, light, and location of individuals in a building, chemical/gas sensor, structural health monitoring, and more. Join us to share your research in this area and to freely discuss and network with colleagues from around the globe interested in energy harvesting solutions.

This 4<sup>th</sup> annual meeting will feature plenary lectures, invited talks, and contributed talks within the following topical areas:

- Energy harvesting (e.g., piezoelectric, inductive, photovoltaic, thermoelectric, electrostatic, dielectric, radioactive, electrets)
- Energy storage (e.g., supercapacitors, batteries, fuel cells, microbial cells)
- Applications (e.g., structural and industrial health monitoring, human body network, wireless sensor nodes, telemetry, personal power)
- Emerging energy harvesting technologies (e.g., perovskite solar cells, shape memory engines, CNT textiles, thermomagnetism, bio-based processes)
- Energy management, transmission, and distribution; energy-efficient electronics for energy harvesters and distribution
- Fluid-flow energy harvesting
- Solar–thermal converters
- Multi-junction energy harvesting systems
- Wireless power transfer

### EHS 2021 CO-CHAIRS



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### EHS 2021 TECHNICAL PROGRAM

- S1:** Joint with **MCARE Symposium 6: Materials, Components and Devices for Self-powered Electronics**
- S2:** Integrated Energy Harvesting and Storage Systems for Wearables and IoT
- S3:** Multi-functional Energy Conversion Materials and Devices for Energy Harvesting and/or Sensing
- S4:** Joint with **MCARE Symposium 3: Challenges in Thermal-to-Electrical Energy Conversion Technology for Innovative Novel Applications**
- S5:** Special Symposium — Celebrating 20 years of Energy Harvesting

#### SYMPOSIUM 6:

**Special Symposium—European Energy Harvesting Workshop with Special Honor to Professor Pim W.A. Groen**

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- Additive Manufacturing of High and Ultra-High Temperature Ceramics and Composites: Processing, Characterization and Testing

#### ARTIFICIAL INTELLIGENCE

- Materials Informatics for Images and Multi-dimensional Datasets

#### BIOMATERIALS

- Porous Materials for Biomedical Applications
- Next Generation Biomaterials

#### CERAMIC AND GLASS MATERIALS

- Engineering Ceramics: Microstructure-Property-Performance Relations and Applications
- Glasses and Optical Materials: Current Issues and Functional Applications
- Ceramics and Glasses Modeling by Simulations and Machine Learning
- Manufacturing and Processing of Advanced Ceramic Materials
- Ceramic Matrix Composites
- *Journal of the American Ceramic Society Awards Symposium*
- Phase Transformations in Ceramics: Science and Applications
- Preceramic Polymers; Synthesis, Processing, Modeling, and Derived Ceramics
- Solid-state Optical Materials and Luminescence Properties
- Thermal Shock Resistance of Ceramics and Composites

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- Advances in Dielectric Materials and Electronic Devices

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## FUNDAMENTALS AND CHARACTERIZATION

- Emergent Materials under Extremes and Decisive In Situ Characterizations
- Grain Boundaries, Interfaces, and Surfaces in Ceramics: Fundamental Structure-Property-Performance Relationships
- Materials vs. Minerals: Bridging the Gap between Materials Science and Earth and Planetary Science

## MATERIALS-ENVIRONMENTAL INTERACTIONS

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- Coatings to Protect Materials from Extreme Environments
- Progressive Solutions to Improve Corrosion Resistance for Nuclear Waste Storage
- Thermodynamics of Materials in Extreme Environments

## NANOMATERIALS

- Controlled Synthesis, Processing, and Applications of Structural and Functional Nanomaterials
- Mechanistic Insights into the Synergistic Properties of Nanocomposites
- Nanotechnology for Energy, Environment, Electronics, Healthcare and Industry

## PROCESSING AND MANUFACTURING

- 13<sup>th</sup> International Symposium on Green and Sustainable Technologies for Materials Manufacturing and Processing
- Processing and Performance of Materials Using Microwaves, Electric and Magnetic Fields, Ultrasound, Lasers, and Mechanical Work – Rustum Roy Symposium
- Synthesis, Characterization, Modeling and Applications of Functional Porous Materials

## SPECIAL TOPICS

- 50 Years of Characterizing Structural Ceramics and Glasses: Recognizing the Contributions of George Quinn
- Edward Orton Jr. Memorial Lecture
- GOMD Alfred R. Cooper Award session
- ACerS Education and Professional Development Symposium
- ACerS Frontiers of Science and Society – Rustum Roy Lecture
- ACerS Richard M. Fulrath Award Session
- ACerS Robert B. Sosman Award Symposium
- ACerS/EPDC: Arthur L. Friedberg Ceramic Engineering Tutorial and Lecture
- Research Lightning Talks
- Online Teaching Best Practices for the COVID Era and Beyond



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PACRIM

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- S2:** Frontier of modeling and design of ceramics and composites
- S3:** Advanced structure analysis and characterization of ceramics

### Innovative Processing and Manufacturing

- S4:** Novel, green, and strategic processing and manufacturing technologies
- S5:** Polymer derived ceramics (PDCs) and composites
- S6:** Advanced powder processing and manufacturing technologies
- S7:** Synthesis, processing, and microstructural control of materials using electric currents, magnetic fields, and/or pressures
- S8:** Porous ceramics: Innovative processing and advanced applications
- S9:** Additive manufacturing and 3D printing technologies
- S10:** Sol-gel processing and related liquid-phase synthesis of ceramics
- S11:** Layered double hydroxides: Science and design of binding field with charged layers
- S12:** Specific reaction field and material fabrication design

### Nanotechnology and Structural Ceramics

- S13:** Novel nanocrystal technologies for advanced ceramic materials and devices
- S14:** Functional nanomaterials for energy harvesting and solar fuels
- S15:** Engineering ceramics and ceramic matrix composites: Design, development, and applications
- S16:** Advanced structural ceramics for extreme environments
- S17:** Multifunctional coatings for structural, energy, and environmental applications
- S18:** Advanced wear resistant materials: Tribology and reliability
- S19:** Geopolymers: Low energy and environmentally friendly ceramics

### Multifunctional Materials and Systems

- S20:** Multiferroic materials, devices, and applications
- S21:** Crystalline materials for electrical, optical, and medical applications
- S22:** Microwave dielectric materials and their applications
- S23:** Transparent ceramic materials and devices

### Ceramics for Energy Systems

- S24:** Solid oxide fuel cells and hydrogen technologies
- S25:** Direct thermal to electrical energy conversion materials, applications, and thermal energy harnessing challenges
- S26:** Materials for solar thermal energy conversion and storage
- S27:** Advanced materials and technologies for electrochemical energy storage systems
- S28:** Atomic structure and electrochemical property diagnosis toward full crystal rechargeable batteries
- S29:** Ceramics and ceramic matrix composites for next generation nuclear energy
- S30:** High temperature superconductors: Materials, technologies, and systems

### Ceramics for Environmental Systems

- S31:** Advanced functional materials, devices, and systems for environmental conservation, pollution control, and critical materials
- S32:** Ceramics for enabling environmental protection: Clean air and water
- S33:** Photocatalysts for energy and environmental applications
- S34:** Glass and ceramics for nuclear waste treatment and sequestration

### Biomaterials, Biotechnologies, and Bioinspired Materials

- S35:** Advanced additive manufacturing technologies for bio-applications; materials, processes, and systems
- S36:** Advanced multifunctional bioceramics and clinical applications
- S37:** Material and technology needs for medical devices, sensors, and tissue regeneration
- S38:** Nanotechnology in medicine
- S39:** Biomimetics and bioinspired processing of advanced materials

### Special Topics

- S40:** 6<sup>th</sup> International Richard M. Fulrath Symposium, "Frontiers of ceramics for a sustainable society"
- S41:** Advancing the global ceramics community: fostering diversity in an ever-changing world
- S42:** Young Investigator Forum: Next-generation materials for multifunctional applications and sustainable development, and concurrent societal challenges in the new millennium



## 56<sup>th</sup> Refractories Symposium honors Richard C. Bradt despite pandemic

Credit all images: ACerS

The emerging coronavirus pandemic forced cancellation in March 2020 of the 56<sup>th</sup> Annual St. Louis Section/Refractory Ceramic Division Refractories Symposium. Unfortunately, the pandemic situation precluded an in-person gathering in 2021, too, prompting organizers to proceed with the symposium in an online virtual format on March 24–25, 2021.

“Even though we couldn’t be in person this year, the Refractories family still reunited for a fun two days. Each speaker is a familiar face for most of the attendees, which I believe helped the audience stay focused and connected to the virtual meeting,” says co-organizer Kelley Wilkerson, assistant teaching professor at the Missouri University of Science and Technology.

The 56<sup>th</sup> symposium honored the late Richard Bradt, one of the great luminaries in the refractories industry. Speakers reflected on the impact he had on them personally and his tremendous global influence. Bradt was an ACerS Distinguished Life Member and Fellow of The American Ceramic Society.

Charles Semler of Semler Materials Services spoke about Bradt’s “early” years, comprising the first 30 or so years of his career. From the beginning, Bradt’s research focused on structure–property relationships with an emphasis on mechanical properties, especially the application of fracture mechanics to brittle materials. He applied rigorous quantitative methods, used statistics, looked for correlations, and adopted modeling and visual imaging to improve understanding of refractories.

“Dick was highly instrumental in pushing our industry into the modern era,” says Semler.

Despite being online, nearly 180 people registered for the event to hear 19 talks over two days. Highlights included the presentation of the St. Louis Section’s Theodore Planje Award for 2020 and 2021. Tom Vert, retired from Dofasco/Arcelor Mittal and



Jeff Smith (left) introduces 2020 Theodore Planje Award winner, Tom Vert, who is displaying his award.

president of UNITECR 2021 (now 2022), gave the 2020 Planje Award lecture. His engaging talk, titled “Top ten things they don’t tell you in university about the refractory industry,” noted that “what happens in the lab and in the steel plant are two different things.” His tips covered a wide range, including that customers can be grumpy, never check luggage, teamwork is essential, there are no friends during breakout investigation, and lifelong friendships are inevitable.

The 2021 Planje Award winner, Ruth Engel, Refractory Consulting Services, also worked in the steel industry. Her talk, “The path to career planning is SERENDIPITY,” highlighted key nodes and lessons from her 25-year career as a refractories engineer with Armco/AK Steel. She says, “One thing that has never changed is that refractories are always blamed for the operations problems. When you have a refractory problem, the operation stops.”

RCD also presented its Allen Award to Bruno Luchini, Joern Grabenhorst, Jens Fruhstorfer, Victor C. Pandolfelli, and Christos G. Aneziris for their paper, “On the nonlinear behavior of Young’s modulus of carbon-bonded alumina at high temperatures,” published

in the January 2018 issue of *JACerS* (pp. 4171–4183).

Next year, the 57<sup>th</sup> Refractories Symposium will be held in conjunction with UNITECR in Chicago, Ill., March 15–18, 2022 (originally scheduled for Sept. 14–17, 2021).

“Missing the symposium in 2020 meant we had a lot to do this year, and it was amazing to work with the Society, PCSA, and Bravura to make it happen. I am so thankful for everyone’s hard work to make this event a success, and I can’t wait to see everyone in person again soon,” says Wilkerson. <sup>100</sup>



Ruth Engel’s first hardhat. Engel was the 2021 St. Louis Section Theodore Planje Award winner.

## Calendar of events

### April 2021

**25–30** ➡ International Congress on Ceramics (ICC8) – Bexco, Busan, Korea; [www.iccs.org](http://www.iccs.org)



**27–28** Glass International Digital Forum – VIRTUAL EVENT ONLY;

<https://www.glass-international.com/glass-international-digital-forum>

### May 2021

**3–7** 6<sup>th</sup> International Conference on Competitive Materials and Technology Processes (ic-cmtp6) – Hunguest Hotel Palota, Miskolc-Lillafüred, Hungary; [www.ic-cmtp6.eu](http://www.ic-cmtp6.eu)

**16–19** ➡ Ultra-high Temperature Ceramics: Materials for Extreme Environment Applications V – The Lodge at Snowbird, Snowbird, Utah; <http://bit.ly/5thUHTC>

### June 2021

**21–24** ➡ The NSMMS & CRASTE Joint Symposia – Bethesda North Marriott Hotel & Conference Center, Rockville, Md.; <https://www.usasymposium.com/space/default.php>

**28–30** MagForum 2021: Magnesium Minerals and Markets Conference – Grand Hotel Huis ter Duin, Noordwijk, Amsterdam; <http://imformed.com/get-imformed/forums/magforum-2020>

### July 2021



**19–22** Materials Challenges in Alternative & Renewable Energy 2021 (MCARE 2021) combined with the 4<sup>th</sup> Annual Energy Harvesting Society Meeting (EHS 2021) – VIRTUAL EVENT ONLY; <https://ceramics.org/mcare2021>

### August 2021

**31–Sept 1** 6<sup>th</sup> Ceramics Expo – Cleveland, Ohio; <https://ceramics.org/event/6th-ceramics-expo>

### October 2021

**12–15** ➡ International Research Conference on Structure and Thermodynamics of Oxides/carbides/nitrides/borides at High Temperature (STOHT) – Arizona State University, Ariz.; <https://mccormacklab.engineering.ucdavis.edu/events/structure-and-thermodynamics-oxidescarbidesnitridesborides-high-temperatures-stoht2020>

**17–21** ACerS 123<sup>rd</sup> Annual Meeting with Materials Science & Technology 2021 – Greater Columbus Convention Center, Columbus, Ohio; <https://ceramics.org/mst21>

**20–21** ceramitec conference 2021 – Messe München; Munich, Germany; <https://www.ceramitec.com/en/trade-fair/ceramitec-conference>

**18–20** Fluorine Forum 2021 – Pan Pacific Hanoi, Vietnam; <http://imformed.com/get-imformed/forums/fluorine-forum-2020>

**25–27** China Refractory Minerals Forum 2021 – InterContinental, Dalian, China; <http://imformed.com/get-imformed/forums/china-refractory-minerals-forum-2020>

### November 2021

**1–4** ➡ 82<sup>nd</sup> Conference on Glass Problems – Greater Columbus Convention Center, Columbus, Ohio; <http://glassproblemsconference.org>

### December 2021

**12–17** 14<sup>th</sup> Pacific Rim Conference on Ceramic and Glass Technology (PACRIM 14) – Hyatt Regency Vancouver, Vancouver, British Columbia, Canada; [www.ceramics.org/PACRIM14](http://www.ceramics.org/PACRIM14)

### January 2022

**18–21** Electronic Materials and Applications 2022 (EMA 2022) – DoubleTree by Hilton Orlando at Sea World Conference Hotel, Orlando, Fla.; <https://ceramics.org/ema2022>

**23–28** 46<sup>th</sup> International Conference and Expo on Advanced Ceramics and Composites (ICACC2022) – Hilton Daytona Beach Oceanfront Resort, Daytona Beach, Fla.; <https://ceramics.org/icacc2022>

### March 2022

**15–18** 17<sup>th</sup> Biennial Worldwide Congress Unified International Technical Conference on Refractories – Hilton Chicago, Chicago, Ill.; <https://ceramics.org/unitecr2021>

### May 2022

**22–26** Glass and Optical Materials Division Annual Meeting (GOMD 2022) – Hyatt Regency Baltimore, Baltimore, Md.; <https://bit.ly/3ftnJql>

### June 2022

**22–26** ACerS 2022 Structural Clay Products Division & Southwest Section Meeting in conjunction with the National Brick Research Center Meeting – Omni Charlotte Hotel, Charlotte, N.C.; <https://bit.ly/31zyfob>

### July 2022

**24–28** Pan American Ceramics Congress and Ferroelectrics Meeting of Americas (PACC-FMAs 2022) – Hilton Panama, Panama City, Panama; <https://ceramics.org/PACCFMAs>

### July 2024

**14–19** International Congress on Ceramics – Hotel Bonaventure, Montreal, Canada; [www.ceramics.org](http://www.ceramics.org)

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## Joining of similar and dissimilar materials

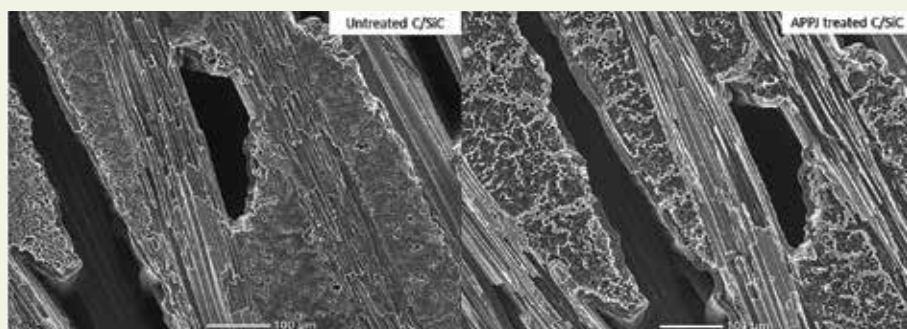
An oft-cited adage in industry is “whenever possible, avoid joining.” Often the discontinuity formed in a component during joining results in the creation of a weaker area, leading to lower performances there compared to the surrounding material. However, joints often cannot be avoided, and so different strategies must be proposed.

It is possible to choose between mechanical joining, such as clasps and latches; direct joining, i.e., welding; or indirect joining, such as brazing or adhesive joints. Any of these techniques can be used to join similar or dissimilar materials, but when the latter are bonded together, some additional challenges arise because of their different physical and/or chemical properties.

Technical ceramics often need to be joined with themselves or integrated with other materials to develop devices that operate in challenging environments. Their intrinsic brittleness makes the process of preparing them for mechanical joining complex; furthermore, this choice would not be effective if sealing is required by the application.

Few solutions for direct joining of technical ceramics exist. One option is to carry out direct bonding, but this option involves solid-state diffusion. For ceramics, solid-state diffusion requires high temperatures and pressures plus extended times, making the process unaffordable. Some works on laser welding of ceramics have been recently published, but large-scale industrialization of this process is not yet achieved.

Indirect joining is a common choice for joining ceramics and ceramic-based materials. Glue can be a solution for low-temperature applications; for high-temperature applications, brazing alloys or glass-ceramics should be considered. Reactive brazing alloys lead to the formation of a reaction layer at the ceramic-alloy interface, which improves the



**Surface of a carbon fiber reinforced silicon carbide (C/SiC) specimen before the atmospheric-pressure plasma jet treatment (left) and after the treatment (right).**

performance of joints. Glass-ceramics provide the advantage of offering the desired properties for the application, though achieving this benefit requires designing an appropriate chemical composition and choosing a proper devitrification process. For dissimilar joints, the joining material must provide a well-bonded interface on both materials and should minimize the difference of the coefficient of thermal expansion, thus reducing detrimental residual stresses.

For more than 25 years, my research group has actively researched joining solutions for ceramics. We have developed a strong knowledge on glass-ceramics as joining materials<sup>1</sup> and have designed new joining processes, for example, the refractory metals-wrap technique, which provides a joining material that consists of a silicon matrix dispersed with reaction-derived silicide particles.<sup>2</sup>

As a second-year Ph.D. student, my doctoral research activity focuses on surface modification of ceramic matrix composites (CMCs) and ceramics to enhance the joint strength. Concerning CMCs, the objective is to induce a selective removal of fibers, creating a brush-like structure on the surface. In a previous work,<sup>3</sup> this type of structure was generated through a thermal treatment, but a detrimental effect on mechanical properties was reported.


To prevent negative effects on mechanical properties, we now are exploiting atmospheric-pressure plasma jet (APJ) to confine the treatment only to the surface. Furthermore, this technique is more ecofriendly compared with other traditional techniques, quite

inexpensive, and it can be applied to large components and/or large batches.

To date, we have worked on carbon fiber reinforced carbon and carbon fiber reinforced silicon carbide, obtaining good results both in term of selective removal and increment of the surface area (Figure 1). Future activities will focus on assessing the effectiveness of this treatment as joint strength enhancer when brazing alloys are used as joining materials.

## References

- <sup>1</sup>M. Ferraris, V. Casalegno, F. Smeacetto, M. Salvo. “Glass as a joining material for ceramic matrix composites: 25 years of research at Politecnico di Torino,” *Int. J. Appl. Glas. Sci.* **11** (2020) 569–576. <https://doi.org/10.1111/ijag.15032>.
- <sup>2</sup>P.K. Gianchandani, V. Casalegno, M. Salvo, M. Ferraris, I. Dlouhý. “Refractory metal, RM – wrap’: A tailorable, pressure-less joining technology,” *Ceram. Int.* **45** (2019) 4824–4834. <https://doi.org/10.1016/j.ceramint.2018.11.178>.
- <sup>3</sup>F. Valenza, V. Casalegno, S. Gambaro, M.L. Muolo, A. Passerone, M. Salvo, M. Ferraris, “Surface engineering of SiC f /SiC composites by selective thermal removal,” *Int. J. Appl. Ceram. Technol.* **14** (2017) 287–294. <https://doi.org/10.1111/ijac.12618>.

*Alessandro De Zanet is a Ph.D. student in materials science and technology at Politecnico di Torino, Italy. His activities are carried out under the direction of professors Valentina Casalegno and Milena Salvo. In his spare time, he enjoys reading nonfiction books on social sciences and innovation, playing boardgames, and walking.* 





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