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Joining of Advanced Materials by Plasticity "An enabling technology"

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This presentation does not contain any proprietary or confidential information

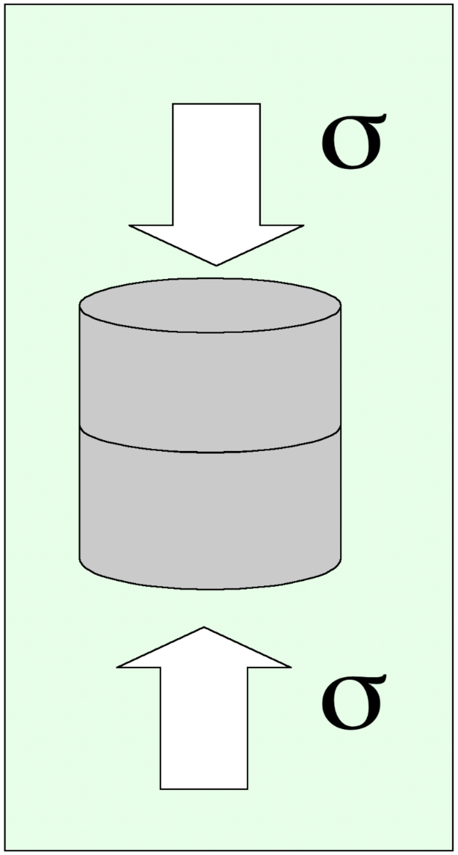
Purpose of Work

Joining by techniques such as brazing with a metal or glass changes the composition of the joint and, in general, reduces the strength of the joint

Possible applications: valves, sensors, fuel cells, optical, biomaterials, etc.

- Determine if advanced materials can be joined by plasticity
  - Assumption - grain boundary sliding (GBS) produces rotation and interpenetration of grains
- Determine the joint properties
  - Microstructure, strength, fracture, electrical, optical properties
- Test the assumption of GBS
  - Grain rotation experiments, electron back-scattered diffraction and high-intensity X-ray diffraction (Advanced Photon Source)
- Application
  - Oxygen sensor with internal reference

Approach



Experiments

- Relatively low joining temperatures:

  $1100 \leq T \leq 1350^{o}C$.

- Moderate strain rates in constant-strain-rate tests (Ar or air):

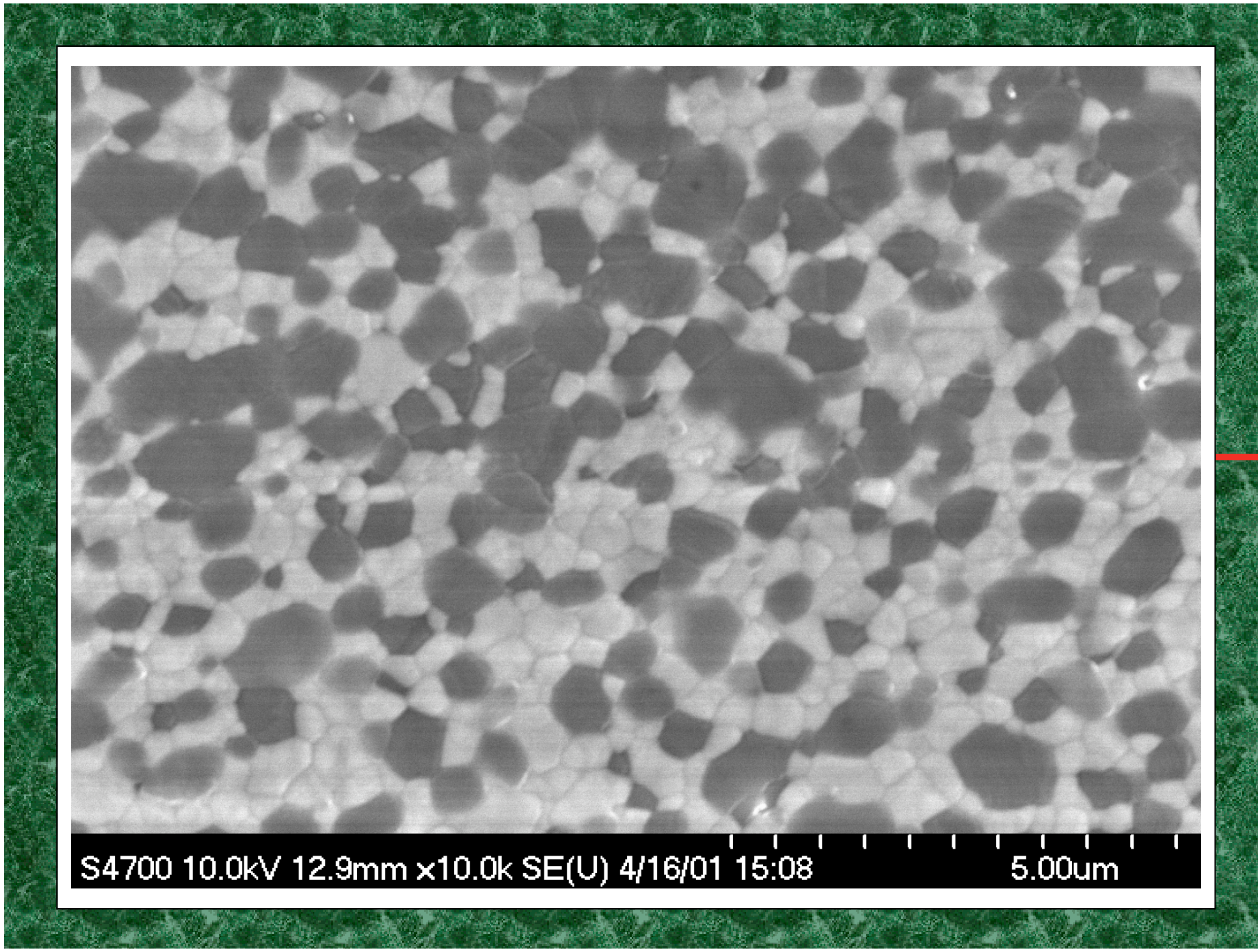
  $\dot{\varepsilon} \approx 10^{-5}\ s^{-1}$.

- Near-net-shape process:

  $\varepsilon_{max} < 10\ \%$.

No special surface preparation required

Microstructure

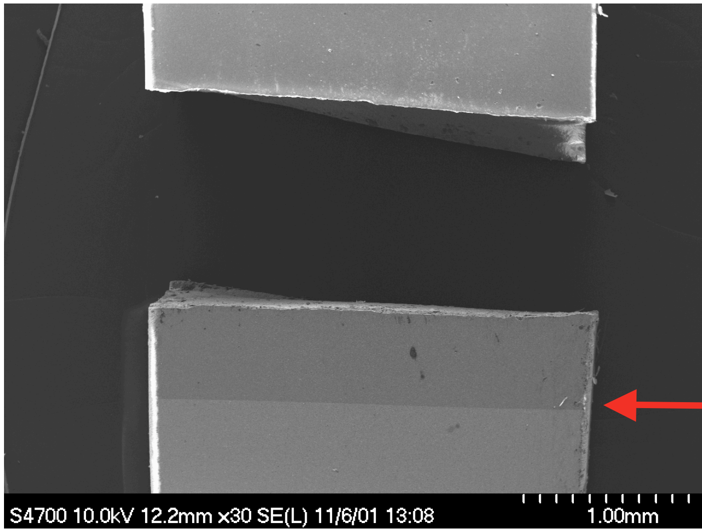


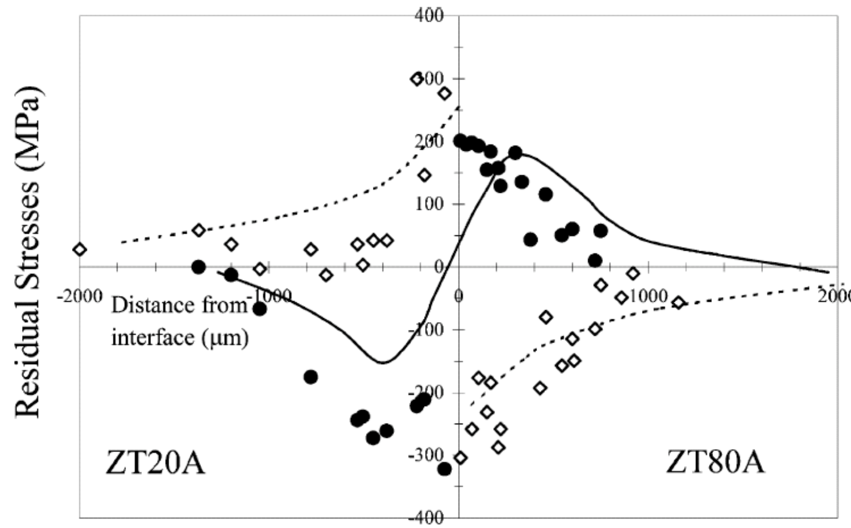
Y-ZrO_2 + Al_2O_3

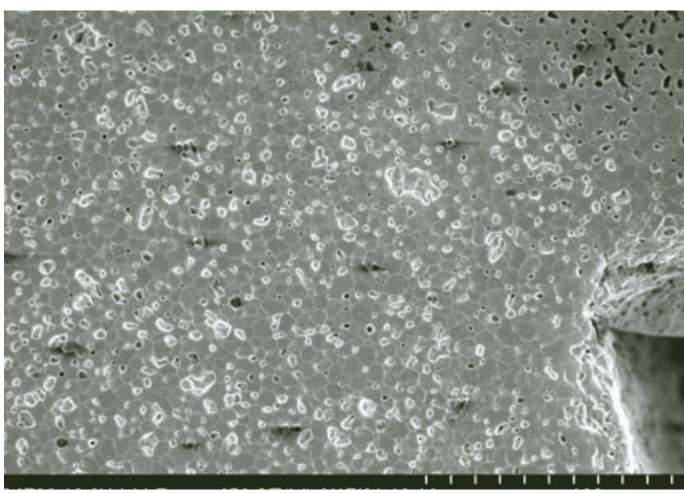
Joining plane

No porosity

Joint Properties – Fracture and Electrical Properties







FEA calculated residual stresses shown as lines, fracture occurs at position of maximum tensile stress, <u>NOT</u> at interface

LSM/LSM- 1200°C, 1.6 MPa, 3 minutes

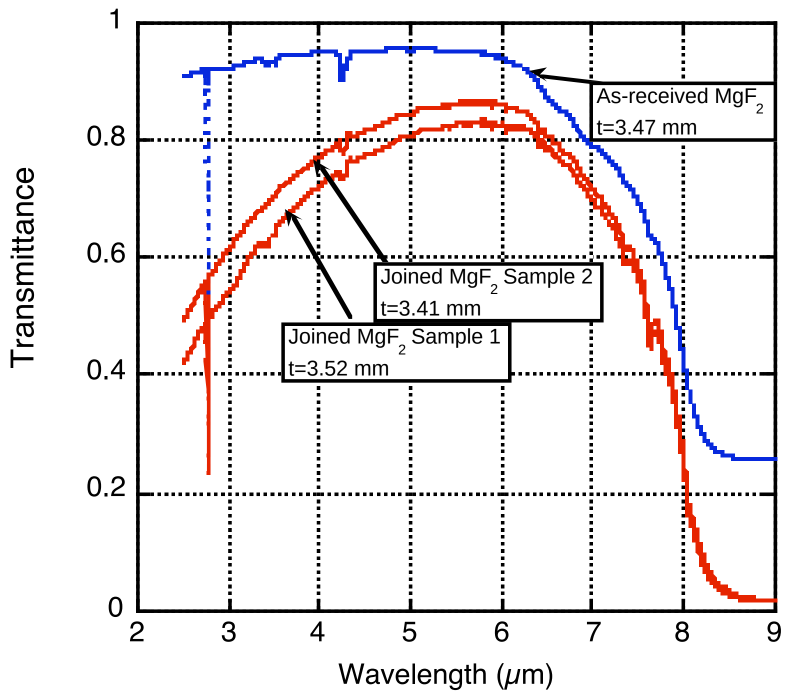
Bulk resistivity = 80 ± 10 ohm–cm

Joined resistivity = 75 ± 10 ohm–cm

No change in resistivity resulting from interface

Optical Properties

Integrated transmission of joined samples is ≈ 80% unjoined, no correction for length differences

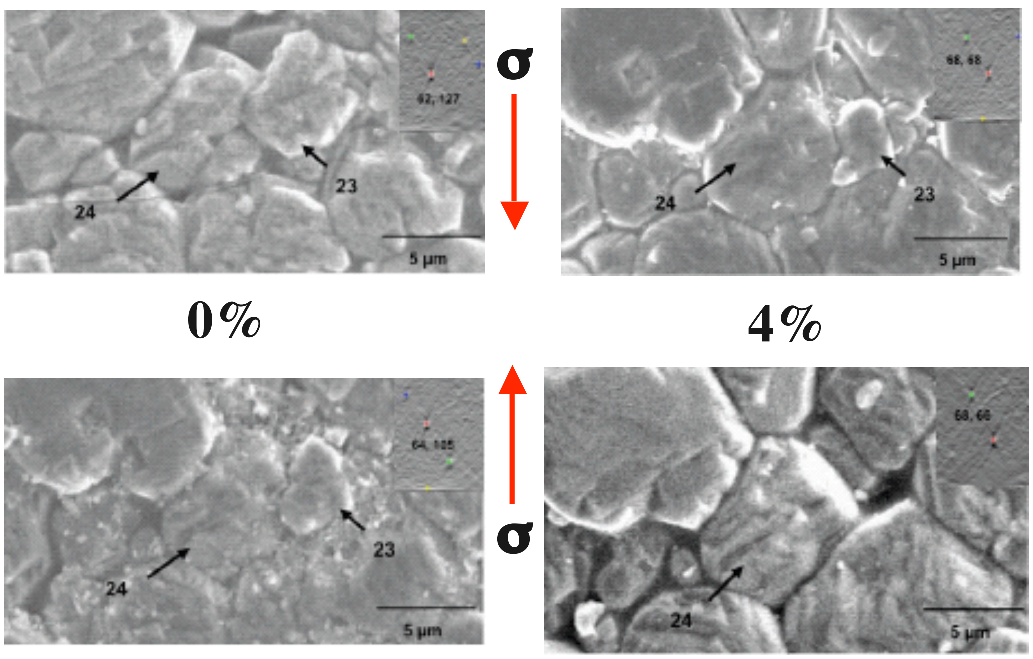


Materials Joined by Plasticity

- *Ceramics (structural/optical)*
- *Composites (Whisker-reinforced)*
- *Intermetallics*
- *Cermets*
- *Biomaterials*
- *MMC*

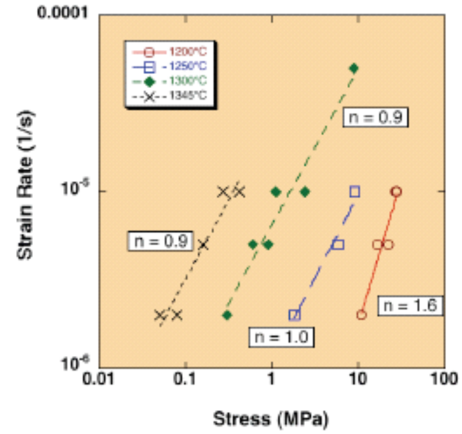
Mechanism – Grain Boundary Sliding

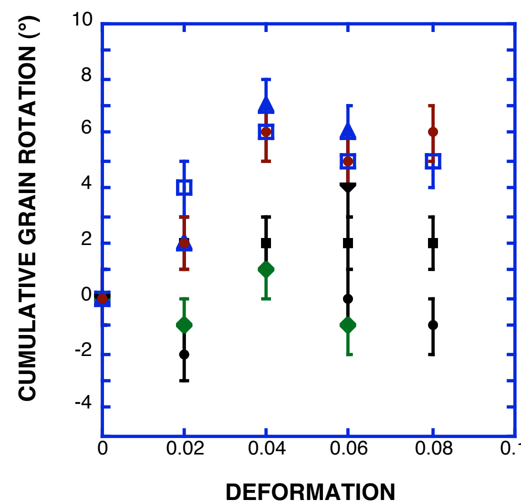
- ***If deformation is via GBS, and no cavitation, then grains must rotate and can interpenetrate to form a "perfect" bond***
  - ✓ **Fabricate high-density $SrTiO_3$ with ≈ 6μm equiaxed grains**
  - ✓ **Establish that high-temperature deformation is via GBS by measuring deformation and microstructure changes**
  - ✓ **Measure grain rotation by electron back-scattered diffraction and X-ray diffraction using APS as a function of strain at 1300°C**



0% **4%**

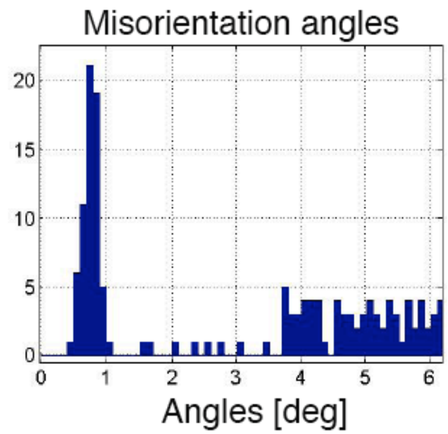
2% **8%**

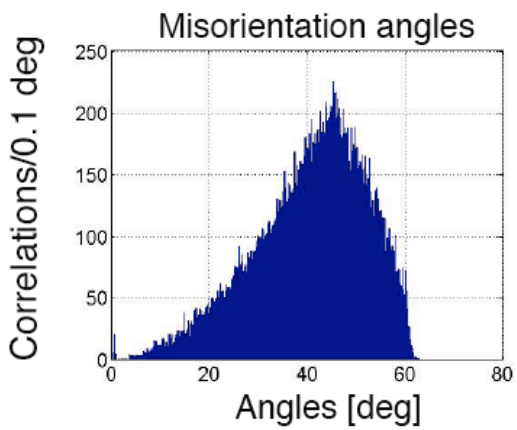




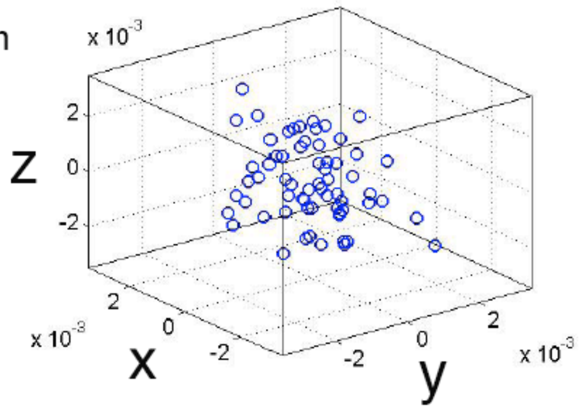
X-ray Diffraction using Advanced Photon Source

- Loading2: 0.05% -> 0.45%
- ≈ 220 grains (completeness > 0.4), 64 grains correlated





Misorientations in Rodrigues space



- Grain rotations < 0.4 deg / 0.4%
- track rotation paths

Gifkin's modification of Ashby-Verrall is best description

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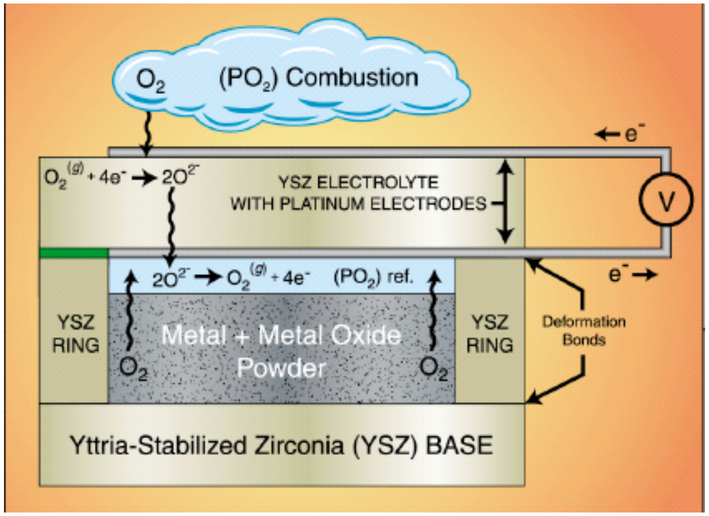
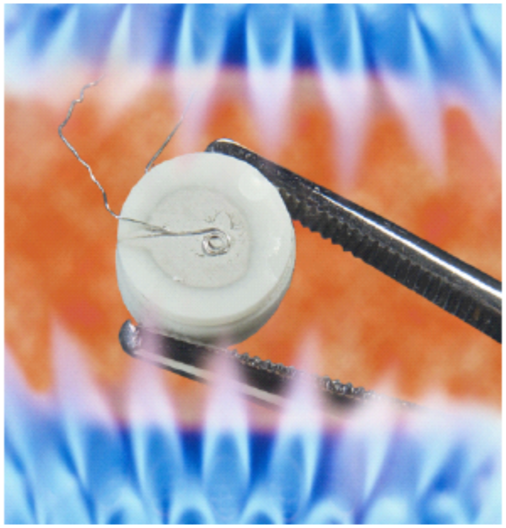
Application to Oxygen Sensors

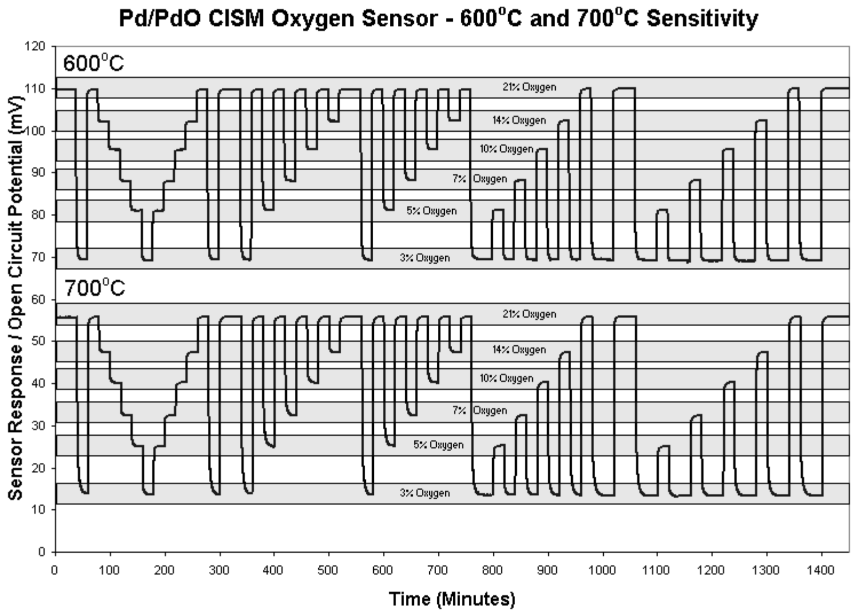
2% error in flue gas composition results in 0.5% decrease in fuel efficiency (inefficiency estimated at $409M for coal-fired plants, 97) – total expenditures on gas sensors - $880M (2003)

- Optical
  - Requires line of sight to chamber
  - Expensive equipment
- Thin-film semiconductor
  - Temperature limited
  - Non–linear response
  - Sensitive to impurities
- Internal reference
  - Sealing is **huge** problem
    - *Metal housing limits temperature, increases expense*
    - *Metal seals change oxygen ion conductivity, limit temperature*
    - *Glass seals fatigue due to thermal cycling, limited operational temperature*

Technical Accomplishments – Oxygen Sensor







- *Stable for months*
- *Very small*
- *No complex electronics*
- *Inexpensive*

Summary

Bridged the gap between basic and applied science/technology

- *Produced pore–free, strong joints using plastic deformation in a variety of advanced materials (US patent 6,974,070&one applied)*
- *Deformation is via grain–boundary sliding and rotation, resulting in interpenetration of two pieces to join*
- *Used technique to achieve a gas–tight seal allowing the production of a miniaturized oxygen sensor with an internal reference (R&D 100 and 2 patent applications)*
- *Shown that sensor is gas tight and responsive, follows Nernst*

▪ Technology Transfer

Negotiations for license for sensor is ongoing

FUTURE DIRECTIONS

- Replacement of Pt interconnect with a conducting ceramic
- Conversion of oxygen sensor to oxygen/NOx sensor–for emissions control
- Joining of bipolar plate to electrolyte
- Joining of metal or intermetallic to ceramic

Publications, Presentations, Patents (Reference for reviewers)

- Publications
  - 22 published papers in peer-reviewed journals
    - 3 more in press
- Patents
  - 1 patent granted, 3 applications filed, additional invention report
- Presentations
  - 17 presentations at international meetings (3 invited, 1 keynote)
- Awards
  - R & D 100
- Education
  - 2 Post docs, 1 Ph.D. (2006), 1 Ph.D. student