Outreach and Highlights of Research for Non-technical Audiences
New Routes to Superstretchy Ceramics
Martha Mecartney, PI        DMR Grant 0207197

Usually we think that ceramics are brittle and break if we try to bend them. Researchers at the University of California, Irvine in collaboration with Professor Trudy Kriven at the University of Illinois have found a way that ceramics when heated can be deformed or stretched extensively without breaking.

The key is to make the ceramic out of at least three different compositions (phases). By using novel processing techniques, very small grains (crystals) of each composition can be made. These small grains can slide by each other when the material is heated, making it easy for these materials to be stretched over 100%.

At room temperature, however, these materials are very strong and difficult to break. This work has found that superstretchy ceramics can be made out of almost any three different compositions - alumina/zirconia/mullite (aluminosilicate) and alumina/zirconia/nickel spinel are just two examples from this work.
Superplastic Deformation of a Ceramic Material

\[ \dot{\varepsilon}_0 = 1.0 \times 10^{-4} \, \text{s}^{-1} \]

Strain to Fracture: \(~520\%~\)

8 mol\% \(Y_2O_3\) Cubic Stabilized \(ZrO_2\) with 5 wt\% \(SiO_2\) Tested at 1425°C

Martha L. Mecartney, University of California, Irvine
Research sponsored by National Science Foundation under Grant No. DMR - 0207197
Superplastic Ceramic Materials

- Superplastic materials can withstand tensile deformations exceeding 100% elongation.
  - This property is often referred to as superplasticity.

- By dispersing a nanometer scale second phase in yttria stabilized cubic zirconia, this ceramic material was made superplastic.
  - The concept of using a dispersion of two nanoscale phases to enhance superplasticity in ceramics is currently being tested.

- Superplasticity enables superplastic forming (SPF).
  - Using SPF, complex shapes may be fabricated using significantly fewer steps than traditional manufacturing techniques and providing a more economical process.

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Superplastic Deformation of Alumina-based Composites (Alumina-Zirconia-Mullite)

The matrix is alumina and the volume percent of zirconia and mullite are indicated in parentheses in the key.

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Superplasticity in Alumina Composites

- One key requirement for ceramic superplasticity is the retention of a fine grain (crystallite) size during deformation.

- This research investigates the possibility of using $\text{Al}_2\text{O}_3$ dispersed with $\text{ZrO}_2$ and mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) to make superplastic $\text{Al}_2\text{O}_3$.

- With an increase in the amount of the second and third phases, the strain rate becomes more stable, indicating that the tri-phase microstructure helps to limit grain growth.

- Several underrepresented undergraduate students were involved in the research effort and had an opportunity to participate in summer research during 2003. This research experience will help with future engineering employment and enhance their consideration of completing advanced degrees.

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A Comparison of Grain Boundary Widths in Ceramics Determined by Impedance Spectroscopy (IS) and Analytical Electron Microscopy (AEM)

Martha L. Mecartney, University of California, Irvine
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A Comparison of Grain Boundary Widths in Ceramics Determined by Impedance Spectroscopy (IS) and Analytical Electron Microscopy (AEM)

- Segregation at grain boundaries can provide a chemical measure of the width of grain boundaries from compositional scans using analytical high resolution transmission electron microscopy (AEM).
- AEM is difficult, expensive and time consuming.
- An electrical grain boundary width can be determined using impedance spectroscopy (IS), if the grain size of the ceramic is known.
- IS is relatively easy and inexpensive.
- A comparison of the two techniques finds that IS data can be used to predict grain boundary widths, providing a new technique for quickly characterizing the average grain boundary width in a ceramic.

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Metal-like behavior in ceramics is demonstrated above in a highly deformable (superplastic) alumina(A)/zirconia(Z)/mullite(M) fine grain ceramic composite. Dislocations emanating from grain boundaries accommodate grain boundary sliding during high temperature deformation, similar to the phenomenon demonstrated in metals. The existence of a threshold stress in this material has also been documented, additional evidence that high strain rate superplasticity in ceramics may have many parallels to metallic systems. These results may lead to new opportunities to develop novel superplastic ceramics for net shape forming.
The Role of Crystallographic Defects in Ceramic Superplasticity

National Science Foundation Grant 0606063 from the Division of Materials Research
Principle Investigator: Martha L. Mecartney

- NON-TECHNICAL DESCRIPTION: This project studies how defects in materials (primarily misaligned atoms in a line or at a boundary) can be deliberately introduced to make ceramic materials ductile at high temperatures. Potential economic savings are possible for the ceramics industry in the U.S. for use in shape-forming complex oxide ceramic components at high temperatures while retaining excellent room temperature mechanical properties. This research helps train a diverse cohort of graduate and undergraduate students and provides the basis for a new graduate course on Grain Boundaries and Interfaces in Materials that will be introduced into the curriculum.

- TECHNICAL DETAILS: In addition to promoting possible commercial applications of superplastic forming by designing new superplastic ceramics, this research will contribute to understanding the fundamental deformation mechanisms operative in mullite and mullite composites at high temperatures. For the first time the dislocation structure and slip system will be determined for mullite, a material proposed for use in high temperature structural applications due to its excellent thermal stability, high thermal shock resistance, and low thermal conductivity. Students will be trained in the advanced techniques of high resolution atomic force microscopy (AFM) and focused ion beam (FIB) instrumentation to study relative grain boundary sliding and the role of interfacial defects.
Design of Superplastic Ceramics

Fine grain superplastic microstructures allow net-shape forming at high temperatures yet still maintains high strength at room temperature.

Challenge: Design Multi-component Microstructures to Limit Grain Growth but Promote Grain Boundary Sliding

NSF Grant DMR - 0606063
Electron backscattered diffraction on a field emission scanning electron microscope is used to determine the relative orientation of grains in mullite. The color coded images show that the long axis of acicular grains is the c-axis [001], and the sides of the grains are [100], [010] or [110]. This information on how crystals are aligned helps explain the creep resistance of the material at high temperatures.
Nanodimensional Measurements of Superplastic Deformation

PI Martha L. Mecartney, NSF DMR Grant #0606063

This research uses back-scattered electron contrast in the scanning electron microscope (SEM) to identify three different phases in this ceramic composite and atomic force microscopy to measure relative nanometer displacements during superplastic forming. The graph below shows that at the nanoscale, grain boundary sliding is non-uniform in this alumina (A), zirconia (Z) and mullite (M) composite.

Top - SEM of three phase material. Right - AFM image. Below are similar images after 9% strain at 1350°C. The smiling face marks the same grain in all images.
AFM Metrology and Nanoparticles

Navy Contract N00244-05-P-2456
Mecartney Group Research
Solid Oxide Fuel Cell (SOFC) Electrolytes

What role does the chemistry and structure of grain boundaries play in controlling ionic conductivity?

Nanocrystalline Y-CSZ on Si

Lanthanum apatite ceramics

Mecartney Group Research
Could You Put this “Jigsaw Puzzle” Back Together?  Tortuous Grain Boundaries in Mullite
DMR Grant 0207197 has had a strong involvement of underrepresented students in engineering

Joy Trujillo, an undergraduate researcher at the University of California, Irvine, developed new techniques to etch grain boundaries in multiphase materials and a method to characterize complex grain shapes such as the “jigsaw puzzle” structure below. She is a coauthor on a submitted journal publication, and received funding from the American Ceramics Society/NSF to attend the 1st International Congress on Ceramics in Toronto in June 2006 to present a research poster.

Ph.D. students Mai Ng and Lili Taherabadi, who conducted research on this grant, both served as mentors and female role models for the undergraduate students. Lili recently successfully completed her advancement to candidacy exam for the Ph.D. in Materials Science and Engineering at UC Irvine.

Aminah Rumjahn, an undergraduate from the University of California, Davis, conducted summer research on novel milling approaches to make stable nanocrystalline ceramics. She was also supported by the NSF REU “IM-SURE” grant.
Space Shuttle Tiles Stay Cool to the Touch

During Sally Ride Science Day at UC Irvine, the amazing properties of ceramics are demonstrated in a series of workshops by Professor Martha Mecartney and her graduate student Peter Dillon to over 50 middle school students.
Hands-on workshops for Sally Ride Day and for Girls Inc. are part of the PI’s outreach efforts to bring the excitement of materials science to the public and broaden the participation of women and girls in engineering.
The broadest impact is training a new generation of scientists and engineers. Both scientists were both supported by this NSF grant, and advised by Professor Martha Mecartney. Both doctoral students had children while in graduate school, but managed to complete their dissertations within four years of joining the Mecartney research group, demonstrating their successful integration of family life and a career in engineering including obtaining advanced technical degrees.
Outreach activities with Girls Inc. organization demonstrating the magic of ceramic engineering

PI Martha Mecartney and her graduate students Lili Taherabadi, Lynher Ramirez, Chris Hoo, and Kevin Olson involve middle school girls in experiments on the strength of ceramic composites, the insulating properties of space shuttle tiles, and the amazing microstructure of superplastic ceramics as shown by scanning electron microscopy.