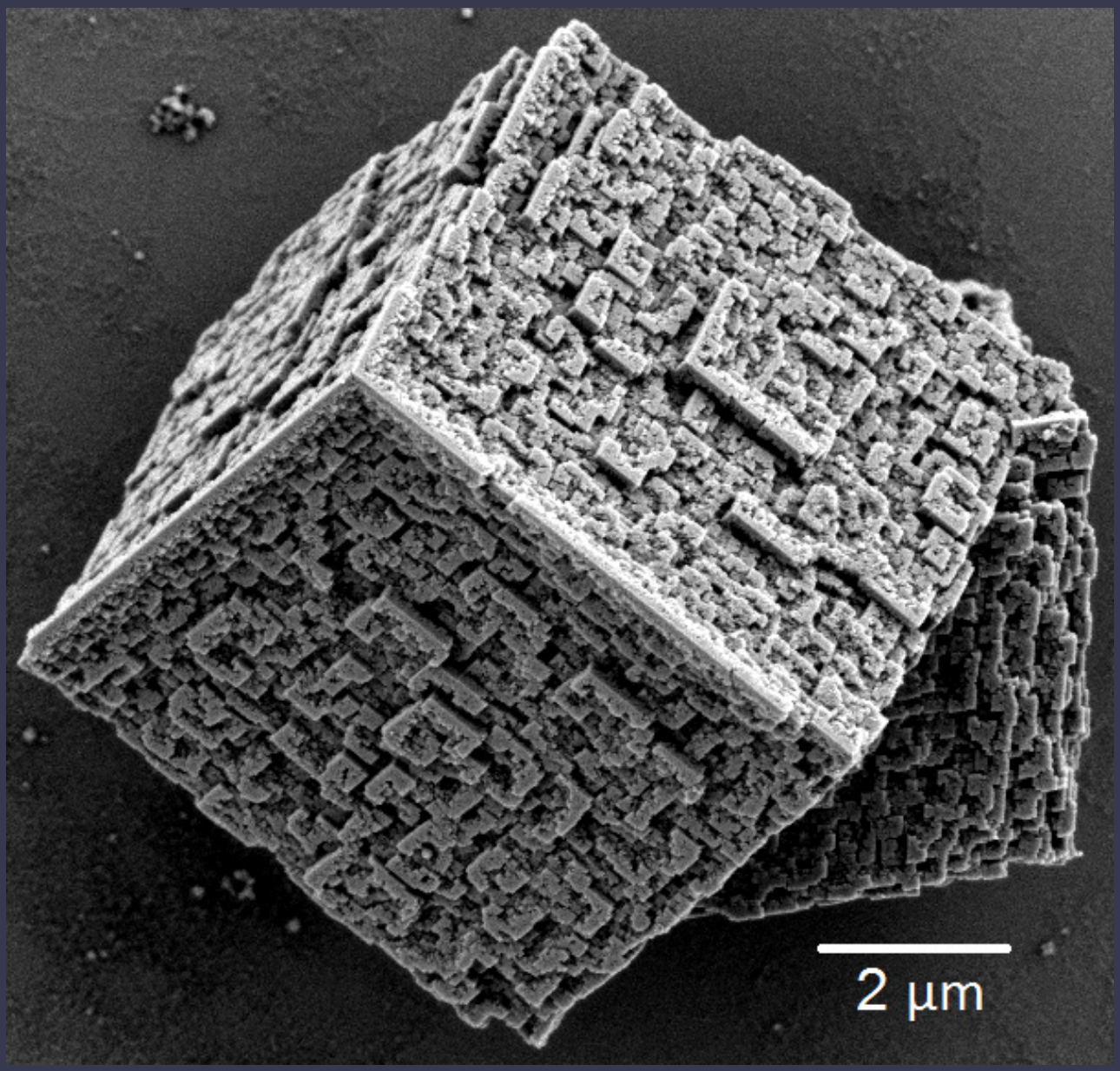
# aacg

## American Association for Crystal Growth

Volume 39, Issue 1, Spring 2014



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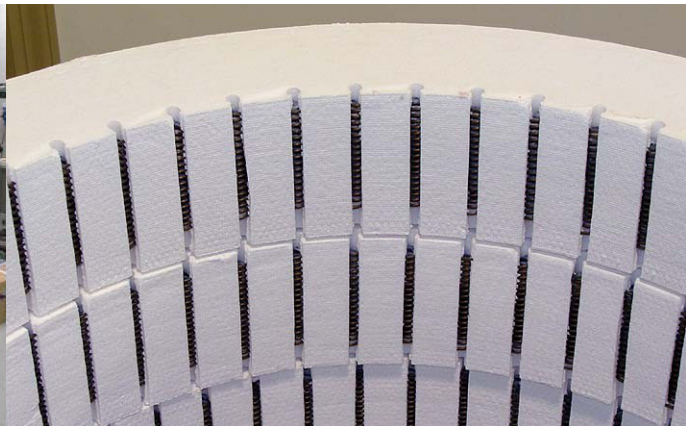

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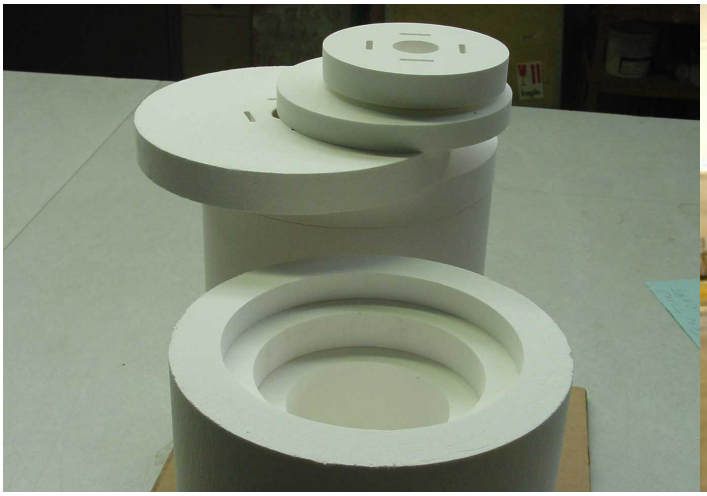

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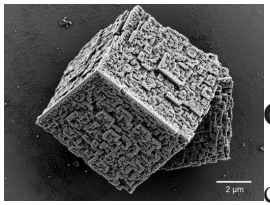

**On the cover:**
"The Borg" - Engineering of calcite crystal faces using a recombinant form of the biomineralization protein AP7; Eric P. Chang and John. S. Evans , Department of Basic Science and Craniofacial Biology – New York University College of Dentistry

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# President's Corner

**AACG president Peter Schunemann**

If you are reading this newsletter you are, I hope, a current and active member of the American Association for Crystal Growth.

As a result of some upheaval in our organization a few years ago (following the sudden and untimely death of our much beloved former AACG President Bill Bonner), our active membership list and our newsletter mailing list became somehow deconvolved. As a result many of you continued to receive the AACG Newsletter without renewing your membership via annual dues or by attending a conference. If that is the case, I encourage you to renew your AACG membership today (by clicking on the membership link at http://www.crystalgrowth.org/ or contacting Shoshana Nash directly at 888.506.1271 or aacg@comcast.net) since future newsletters will only go out to current members.

In addition to the Newsletter, AACG membership includes access to the AACG Member Directory. It has been several years since we sent out a membership roster with valuable contact information, but we will soon be emailing an updated list of current members who choose to be included in the directory.

Our annual scientific conferences are still the primary focus of our organization and remain the best opportunity to network with other experts in our highly specialized field. The meetings alternate between large national conferences (The American Conference on Crystal Growth & Epitaxy, ACCGE) with ~350 attendees in the odd years and a more intimate single session workshop (ACCGE-West) with ~ 75 attendees in the even years. The national meeting rotates between Keystone, CO (2013), Big Sky, MT (2015), and Monterey, CA (2017) whereas the ACCGE-West Meeting is always held at the beautiful Stanford Sierra Camp on Fallen Leaf Lake (2014).

In a unique and multi-disciplinary field like crystal growth, there is simply no substitute for the information and connections that can be gained by attending one of our conferences. New ideas from presentations of cutting-edge results, insights from respected scientists, tips from fellow process engineers, face-time with potential sponsors, relationships with critical material and equipment suppliers – any and all of these can yield dividends that far exceed the cost of attending an ACCGE meeting. Above and beyond the business and career benefits, of course, is the enjoyment we have when we all get together – crystal growers are a fun crowd that doesn't take itself too seriously!

I can personally attest to gaining some invaluable technical guidance at our last crystal growth conference – ACCGE-19 held in Keystone, Colorado last summer. (Many thanks to Dave Bliss, who dedicated much of his first year of "retirement" to organize this highly successful meeting!) I gave a presentation on hydride vapor phase epitaxy (HVPE) of orientation-patterned GaP – an exotic area of research with only a few published papers in the open literature. Afterwards I was talking to our esteemed treasurer, Dave Vanderwater. Based on his years of experience at Lumileds growing GaP by HVPE for LED applications, he suggested that I would probably get much better results at significantly higher growth temperatures (closer to 800°C). I acted on his advice, and my results indeed improved dramatically. The work that Dave and his colleagues in industry had done on GaP HVPE is arguably the most extensive work ever performed in this area and yet it was unpublished – only by presenting a paper and talking to Dave at ACCGE-19 did I gain vital information that saved me many hours of R&D and accelerated me to extraordinary progress. This is a classic example of a trip that paid for itself (and then some!).

Beyond the purely technical interactions that nerdy sci-

# President's Corner (cont.)

entists and engineers thrive on are the deeply personal long-term friendships that are formed in the crystal growth community. These friendships are undoubtedly the most joyful benefit of the AACG experience, and consequently are also the greatest source of loss when these friends pass away. This last year saw the passing of some towering figures in the crystal growth community: Mike Wargo, Dave Brandle, and Paul Bridenbaugh. The brief memorials in this newsletter cannot begin to express our appreciation for them both as crystal growers and as human beings. It was a great privilege to know them, and their presence will be sorely missed. May they continue to inspire us to work, and serve, and interact in a way that honors their memory.

In your service,

Pete Schunemann
AACG President

## Photos from Keystone

Keystone report on Page 20; Photos continue on page 26
*Photos courtesy of Russ Dupuis*

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**Digital Laue detectors – an alternative to high sensitivity films**
for both laboratories and synchrotrons wishing to carry out systematic bulk crystal orientation and micro crystal characterisation.

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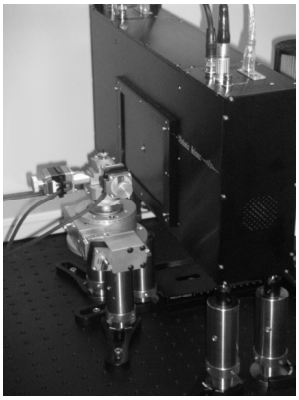
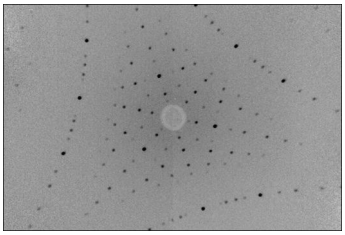
The acquisition software delivers ready to be indexed digital images from PC, to Linux remote controlled machines. Indexation of Laue patterns can be performed off line using dedicated software packages.

An existing Laue set up can be upgraded with turnkey solution including beam delivery: i.e. X-ray source, collimators, goniometer/sample holder up to system installation/integration. Very high resolution detectors allowing strain analysis studies are available on demand.


Laue system

W111 pattern acquired with standard 04x08mm Cu source using 25kV, 25mA and 5 minutes exposure.


Images courtesy SPL, Netherlands

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# Upcoming Events

April 21-25, 2014
2014 MRS Spring Meeting
San Francisco, CA
http://www.mrs.org/spring2014/

May 5-9, 2014
SPIE DSS 2014
Baltimore, MA
http://spie.org/x6765.xml

May 19-22, 2014
2014 International Conference on Compound Semiconductor Manufacturing Technology
Denver, CO
http://www.csmantech.org/

June 8-11, 2014
24th AACGE West Conference on Crystal Growth & Epitaxy
Fallen Leaf Lake, CA
http://www.crystalgrowth.org/23rd-AACGE-Western-Section-Conference.html

June 8-13, 2014
CLEO 2014
San Jose, CA
http://www.cleoconference.org

June 25-27, 2014
56th Electronics Materials Conference
Santa Barbara, CA
http://www.mrs.org/56th-emc/

August 3-8, 2014
Gordon Conference on Defects in Semiconductors
Waltham, MA
http://www.grc.org/programs.aspx?year=2014&program=grs_defect

August 17-21, 2014
SPIE Optics + Photonics 2014
San Diego, CA
http://spie.org/x30491.xml

*Compiled by Mike Snure.*
*Please submit conference information to the editor for inclusion in future issues.*


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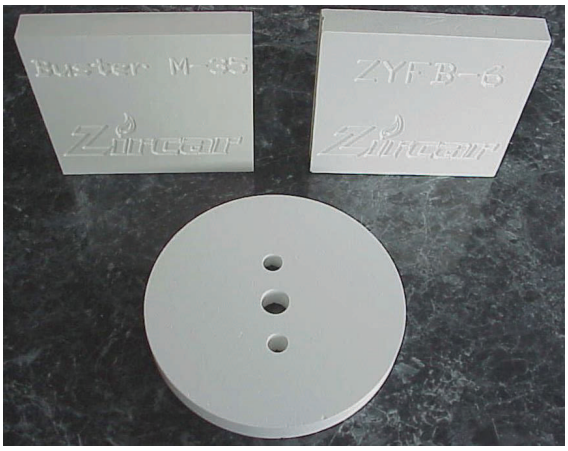


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# Obituary: Michael Wargo

*"I hope they really do name a crater on the moon after Mike Wargo, and it had better be a big one, for he made a huge impression!"*

Michael J. Wargo, Chief Exploration Scientist for NASA's Human Exploration and Operations Mission, died unexpectedly on August 4 at his home in Alexandria, VA. He was 61 years old. Dr. Wargo was the son of Margaret and John J. Wargo of Clairton, PA, both deceased, who themselves were the children of Slovak immigrants. Michael was a leading contributor to NASA's human lunar and planetary exploration program. As a scientific member of many lunar missions, including the Lunar Reconnaissance Orbiter and the LCROSS satellite, Dr. Wargo helped map resources for human missions to the moon and participated in the discovery of ice in the shadows of lunar craters.

In a nearly two-decade career at NASA, he received numerous awards including NASA's Exceptional Service Medal and seven group achievement awards. He was a member of the team planning the next robotic mission to Mars in 2020 and worked gathering crucial scientific information needed to allow humans to be sent safely to the moon, Mars and near-Earth asteroids. Much of his work has helped develop a "roadmap" for human and robotic space exploration for the next two decades.

Dr. Wargo graduated from MIT, with an SB degree in Earth and Planetary Science and received a Doctorate in Materials Science in 1982. At MIT, he was recognized with the John Wulff Award for Excellence in Teaching and the Hugh Hampton Young Memorial Fund Prize for exhibiting leadership and creativity while maintaining exceptionally broad and interdisciplinary interests. He began his career at NASA by turning a fledgling microgravity research division into a worldclass program. NASA drew on Dr. Wargo's ability to explain complex scientific findings in straightforward terms as a spokesman at agency press conferences.

NASA is asking the International Astronomical Union to name a crater on the moon in his honor "so his name will be forever enshrined in the heavens." His colleagues and his friends remember him as inspirational, full of passion and energy, with a booming voice and a great heart. He is survived by his wife, Adele Morrissette of New York, NY, and brothers John, David and Robert, all of whom graduated from MIT.

Gifts may be made to MIT in memory of Michael Wargo for the Department of Materials Science Endowed Fellowship Fund by contacting Bonny Kellerman, bonnyk@mit.edu or at 617-253-9722.

*Obituary published in New York Times*

**Mike was truly a larger-than-life character in the history of the AACG; here are some fond memories shared by AACG members:**

Mike had big heart. He always had a good story to tell, he included everyone, and he was very enthusiastic about life.
- Alexana Roshko

Mike was a great guy. I have many great memories of him at MIT in the 80's. You could always count on Mike to go with you to the Muddy Charles, your party or someone else's. He always kept the conversation lively and I never had to ask him to repeat a darn word no matter how loud the music was. Mike was a HIFI guru. A lot of us would hear him speak the gospel on speakers, amps and turntables. The last time I saw Mike at MIT, he convinced me to go to a party around 4 in the morning while I was walking home along the Charles River. He gave me a ride on his motorcycle to the party and we watched the sun rise. After that I had not seen him for years until I made a visit to NASA to talk about growing some crystals in space. I was meeting with the director and he immediately asked me if I knew Mike. Five minutes later Mike popped in and his trademark voice broke every nano crystal of ice in the room. It was great to see him. I wished our paths had crossed more. He made everything fun! My deepest sympathies go to his family. Know that he had a great impact on us all.
- Rik Riman

Mike had been involved with the microgravity program from his time at MIT. His advisor, August Witt, had been a PI on semiconductor crystal growth experiments on Skylab. As an undergraduate geologist, Mike had taken Witt's well-known introductory chemistry course for non-majors. After graduating, Mike was working as a salesman for Tech HiFi (Tech HiFi stories were a staple of any evening with Mike. I think it was his favorite job.) when Witt came into the store, recognized him, and asked Mike what he was doing there. A little while later Mike was a grad student in Materials Science and Engineering. He stayed in the department for 20 years.
As an LaRC physicist, I did a detail in the Witt Lab with Mike to learn the art and science of crystal growth. Mike started at HQ in 1990-1991 serving as the program scientist for materials science as a JPL detailee - that was a common way to get people on board in the old OSSA.
-Roger Crouch

Michael Wargo and I were graduate students in 1980 in Professor Gus Witt's group at MIT. My office was down the hall from Mike's, and we both taught recitation sections for Gus Witt's massive freshman class in Materials Science. Mike was an early audiophile, having a part-time job selling audio equipment. Whenever I needed to find Mike, I could always locate him because he had a resonant bass voice that could be heard from the far end of the hallway. This voice was extremely helpful in communicating with Professor Witt, who was partially deaf in one ear. Gus Witt relied heavily on Mike, more than anyone else, for connecting with students, checking on experiments, and advising Gus on the latest commercial audio systems.
-David Bliss

I first met Mike Wargo as a freshman at MIT in 1981 when he taught a couple of recitations for 3.091 - fondly known as Sree-oh-nine-vun, the Solid State Chemistry class taught so well by Gus Witt (another founding father of crystal growth who passed away in 2002) that was a great recruiting tool for the Materials Science department.
I'm sure all of us who knew him would agree he was among the most memorable and well-liked characters in the history of the AACG. He was fun-loving, enthusiastic, smiling, warm and friendly, and his deep resonating voice could always be heard above the din at those famous hospitality suites hosted by Johnson-Matthey and Englehard in the "golden age" of crystal growth in the 80s and 90s. During those long evenings he always had a beer in his hand and a story to tell and a small crowd around him to tell it to. For him crystal growth was as much an art as a science, and he took great pride in his ability to stare into a Czochralski furnace and have the intuition and magic touch to tweak the parameters to produce that thing of beauty – a single crystal boule. My most poignant memory of Mike, however, was at one fateful crystal conference where he had to tell all of his friends whom he had funded for years that the microgravity crystal growth program at NASA had come to an end – you could tell it really broke his heart.
I hope they really do name a crater on the moon after Mike Wargo, and it had better be a big one, for he made a huge impression!
-Pete Schunemann

"Come on!" would resonate by the office door in the evening and it would be time for total immersion in English at the Muddy Charles. Mike was always the perfect science teacher and for language: the Pub was the best location! As a newcomer at MIT and not knowing much English it was not always easy but Mike was always there for me, along with David (Bliss) and Christine (Wang). He knew when help was needed and certainly knew how to put a smile on people's faces. I keep very fond memories of those four years. I learned of his passing at a meeting in Washington and on the plane back to San Francisco images flashed back to me: Mike at every corner of Building 13, by his puller, in the corridors, etc… I will never forget his big hug the last time I saw him at a conference and his loud laugh as we recalled old stories.
From that crater he will make us smile again when we look at the moon.
-Edith Bourret

# Obituary: Charles David Brandle Jr.

Charles David (Dave) Brandle Jr., longtime crystal grower and past president of the AACG, passed away September 2, 2013.

Dave grew up in Victoria, Texas and after two years at Victoria Junior College enrolled at The University of Texas at Austin. On graduating in 1963, Dave and his wife Patty relocated to Indianapolis to take a job with Union Carbide's Speedway Research Center. Within the year, Patty gave birth to their daughter Wendy. After two years, Dave returned to UT Austin to pursue a Ph.D. in chemical engineering. His son Chris was born in 1969 and Dave obtained his doctorate in 1970.

The family relocated to the east coast when Dave joined Bell Labs in 1970. In 1975, the Brandles moved to San Diego, where Dave re-joined Union Carbide as Director of Research. Five years later, the family returned to Bell Labs in New Jersey where Dave stayed until his retirement in 2002. After Dave and Patty retired to their home state of Texas, Dave continued consulting for various crystal growth firms around the world.

Much of Dave Brandle's life's work focused on the Czochralski growth of network oxides - notably synthetic perovskites, garnets, and spinels. Many of the materials he developed had significant uses as substrates, lasers, phosphors and electrooptic devices.

Coming to Bell Labs near the beginning of the magnetic bubble effort Dave filled the strong need for a lattice matching garnet substrate with the development of gadolinium gallium garnet (GGG). As a chemical engineer, Dave brought a strong understanding of fluid dynamics and flow that went into process control for diameter control and defect reduction through the starting conditions. In particular he appreciated the need for divalent dopants to control interface stability and prevent corkscrewing.

Dave had an early appreciation for the fact that the stoichiometric composition is not always the congruent composition and he and his collaborators determined the congruent compositions of the rare earth gallium garnets, rare earth scandium gallium/aluminum garnets, lithium niobate, lanthanum aluminate, and many other materials in the course of developing their growth to technologically useful sizes. Dave and longtime collaborator Alex Valentino had a veritable library of garnet and perovskite materials they had grown.

As a supervisor, Dave extended his intellectual reach to a wide variety of crystal growth areas. In his second stint at Union Carbide he had responsibility for growth of sapphire and ruby as well as 3" GGG. In returning to Bell Labs he took over both the Czochralski and liquid phase epitaxy (LPE) efforts. When the magnetic bubble effort burst (got caught between a RAM and a hard disc as they say) he guided the team into electrooptic materials (lithium niobate), single crystal phosphors (Tb- and Ce-doped yttrium orthosilicate and lanthanum magnesium aluminate), and laser hosts (Nd-doped gadolinium scandium gallium garnet). To make sure his team came to talk to him frequently, he always kept a jar of M&Ms on his desk.

In particular, Dave's thesis work on perovskites prepared the team when perovskite substrates were needed for high temperature superconductors. Following a commitment from Bell Labs' management, the team commercialized lanthanum gallate, neodymium gallate, lanthanum aluminate (the subject of Dave's first publication at the Union Carbide Speedway Labs), and developed the new congruent solid solution lanthanum strontium aluminum tantalate (LSAT). When a new material was needed Dave would pull out his old notebooks, which had a wealth of compositions. For quite a period these substrates

were actually sold out of the basic research area of Bell Labs through Nassau Metals, AT&T's arm for metals recovery.

Eventually it was understood that the bismuth-doped rare earth iron garnets developed as thin films for magnetic bubble materials had a far more important application in thick film garnet Faraday rotators for magnetooptic isolators in fiberoptic systems. Under Dave's guidance the team followed the Japanese lead and independently developed a crystal growth platform including up to 5" substituted GGG substrates and film growth in excess of 0.5 mm thick, where the film and substrate are of comparable thickness. In the process the team invented the Latching Faraday rotator that won an R&D 100 award in 1996. This process was transferred to the Reading works and, at the peak, 100 furnaces were growing films for 12 million devices per year under Dave's guidance. Of that time one of the team members Ron Genova wrote: "Dave was a wonderful, gifted man whom I admired and greatly respected. As time goes on, I've realized that you never appreciate how great a group of co-workers is until long after everyone has moved on ... and then it dawns on you how fortunate you were to have been part of an awesome team. I count our days together in the Labs as among the best in my entire career."

Dave was made a Bell Laboratories Fellow in 1998. At the time, this seminal honor had only been given to approximately 160 individuals in the history of Bell Laboratories. In the later years at the Labs, the team significantly also developed rare earth scandium perovskites as substrates for a variety of functional materials and magnesium scandium aluminate as a substrate for gallium nitride.

In his free time, Dave enjoyed gardening (and dueling with woodchucks), woodworking, cheering for the Longhorns, making his famous lemon meringue pies and smoked briskets, and spending time with his family. Dave was a great mentor, collaborator and friend and was very widely loved and respected in the crystal growth community to which he devoted extensive service. Longtime colleague and fellow AACG officer Eric Monberg may have put it best: "David was a classy individual and raised the spirits and IQ of any group that he was a part of. I hope they lay him to rest with his cowboy boots on!"


## Multiwire Laboratories, Ltd.
## MWL 120 Back-Reflection Laue Camera: The Real-Time Advantage

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The MWL120 Real-Time Back-Reflection Laue Camera System (left) provides fast, easy, and reliable crystal orientation. True real-time imaging capabilities combined with motorized sample stages allow for dynamic reorientation. Collection mode provides variable exposure time data collection for high-precision orientation.

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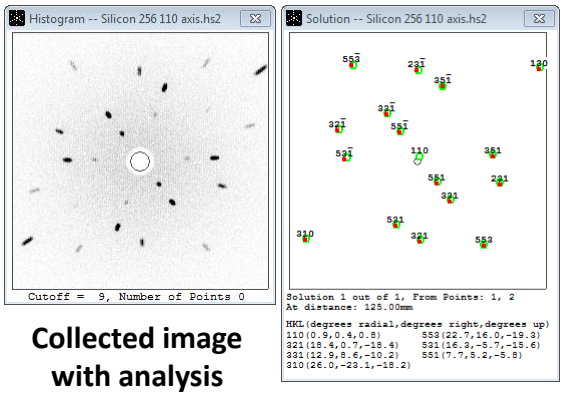


**Collected image with analysis**

Right: Screen captures from real-time mode shows a silicon sample as it is rotated into alignment along the (1,0,0) axis.
Far left: Silicon image collected with 20 second exposure.
Left: Results of automatic point detection and indexing.

For more information, contact:
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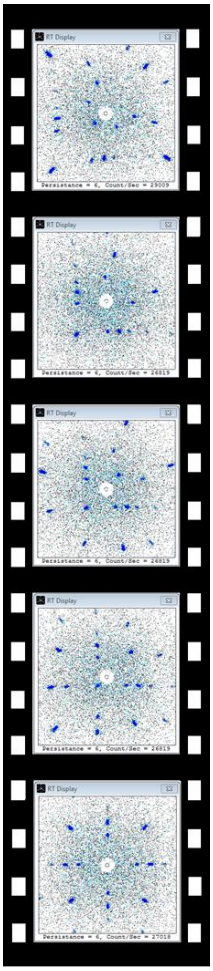


**Real-Time Image Sequence**

# Obituary: Paul Bridenbaugh

The AACG has lost one of its long-time members, Paul Bridenbaugh, who passed away gently and softly in the early evening of June 26, 2013. Paul was born in 1936 and spent an ideal childhood growing up in Point Pleasant Beach, N.J. Paul graduated from Point Pleasant High School as valedictorian, having been class president for his junior and senior years. Paul went on to Rutgers University with a full four-year scholarship and graduated in 1958 from the College of Engineering as a ceramics engineer.

After graduation Paul went to work at AT&T Bell Labs as a materials scientist where he spent 35 years. He was a particular collaborator of George Peterson, Bob Laudise, and Jim Philips. He and George were growing lithium niobate as early as 1964 and they continued to collaborate on fifteen publications on topics ranging from non-linear optical crystals to superregenerative NQR spectrometers.

His Bell Labs work spanned photovoltaics, II-VI crystals, chalcogenide glasses, organic semiconductors, and fluorides for use in lasers, solar electrical generation, light manipulation, and medical imaging.

He was known as a man who, although he was busy with his own job, always took the time to give advice or a word of wisdom to help younger engineers navigate the work of the materials science department. He was the departmental coffee maker and organized liquid nitrogen ice cream parties.

After "retiring" from Bell Labs, Paul continued his career with Coherent Crystal Associates in East Hanover, N.J., and finally with a startup company, Crystal Genesis. In these years he worked extensively with piezoelectric crystal development for sonar applications.

Paul spent his 48-year career doing cutting edge and extensive research and development in crystal growth. His research resulted in the development of a number of published patents. He also authored and co-authored more than 70 peer-reviewed scientific articles in leading scientific journals. In addition to AACG, Paul was also a member of Keramos (Ceramic Honor Society).

He is survived by his wife, Patricia, three daughters, one step-son and 11 grandchildren. He will be missed by all of us.

# Recent Government Contracts Awarded

**NSF SBIR Phase I 2014**
**"An Enhanced Epitaxial Crystal Growth Method To Fabricate MTJ Having Ultra-Low Magnetic Damping"**
**T3Memory USA, Inc.**
**Santa Clara, CA**

**NSF SBIR Phase I 2014**
**"Fully Printed Single Walled Carbon Nanotube Thin Film Transistor Backplanes"**
**Atom Nanotechnology**
**Inglewood, CA**

**NSF SBIR Phase I 2014**
**"Ultra-High-Efficiency Thermal Interface Materials Based on Self-Aligned Graphene Fillers"**
**Quantum Seed LLC**
**Riverside, CA**

**NSF SBIR Phase I 2014**
**"High Efficiency - Low Cost Hybrid Photovoltaic-Thermoelectric Cells"**
**HybridalSol LLC**
**Auburndale, FL**

**NSF STTR Phase I 2014**
**"Efficient Mid-Infrered Interband Cascade Lasers"**
**Applied Quantum Enovation LLC**
**Norman, OK**

**NSF STTR Phase I 2014**
**"Pulsed Laser Polishing"**
**LasX Industries, Inc.**
**White Bear Lake MN**

**NSF STTR Phase I 2014**
**"Low-Cost and Green Deposition of Multifunctional Oxide Films"**
**Solution Deposition Systems, Inc.**
**Santa Barbara, CA**

**DOE SBIR Phase I 2014**
**"Next Generation Semiconductor-Based Radiation Detectors Using Cadmium Magnesium Telluride"**
**Brimrose Technology Corporation**
**Sparks, MD**

**DOE SBIR Phase I 2014**
**"Low cost infrared nanospectroscopy"**
**Anasys Instruments Corp.**
**Santa Barbara, CA**

**DOE SBIR Phase I 2014**
**"Efficient Large-Area X-ray Detectors"**
**Dxray, Inc.**
**Northridge, CA**

**DOE SBIR Phase I 2014**
**"Improved Laser Technology for nonlinear-optical microscopies"**
**Kitware, Inc.**
**Boulder, CO**

**NASA SBIR Phase I 2013**
**"A High Efficiency 1kWatt GaN amplifier for P-Band pulsed applications"**
**Integra Technologies, Inc.**
**El Segundo, CA**

**NASA SBIR Phase I 2013**
**"High Temperature Sensors Using Vertically Aligned ZnO Nanowires"**
**HARP Engineering, LLC**
**Gainesville, FL**

**AF SBIR Phase I 2013**
**"Low Temperature Thermoelectric Refrigeration with HgCdTe-based Superlattices"**
**EPIR Technologies, Inc.**
**Bolingbrook, IL**

**Army STTR Phase I 2013**
**"Conformal Passivation of High Aspect Ratio HgCdTe Surfaces by ALD Using a Novel Cd-Precursor"**
**Radiation Monitoring Devices, Inc.**
**Watertown, MA**

**Army STTR Phase I 2013**
**"Solar Blind MgZnO Photodetectors"**
**Agnitron Technology, Inc.**
**Eden Prairie, MN**

**Navy SBIR Phase I 2013**
**"Gallium Nitride Based Active Electronically Scanned Array (AESA) Technology for High Altitude Periscope Detection"**
**Custom MMIC Design Services Inc.**
**Westford, MA**

**Navy SBIR Phase I 2013**
**"Gallium Nitride (GaN)-based High Efficiency Switch/Transistor for L-Band RF Power Amplifier Applications"**
**Cambridge Electronics, Inc.**
**Cambridge, MA**

**Navy SBIR Phase I 2013**
**"High-Power 3 Micron Fiber Based Laser System"**
**COE Optics, LLC**
**Seneca, SC**

**Compiled by Mike Snure**

# ACCGE-19 Photo Contest

## Sponsored by Journal of Crystal Growth

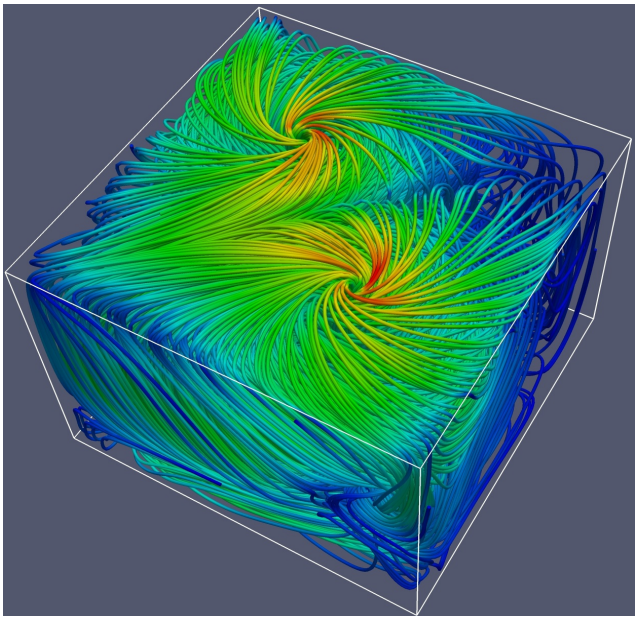
The photo contest at ACCGE-19 was organized by Candace Lynch (Inrad Optics) and Balaji Raghothamachar (Brookhaven National Laboratory). The Journal of Crystal Growth sponsored prizes for winners in 3 categories: natural untouched micrographs or photographs, photographs including digital manipulation (and computational simulations), and student microscopist/photographer. Winners were selected by ballot from among 14 entries.

**First Place, natural untouched micrographs:**

The Borg
Eric P. Chang*1, Jennie A. Russ2, Andreas Verch3, Roland Kröger3, Lara A. Estroff2, John. S. Evans1 Department of Basic Science and Craniofacial Biology – New York University1, Department of Materials Science and Engineering – Cornell University2, Department of Physics – University of York3

Engineering of calcite crystal faces using a recombinant form of the biomineralization protein AP7.

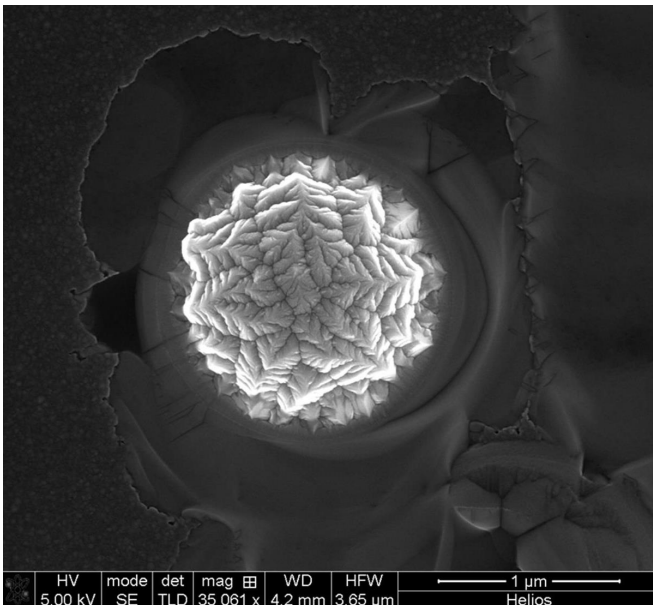
**(image on cover)**



**First Place, photographs including digital manipulation (and computer simulations):**

Hedgehog Tenderness
Daniel Vizman, Alexandra Popescu, Radu Negrila
Department of Physics, West University of Timisoara, Romania

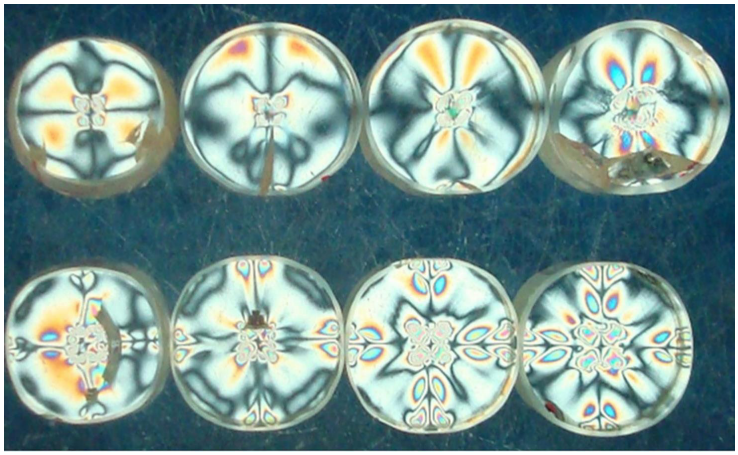
This image represents forced convection generated by a special type of electromagnetic stirring. A square-shaped crucible, characteristic for directional solidification method, is placed in a vertical low intensity magnetic field. The stirring effect is generated by the combination between the magnetic field and an electrical current passing through two electrodes in contact with the free melt surface.




**First Place, student microscopist/photographer:**

GaAs µ-cauliflower
Sema Ermez, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA

The "µ-cauliflower" GaAs crystal was grown by metal organic chemical vapor deposition (MOCVD). Similar growth conditions result in nanowire growth; however, in this example the imbalance of Ga and As precursors (V/

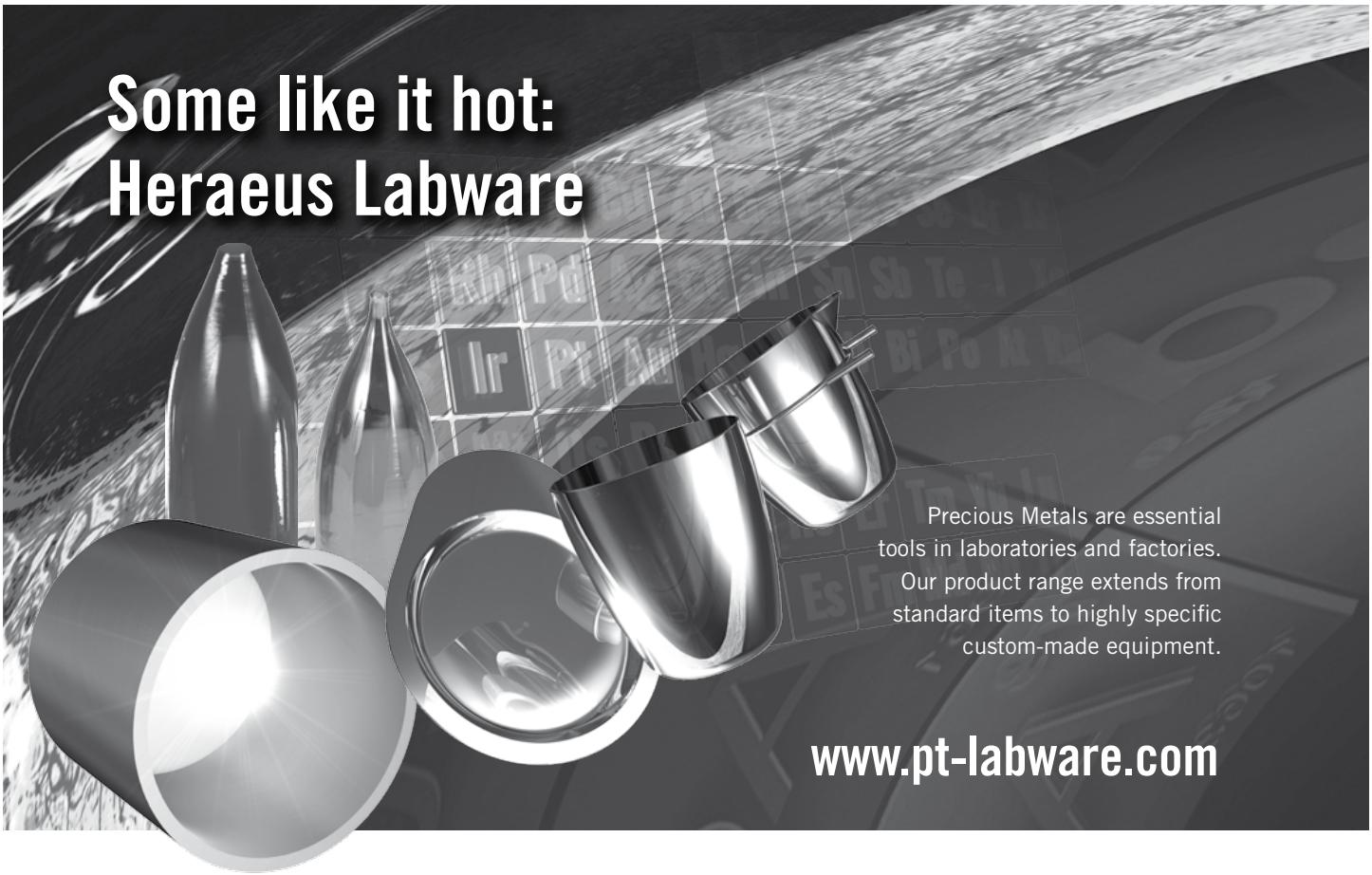


III ratio) during growth resulted in polycrystalline structure.

**Second Place overall:**

Lattice Strain Evolution During Crystal Growth Along Different Crystallographic Directions
Mark Randles and Denys Solodovnikov
Northrop Grumman - SYNOPTICS

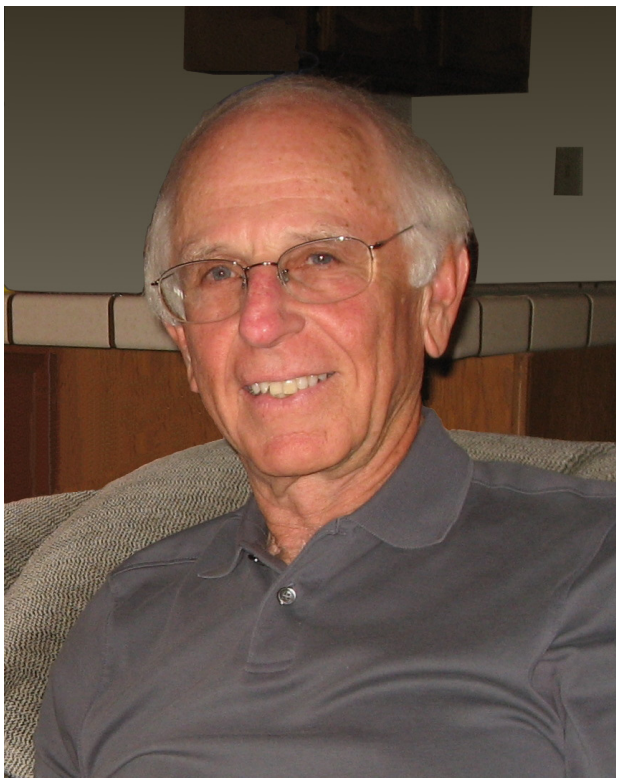
This picture displays lattice strain evolution during single crystal growth of a rare-earth aluminum garnet, as seen through crossed polarizes. Shown are two crystals grown in [110] (top row) and [100] (bottom row) direction, demonstrating how crystal symmetry affects the strain patterns in crystals grown under the same conditions.

# Interview with Don Shaw

**contributed by Dave Bliss**

Don Shaw contributed pioneering work on vapor phase epitaxy of gallium arsenide during the 1960s and 1970s while he was at Texas Instruments (TI). Early in his career, his experimental methods shed light on the mechanism and kinetics of orientation-dependent growth rates. In this interview, Don Shaw describes his early years as a crystal grower at TI and his later transition to management.



DB: How did you get involved with crystal growth?
DS: My PhD research did not involve crystal growth. I got my physical chemistry doctorate at Baylor, and my thesis was on electron microscopy of clay minerals. These are materials of interest to the oil industry. And when I graduated in 1965, it was truly a golden time for a PhD graduate. I had many offers, and in fact one oil company wanted me to simply continue what I had been doing with clay mineral research. I only interviewed with one semiconductor company (TI), and I was really struck by the enthusiasm of the employees and the dynamic nature of an emerging technology. I had no background in crystal growth, but my first project was to develop selective epitaxial growth of Gallium Arsenide. This was part of a program to develop a monolithic GaAs integrated circuit. A very ambitious program and, I should add, very premature.

I think TI's main interest in me was my characterization experience. TI was buying its first electron microprobe when I arrived, and my background in electron microscopy was a good fit. They wanted me to establish an electron microprobe capability. But, although the microprobe had been purchased, it had not yet been delivered. So my first temporary assignment in epitaxial growth soon became permanent. Sometimes it is good to move on to something new.

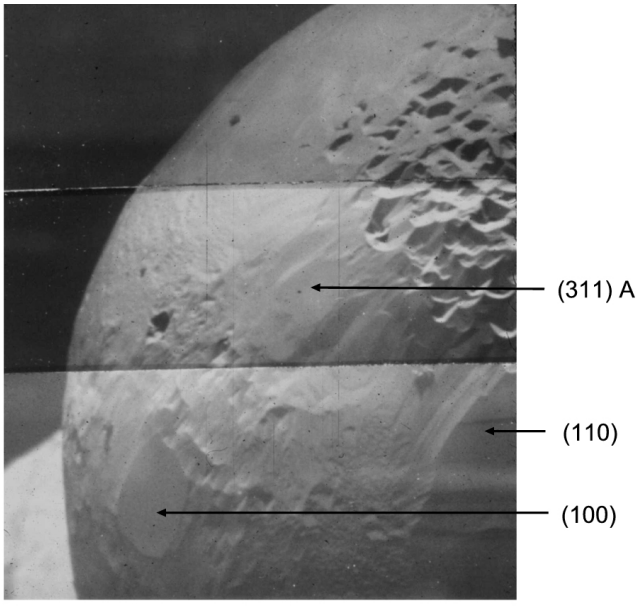



Figure 1. Scanning electron micrograph of epitaxial GaAs on spherical GaAs substrate ( x 66).

DB: Did you have to build your own reactor?
DS: Yes, I did build it, but there were a couple of others that had been built, so I had a model to go by. It was a horizontal tube reactor using the arsenic trichloride and gallium system. Others, for example, the RCA Lab, were working on a different system using arsine, but that would not meet our objectives. Right at that time the Gunn Diode had been invented. This was one of the first commercially successful GaAs microwave devices. A critical factor to success with making Gunn Diodes is the purity of the material. One could never achieve the high purity using arsine, certainly not the arsine that was available then. So the arsenic trichloride process was the method of choice.

DB: Were you tasked to grow FETs?
DS: The GaAs FET had not been introduced at that time. The term we used was "functional electronic block". It was envisioned that we would take semi-insulating GaAs substrates, etch holes in the wafer, and then deposit semiconducting material in the holes to bring it up to the level of a planar integrated circuit. In fact the dream was that after the initial deposition, you could then deposit semi-insulating material on top and thereby build up a three-dimensional integrated circuit. In this dream we weren't too concerned about heat dissipation. It was a very ambitious AFRL program, although not totally successful in the beginning. However, this effort eventually led to the breakthrough of the monolithic microwave integrated circuit (MMIC), one of TI's major success stories.

I continued to work on epitaxial materials, and at one point, I was the only person in the research lab doing epitaxial GaAs growth. We had programs with the Air Force Lab and with the Office of Naval Research. The main driver was to develop the MMIC, but a step along the way was the GaAs FET.

DB: Did you buy Chromium-doped GaAs substrates or grow them in-house?
DS: Not me personally, but TI was growing these Cr-doped substrates in the basement of the research lab, and I worked pretty closely with those colleagues. TI had a patent on Cr-doped semi-insulating substrates, which probably expired about the time they became really useful. We were using a system called "Syringe-type Czochralski" before the days of LEC. It worked by using a seal to maintain the arsenic overpressure. Later, the person who did much of that work, Jack Morgan, went on to start his own company, and eventually TI bought that company back.

DB: Who do you recall from the early days of GaAs development at TI?
DS: There was Glen Westfall and Andy Purdes, who later took over the management of the GaAs activity. Yung Chung Kao and Hung Dah Shih were MBE growth specialists. Ed Beam, another MBE grower, is now at Triquint. Fred Doerbeck and I worked together on epitaxial growth, and then Fred later moved from the research lab to a development organization, where he got involved in LEC growth. Of course the most famous crystal grower at TI was Gordon Teal. His success and the success of his group on growth of semiconductor crystals is what really put TI on the map. When I joined TI he was a respected emeritus research leader, no longer active in crystal growth, but I occasionally had opportunities to talk with him when he would visit the research library.

**Table 1**

| Orientation | Deposition rate (microns $h^{-1}$) |
|---|---|
| {001} | 29 |
| {115} | 32 |
| {113} A | 34 |
| {112} A | 67 |
| {111} A | 90 |
| {221} A | 92 |
| {331} A | 70 |
| {551} A | 51 |
| {110} | 14 |
| {331} B | 15 |
| {221} B | 20 |
| {111} B | 6 |
| {112} B | 24 |
| {113} B | 37 |

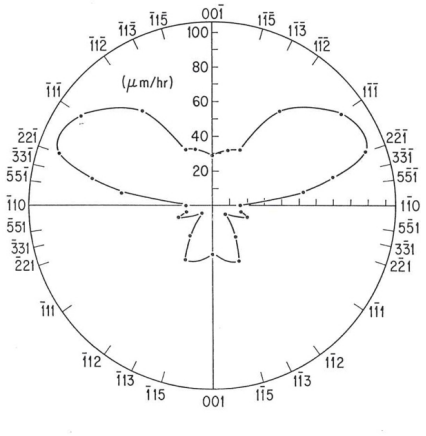



Figure 2. Polar diagram of deposition rate vs. crystallographic orientation.

DB: You conducted a rather unique experiment to determine the orientation-dependence of the growth rate of GaAs. What was the challenge there?
DS: That was an interesting time. If you grow epitaxially on a GaAs sphere, it will grow in all directions simultaneously (Figure 1). It was particularly important to the VPE research I was doing because of the large number of variables in the process. We needed a way to determine the different growth rates under identical conditions. It turned out that fabricating the sphere was not so hard, but the real challenge was that mechanical fabrication would damage the surface, which is bad news for epi-growth. To remove the damage, you can etch it away, but most etchants are orientation-dependent themselves. With most etchants, you may start with a sphere, but end up with a faceted polyhedron. The challenge in doing this experiment was to find an isotropic etchant, one that was basically diffusion limited.

DB: Where did you present the paper?
DS: That is another story. I was told that the International Symposium on GaAs would be held on the island of Bermuda. It was a great opportunity for a third-year employee, and the company seemed amenable to sending me. But for some reason, right at the last minute, the conference orga-

nizers decided not to hold the meeting in Bermuda, and instead, of all places, they decided to hold it in Dallas. This was only seven miles from where I worked. The paper was published in 1968 by the IOP for the second international symposium on GaAs.[1]

DB: Did you eventually move into management?
DS: I was quite happy working in the lab, and TI seemed satisfied with my performance, but then one of the managers in the materials area died, and they asked if I would expand my efforts to include management of his programs. That was a shift in my career and my activities. I continued my VPE work, but I assumed responsibility for bulk materials, for epitaxial growth of garnets for magnetic bubble memories, and another program on mercury cadmium telluride epitaxial growth.

A few years after I became the manager of this group, I was asked to form and direct a new materials science laboratory in Dallas. This laboratory had a broad range of activities, including materials preparation, characterization, and development of new semiconductor processing approaches. I spent nearly a decade as the director of that laboratory. With every year, I did less in the laboratory, and more administrative work. And then to my great surprise, at the end of 1990, TI decided to establish their first international R&D center in Japan. I was asked to be its founding director, so that was a major change. My wife and I moved to Japan in 1991.

DB: Did you have to learn Japanese?
DS: The answer to that is, no! But I certainly did try. I learned enough to communicate with the taxi driver, and so forth, but almost always, my Japanese was inferior to my colleagues' English.

DB: Who did you recruit to work for you?
DS: The new research center that I became responsible for was a beautiful affair. It was a brand-new six-story building, but empty. I felt like a librarian who had no books. We had one senior Japanese researcher, who served in the role of a branch manager, and we brought another senior person from the US. Otherwise, we recruited from the universities. Our objective was to recruit talented young researchers, and that was my main job. Recruiting new PhDs in Japan is quite different than in the US. The role of the graduate's advisor in determining his initial employer is considerably greater in Japan. In addition to speaking to potential employees, we especially needed to communicate with their advisors.

DB: What was the Japanese work environment like?
DS: We didn't sing the company song in the research lab, but there were many differences, as you can imagine. But I did not find any differences between the capabilities of Japanese and American researchers. There was a difference in style and culture. At about the time I moved to Japan, the management philosophy in the US was management by support, that is, by walking around to get close to the employees. I really enjoyed that with my organization in Dallas, where I could walk into the lab and chat a while with the researchers while they were working. But in Japan, that technique proved to be difficult, because whenever I walked into the laboratory, almost immediately, the researchers would just stop. They would assume a sort of formal stance, as if a shogun had walked in the door. That was one big difference I noticed, but the strength of the researchers was certainly equivalent to the US.

DB: Was the idea of an international research center a success for TI?
DS: Yes, we had success in several areas like digital image compression and speech recognition. We did have a materials research program on high-k dielectrics for silicon, which was closely coordinated with the research lab in Dallas, where the main activity was. I was in Japan for over three years, and soon after I returned to the US, there was a distinct change in philosophy at TI and also at many other industrial organizations. There was more emphasis on design, rather than fundamental development. After I returned to the US, the lab in Japan continued to do research, but more directed to design.

DB: What happened to the technologies that were developed by TI? Were they spun off?
DS: Most of the military-related business was sold to Raytheon. Then most of the III-V microwave component ended up at Triquint, here in the Dallas area. Many of the materials and device people are there now. Another spin-off was Yung Chung Kao's MBE group, which re-formed as IntelliEPI. And the IR sensor company, DRS, was formed when a whole group spun off from TI but remained in the same building they had when TI got out of the business.

DB: Looking forward, do you think there are still opportunities for young people coming into the field of crystal growth?
DS: I think the answer is a clear yes, but there has been a change of emphasis that I perceive with the development of nanotechnology. Maybe I'm just an eternal optimist, but I see some of the new developments as very exciting. There certainly are many opportunities in the area of crystal growth. Of course the decline of the major research labs, like Bell Labs and RCA, has had an impact on the US position.

DB: Could there ever be a substitute for those big laboratories?
DS: I could certainly see advantages in having a national research center, as a means of generating a supply of crystals for smaller organizations that do not have the capabilities to produce them. But on the other hand, the success of our III-V program at TI was really made possible because of the day-to-day interaction between crystal growers and device developers. We were in the same building where we could interact with each other. People from Device Fabrication could come and talk to us about problems with the crystals. There are tremendous advantages to being vertically integrated. And the interactions go beyond the walls of any one company. If I could think about one event that was important to my career, it was the Gordon Research conference.

I remember attending Gordon conferences on electronic materials and on crystal growth early in my career. Many of the people I had lasting relationships with in the field I met at Gordon Conferences. I can't think of a better forum for stimulating discussions, and discovering new scientific trends, and forming collaborations.

1 IOP Conference series #7 p 50, Proceedings of the 2nd International symposium on GaAs 1968

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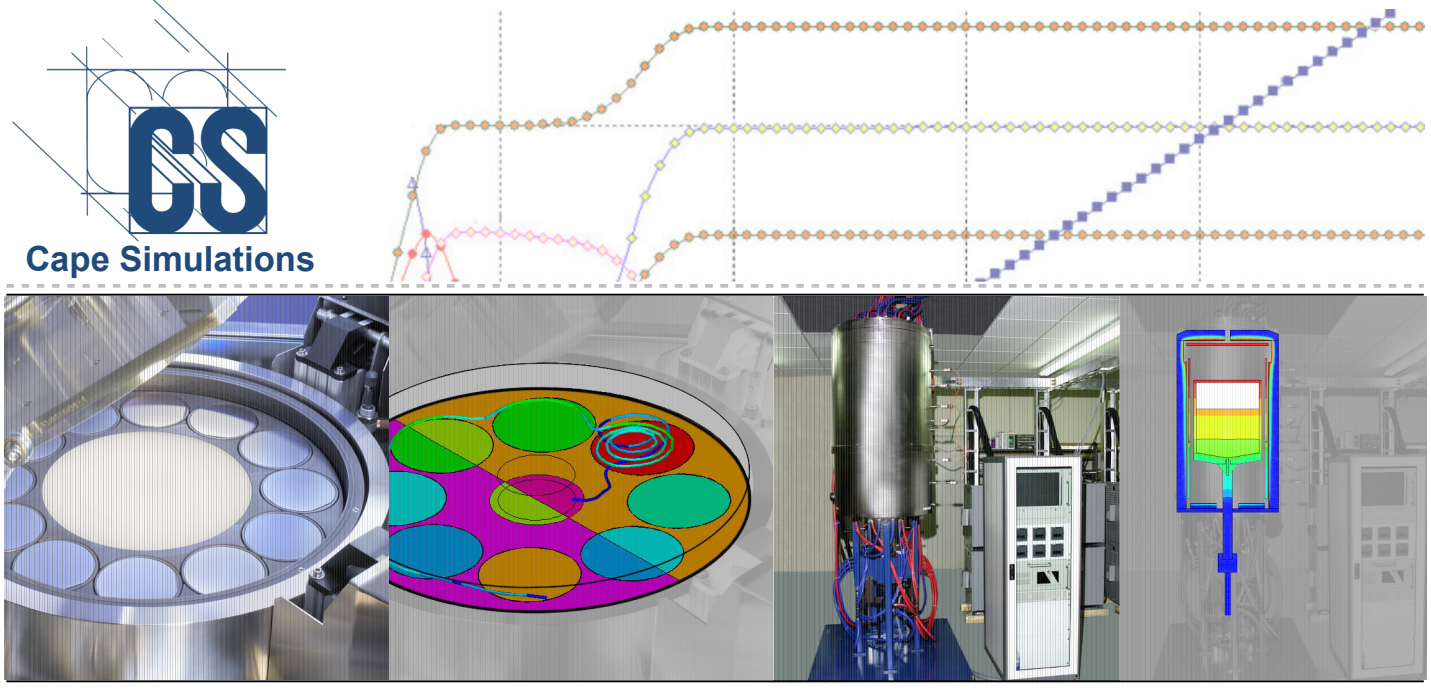

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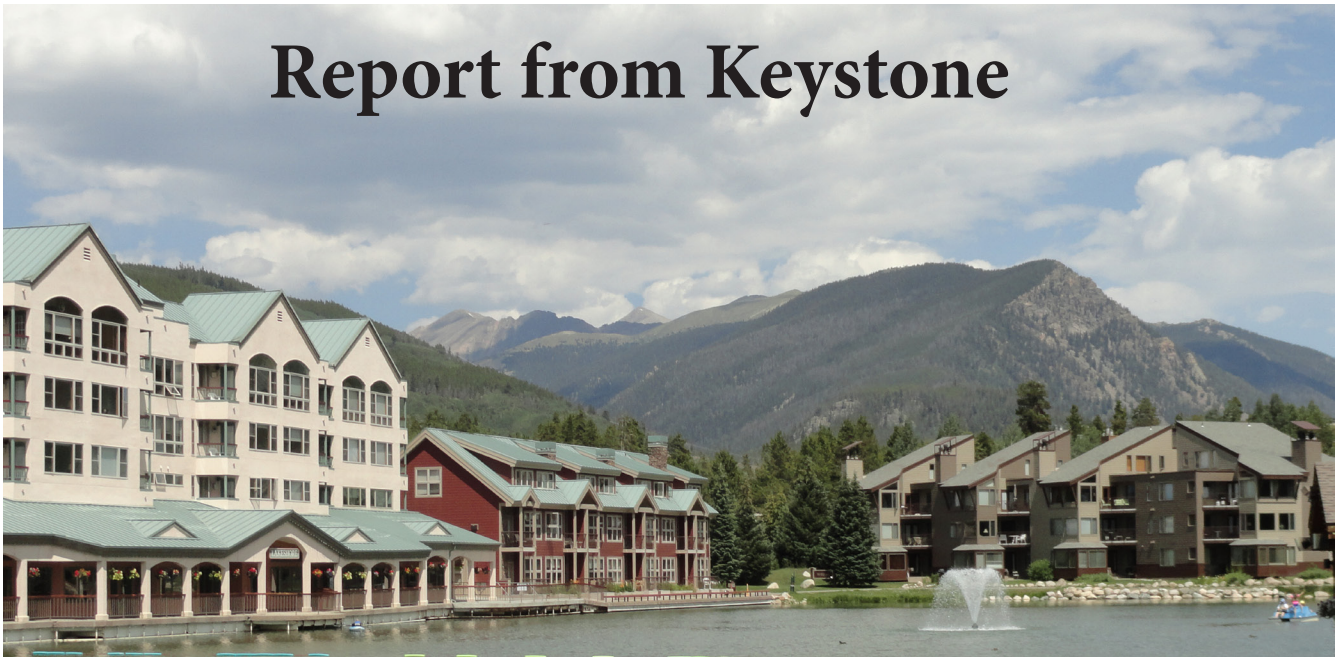
# Report from Keystone



The "Nineteenth American Conference on Crystal Growth and Epitaxy" was held jointly with the "Sixteenth US Biennial Workshop on Organometallic Vapor Phase Epitaxy" in Keystone, Colorado, from July 21-26, 2013. The conference was co-chaired by David Bliss (independent consultant), Russ Dupuis (Georgia Institute of Technology), and Chris Wang (MIT Lincoln Laboratory). It was an exciting week in which crystal growth experts from all parts of the world engaged together in lively discussions and social events.

The conference began with two plenary lectures on current commercial conditions in crystal growth. Dr. Ted Ciszek spoke on "Photovoltaic materials and crystal growth as influenced by changing market dynamics", and Dr. Mike Krames from Soraa, Inc., spoke on "Solid-state lighting employing gallium nitride substrates". On Tuesday morning the two winners of the AACG Award and the Young Author Award gave talks at a plenary session. Jim De Yoreo, the AACG Award winner, spoke on "Physical insights into nature's way of making materials" and Young Author Award winner Colin McMillen spoke on "Hydrothermal solutions: unique approaches to challenging crystal growth problems". The social highlight of the week was a Wild West Barn Barbeque banquet at the Keystone Stables, complete with "steer roping" instructions on-site. In addition to the oral talks described here, there were 43 posters presented at an eventful session on Tuesday afternoon.

The joint forum had a twofold goal: to provide a broad picture of recent developments and accomplishments in the field, as well as to provide a venue for newcomers to the field to familiarize themselves with crystal growth science and technology. Topical sessions covered specific aspects of experimental and industrial growth processes, as well as fundamental theory, characterization and applications.

The conference brought together 325 participants from academia, industry and national laboratories from 18 countries. After a thorough review process, the program committee selected over 300 papers to be presented at the conference, of which 4 were plenary papers, 47 were invited papers, 250 contributed papers and 11 late-news papers. At the conference 190 oral papers were presented in three parallel sessions and 43 papers were presented during a poster session.

OMVPE Workshop - Robert Biefeld, Sandia National Laboratories

The Workshop was co-chaired by Professor R. D. Dupuis, Georgia Institute of Technology, and C. A. Wang, MIT Lincoln Laboratory and the program co-chairs were A. A. Allerman and R. M. Biefeld both from Sandia National Laboratories. The Workshop consisted of three separate sessions and five joint sessions with ACCGE. The most popular topics in these eight sessions were nitride and photovoltaic materials presentations. Nitrides were the focus of 14 out of the 53 talks and 12 were on photovoltaic materials. There was also renewed interest in narrow gap materials and devices, with 8 talks on these materials.

Two-Dimensional Materials Symposium - D. Kurt Gaskill, U.S. Naval Research Laboratory, Washington, DC

The first 2-Dimensional Materials Symposium was held in conjunction with ACCGE-19 at Keystone, CO this past July. Graphene, a single layer of carbon atoms bonded in a planar hexagonal configuration, is a well-known example of a 2D material and a subject of intense world-wide research over the last 9 years. Recently, research into other 2D materials is gaining international interest since ultra-thin two-dimensional layered materials offer the potential for materials properties that far exceed those of their bulk-like crystal counterparts. The goal of the symposium was to bring together leading researchers actively investigating these materials to identify breakthroughs as well as issues that may inhibit further development. The symposium consisted of 24 presentations, 10 of which were invited, on the synthesis, properties, and applications of 2D materials. Presentations focused on graphene, boron nitride, the metal chalcogenide materials $MoS_2$ and $WS_2$, silicene (a two-dimensional form of silicon), and $Y_2O_3$.

The meeting had many highlights – here I discuss two presentations that were of great interest to the audience. Jeong Moon of HRL presented recent work on graphene field effect transistors (FET) and presented measurements of properties that may lead to disruptive technological applications. For example, graphene frequency mixers have performance superior to the current state-of-the-art (dominated by III-V semiconductors). In addition, the performance of high dynamic range radiometers was encouraging as the dynamic linear range of graphene is 60 dB whereas the range for Si is 20 dB and GaAs is 35 dB. Lastly, he described encouraging results, which may herald potential logic applications, where a graphene FET-structure was exposed to fluorine and enabled a bias to control the current with resulting on/off ratio of about 100,000 in a normally off configuration.

Jim Hone of Columbia presented recent work on synthesizing $MoS_2$ using a CVD-like process. Here, the synthesis process uses the transport of $MoO_3$ and S to a $SiO_2$/Si substrate held at 650°C in an $N_2$ carrier gas. Synthesis conditions could be tuned to yield up to 100 micron triangular deposits

that can be 1 or 2 monolayers (or thicker). Although growth was not well-controlled in thickness uniformity, samples still demonstrated that the band gap goes direct to indirect as the thickness changes from 1 ML to 2 ML. The mobility was shown to be greatly dependent upon encapsulation with $HfO_2$ for reasons not well understood and this effect can explain recent publications by others on gated properties. Ohmic contacts are still an issue for devices.

To foster interaction, an informal cook-out and discussion took place the evening of the first day of the symposium at the apartment of Dr. Vanya Darakchieva from University of Linköping. This led to wonderful discussions of the first day's presentations and research by the participants over a meal of grilled foods that continued for several hours. It is our hope that the positive interactions fostered by this first Symposium will be continued onto future symposia!

Bulk Crystal Growth - Aleksandar Ostrogorski, Illinois Institute of Technology

Six sessions covered a wide range of topics from Kyropoulos growth of sapphire to novel relaxor piezoelectric crystals. Invited talks were given by Roberto Fornari from the Leibniz Institute and Jan Seebeck from Fraunhofer. Fornari spoke on High-quality $In_2O_3$ and β-$Ga_2O_3$ grown from the melt, and Seebeck talked about numerical simulation of melt flows during solidification using magnetic fields induced by AC operated heaters. There were several papers modeling thermal flow and interface shape during bulk growth under a variety of growth conditions, since the modeling symposium was folded into the other sessions of the conference. Several papers focused on defect control during Bridgman or VGF growth of GaAs.

Correllated Electron Crystals Symposium - Athena Safa-Sefat, Oak Ridge National Laboratory

The Correlated Electron Crystals Symposium of 2013 had a focus on superconducting materials in film or bulk single crystal forms. This symposium brought together several of the leading materials researchers who are actively investigating superconducting materials, in order to describe synthesis breakthroughs to improve the properties of known superconductors and to further the understanding of their fundamental behavior.

Single-crystal growth techniques such as optical float-zone technique specifically in relation to the old cuprates, and flux-growth and Bridgman techniques for the synthesis of iron-based superconductors (FeSC) were discussed. The film deposition of FeSC, using the deposition techniques such as pulsed laser deposition and molecular beam epitaxy of the selenides, was discussed. The topics of invited talks included the synthesis, bulk structure and properties, and local and surface structures of new or improved superconducting materials such as Pr-doped $CaFe_2As_2$, Ti-based oxides of $BaTi_2Pn_2O$ (Pn = P, As, Sb), Co-doped $BaFe_2As_2$, and K-doped $BaFe_2As_2$. A few related topics of iridates and topological superconductors were also reviewed. The invited speakers included Geetha Balakrishnan, Arnold Guloy, Janusz Karpinski, Minghu Pan, Weidong Si, Claudia Cantoni, Kris Gofryk, Bayram Saparov, Jiaqiang Yan, and Lili Wang.

Fundamentals of Crystal Growth - Simon Brandon, Technion Institute, Israel

A total of 4 invited talks, 15 contributed oral papers and 7 posters were accepted for presentation in this symposium. The oral contributions were arranged in 4 separate sessions, 3 on Monday and 1 on Tuesday. The first session on Monday morning was opened by E. Heike from the University of

Bayreuth in Germany who gave an invited talk in which she demonstrated the efficiency of her combined kinetic Monte Carlo and phase field method when modeling epitaxial growth. Additional presentations in this session focused on defects, dopants and stress in silicon where, for example, G. Samanta from SunEdison discussed his efficient computational approach to the calculation of microdefects during the Czochralski growth of silicon. The following session began with an invited contribution presented by T. Sinno from the University of Pennsylvania who described his recent work on modeling colloidal systems. He based his analyses of phase behavior and assembly of particles into binary colloidal superlatices on actual well characterized systems involving micro and nano scale spheres. Attractive interactions were created between these spheres by grafting single-stranded DNA oligomer brushes on their surfaces.

The last Monday session was initiated by M.E. Glicksman who gave an invited talk in which he described an intriguing model involving surface heat conduction along curved (and therefore non-isothermal) melt/crystal interfaces. Specifically, he presented evidence that this phenomenon is associated with dendritic branching. Another interesting contribution in this session was that of L. Zepeda-Ruiz from Lawrence Livermore National Laboratory (LLNL) who described his work on molecular dynamics modeling of hydrogen isotope crystallization, relevant to the improvement of material used in ignition experiments on the National Ignition Facility at LLNL. The final session of this symposium, that took place on Tuesday morning, included the invited contribution of R. Madhav from the Indian Institute of Technology in Kanpur who presented his work on modeling the impact of impurities on step-flow in solution growth systems.

III-V Nitride and Other Wide Bandgap Semiconductors - Andrew Allerman (Sandia National Laboratories) and Dirk Ehrentraut (Soraa, Inc.)

The symposium on III-V Nitride and Other Wide Bandgap Semiconductors was composed of two sessions. The first session had 6 talks scheduled of which the last was cancelled. Russell Dupuis (Georgia Tech) opened this session reporting some results on light emission from InGaN HBTs. Here the light is emitted from the base emitter and base collector junction at wavelengths stretching from the

UV well into blue light. Dupuis gave a second paper in this session where UV lasing has been demonstrated for a MQW structure homoepitaxially grown on an AlN substrate from Hexatech (NC). The lowest emission threshold was 221 kW/cm$^2$ at 245.3 nm. Yamamoto et al. gave some insights into their work on in situ X-ray diffraction (XRD) monitoring of OMVPE during the growth of InGaN/GaN superlattices. Using a similar experimental approach for in situ XRD, Highland showed results from their work on controlling growth modes on m-plane and c-plane single crystal thick films.

The second session was opened with the invited talk on the scalable, high-rate ammonothermal growth of bulk GaN by D. Ehrentraut and M.P. D'Evelyn (Soraa, inc.). Growth of two inch crystals and high growth rates up to 40 µm/h has been demonstrated. M. Bickermann et al. (IKZ Berlin) presented current topics on the sublimation growth of bulk AlN. He informed that a 65 million euro EU-program on deep UV devices ("Advanced UV for Life") is about to start next year (2014-2020). Next was C.-A. Liu (Taiwan) with the growth

and characterization of nonpolar InN on LiGaO2 by halide vapor phase epitaxy. Since InN decomposes in H2 at 350 °C the move to N2 as carrier gas was made. Best films have been grown with the III/V ratio = 110 at 700 – 720 °C in the growth zone at rates up to 1.5 μm/h. Finishing the session was Ohno (Tohoku University) with his talk on slip systems in wurtzite ZnO single crystals at elevated temperatures.

Photovoltaic Materials - John Geisz, National Renewable Energy Laboratory

The Materials for Photovoltaics and Energy Technology Symposium highlighted the state of silicon markets with a plenary by Ted Ciszek and an invited talk by Joel Kearns from Sun Edison (formerly MEMC) on continuous Czochralski growth. Other talks focused on understanding challenging aspects of silicon and III-V crystal growth for photovoltaic applications.

Nonlinear Optic and Laser Host Materials Symposium: Kevin Zawilski, BAE Systems

The session on non-linear optic and laser host materials was chaired by Kevin Zawilski of BAE Systems and Dieter Jundt of Gooch and Housego and ran for 4 sessions during the first two days of the conference. Invited talks were given by Peter Schunemann from BAE Systems who talked about non-linear optical materials for mid-IR frequency conversion, Joe Kolis from Clemson University who talked about multifunctional materials grown via hydrothermal growth as solid state laser hosts, and Romain Gaume from the University of Central Florida who talked about Laser Induced Breakdown Spectroscopy (LIBS) and optical ceramics as laser hosts.

Despite several talks being cancelled due to sequestration-related travel restrictions, the sessions were well-attended and featured many high-quality talks on a wide variety of growth techniques and materials systems. Apart from the invited speakers, topics ranged from quasi-phase-matched materials grown through vapor epitaxy, to large LBO crystals grown through top-seeded solution growth, to crystal fibers grown through laser heated pedestal growth, and to new materials such as $BaTeMo_2O_9$.

*Photos courtesy of Merry Koschan*

# Executive Committee Update

We are pleased to announce the results of the Executive Committee elections of the American Association for Crystal Growth. The Executive Committee for 2013-2015 will be made up of the following individuals. The starred names are the newly elected/re-elected members.

*Banish, Gordon
Biefeld, Robert (Vice President)
Boatner, Lynn A.
*Bourret, Edith
Cederberg, Jeff
*Dabkowski, Antoni
Derby, Jeffrey J.
DeYoreo, Jim
*Dhanaraj, Govindhan
Feigelson, Robert S.
Fratello, Vincent J.
*Geisz, John
Higgins, Bill
Jackson, Kenneth A.
*Kuech, Thomas F.
Licht, Steve
Lynch, Candace
*Mnushkina, Irina
Motakef, Shari
*Orme, Christine
Ostrogorsky, Aleks
Redwing, Joan (Secretary)
Roshko, Alexana
Schunemann, Peter (President)
Skowronski, Marek
Vekilov, Peter
Vanderwater, David (Treasurer)
*Wang, Christine A.
Watkins, Simon
*Yeckel, Andrew
*Zawilski, Kevin
*Zepeda-Ruiz, Luis

We would like to express our strong thanks to the outgoing ExecComm Members, Dave Bliss, Darrell Schlom, Rose Scripa, and Bob Sekerka, who are all long-term volunteers. We would also like to thank those who were not elected this round for their willingness to serve. Be assured that the organization will find a place for their talents. The AACG would not exist without the extensive activities of our volunteers and the support of their employers and families. The service of these people is truly the heart and soul of the organization.

The Nominations and Elections Committee
Vince Fratello
Jeff Derby
Andrew Yeckel

# Membership Information

**Individual member benefits include:**

- Annual AACG Newsletter
- Annual roster of current members with contact information
- Important conference announcements
- Discounted conference registration
- Discounted membership rates for retired members

**Company membership benefits include:**

- All of the individual membership benefits
- Listing as a Corporate Affiliate on the website and in the newsletter
- Use of the AACG roster for business-mailing purposes

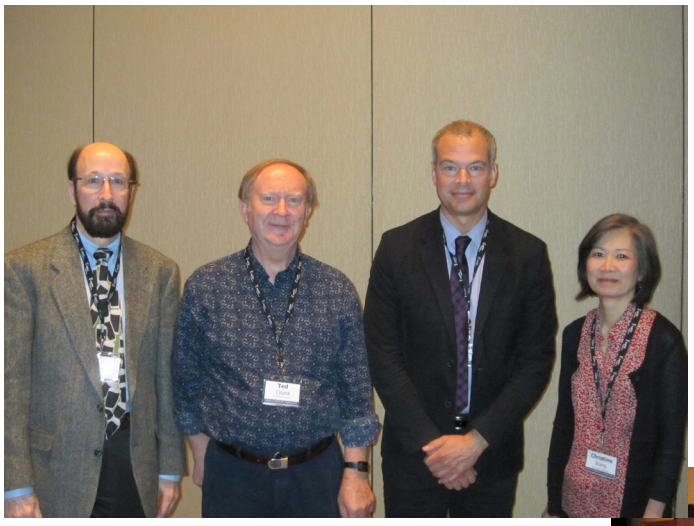
The AACG has approximately 600 members. The largest fraction is from the United States, but many foreign countries are represented. Our membership includes engineers, scientists, educators, technologists, marketing representatives, and students. All share a strong interest in one or more facets in the field of crystal growth and epitaxy.

Membership in the AACG is open to everyone with a professional interest in crystals, thin films, crystal growth, epitaxy, and characterization. This includes theoretical and experimental work on the growth and properties of single crystals in bulk, film, or particulate form, crystallization phenomena, crystal/thin film production, and related applications.

**Email aacg@comcast.net to request an online membership invoice or visit www.crystalgrowth.org for more information.**

# Photos from Keystone

**Photos courtesy of Russ Dupuis**



# Induction Heating Power Supplies

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# Inaugural meeting of the southeast section of the American Association for Crystal Growth

## Contributed by Merry Koschan

Enthusiastic crystal growers launch new section!

East Tennessee is one of the most active areas in the U.S. for crystal growth; this will be a great opportunity for technical and social exchange.

On November 7, 34 people gathered at the University of Tennessee for the first meeting of the new AACG southeast section. The attendees included those from industry, academia, and a national lab; the mix ranged from long-time professionals to students, and interests ranged from crystal growth to characterization to crystal processing and device fabrication.

The meeting was sponsored in part by the UT Office of Research via funding for the Tennessee Crystal Synthesis Initiative Organized Research Unit, which is co-directed by Dr. David Mandrus and Dr. Veerle Keppens.

The program started with a buffet dinner, followed by a presentation by Merry Koschan outlining a brief history of AACG and our local section; current plans are for three meetings per year, in the fall, winter and spring. The technical part of the meeting was a tour of the Scintillation Materials Research Laboratory (SMRC) at the University of Tennessee.

The SMRC is dedicated to the development and discovery of new scintillation materials. It was originally founded by an agreement between UTK and Siemens Medical Solutions in 2005, and has since had research projects with a wide range of entities, from Siemens to the Department of Energy, the Department of Homeland Security, the National Science Foundation, RMD, and Nucsafe. The research center currently has four large laboratories: two dedicated to the Bridgman growth of hygroscopic metal halides, one focused on high temperature oxide growth, and the fourth is fully equipped for characterization of scintillation and optical properties.

Please visit the AACG website at www.crystalgrowth.org for conference updates, career opportunities, and a new members' only portal.

# Crystal Growth Award - James De Yoreo

The American Association for Crystal Growth Award was established to recognize an individual's outstanding contributions to the field of crystal growth and epitaxy. The first award was given to Sir Charles Frank in 1978. The award is presented at the national meeting. The 2013 AACG Crystal Growth Award was presented to Dr. James DeYoreo, currently Chief Scientist, Pacific Northwest National Laboratory, for his extraordinary contributions in elucidating mechanisms of nucleation, self-assembly, and crystal growth of biomaterials. His pioneering work in direct visualization of crystal growth using in situ atomic force microscopy and in situ transmission electron microscopy has allowed quantification of the crystal growth process at the nanoscale and provided unique insights in the kinetic processes underlying biomineralization and bimolecular assembly.

Jim De Yoreo is the Chief Scientist for Materials Synthesis and Simulation Across Scales at Pacific Northwest Laboratory. He received his Ph.D. in Physics from Cornell University in 1985. Following post-doctoral work at Princeton University, he became a member of the technical staff at Lawrence Livermore National Laboratory (LLNL) in 1989, where he held numerous positions including Group Leader for Biophysical and Interfacial Sciences, Director of the Biosecurity and Nanosciences Laboratory, and Deputy Director of the Laboratory Science and Technology Office. He joined Lawrence Berkeley National Laboratory in 2007 where he served as Deputy Director and Interim Director of the Molecular Foundry, as well as Deputy Director of the Center for Nanoscale Control of Geologic $CO_2$.

DeYoreo is a member of the MRS, APS, ACS, and AACG, where he sits on the Executive Committee. He is Associate Editor in Chief for Frontiers of Materials Science and Chairs the Advisory Board for "Hard Soft Interfaces: From Understanding to Engineering" a UK Engineering and Physical Sciences Research Council Consortium. He has served as President of the Materials Research Society (MRS), on the MRS Board of Directors, and on committees for the National Academy of Sciences, the Department of Energy, and Congress.

De Yoreo's research has spanned a wide range of materials-related disciplines. Most recently, he has focused on in situ AFM and TEM investigations of interactions, assembly, and crystallization in biomolecular and biomineral systems. De Yoreo has authored, co-authored, or edited over 175 publications and patents. He is a recipient of the Laudise Prize of the International Organization for Crystal Growth, an R&D 100 Award, and the LLNL Science and Technology Award, and is a Fellow of the American Physical Society.

# Young Author Award - Colin McMillen

The American Association for Crystal Growth Young Author Award is presented to young persons (under 35 years of age) who have shown outstanding achievement in the field of crystal growth and epitaxy, primarily through published papers. The 2013 award was given to Dr. Colin McMillen of Clemson University.

This 2013 award recognizes his accomplishments in the bulk and epitaxial growth of oxide materials. His work has spanned the growth of new lasing materials, the hydrothermal growth of the deep-UV non-linear optical crystals, and a variety of multifunctional crystals.

Dr. McMillen received a Bachelor of Science degree in Chemistry from Clemson University in 2002, and subsequently earned his Ph.D. in Chemistry from Clemson University in 2007, studying inorganic chemistry in the laboratory of Prof. Joe Kolis. As a research associate with the Center for Optical Materials Science and Engineering Technologies (COMSET), his experience in descriptive inorganic chemistry and hydrothermal crystal growth spanned the range of deep-UV NLO materials, oxide laser hosts and multifunctional single crystals.

In addition to his crystal growth interests, Colin presently serves as crystallographer and Director of the Molecular Structure Center in the Department of Chemistry at Clemson.

# Experiences of a Young Researcher in Hydrothermal Crystal Growth

**Colin D. McMillen, Clemson University**

**2013 AACG Young Author Award lecture, presented at ACCGE-19 in Keystone, CO**

You can always identify someone who has experimented in the field of hydrothermal crystal growth by the crystals they keep on their office windowsills – usually displaying several quartz points and at least one faceted and pleasantly dappled synthetic quartz boule. Such was the case when, as an undergraduate, I wandered into a faculty office to discuss my new research project entitled "Chemistry Under Unusual Reaction Conditions" (not to be interpreted as "Chemistry for Unusual Researchers").[1] In the years following, I have learned that the "unusual" reaction conditions afforded by hydrothermal fluids can be applied to many crystal growth approaches (synthesis, bulk growth, epitaxial growth) for a wide variety of chemical systems. This piece briefly shares some of the author's personal experiences in extending the hydrothermal technique beyond the quartz boules that sit on our windowsills.

Of course, the mineralogists who pioneered crystal growth from hydrothermal fluids already discovered that the technique was not to be confined to a singular system or approach! Researchers including Schafhautle, Bunsen, Friedel (Charles and Georges), von Chroustschoff, Gorgeu, Doelter, Spezia, Baur and many others prepared crystals of nearly one hundred minerals from 1845-1912.[2] Their progress using rudimentary equipment such as unshielded glass tubing, welded gun barrels and simple steel containers was an impressive feat, and this early work still serves as inspiration for synthetic surveys. The next fifty years of hydrothermal research would benefit from great technological advances made by several crystal growth giants. Names such as Morey, Bridgman, Tuttle, Roy and Laudise would develop the autoclave technology to safely contain fluids at much higher temperatures and pressures, while also advancing experimental design to enable new approaches to synthesis as well as bulk crystal growth.[3-5] Much of the work in hydrothermal crystal growth since then is clearly traceable to these roots.[6-10]

Reviews by Laudise[11] and Rabenau[12] were my first reading assignments in the field, as anyone new to the technique will first learn about autoclave sealing mechanisms, precious metal liners and using mineralizers to dissolve reactants and crystallize products. Thus began my initial foray into the phase space of borate- and beryllate-based crystals, with a goal of synthesizing anhydrous, noncentrosymmetric (NCS) crystals for use as deep-UV non-linear optical (NLO) materials. A hydrothermal approach here would be interesting given the complex melting behavior of borates (often viscous, melting incongruently or forming glasses). A number of novel compounds were prepared, making for interesting studies in crystallography, but none quite fit what was ideal for a NLO material. Many of the NCS crystals possessed OH-groups which limit thermal stability and play havoc with thermo-optic coefficients.[13] Those that were anhydrous were either centrosymmetric (for example $LiBeSr_2B_3O_8$), had poor morphology ($Sr_2Be_2B_2O_7$) or were not phase-matchable ($\gamma$-$LiBO_2$), despite having many other encouraging properties.[14-15] However, concurrent projects in the hydrothermal growth of fluorides provided some hope, as the metal-fluoride bonds appeared to resist hydrolysis when grown from aqueous alkali fluoride mineralizers.[16-17] The logical consequence of this was that OH-containing crystals could instead be prepared as fluoride analogs ($Sr_3B_6O_{11}(OH)_2$ vs. $Sr_3B_6O_{11}F_2$, for example).[18-19] Indeed, these borate fluorides are new, potentially interesting NLO materials.

Likewise, NCS fluoroberyllium borates such as $KBe_2BO_3F_2$ (KBBF), $RbBe_2BO_3F_2$ (RBBF) and $CsBe_2BO_3F_2$ (CBBF) were also amenable to hydrothermal synthesis.[20] These crystals in fact formed as moderately-sized (up to 5 mm) crystals simply by spontaneous nucleation. This immediately enabled seeded bulk growth reactions. Steady progress in determining the solubility behavior of these compounds has led to the optimization of the growth conditions and the transition from laboratory to commercial growth scales. Optical quality KBBF and RBBF crystals up to 1.5 cm in diameter can now be grown hydrothermally (Fig. 1). [21] The synthetic approaches used to crystallize these borates and borate fluorides, as well as mixed oxyanion species such as the beryllium borates have been extended to phosphates, silicates, carbonates, vanadates and combinations thereof.[22] Be warned of the temptation to easily "design" hydrothermal reactions involving more and more components, but be encouraged that the resulting crystallography will also be a lot of fun.

\

Figure 1: Hydrothermally-grown RBBF crystal (Advanced Photonic Crystals, Fort Mill, SC).

But what if complexity and new materials synthesis are not the goals? Could the hydrothermal method be useful for compounds with simple compositions that just resist crystallization by other means? These questions led to the study of rare earth oxides such as $Sc_2O_3$ and $Lu_2O_3$, which are promising laser hosts for high average power lasers due to their high thermal conductivities, robust nature and desirable optics. Of course, when a material resists crystallization (these materials melt above 2450 °C) that means there are no convenient seed crystals from which to start. With suitably basic mineralizers (20 M KOH(!)) small (0.5 mm) crystals were obtained hydrothermally at 650 °C, a relatively low temperature compared to the melting point. Considerable effort was spent "bootstrapping" the nucleation reactions to obtain seeds that could be tied onto a ladder for growth. In the case of lutetia, only one 2 $mm^3$ "seed" crystal was ever obtained (scandia proceeded more easily[23]).

By some combination of good design and divine fortune, the first hydrothermal $Lu_2O_3$ growth experiment turned the 2 $mm^3$ seed into a 4 $mm^3$ crystal, which could then be cut into more seeds. Each growth run brought incremental progress to 6 and 8 mm crystals, and generated more seeds. Experiments could then be devoted to optimizing clarity, improving the growth rate and doping with laser ions of interest such as $Yb^{3+}$ and $Er^{3+}$.[24] While it took a few years to traverse from 0.2 mm to 2 mm to larger crystals, and a few more years to develop some interesting spectroscopy,[25] being able to trace each hydrothermal $Lu_2O_3$ crystal back to the single 2 mm cube provides its own satisfaction. Work here is ongoing to scale to larger crystals.

Of course, not all applications call for bulk three dimensional crystals (thin disks, waveguides, microlasers and multifunctional crystals, for example). In such cases where crystalline layers of ~50 microns to a few millimeters are needed, the use of hydrothermal methods as an epitaxial growth technique, although less common, can be a unique but suitable approach. Slower growth rates associated with hydrothermal growth can even be a benefit in these cases by leading to enhanced control at the growth interface with the substrate. Where previous studies focused on synthesis of new materials and growth of difficult-to-grow crystals, this study sought to apply fresh growth perspectives to a well-known material, YAG. YAG was particularly appealing because of its variety of coordination sites, its ability to support rare earth and transition metal dopants and the ready availability of substrate crystals (a value truly appreciated after self-generating $Lu_2O_3$ seed crystals). Such a system seems ideal for creative, customized crystal growth (Fig. 2).

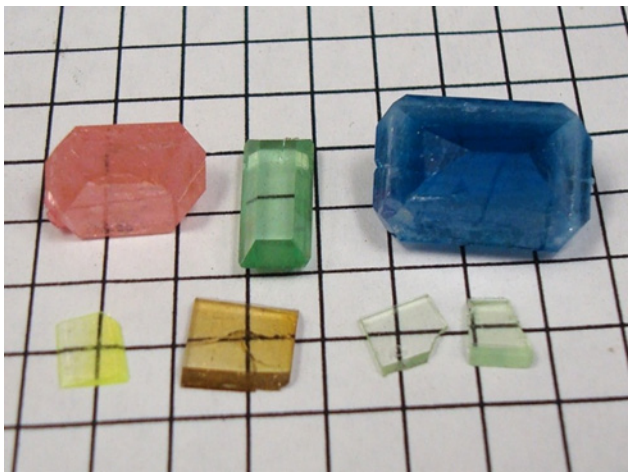

Figure 2: Hydrothermally-grown garnets.

Learning to do hydrothermal growth above 600 °C during the study of the rare earth oxides has proven quite useful here, as the aluminate garnets seem to grow best using a thermal gradient of 620-650 °C with 2 M $K_2CO_3$ as a mineralizer, resulting in layers up to 3 mm in thickness.[26] Simple doping of trivalent rare earth and transition metal ions was studied, followed by more complex schemes utilizing charge-compensating ions in the lattice to stabilize ions such as $Cr^{4+}$ and $Co^{2+}$, which are known to Q-switch 1.0 and 1.5 micron lasers. Crystals were then grown with dopants in multiple layers using consecutive growth runs. Of course, the new growth entirely encompasses the substrate, but with some fabrication between growth runs a variety of single crystals with multiple zones of functionality can be prepared. Alternatively, several dopants can also be co-doped into a single

layer during a single growth experiment. Preliminary results suggest there may be significant advantages to the "self-Q-switching" enabled by crystals such as Ca,Cr,Nd:YAG and Ca,Cr,Yb:YAG (Fig. 3) grown in this way.

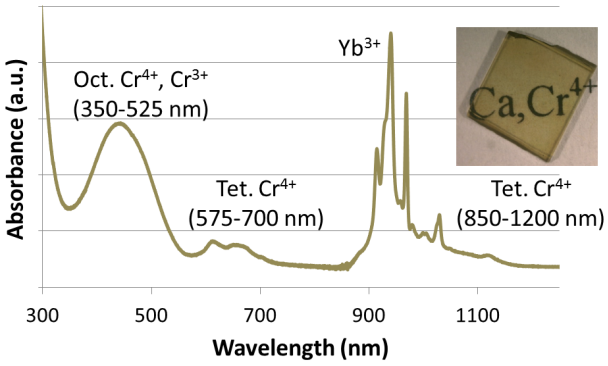


Figure 3: Absorption spectrum of Ca,Cr,Yb:YAG for self-Q-switching applications.

Using a hydrothermal heteroepitaxial approach, crystals of LuAG were similarly grown on YAG substrates.[27] This has enabled further homoepitaxial growth of LuAG, and direct extension of many of the doping schemes explored so far for YAG. LuAG may hold further promise by maintaining higher thermal conductivities upon doping, or as a denser host material for scintillator ions in gamma ray detection. It also opens the door for additional possibilities in waveguide and anti-waveguide crystals (Fig. 4). Indeed, classic materials like the garnets continue to provide new inspiration for crystals growers.

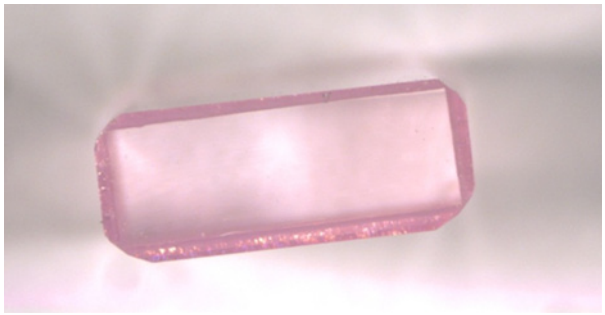

Figure 4: As-grown Er:LuAG on YAG – a precursor to the hydrothermal growth of waveguides.

As a crystal grower and a crystallographer I continue to enjoy both great challenges and rewarding experiences doing "Chemistry Under Unusual Reaction Conditions." Much work remains to build upon these humble contributions. But there are certainly plenty of new systems and new approaches to explore within this classic crystal growth technique to try to fill the spot next to the quartz crystal on our windowsills.

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# Position Openings

**CRYSTAL GROWTH PROCESS ENGINEER**

Integrated Photonics, Inc. (IPI) is a small, private company, located in suburban Hillsborough, NJ. IPI specializes in the development of oxide crystals primarily for use in fiber optic telecommunication components. We ship materials worldwide.

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- monitoring yield and other process parameters for continuous process development and improvement
- documenting crystal growth processes and training process operators

Candidates should have an MS (preferred) or BS in Materials Science and Engineering, Chemical Engineering, Physics, or related science degree, with at least five years of hands-on experience in process development in a crystal growth production environment. Working knowledge of mechanics and electronics is highly desirable. Excellent computer skills and familiarity with using data analysis to drive process improvements is required. This position requires the candidate to be hands-on as well as flexible in taking on multiple roles in a small company.

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Job Responsibilities:
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• Optimize precursor preparation process as needed
• Develop new crystal composition with improved properties such as increased thermal and electrical stability, enhanced mechanical quality factor and key dielectric and piezoelectric properties

Mike Dickinson
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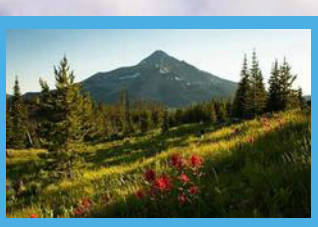
# ACCGE-20/OMVPE-17
# August 2-7, 2015



## CONFERENCE ANNOUNCEMENT:

**The 20th American Conference on Crystal Growth and Epitaxy (ACCGE-20)**

*held jointly with*

**The 17th U.S. Biennial Workshop on Organometallic Vapor Phase Epitaxy (OMVPE-17)**

*to be held in*

**Big Sky, Montana at Big Sky, Montana Resort**

**Visit www.crystalgrowth.org for more information.**

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June 15 - 19, 2014
Berlin, Germany

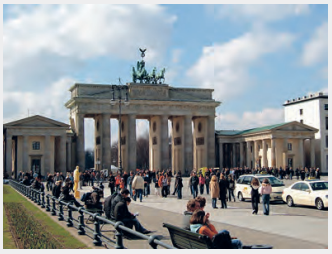
### Important Dates

Registration and abstract submission starts: Oct 15, 2013

Abstract submission deadline: Mar 1, 2014

Abstract acceptance notification: Mar 20, 2014

Early registration ends: Apr 15, 2014



### Location

The workshop will be located at the Novotel Am Tiergarten, located in the inner city of Berlin, the vibrant capital of Germany, with a lot of sightseeing opportunities nearby.

For details visit http://iwcgt-6.ikz-berlin.de.

### Conference Contact

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### Exhibition and Sponsoring Contact

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International Organization for Crystal Growth
http://www.iocg.org/

GERMANY
JUNE 15 - 19

**6th International Workshop on Crystal Growth Technology**

IWCGT-6

1st Announcement

BERLIN 2014

**Topical Sessions**

Advances in bulk crystal growth of semiconductor and photovoltaic materials

Optical and laser crystals

Scintillators, piezo- and magnetoelectrics

Substrates for wide band-gap and oxide semiconductors

Growth control, quality assurance & management of resources

Crystal shaping and layer transfer technologies

Frontiers in crystal growth technology

**Panel Discussion**

Advancing new growth technologies to industrial application

iwcgt-6.ikz-berlin.de

# A way to new crystals

SCIDRE
SCIENTIFIC INSTRUMENTS DRESDEN GMBH

## The high pressure crystal growth furnace by ScIDre

The HKZ offers a broad range of unique properties:

- pressure range from $10^{-3}$ mbar to 150 bar
- several gases like argon, oxygen, nitrogen[+] and other
- high pressure cleaning device for argon
- static atmosphere or gas flow from 0.2 $mlmin^{-1}$ to 1 $lmin^{-1}$

- optical heating with two ellipsoidal mirrors in a vertical alignment
- lamp power 3, 5, 7 kW
- smooth energy adjusting via power shutter
- temperatures up to 3000°C
- patented temperature measurement during crystal growth[+]

- growth speed from 0.1 to 200 $mmh^{-1}$ and fast manipulation for setup
- rotational speed adjustable between 0 and 150 rpm

[+]option

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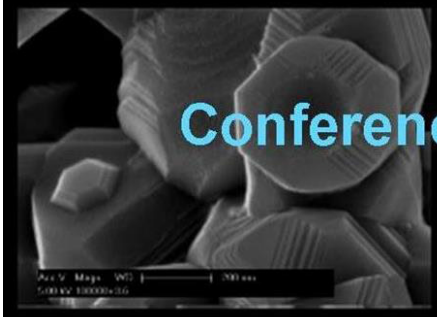
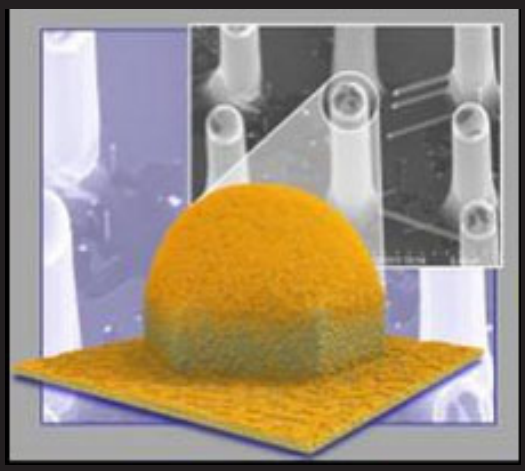


# 24th AACGE Western Section Conference on Crystal Growth & Epitaxy

**Stanford Sierra Camp, Fallen Leaf Lake, CA**

**June 8-11, 2014**



The 24th AACGE Western Section Conference on Crystal Growth and Epitaxy, to be held at the Stanford Sierra Camp, Fallen Leaf Lake, CA, June 8-11, 2014, is situated on the shores of a stunningly beautiful, clear, secluded alpine lake located in a glacially carved valley. The format and the atmosphere of the conference are designed to provide a comfortable and relaxing atmosphere while maximizing interactions amongst specialists and the wider crystal growth community.

The 24th ACCGE West Conference will provide a forum for the presentation and discussion of recent research and development activities in all aspects of epitaxial thin film, bulk and nanocrystal growth; 4 different focused sessions will be held:

- Biocrystallization
- Energy
- Fundamentals
- Modeling

Sessions will discuss experimental and industrial growth processes as well as characterization and applications. The conference will include both oral and poster sessions as well as plenary and invited speakers to provide a broad picture of developments in the field.

Session organizers include:

| | |
|---|---|
| Biocrystallization: | David Kisailus and Andrzej Wierzbicki |
| Energy: | Sean Hearne and Andrew Ichimura |
| Fundamentals: | James DeYoreo and Jeff Rimer |
| Modeling: | Moneesh Upmanyu and Luis Zepeda-Ruiz |

The camp facilities include two to three bedroom condominium style units with private decks and excellent views of Fallen Leaf Lake and the Sierra Nevada Mountains. Meals are provided in a common dining room, making it attractive for entire families to attend. The conference schedule generally includes formal sessions in the mornings and evenings, leaving the afternoons free to explore the adjacent wilderness area on foot, relax by the lake, or take the trip into nearby South Lake Tahoe to experience the casinos. Recreational facilities at the camp include: basketball, volleyball, tennis courts, horseshoes, beach access, and boats are available. There is no charge for the use of sailboats, canoes, rowboats or kayaks.

For more information, to submit an abstract, or to register, please visit www.crystalgrowth.org