CERAMIC TECH CHAT

Episode 28

Title – "Materials research for space and in space: Rick Weber (E28)"

INTRO

De Guire: "I'm Eileen De Guire, and this is Ceramic Tech Chat.

While scientists spend years honing techniques and processes to advance scientific knowledge, sometimes a scientist's truest test is their ability to think outside the box when confronted with unexpected challenges."

Weber: "A few years ago, I was measuring up in our lab for some equipment, and I had a colleague with me. And we thought we got all the measurements we needed, and we got back, and we were missing a few. Well, she was actually wearing a polka dot shirt, so we were able to measure the spacing of the polka dots. We had a bunch of photos, and we measured the spacing of the polka dots on the shirt and then that made a good ruler, so we could figure out the size of the places that we hadn't measured."

De Guire: "That's Rick Weber, president and founder of Materials Development Incorporated in Evanston, Illinois. The company develops instruments for measuring thermophysical properties in extreme environments and provides technical support to clients working with advanced materials. And MDI conducts its own materials research too.

We talked to Rick about what's involved in developing instruments capable of testing materials in extreme environments. We'll also look at one extreme environment that Rick's company is conducting research for—human exploration of space."

(music)

SECTION 1

Weber: "I formed the company in 2006. I'd been involved with a precursor company that was actually sold to a Fortune 50 in 2006. A nice exit. So, I wanted to continue some of the things that we did there. And MDI's focus is really on materials in extreme and nonequilibrium relations. So, in many cases, we look at liquids or glasses, a supersaturated solution, supercooled liquids, things that pertain to often glass formation. We develop instruments, and we also provide research services. So, we have a mix of things.

We currently have six employees. We've got a very nice team, actually. Very capable team, very enthusiastic. And we have a lot of collaborators and people who work with us to provide special services. So, we can put together an expanded team fairly quickly. And of course, we have really good customers, too, which we really appreciate."

De Guire: “So, can you tell us a little bit more about the instrument side of the business? What kind of instruments you make, who buys them, what are the challenges in building them?”

Weber: “Yes. We really focus on materials in nonequilibrium and extreme conditions, so our niche is containerless processing. So, we use these noncontact instruments, sometimes called levitation. And we focus on aerodynamic levitation, which is useful for high temperatures. Combined with laser beam heating, we can reach temperatures of over 3,000°C in some cases, although a lot of the work we do is in the mid-temperature range, 1,500°C to 2,500°C. And we also use acoustic levitation, which is for more ambient temperature solutions.

The challenges are really to develop an instrument that’s easy for the user. That’s really our value add. The techniques are not particularly new in the fundamental sense, but the way that we configure the instruments and the way we evolve the instruments through collaborations. We do a lot of work with people at the Advanced Photon Source, Chris Benmore, for example, doing experiments that enabled us over the years to really refine the performance of the instrument and optimize it. So, that’s sort of where we are on those.

Our customers are quite a wide range. There are several of these in national labs, at user facilities, there are several in universities, and there are some in industry. So, we really cover, you know, the three main areas of federal labs, commercial labs, and university.”

De Guire: “And I’m just curious, how big is a levitation sample?”

Weber: “Levitation, to levitate something, you need to overcome its weight. You know, that’s quite doable. I mean, if you imagine a 747, that’s essentially aerodynamic levitation. So in that sense, they could be very large. The challenge with liquids is the droplet needs to hold together, and that’s the job of surface tension. So our typical sample would be around the size of a lentil, around three millimeters. So it’s enough to produce a droplet that one can probe and measure properties and structure, and also produce a piece of material that can be investigated by optical spectroscopy or mechanical properties, for example. But typically on the order of three millimeters.”

De Guire: “Okay. So that’s big enough to see and manage. How does one get the sample into the levitation? So, at those temperatures, you must have some kind of precursor material that you’re working with. Is it already compounded and reacted? Or how does that work?”

Weber: “Yeah, that’s a good question. It started off that we would actually have spheres machined and that’s quite expensive, especially from hard materials, you know, something like an aluminum silicate or YAG [yttrium aluminum garnet] type material. And a number of years ago, probably around the late ’90s, we figured out a way to just melt material using a laser on a copper half. So it’s very, very similar like to the arc melters that metallurgists use to make alloys. Except this is done under a cover gas that might be argon or air or oxygen, for example, even a redox mixture.

So we'll take a powder mixture, typically 325 mesh powders, blend them in a ball mill, and then fuse those powders together on the half and make little chunks. And then the chunks, due to the surface tension, actually spheroidize quite well. They don't need to be perfect spheres. If you have something that's sort of like a potato shape, that will work okay. You just don't want pointy bits. If you have a pointed tip, it's not very good for aerodynamic levitation and tends to aim downwards and sort of drop.

So, we can cover a phase diagram pretty effectively. I mean, we might be going across a binary in 10 mol percent steps, and it's fairly straightforward to blend up compositions and produce them. Those are then placed at room temperature. They're placed in the levitation device, in the aerodynamic nozzle actually, and then the gas flow lifts them, and then we start to heat them. Typically for oxides, we use a carbon dioxide laser, 500 watts CO_2 laser, very good source. It provides directed energy heating. So the sample gets very hot; the surroundings don't. And the wavelength of the lasers, 10.6 microns, so it doesn't interfere with optical diagnostics and temperature measurement and other measurements you might want to do.

In the case of the acoustic levitator, we would introduce a sample with a syringe typically because it would already be liquid."

De Guire: "Ah, okay, alright. So obviously that would be a low-temperature kind of material test?"

Weber: "Yeah, it's a solution, probably an ethanol or something like that."

De Guire: "Okay. So you mentioned MDI's research focus is materials for extreme conditions and nonequilibrium environments. So, why that focus? What makes those materials so interesting?"

Weber: "We like to work at the interface between basic and applied research, and the containerless techniques do two things. At high temperature, they eliminate contact with the container, so chemical reactions are avoided. And one project that we worked on, still an active project, is molten nuclear fuels. For example, UO_2 [uranium dioxide] melts at almost 3,000°C. And not only is it very hot, but it's also a very chemically reactive melt. So, containing a melt like that is very challenging. So, levitation avoids the need for a container and avoids the contamination.

In the context of glass formation, which is an area we're very interested in, lack of a container or avoiding a container removes extrinsic heterogeneous nucleation, so one can super cool liquids in a way that's not always possible, almost never possible, in a container. For example, something like molten YAG, $Y_3Al_5O_{12}$, supercools 600 or 700 degrees. Equilibrium melting point's about 1,970°C. One can cool it down to about 1,300°C and it's still a liquid. So one's accessing different kinds of properties and structure in that metastable state.

What motivates us there is looking for new kinds of glass. The majority of glasses are made from what are called strong liquids. Professor Austen Angell's classification of liquids, the strong liquids are the ones where a log viscosity versus inverse temperature plot is pretty straight. And there aren't actually very many liquids like that. Silica is an example, and maybe germania and boria. The classic backbone of the glass industry materials. And they're very important materials because they're good glass formers and they can be worked. But those materials are a very energetically ambivalent about the state. There isn't much energetic dimension in the glass, in the crystal, and there isn't much structural change between the glass and the crystal typically. So, they're very stable as glasses.

And in searching for new glasses and benchmarking, we look at more fragile liquids. These are ones where the viscosity doesn't change much when you supercool the liquid. I talked about YAG. Six hundred or 700 degrees below the melting point, it's still not very viscous. But all of a sudden, if you cool it another hundred degrees, there's an enormous change in viscosity, many, many orders of magnitude, and you can cool it to a glass. So that provides a way of benchmarking an extreme end member of the glass family and then characterizing it. And the value of doing that is to see how good can you do? You know, what performance can you get when you go out to those extreme corners of the system, and how can those be used to play back into functional glasses to improve properties."

(music)

SECTION 2

De Guire: "I understand you have a few experiments that are active right now on the International Space Station."

Weber: "Yeah. We're very happy to have these, actually. We have two projects, both on ISS. There's a very long history to these. The first experiments on glass in space were done in the mid-1980s on the space shuttle by Professor Delbert Day and Dr. Chandra Ray at Rolla, now Missouri University of Science and Technology. And they melted some glass in an acoustic levitator, some calcia–gallia–silica. And that was right around the time I was actually getting involved with containerless processing. So I found those experiments very exciting and was always keen to do experiments of that kind.

So, we've worked with NASA over a number of years. We currently have two experiments on the station, as I said. They both use the JAXA furnace, Japanese Aerospace Exploration Agency furnace, in collaboration with NASA as well. And it's called the ELF, the Electrostatic Levitation Furnace. So we're doing levitation in microgravity, which sounds very exotic. But what it achieves is, in microgravity, you don't have any buoyancy, so there's essentially no convection. So the transport processes are diffusion controlled, and that allows one to measure transport properties in a supercooled liquid. And that's a very challenging thing to do. There really isn't a way to do it on the ground for very many systems. A few ambient-temperature systems have been studied in that way, but when you're talking about an oxide melt at 1,500°C that's supercooled, it's tricky. So, the ELF enables us to do that.

And we get a lot of support. We work with the team at JAXA through NASA. We actually use often a Teams meeting, and then the controlling the instrument on the space station from the control room in Tsukuba. So we make the samples, we develop the research plan, we produce the samples and characterize them, and then we send the samples to JAXA, and they eventually go up on a SpaceX flight to the low-Earth orbit ISS."

De Guire: "So, what are the specific experiments, and what materials are you investigating up there?"

Weber: "We selected a range of compositions that cover a variety of different fragilities. Some of the constraints are practical because we want samples that are not too volatile So, for example, sodium silicate would not be a good candidate because the sodium tends to evaporate. So we have a family of two broad groups really. We have some titanates. Did a lot of work on rare earth titanates because they're reluctant glass formers but they make interesting glasses. They actually have an octahedral network, unlike a silicate typical glass former would have a tetrahedral species. So, we have a family of those. Some of them are good glass formers and some of them aren't. So, we're looking at why the viscosity changes with temperature. Other compositions are aluminates. We've got some rare earth aluminates and some group two aluminates, such as calcium and magnesium aluminate. The kinds of materials that one might find in geological systems, for example, very often. And very widely used commercially as well. So we then study the thermophysical properties of those liquids over a range of temperatures. And then, if we recover glass that's brought back, then we'll characterize the glass along with glass made on the ground where you have gravity affecting buoyancy. So, different kind of flow dynamic in the melt."

De Guire: "It seems like these experiments might also be extremely useful for probing phase diagrams and phase relationships."

Weber: "It is, yes. I mean, where we start with almost any set of experiments is to go to the phase diagram. And in fact, we use the compilation [ACerS-NIST Phase Equilibria Diagrams]. I mean, it used to be that we would go up to the Northwestern library and pull out the blue books and photocopy. And then we'd go and dig out the reference papers for the original work and we would go from there. Fortunately, we have the very nice system that ACerS has, the online or, in fact, on a CD-ROM in our case.

So, we look at the phase diagrams, and we look for things that are interesting. And by that I mean anomalies. If it's a simple solution all the way across, that's one case. But we might typically be looking for a miscibility gap or a eutectic or line phase indicating that there's some chemistry going on in the melt, and those are often interesting areas to look at. Particularly eutectics if one's interested in glass because, in some sense, they're already supercooled. They have a propensity to be stable as liquids at relatively low temperatures compared to the end members.

So, although we're not directly measuring phase diagrams, we use phase diagrams a lot, and occasionally we learn things that allow us to slightly modify an existing phase diagram. Maybe something was a bit contaminated or a measurement was a bit off. And so sometimes we get some new data that can help to shed light on that."

De Guire: "Right, well, certainly at the temperatures you're working at, most materials will react with whatever container they're in, and that's probably going to skew the phase relationships that are uncovered. But you've gotten around that.

So, you're collecting a lot of thermophysical data, which is really key to doing good modeling, of course. But then, when your samples come home, what's the plan for them when they come home?"

Weber: "They're pretty high-value samples. We might, for a good composition, have somewhere between fifty and one hundred and fifty milligrams of glass. So, the first thing we do is weigh them accurately so we have a reference weight compared to the start mass for density calculations. And then we take some photographs and do a bit of optical microscopy on the whole bead. They typically start out at two to two and a half millimeters in diameter. Then we would normally send them for X-ray diffraction at APS [Advanced Photon Source at Argonne] to confirm that they're amorphous. We might also do tomography at APS to look for bubbles and internal structure. We might also do neutron diffraction at the Spallation Neutron Source at Oakridge because that provides a different measurement of structure. It's sensitive to the lighter elements like oxygen. Someone can get a full picture of the atomic structure. And possibly NMR, for example, also nondestructive. So, anything nondestructive that's related to the type of material. And then we move on to the more destructive tests. And, actually, the final destination for the samples will be the Materials of the Universe Lab at Arizona State University, Professor Navrotsky's lab, and they will do solution calorimetry and measure the enthalpy of vitrification. And at that point the sample will be dissolved, so it won't exist anymore in its original form."

De Guire: "You're really taking a really comprehensive look at the structure and thermophysical properties. You're collecting every bit of data that's possible from these compositions."

Weber: "That's the goal, yes, really getting the most value out of it because there's a significant investment in the samples. We have this up mass and down mass launch costs, and a lot of time and energy put into them for a lot of people. So, yeah, we really want to get the most out of them."

(music)

BREAK

De Guire: "ACerS and the National Institute of Standards and Technology just released Version 5.0 of their Phase Equilibria Diagrams Database. Besides new phase diagrams and commentaries, the new version features an all-new software interface for easy, quick, and secure navigation. Learn more about the ACerS-NIST Phase Equilibria Diagrams at ceramics.org/buyphase."

SECTION 3

De Guire: "So what's the biggest challenge of doing materials research in space?"

Weber: "I think it's that you have to really prepare well ahead of time. You have to anticipate all sorts of things. And you really don't know until you run a particular sample the kinds of things you might encounter. I mean, you can have a very clear idea of what you're trying to measure and the temperature range you want to study and those kinds of things. But we had one sample, for example, that we thought would melt very easily. And it turned out that it wasn't absorbing the laser (it's heated with a laser beam). But it was fluorescing. It contained some ytterbium. So, the sample lit up blue from the fluorescence of the Yb^{3+}, but it didn't heat, and eventually it exploded, basically. It was because the heat was transmitting to the center of the sample where the four heating laser beams intersect. And when it coupled, it suddenly took off and popped. So, that was an interesting and surprising result.

There's a time lag between when you implement a command. The commands are implemented from the JAXA control room in Tsukuba near Tokyo. And it takes about four seconds to relay up and to be processed and for something to happen. So if you want to change laser power or gas pressure or something like that, there's this brief delay. And one needs to factor that in and really sort of anticipate the next move.

The other small challenge is that the space station orbits around 200 miles up and it talks to the ground by satellites and by pickup stations. On about every hour, there's a region where it's no longer connected. So you can have outages that run from a few seconds to maybe as much as 20 minutes occasionally. So, one needs to factor those in so ideally you want to time an experiment so you haven't got a sample in its ideal state for the measurement just as you go dark. So, you need to sort of have that timeline on the screen, really, along with the experiment to keep an eye on where you are relative to an outage."

De Guire: "So, you're monitoring the experiment and maybe controlling the experiment while it's actually happening. But it all controls through Japan and you're in the United States, so there's a lot of timing issues, it sounds like."

Weber: "We're use the chat dialogue a lot because the operator has a lot to handle sometimes. You know, you're dealing with multiple controls. So we'll use the chat box a lot to talk about, 'Okay, when this reaches this temperature, please do this, please do that.' That's turned out to be very good.

It also provides the documentation, because that is another part of the challenge. We get a lot of data, and a big part of the job on the ground is station analysis, particularly images, to extract volumes and oscillation frequencies and things of that nature. So, we really have to have good notes so we know where we are relative to all the other measurements that have been made temperature, pressure, what have you."

De Guire: "Right. So, what do you think the future for materials science research in space looks like?"

Weber: "I think it's going to have two avenues. I would mention that right now actually NASA is undergoing what's called the decadal review, and that's a process where an external panel reviews areas in materials science that they're going to recommend NASA would focus on.

I think the areas that would be useful are sort of two ends of the spectrum. One is basic research, continuing the kinds of things they've done very well and have established capabilities for. Because that provides fundamental information about how materials can be processed in microgravity. The more applied area is that human exploration of space is expected to be going to include actually inhabiting planets or inhabiting parts of the solar system, at least the moon and then potentially Mars. So, utilizing local materials is important. So, characterizing those materials. There's a lot of glasses out there, for one thing. Natural glasses. And how can you harvest those and turn them into useful products? Is additive manufacturing a good way to make glass components in a low-gravity situation? How does that process work when you don't have a gravity vector to condense the material or pull it down into the melt pool, for example? So, the practical sort of engineering aspects.

And I think both of those need to be studied in parallel so you're building up knowledge from the fundamental level and the applied level."

De Guire: "Right, right. So I'd like to switch gears just a little bit now and talk a little bit about the role of The American Ceramic Society in your career and in your business."

Weber: "I came to the U.S. in '86, and I joined ACerS not long after that, in the late '80s. And I soon got interested in nonequilibrium materials because I was working for a small company that was doing instrumentation, noncontact instrumentation, way back. So, I've been involved with GOMD [Glass & Optical Materials Division] for a long time. I think I've been to probably close to 20 GOMD meetings. I missed two or three for different reasons but try to go every year. It's one of my favorite meetings.

ACerS is a really nice society, I think, because everyone I've met has been pleasant. It's a very pleasant community. So I've made a lot of friends through ACerS and had a lot of opportunities for professional development through attending meetings and workshops. So, I think it's a very good organization.

Some of the peripheral things that ACerS does is very useful. For example, curating a phase diagrams atlas is a valuable resource, and I hope the glass database will come to fruition at some point as well. Because those types of things are useful, but they're not the kinds of things that typically a highly profit-motivated business is going to take on. You need an organization like ACerS to kind of manage those things, and then people will subscribe to them. But they're not going to happen on their own. So, that's a high-value add thing."

De Guire: "Great."

(music)

CONCLUSION

De Guire: "In the push to develop materials capable of handling ever more extreme environments, the parallel development of instruments capable of properly testing these materials will help ensure that we have what we need to continue exploration of the outer space frontier."

I'm Eileen De Guire, and this is Ceramic Tech Chat."

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"Visit our website at ceramics.org for this episode's show notes and to learn more about Rick Weber and Materials Development Incorporated. Ceramic Tech Chat is produced by Lisa McDonald and copyrighted by The American Ceramic Society.

Until next time, I'm Eileen De Guire, and thank you for joining us."