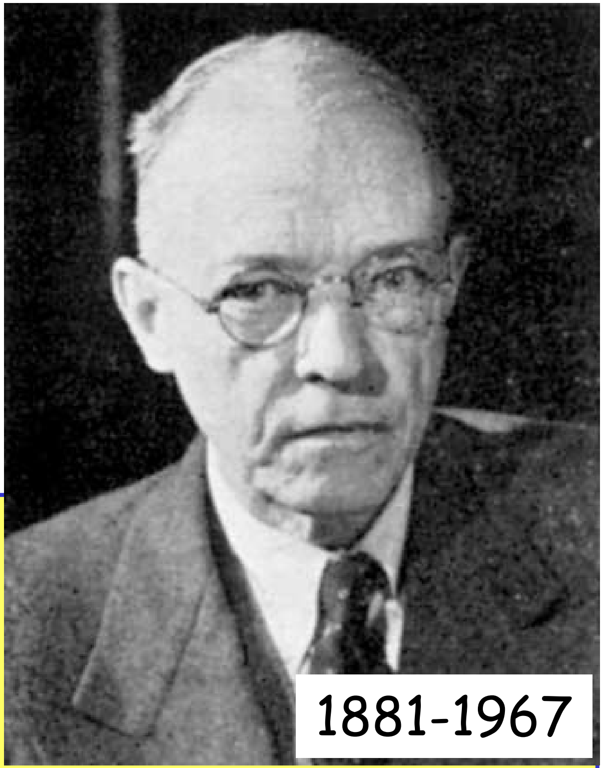
Robert B. Sosman

Physical Chemist
President, The American Ceramic Society, 1937–38
Edward Orton Jr. Lecturer, 1937
Albert V. Bleininger Award, 1953
Ross C. Purdy Award, 1957
John Jeppson Medal, 1960



1881-1967

B.Sc., Ohio State University, 1903
Ph.D., M.I.T., 1907 (1st yr.; 1 of 3)
Arthur D. Little, 1906-1908
Geophysical Laboratory, 1908-1928
U.S. Steel Corp., 1928-1947
Rutgers University, 1947-1962
The Properties of Silica, 1927; *The Phases of Silica*, 1965

Hiker, 7th to complete Appalachian Trail (Maine-Georgia)
Dancer & Dining, *Gustavademecum for the Island of Manhattan*

James B. Austin, Proc. Geological Society of America, 251-258 (1968).

Microstructural Stresses: Networks, Structural Reliability, & Antiquity Preservation

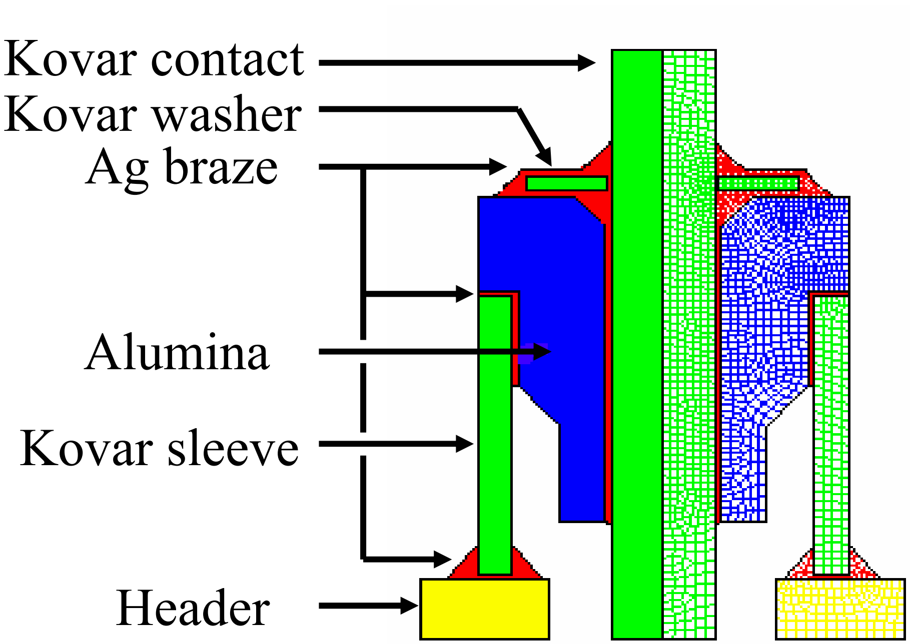
Edwin R. Fuller, Jr.
National Institute of Standards and Technology
Gaithersburg, MD 20899-8522, U.S.A.

<edwin.fuller@nist.gov>

National Institute of Standards and Technology

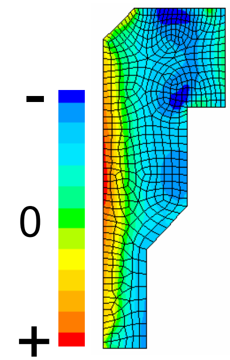
Robert B. Sosman Memorial Lecture
106th Annual Meeting of
The American Ceramic Society
Indianapolis, IN - April 21, 2004

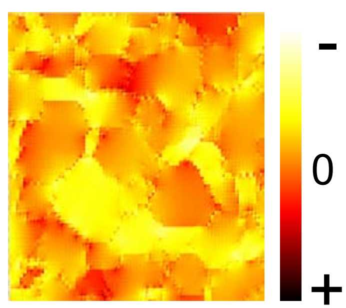
Predict Reliability of Ceramic-Containing Components



Residual Stresses due to:

thermal expansion mismatch	thermal expansion anisotropy

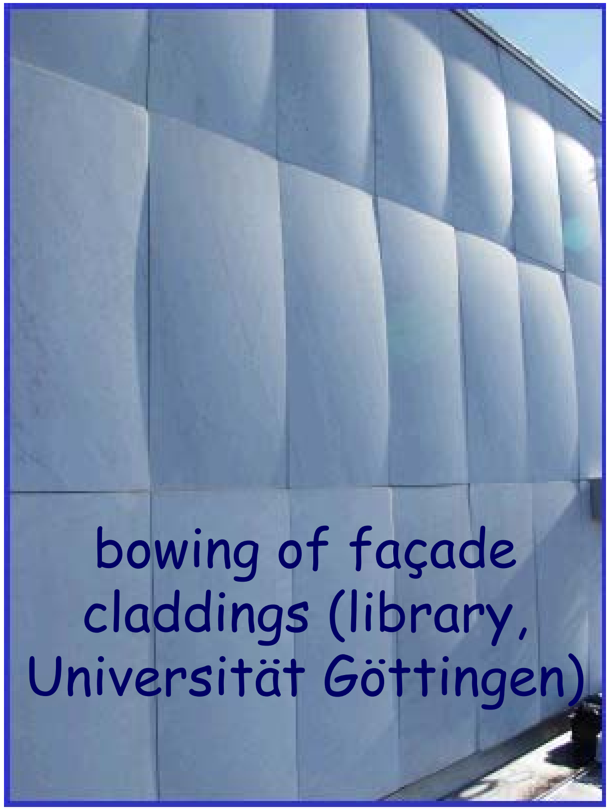


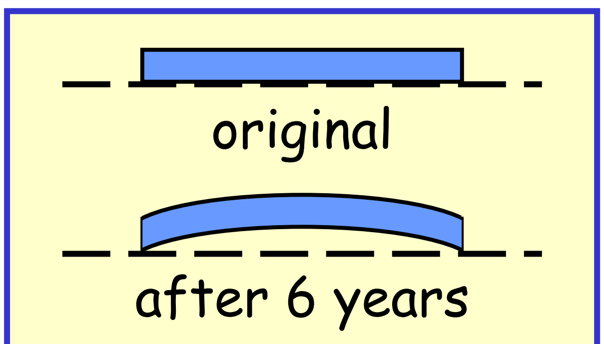


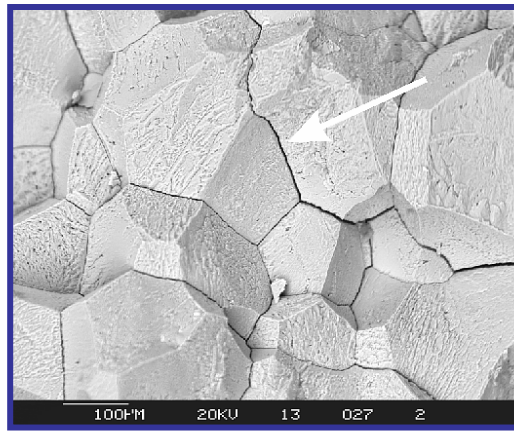
residual stresses can cause spontaneous microcracking and influence R-curve behavior & crack propagation under applied loads

Venkata R. Vedula, Shekhar Kamat & S. Jill Glass, Sandia National Laboratories

Thermal Degradation of Decorative Marbles





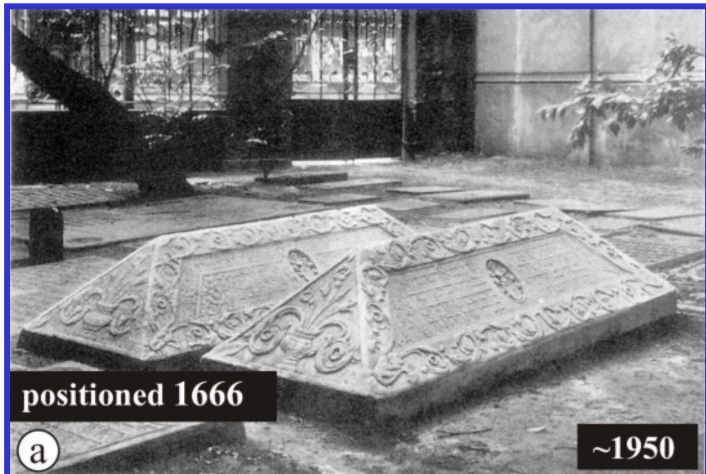


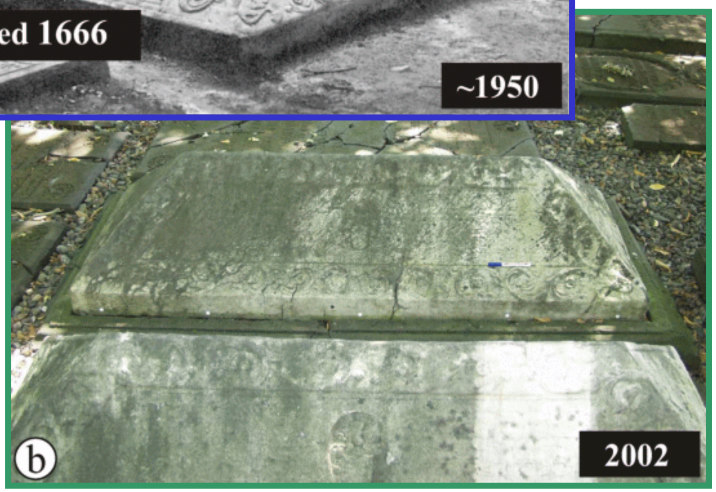
granular disintegration

Thomas Weiß and Siegfried Siegesmund, Universität Göttingen, Germany

Preservation of Antiquity Artifacts

Jewish cemetery in Hamburg Altona (marble tombstones)





Siegfried Siegesmund, Thomas Weiß & Joerg Ruedrich, Universität Göttingen, Germany

Getty Conservation Institute museum, Los Angeles

Microstructural Stresses

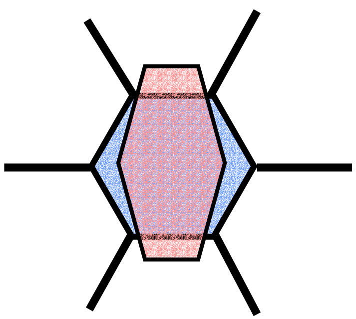
Objective: *to elucidate the influence of various types of texture on microstructural residual stresses and related ensemble physical behavior and properties of polycrystalline ceramics*

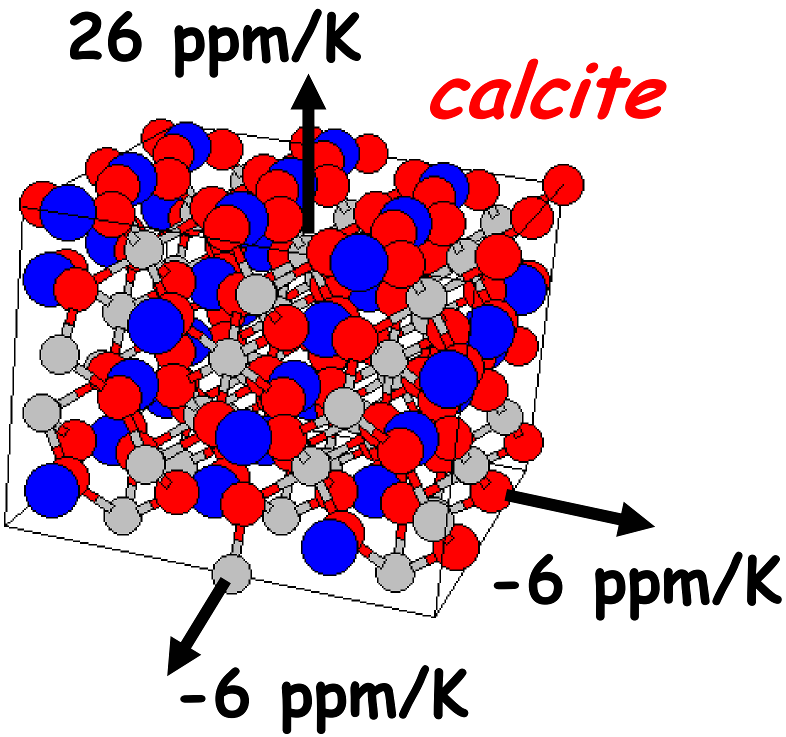
Contents:

- Residual-Stress Networks in Polycrystalline Ceramics
- Microstructural Finite-Element Analysis
- Statistical Description of Microstructure and Texture
- Residual Stress and Physical Property Simulations
- The *Old* and the *New*

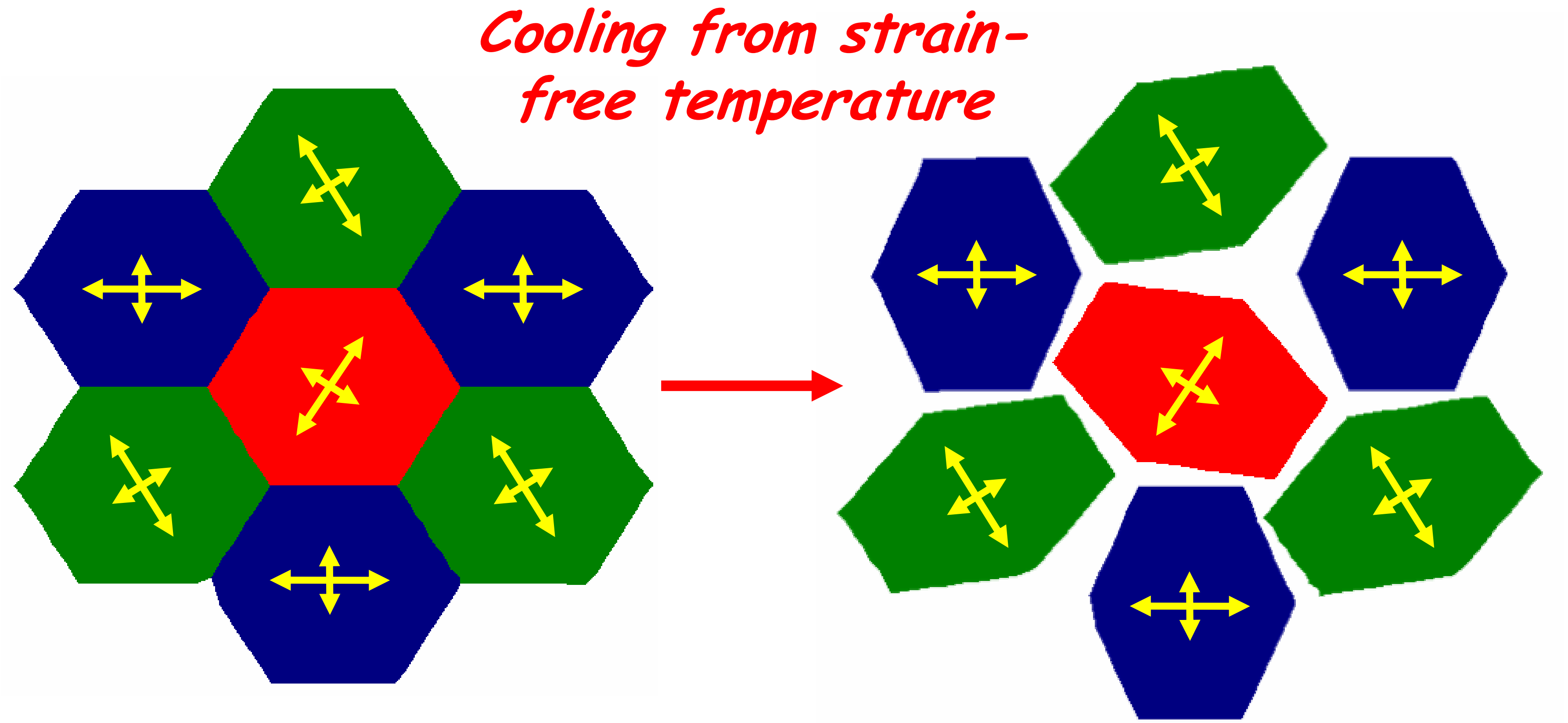
Origin of Microstructural Stresses

Internal microstructural stresses arise due to **T**hermal **E**xpansion **A**nisotropy (**TEA**) misfit between adjacent grains upon heating or cooling

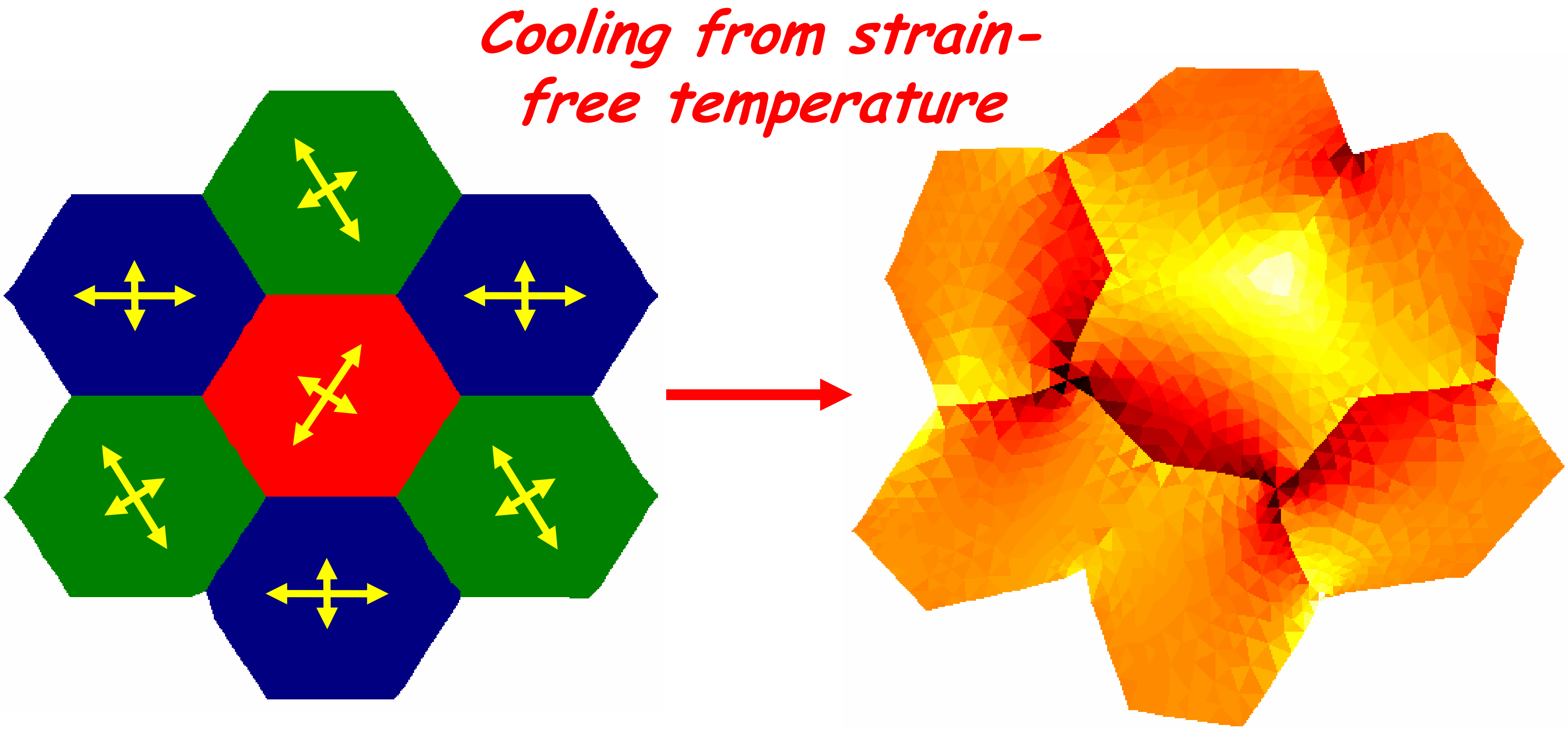




Thermal Expansion Anisotropy Misfit Strains in a Microstructure

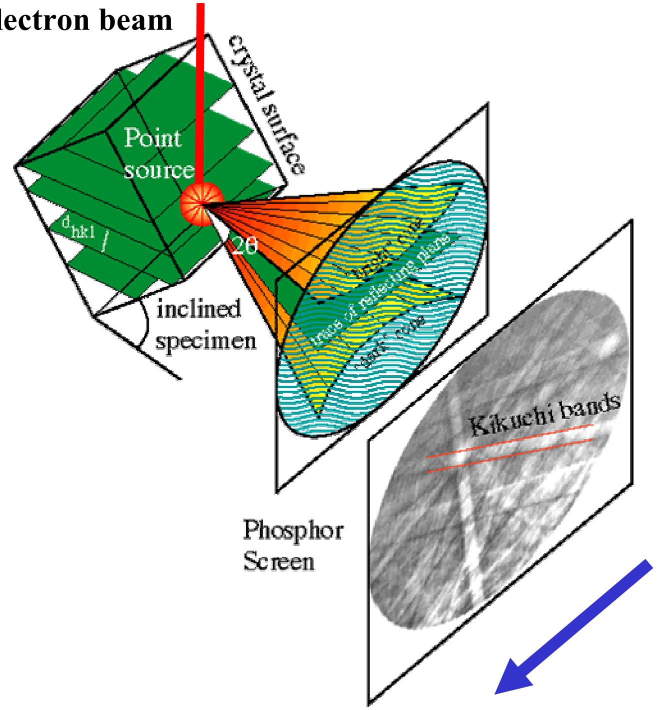


Thermal Expansion Anisotropy Misfit Strains in a Microstructure

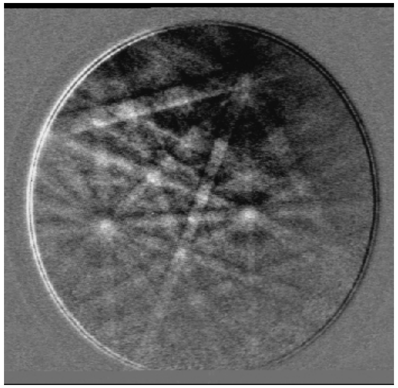


¤ Microstresses are independent of grain size ¤

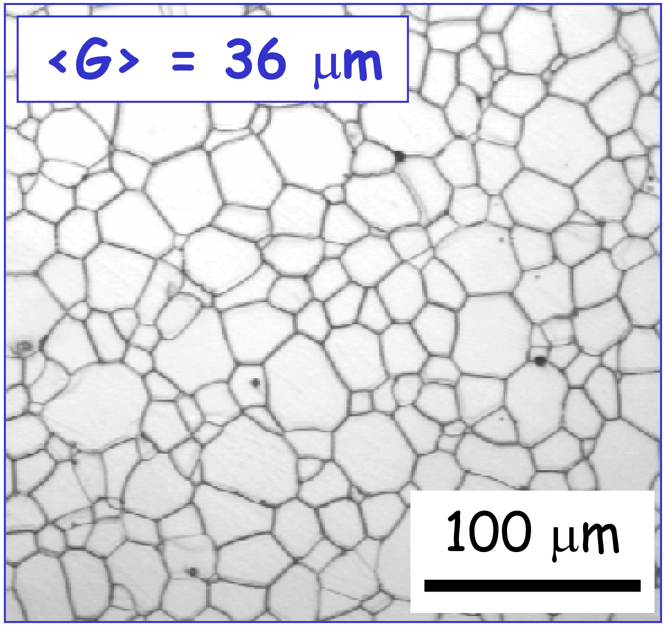
Grain Orientations from EBSD



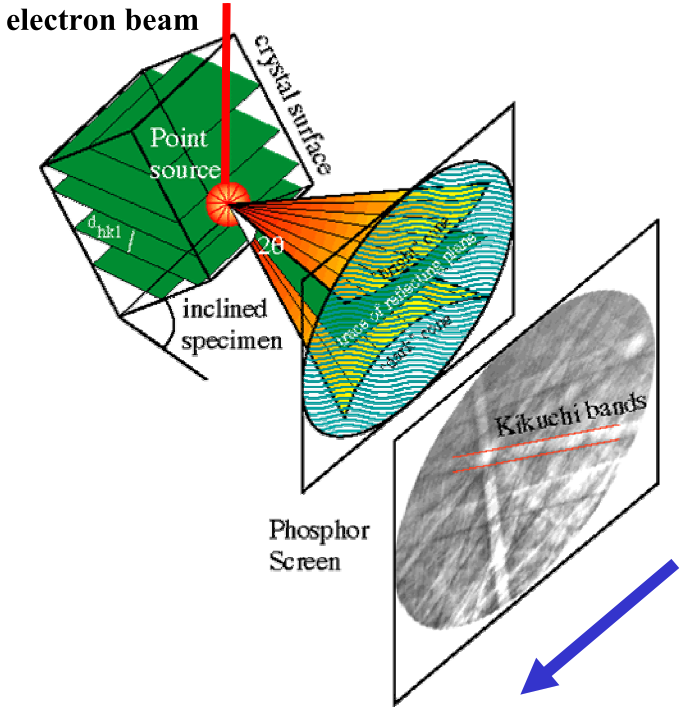
Kikuchi bands



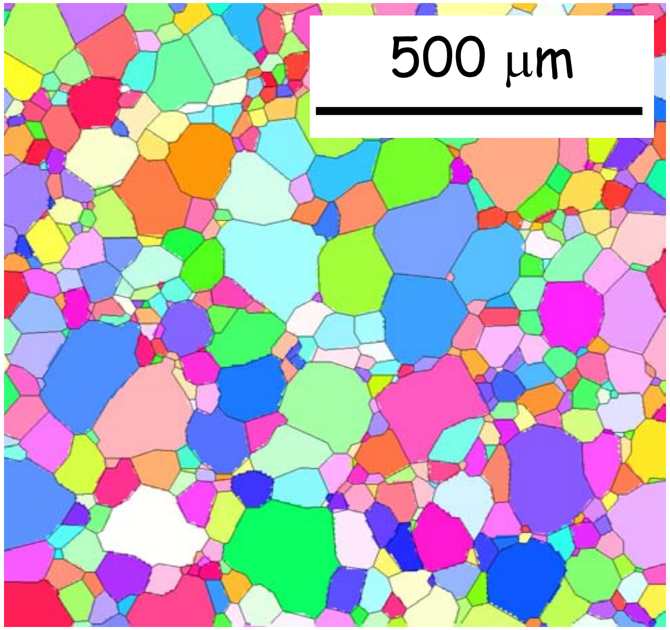
Electron Back-Scattered Diffraction



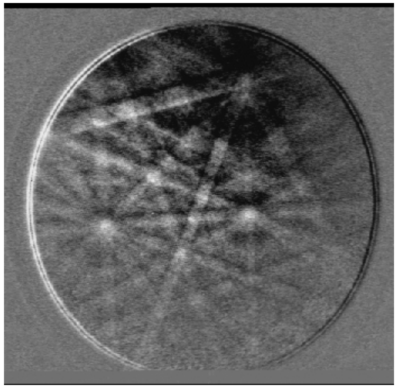
Grain Orientations from EBSD

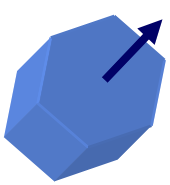


Electron Back-Scattered Diffraction

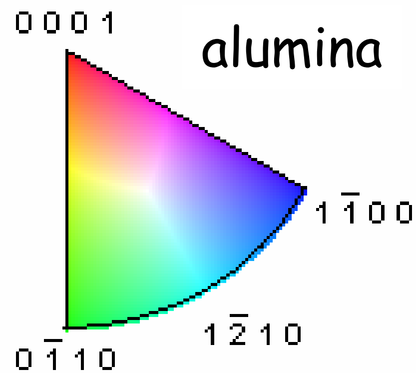


Kikuchi bands

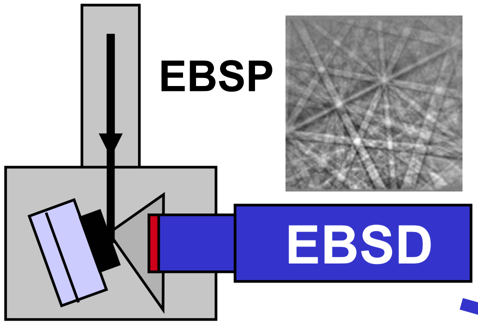




alumina

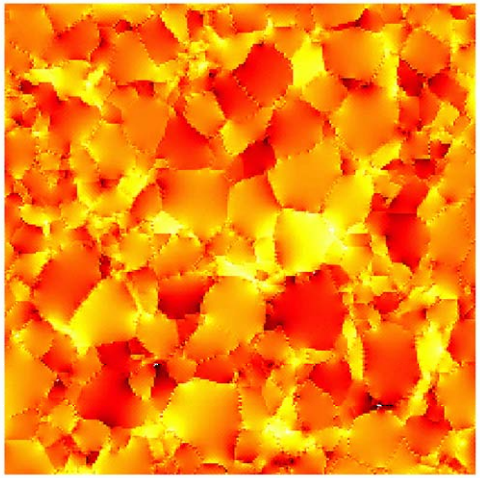


Calculation of Microstructural Residual Stresses



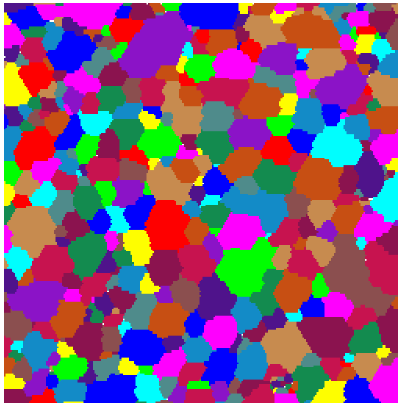
Grain Orientations

microstructural finite element analysis



Microstructural Residual Stresses

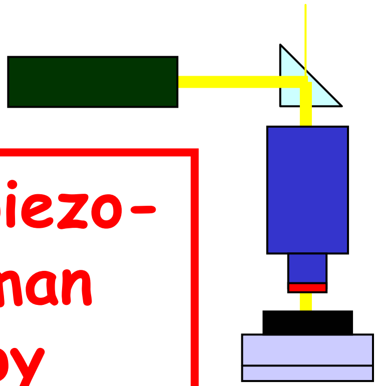
Microstructure



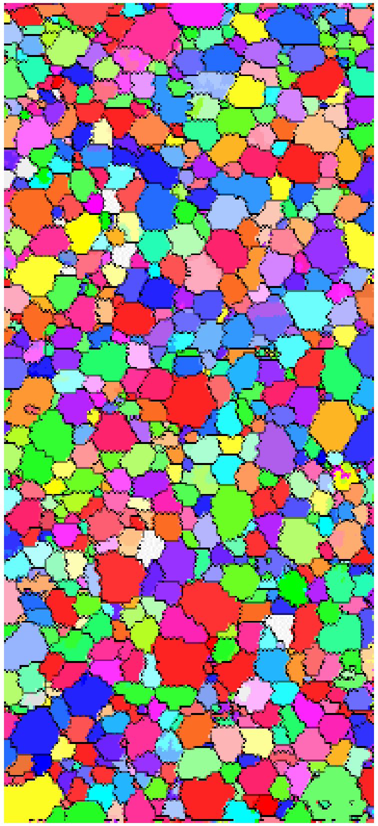
Thermoelastic Properties

C_{11}, C_{12}, C_{13},
C_{33}, C_{14}, C_{44}
α_{11}, α_{33}

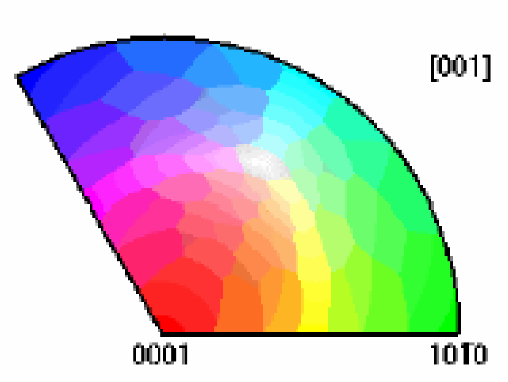
measure with piezo- or micro-Raman spectroscopy



Stress Networks in Alumina

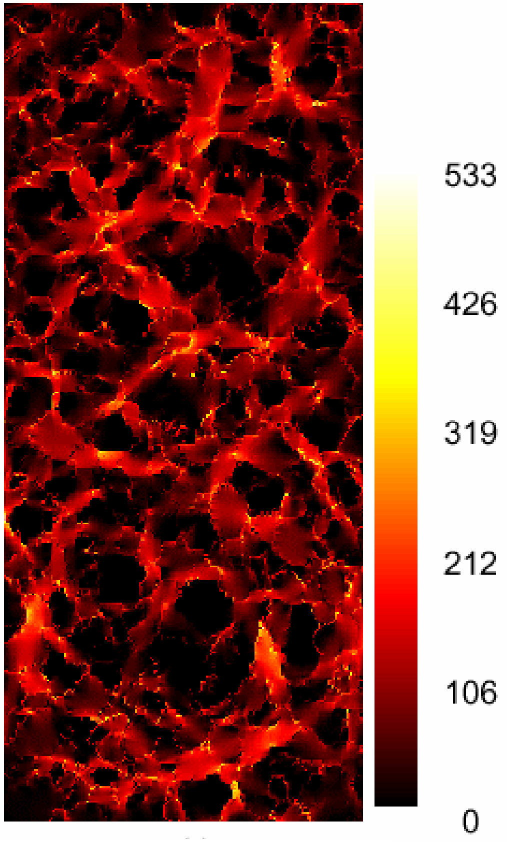


untextured EBSD microstructure



Grain Normals

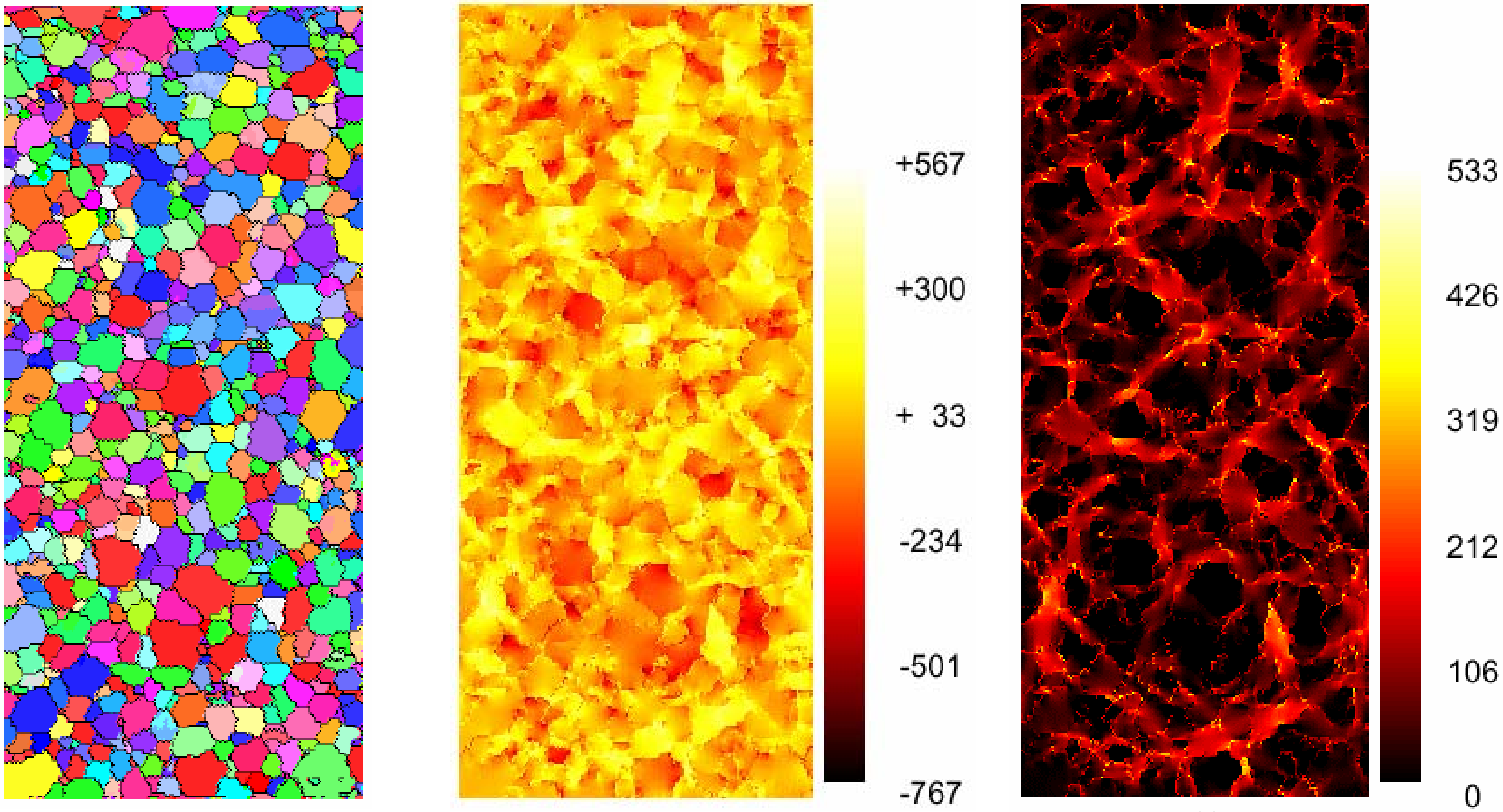
Residual Stress Distribution upon cooling by 1500°C



Max. Principal Stress (MPa)

V.R. Vedula, S.J. Glass, D.M. Saylor, G.S. Rohrer, W.C. Carter, S.A. Langer, and E.R. Fuller, Jr., J. Am. Ceram. Soc., 84 [12], 2947-2954 (2001).

Residual Stress Distribution in Untextured Alumina (ΔT = -1500°C)



EBSD microstructure

Hydrostatic Stress (MPa)

Max. Principal Stress (MPa)

Microstructural Stresses

Contents:

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  Physical Property Simulations
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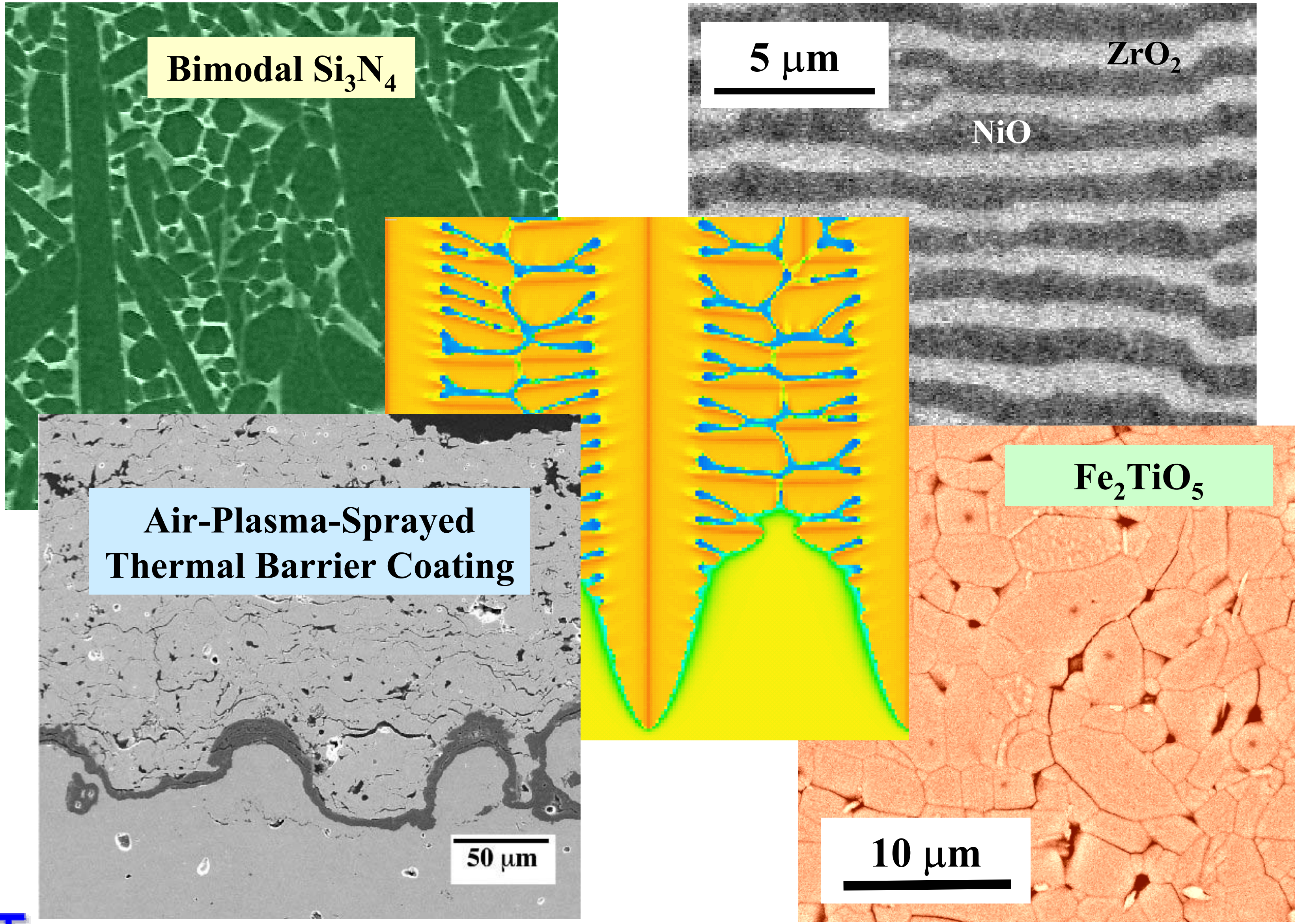
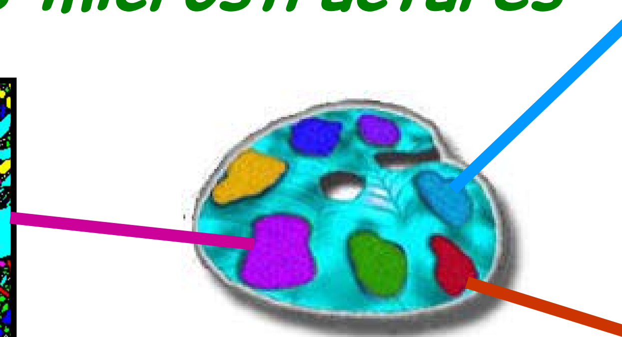
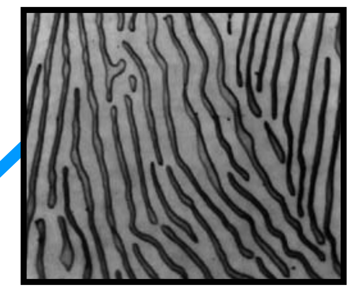
Material Microstructures: Heterogeneous & Stochastic



Object Oriented Finite Element Analysis for Materials Science and Engineering

Public domain software to simulate and elucidate macroscopic properties of complex materials microstructures





http://www.ctcms.nist.gov/oof

INDUSTRY WEEK IW 1999 Technologies of the Year Award

Building a Microstructural Model

Experiments

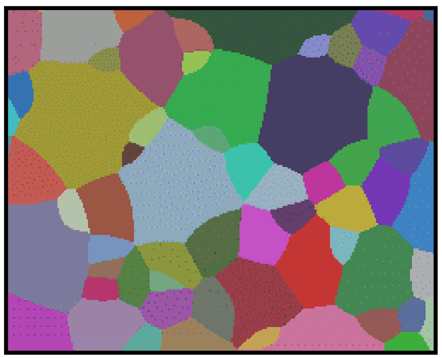
Simulations

Microstructure Data (micrographs)

Fundamental Materials Data

Materials Physics

easy-to-use Graphical User Interface (GUI) - ppm2oof



Object Structure Isomorphic to the Material

Finite Element Solver

easy-to-use Graphical User Interface (GUI) - oof

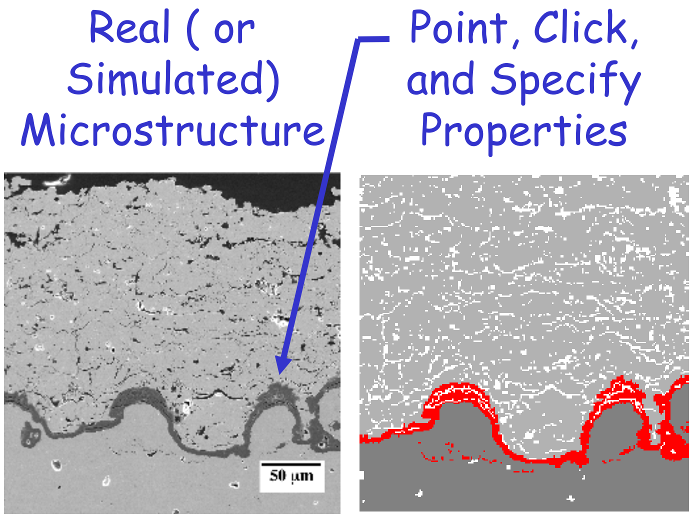
Virtual Parametric Experiments

Visualization of Microstructural Physics

Effective Macroscopic Physical Properties

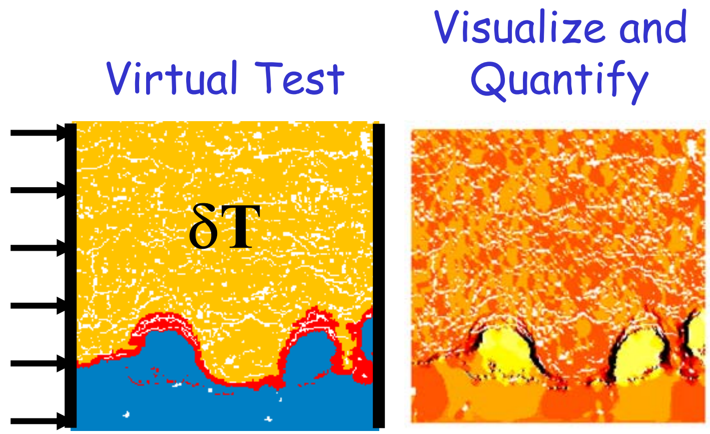
Finite Element Analysis of Real Microstructures

a tool for materials scientists to design and analyze advanced materials



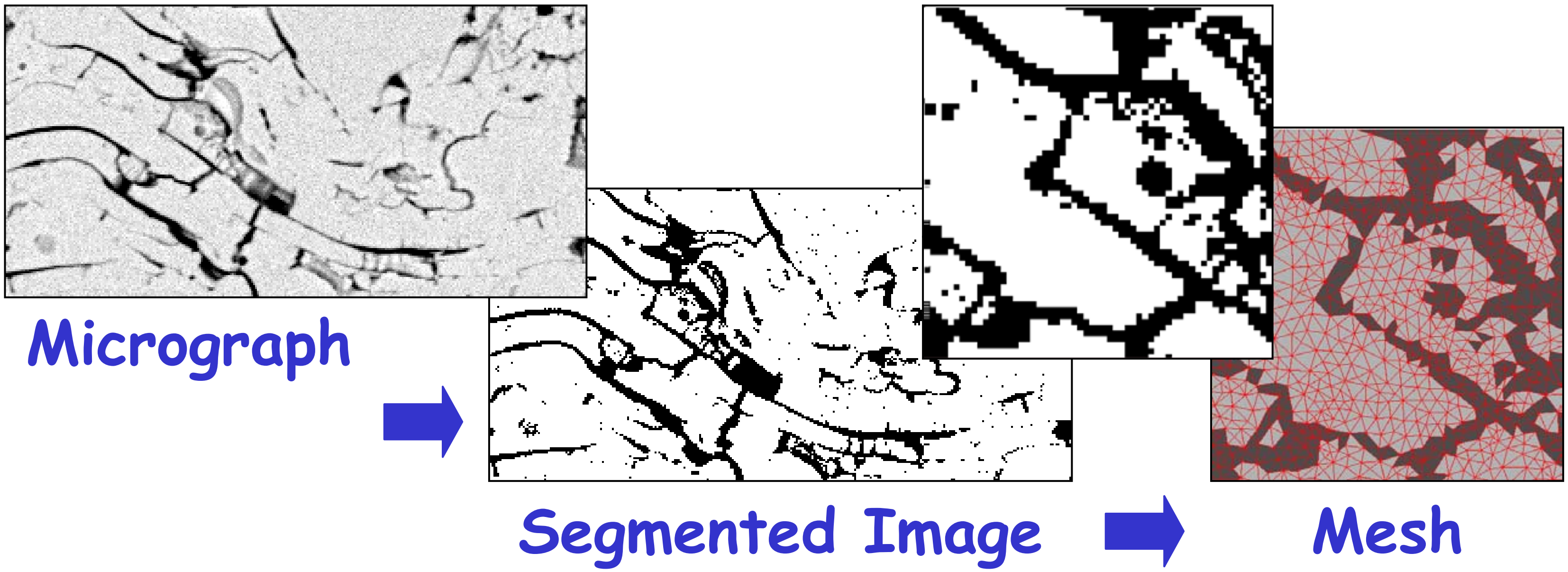
ppm2oof: a tool to convert a micrograph or image of a complex, heterogeneous microstructure into a finite element mesh with constitutive properties specified by the user.

oof: a tool to perform virtual experiments via finite element analysis to elucidate microstructural properties and macroscopic behavior.



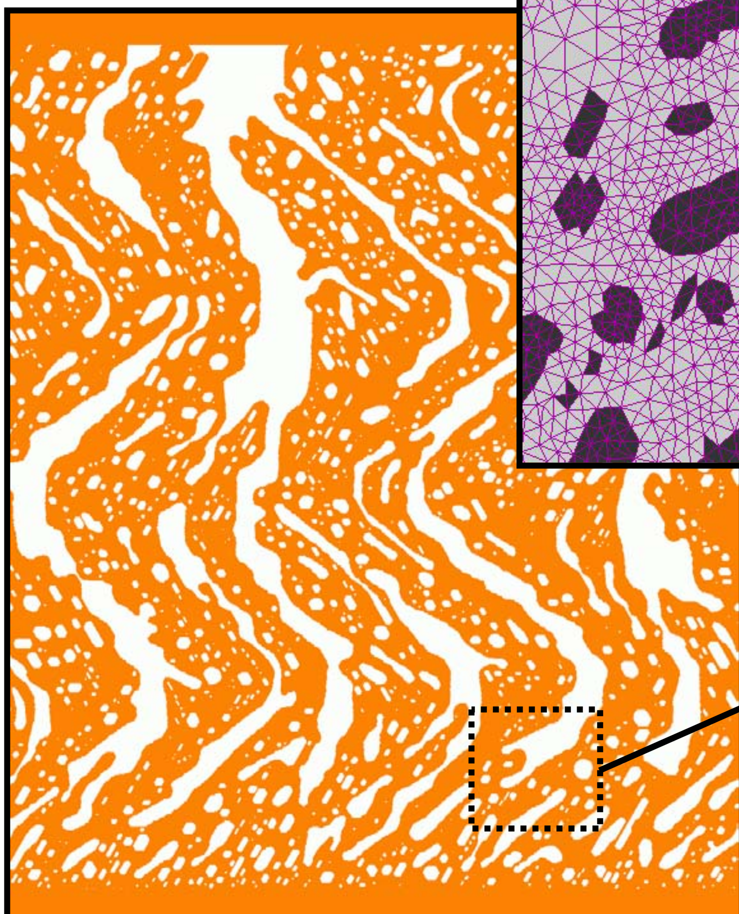
oof2abaqus: converts PPM2OOF or OOF data files into input files for ABAQUS™.

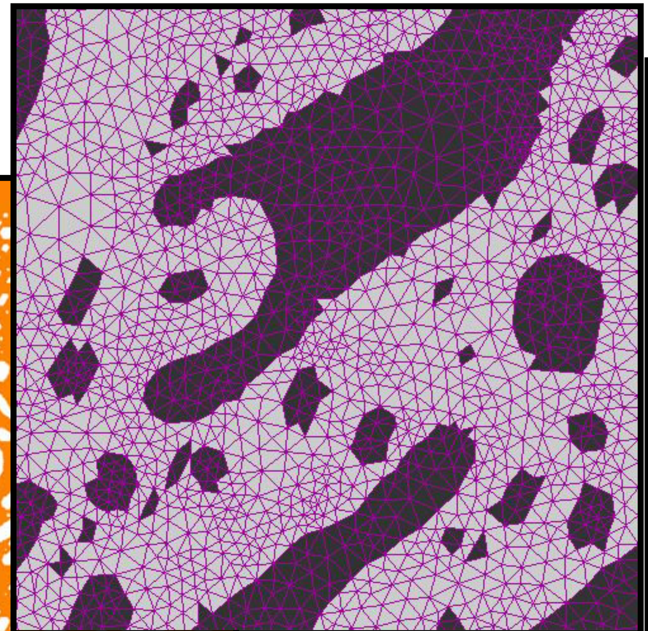
PPM2OOF Tool

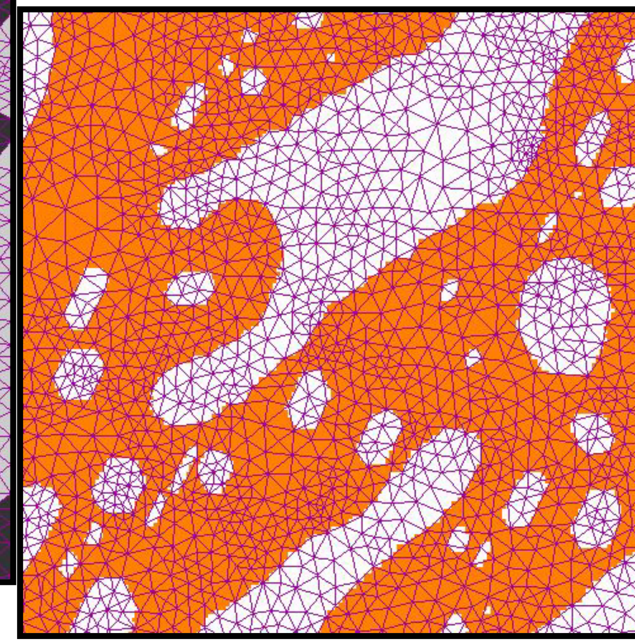


- Convert micrograph to ".ppm" (portable pixel map) file
- Select & identify phases to create segmented image
- Assign constitutive physical properties to each phase
- Mesh in PPM2OOF via "Simple Mesh" or "Adaptive Mesh" – multiple algorithms that allow elements to adapt to the microstructure

Adaptive Meshing by Components

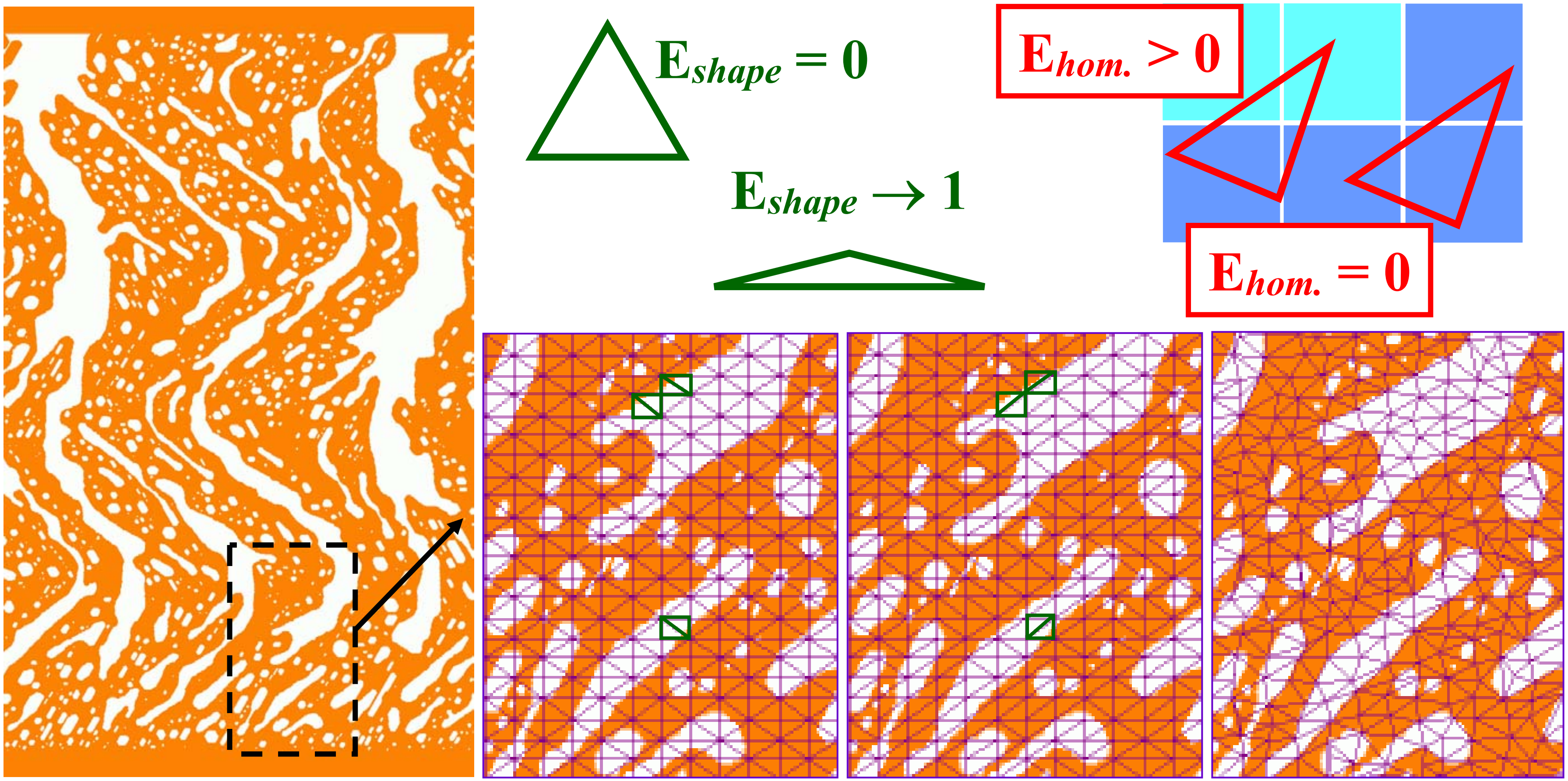






Generate a finite-element mesh following the material boundaries

Adaptive Meshing by Components: refine elements and move nodes via Monte Carlo *annealing* to reduce $E = (1-\alpha)E_{shape} + \alpha E_{homogeneity}$



image

create mesh

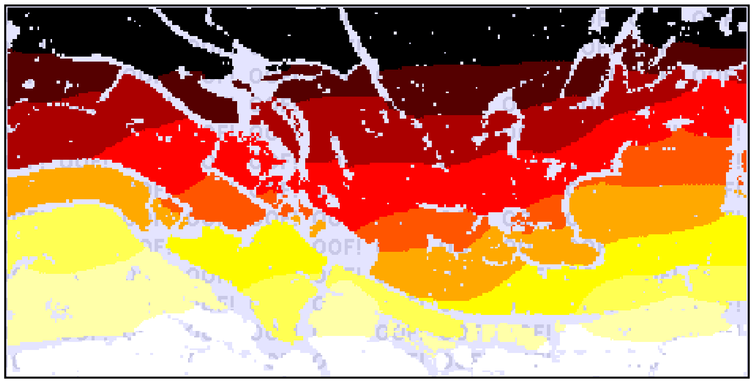
swap worst to reduce E

anneal to reduce E

OOF Tool

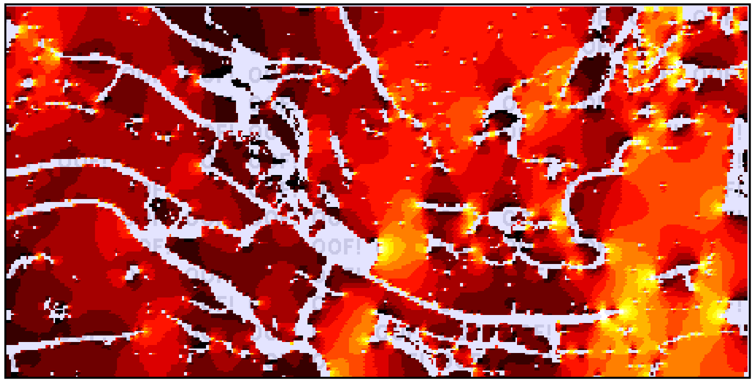
Virtual Experiments: Temperature Gradient

To + δT



To - δT

Visualize & Quantify: Heat Flux Distribution



Perform virtual experiments on finite-element mesh:

- To determine effective macroscopic properties
- To elucidate parametric influences
- To visualize microstructural physics

OOF 2.0 Current Development Effort

Stephen A. Langer, Andrew C. E. Reid
Seung-Ill Haan, and Edwin Garcia

- Extensible and more flexible platform
- Enhanced image analysis tools
- Expanded element types
- Generalized constitutive relations (elasticity, piezoelectricity, etc.) w/coupling between fields

Constitutive Eq.: $\Psi = \sum c \bullet \nabla \phi$	Equil. Eq.: $\nabla \bullet \Psi = f$

- Linear and nonlinear solvers

  with automatic mesh refinement

  – ppm2oof and oof are now combined

Planned Additions

- 3-dimensional finite element solver
- Time-dependent solver
- Plasticity

OOF Development Team

Microstructural Stresses

Contents:

- Residual-Stress Networks
  in Polycrystalline Ceramics
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  of Microstructure and Texture
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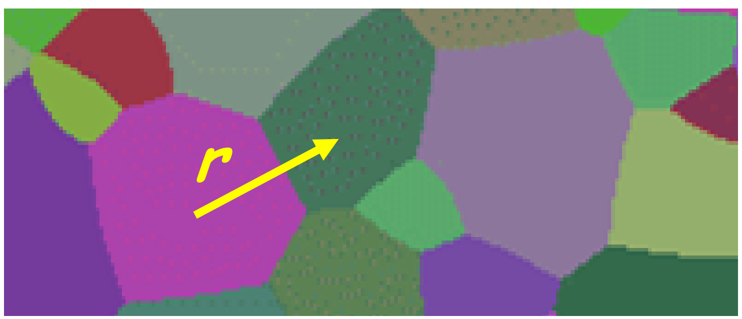
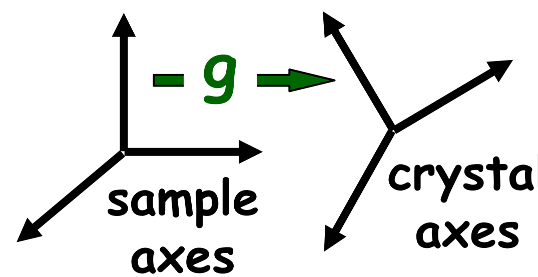
  Physical Property Simulations
- The *Old* and the *New*

Statistical Description of Microstructure

Microstructure Descriptor: $h = \{c, \phi, g, \ldots\}$

- composition: c
- phase: ϕ
- crystal orientation: $g = \{\varphi_1, \Phi, \varphi_2\}$





Microstructure Statistics

- 1-point statistics: $f_1(h)$
- 2-point statistics: $f_2(h, h' \mid r)$

Grain-Boundary Statistics: $S(\Delta g, n)$

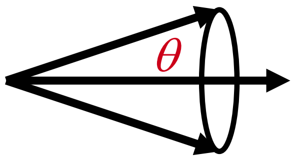
adapted from Surya R Kalidindi

Types of Texture

- **Crystallographic Texture**
  - **O**rientation **D**istributions **F**unctions (ODF) of grains: $g = \{\varphi_1, \Phi, \varphi_2\}$
    - Random in Euler Space (uniform)

      $\varphi_1 \in \{0, 2\pi\}$
      $\cos[\Phi] \in \{-1, 1\}$
      $\varphi_2 \in \{0, 2\pi\}$

    - March-Dollase Fiber Texture

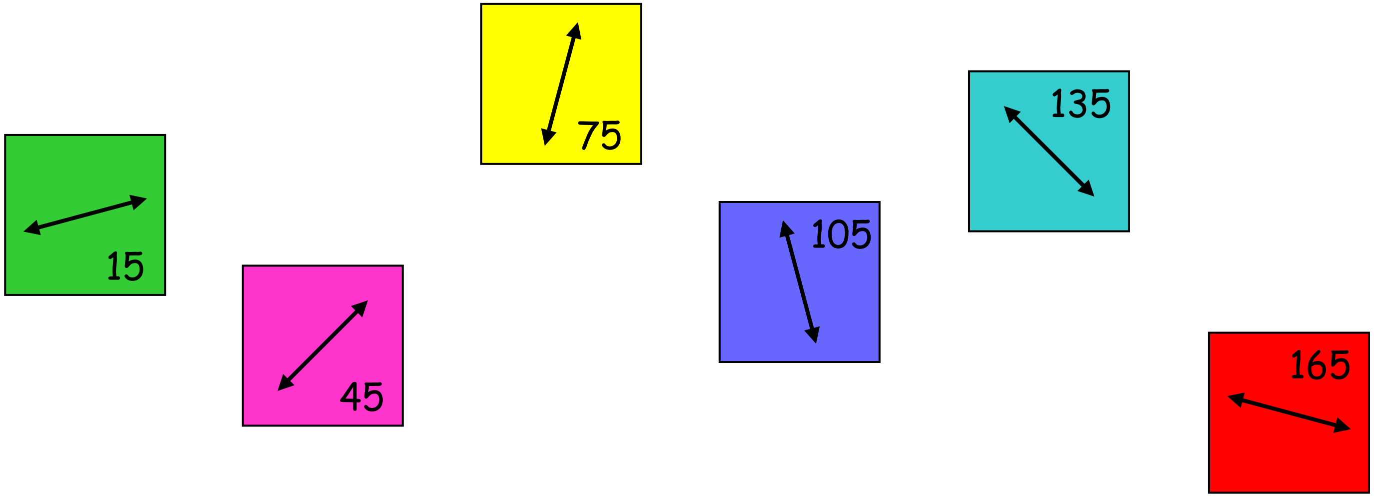


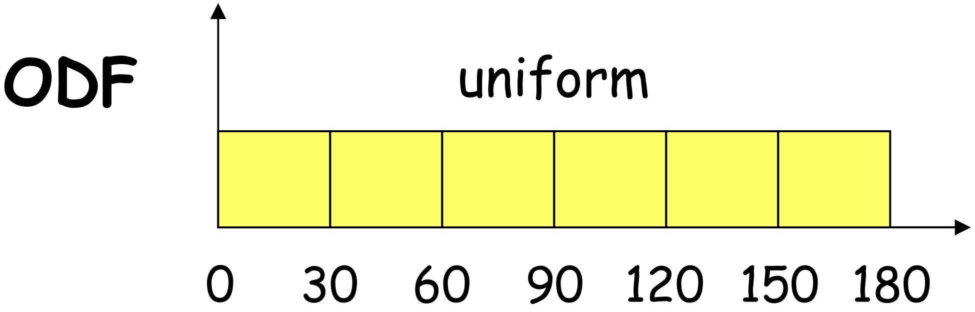
$$f(M,\theta) = M/[\cos^2(\theta) + M \sin^2(\theta)]^{3/2}$$

M: max. **MRD**

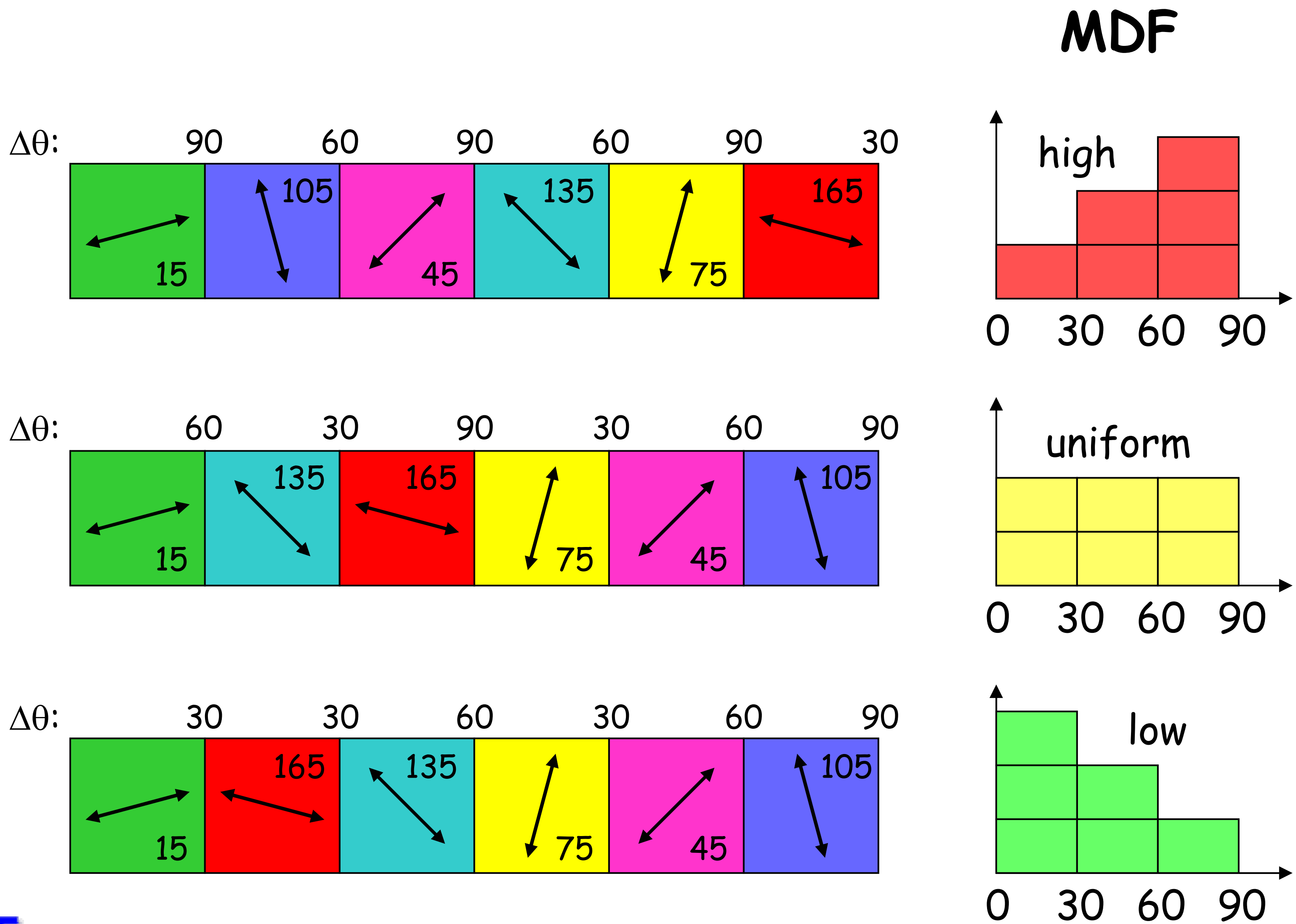
    - Others ODF's
  - Intercrystalline **M**isorientation **D**istributions **F**unctions (MDF): uniform and *high* & *low*
  - Orientation Correlation Functions (k-point statistics)
- **Grain-Shape Morphologic Texture**

Uniform Crystallographic Texture





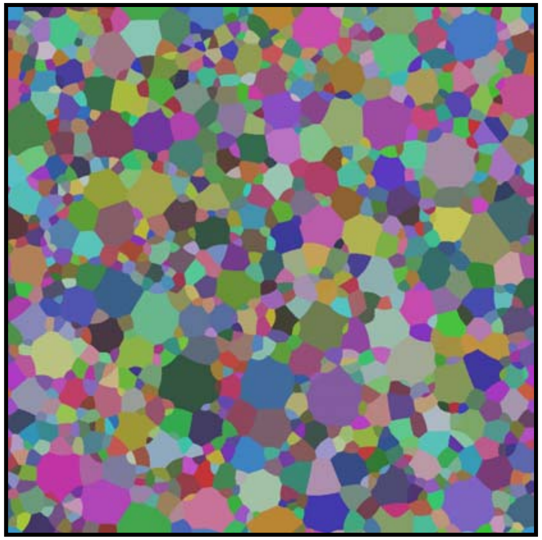
Types of Misorientation Texture



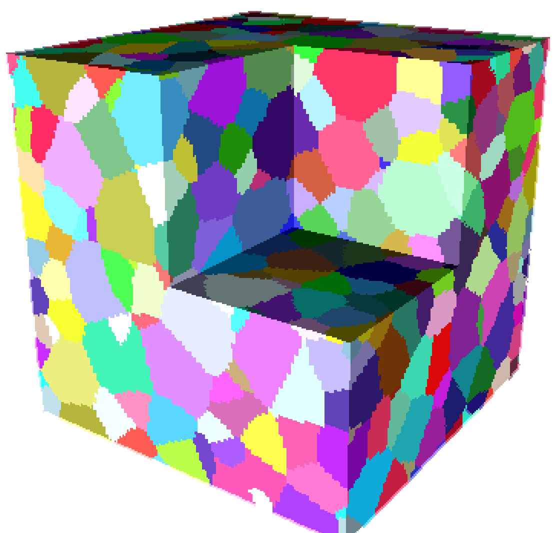
Microstructural Stresses

Contents:

Simulation Model Microstructure



2-D:
924 grains
1000^2 pixels



3-D:
422 grains
100^3 voxels

Grains:

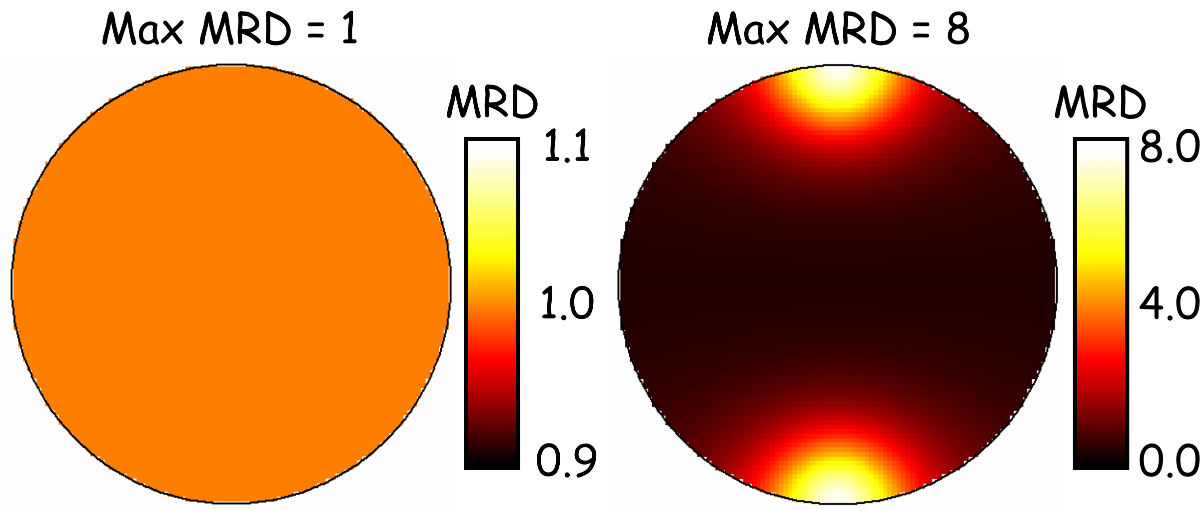
- same composition: either calcite, dolomite, or alumina
- same thermoelastic properties

Orientations:

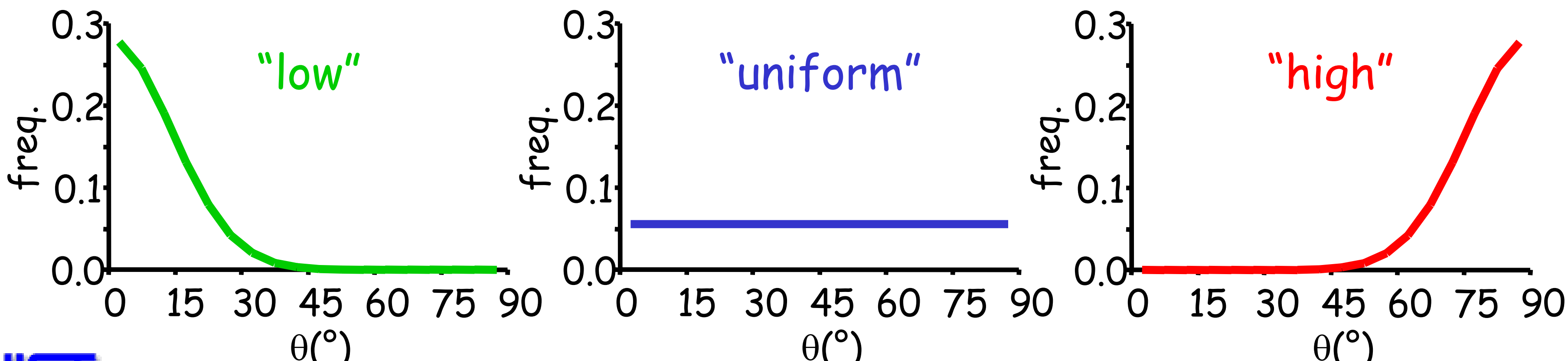
- *different* crystallographic orientations for all grains
- random or c-axis textured distribution of crystalline orientations
- uniform, and high and low distribution of intergranular misorientations

Microstructure Crystallography

Crystalline Orientations Distributions:

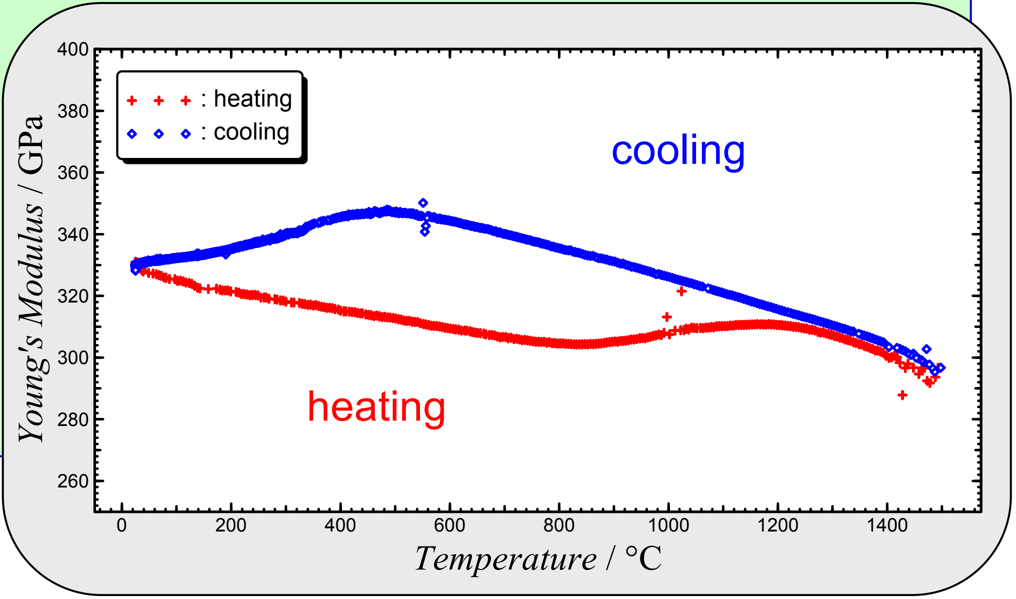


Intergranular Misorientation Distributions:



Phenomena Influenced by Texture

- Microstructural Residual Stresses
- Elastic Strain Energy Density (a measure of microcracking propensity)
- Anisotropy in *Bulk* Thermal Expansion Coefficient
- Anisotropy in *Bulk* Elastic Modulus
- Microcracking and its influence on properties

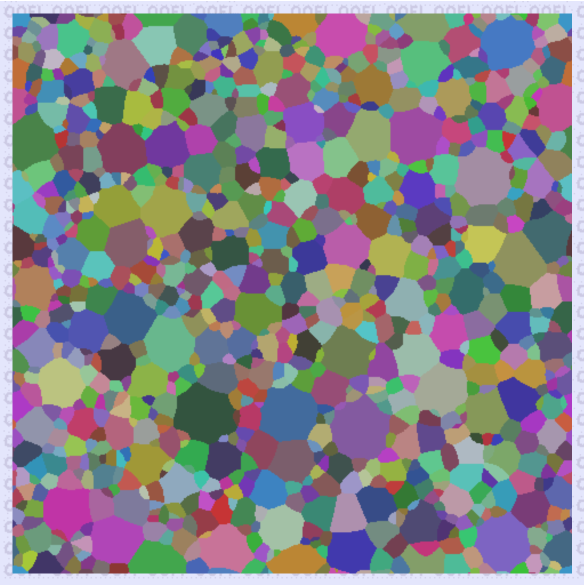


S. Galal Yousef & J. Rödel
T U Darmstadt

Influence of Grain Misorientation Distribution Function

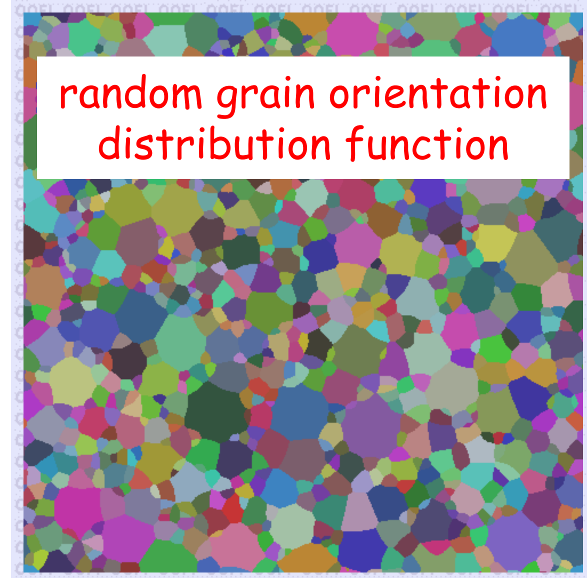
for a random grain orientation distribution function

on residual stresses in polycrystalline calcite upon heating by 100 °C

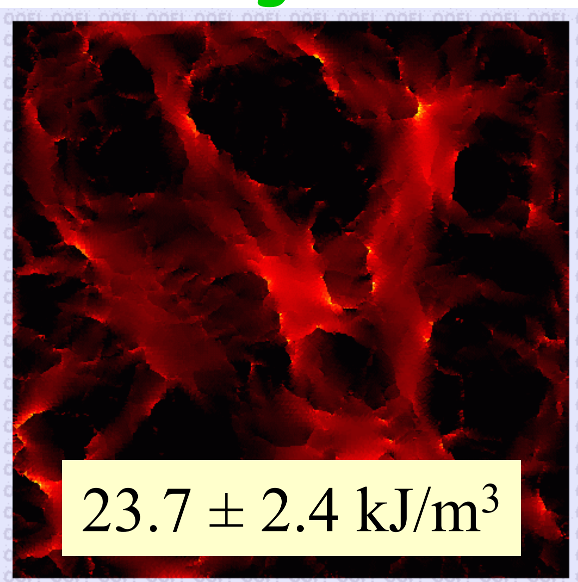


Influence of Grain Misorientation Distribution Function

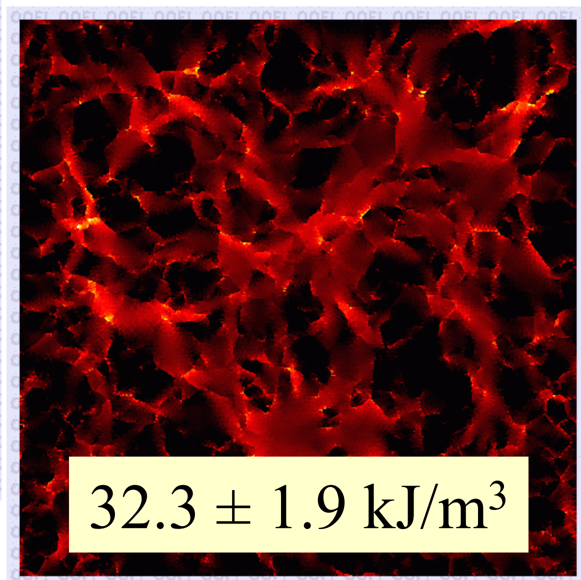
maximum principal stress for a random grain orientation distribution function

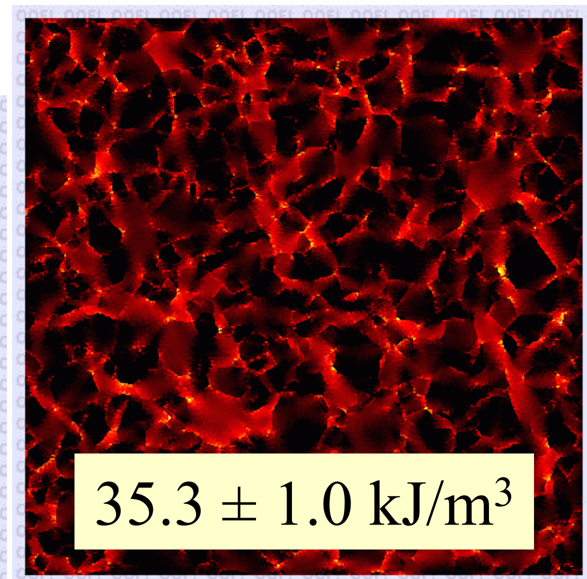


low-angle GB's



uniform

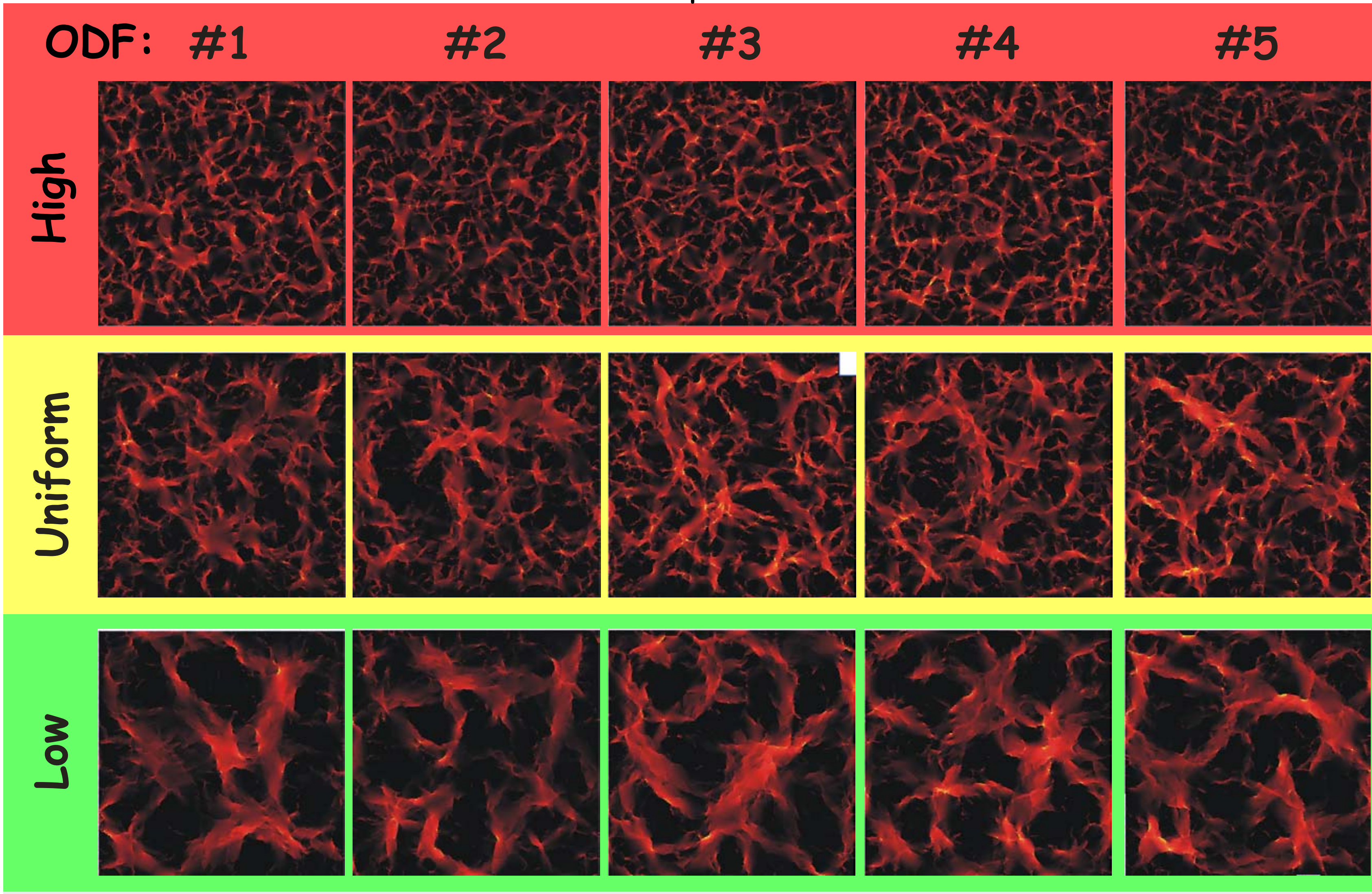




high-angle GB's

Calcite Results

5 replications



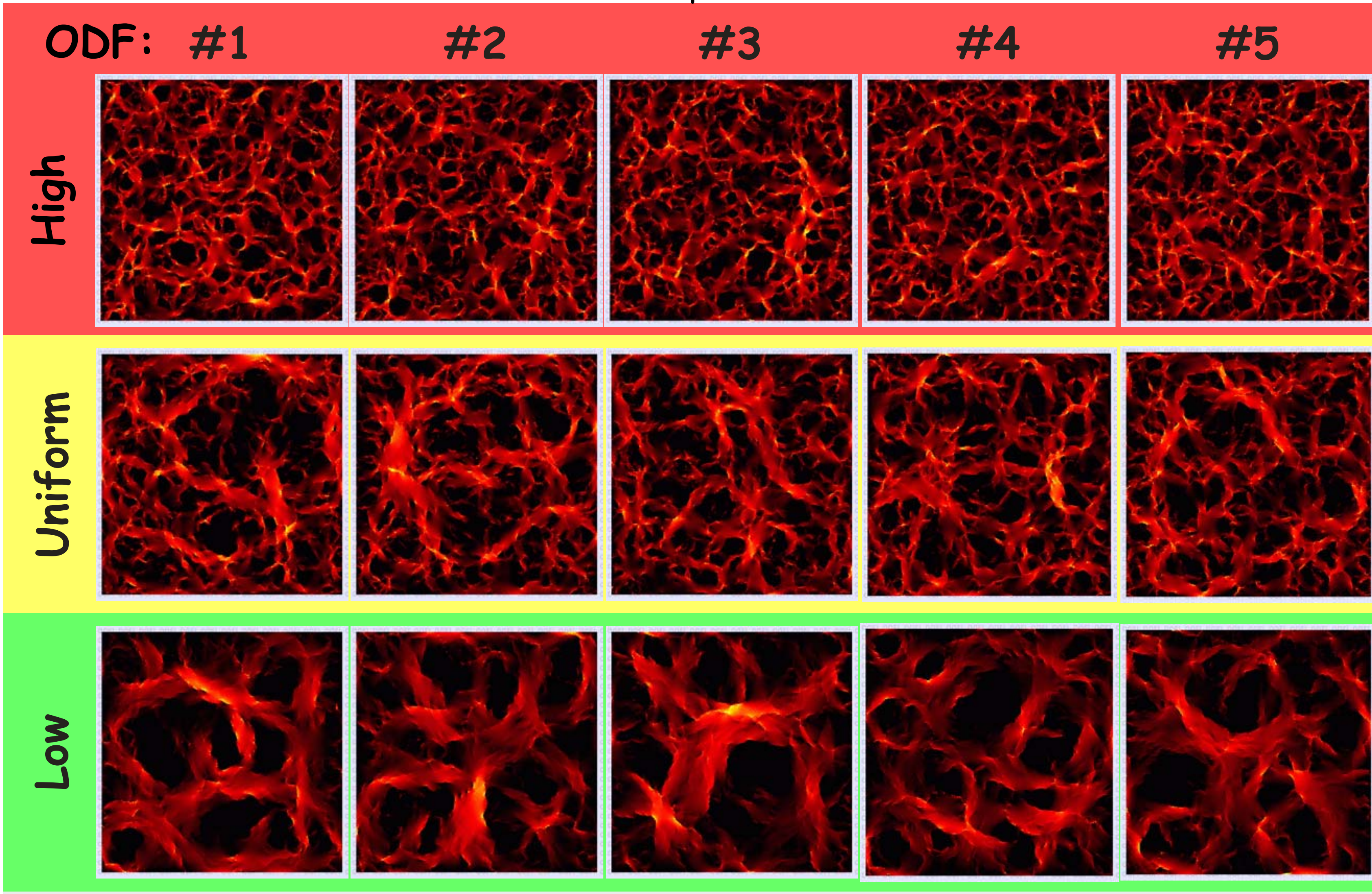
MDF

Maximum Principal Stress upon heating +100°C

for 5 random orientation distributions of ***calcite*** grains

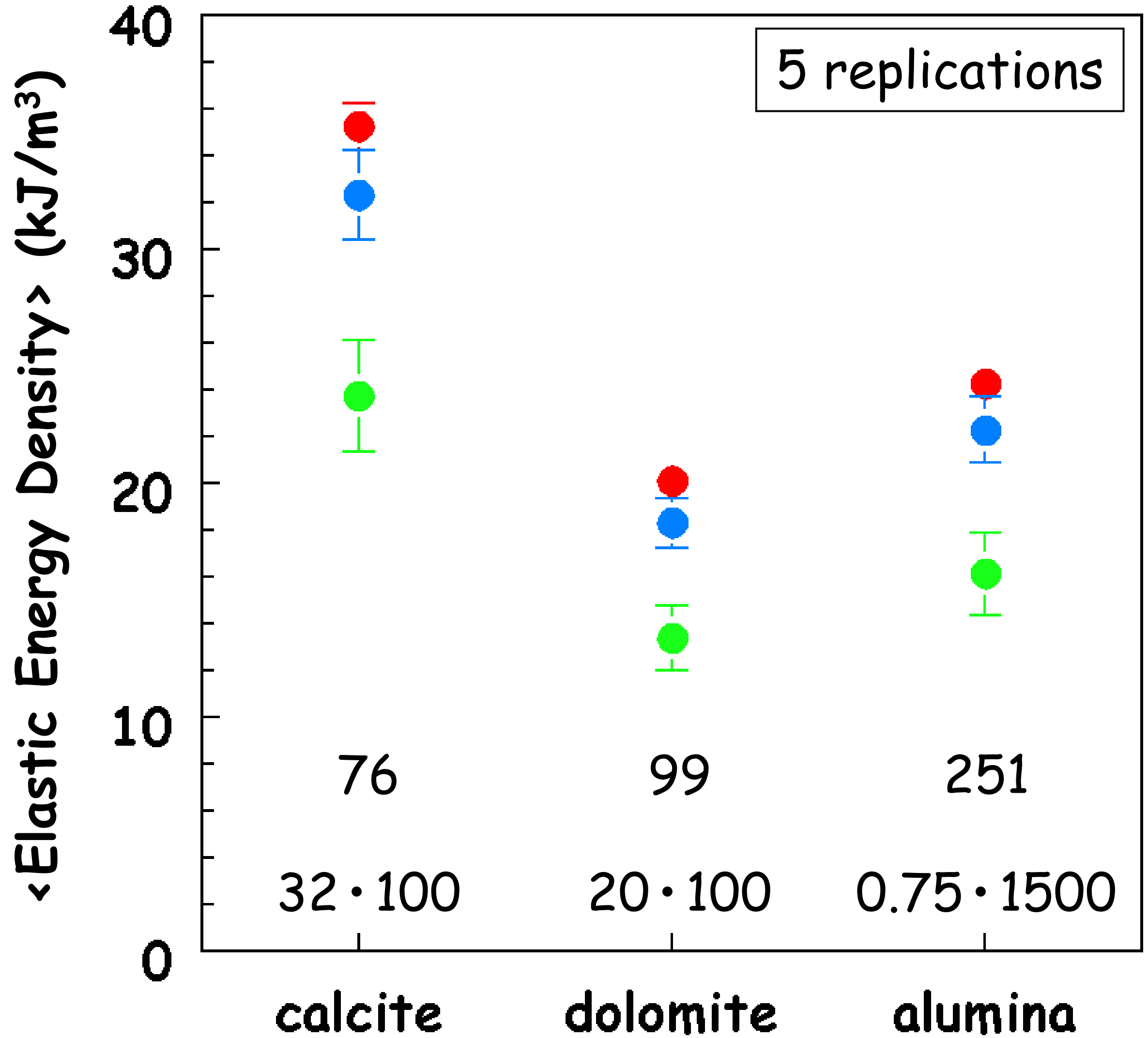
Alumina Results

5 replications



Maximum Principal Stress upon cooling 1500°C for 5 random ODF's orientation distributions of ***alumina*** grains

Elastic Strain Energy Density



ODF:
random
(MRD=1)

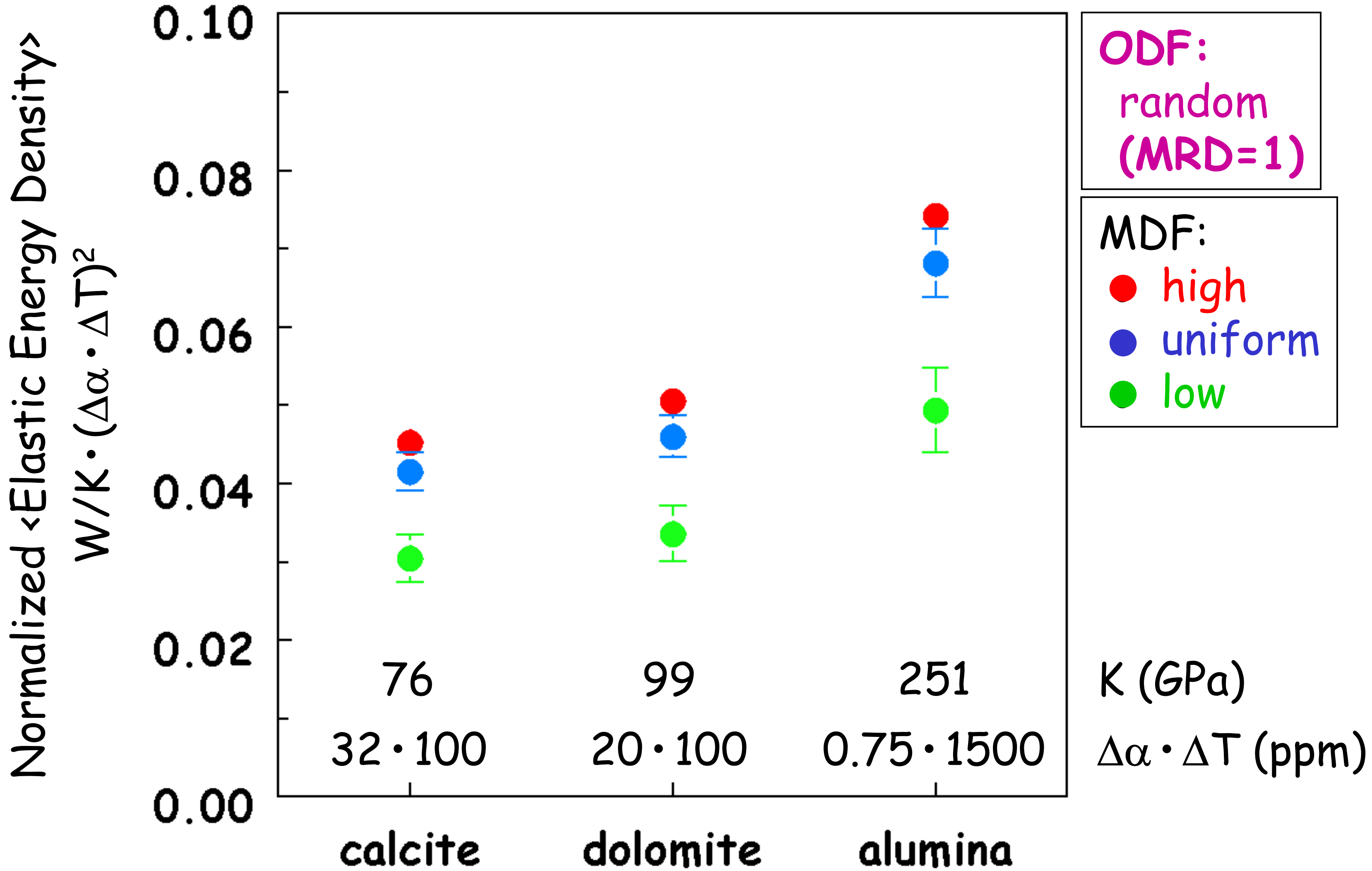
MDF:
- high
- uniform
- low

K (GPa)

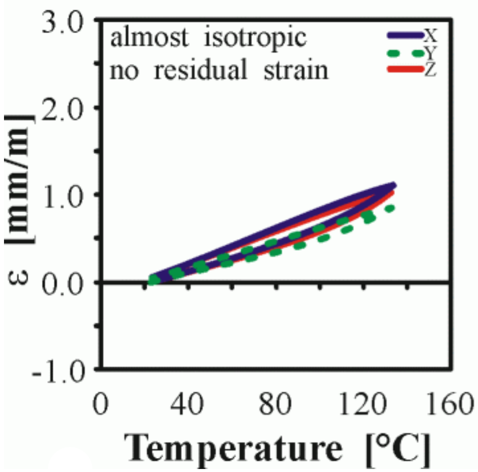
$\Delta\alpha \cdot \Delta T$ (ppm)

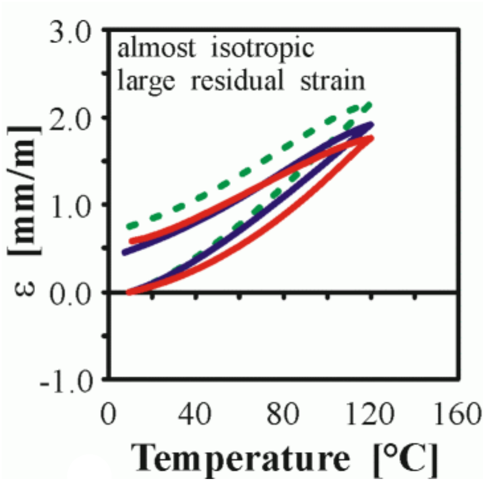
$\Delta\alpha = \alpha_3 - \alpha_1$

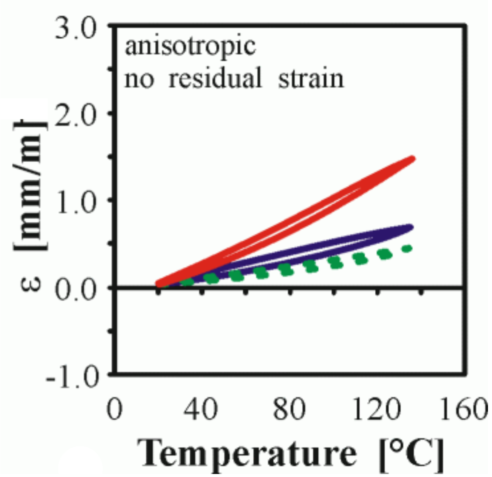
Normalized Elastic Energy Density

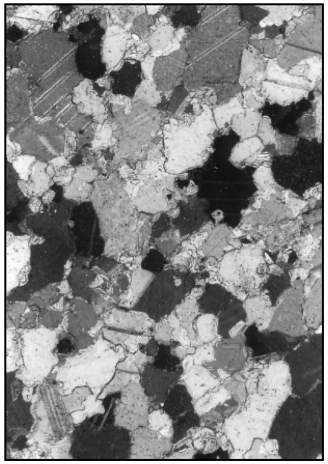


Types of Thermal Expansion









Carrara (Italy)

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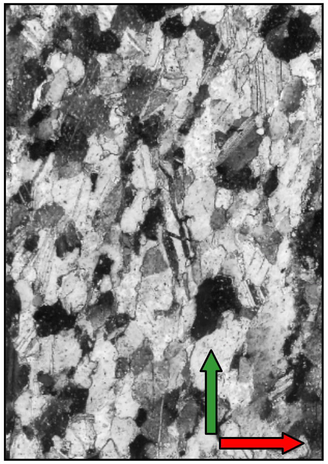
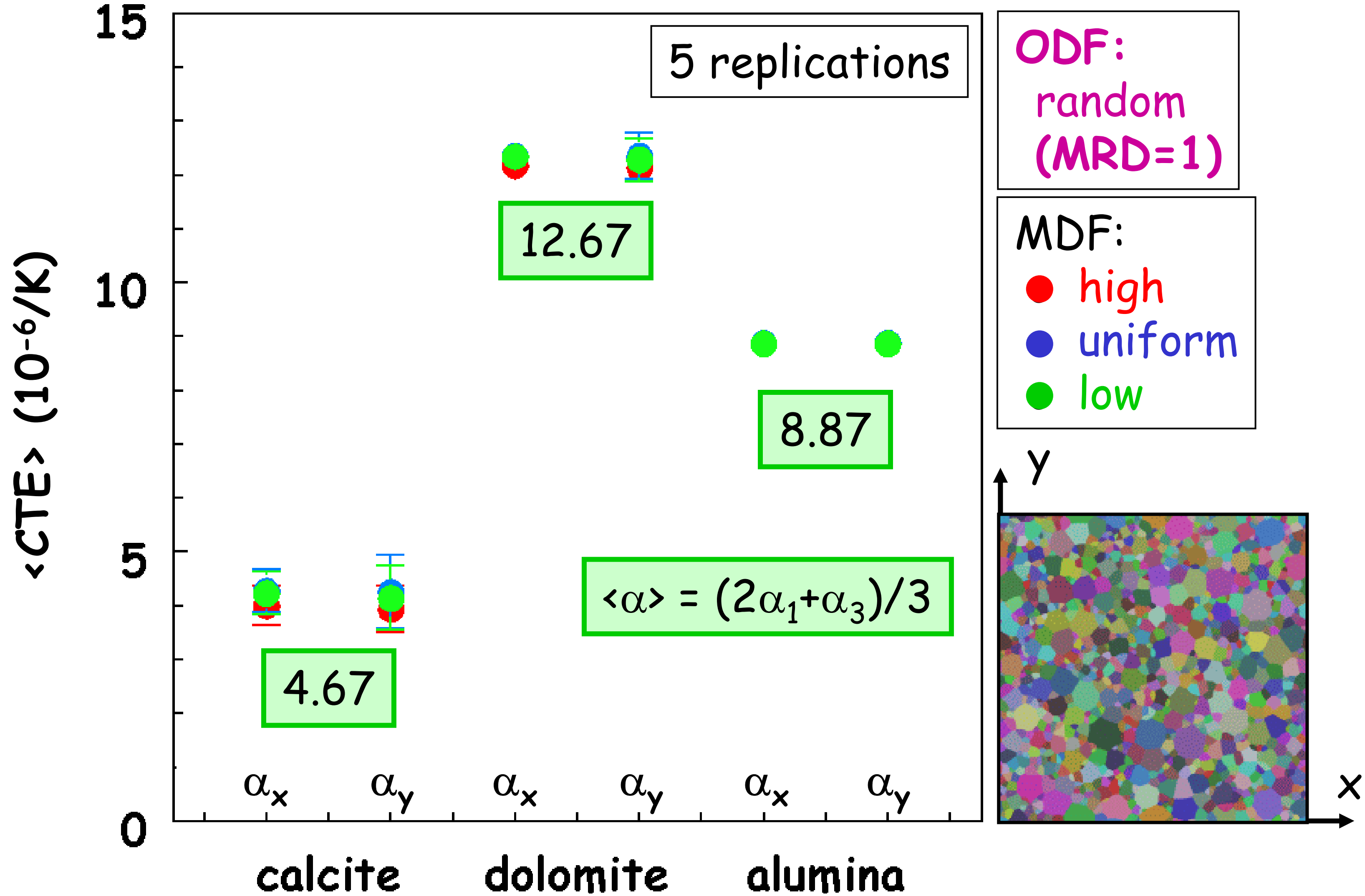
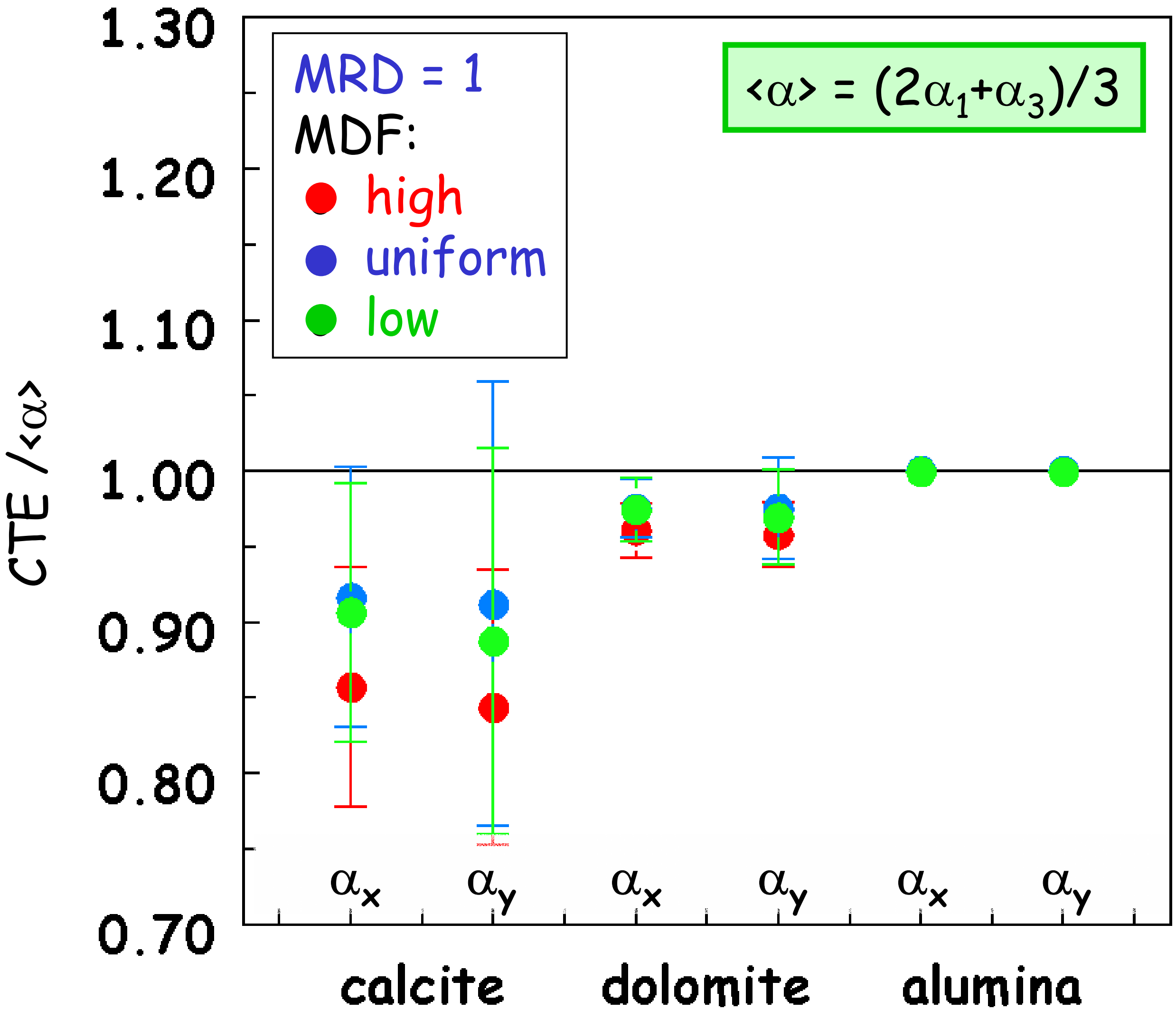


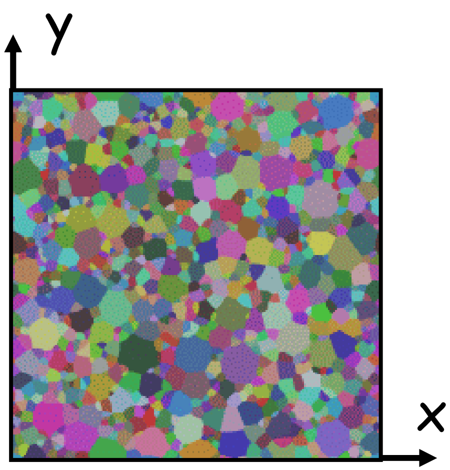
Kauffung (Poland)

Bulk Coefficient of Thermal Expansion



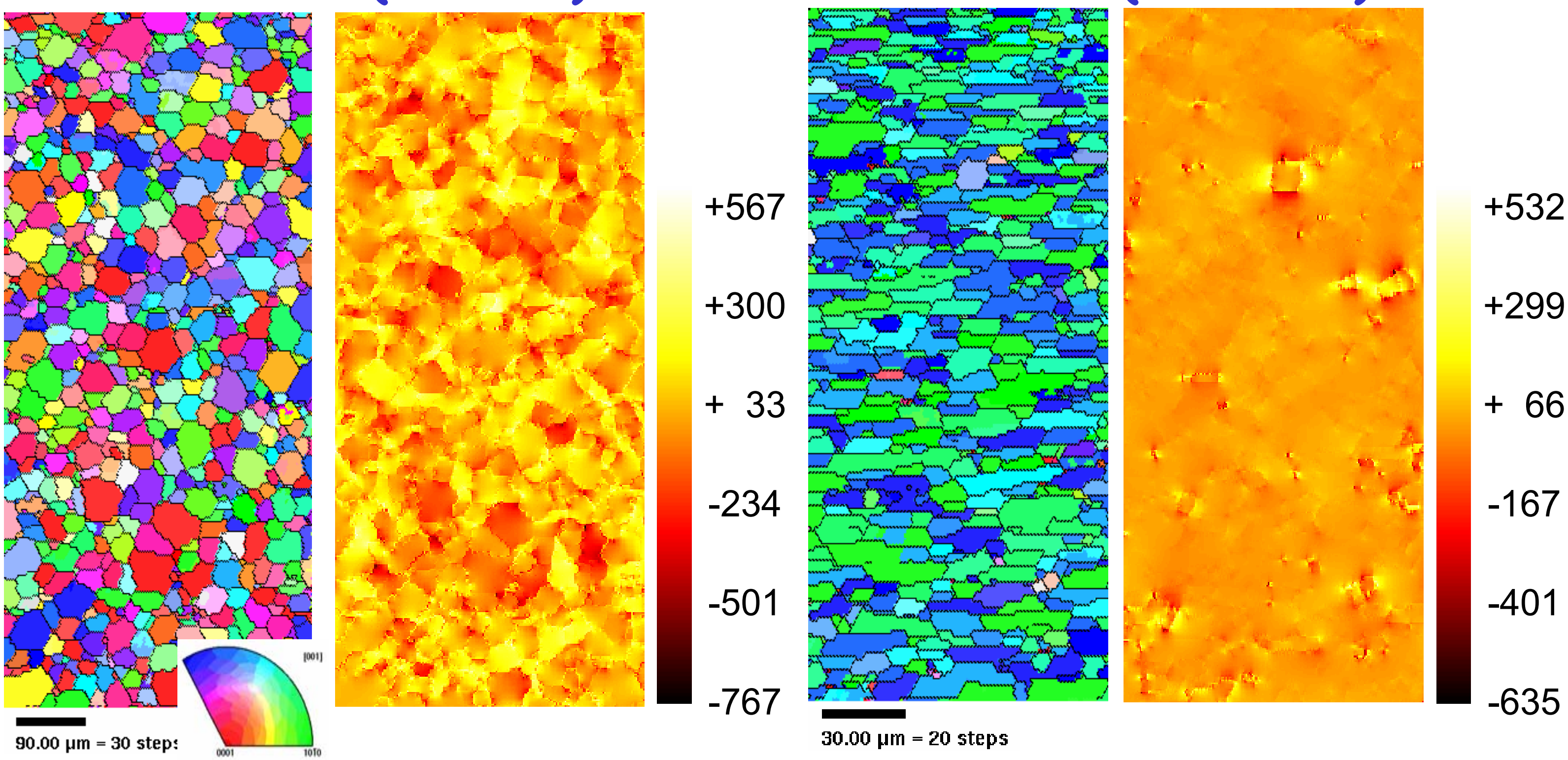
Normalized Bulk CTE





Residual Stresses in Alumina with Crystallographic Textured

Untextured (MRD=2) — Textured (MRD=90)



Hydrostatic Stress, $(\sigma_{11} + \sigma_{22})$, for $\Delta T = -1500\ °C$

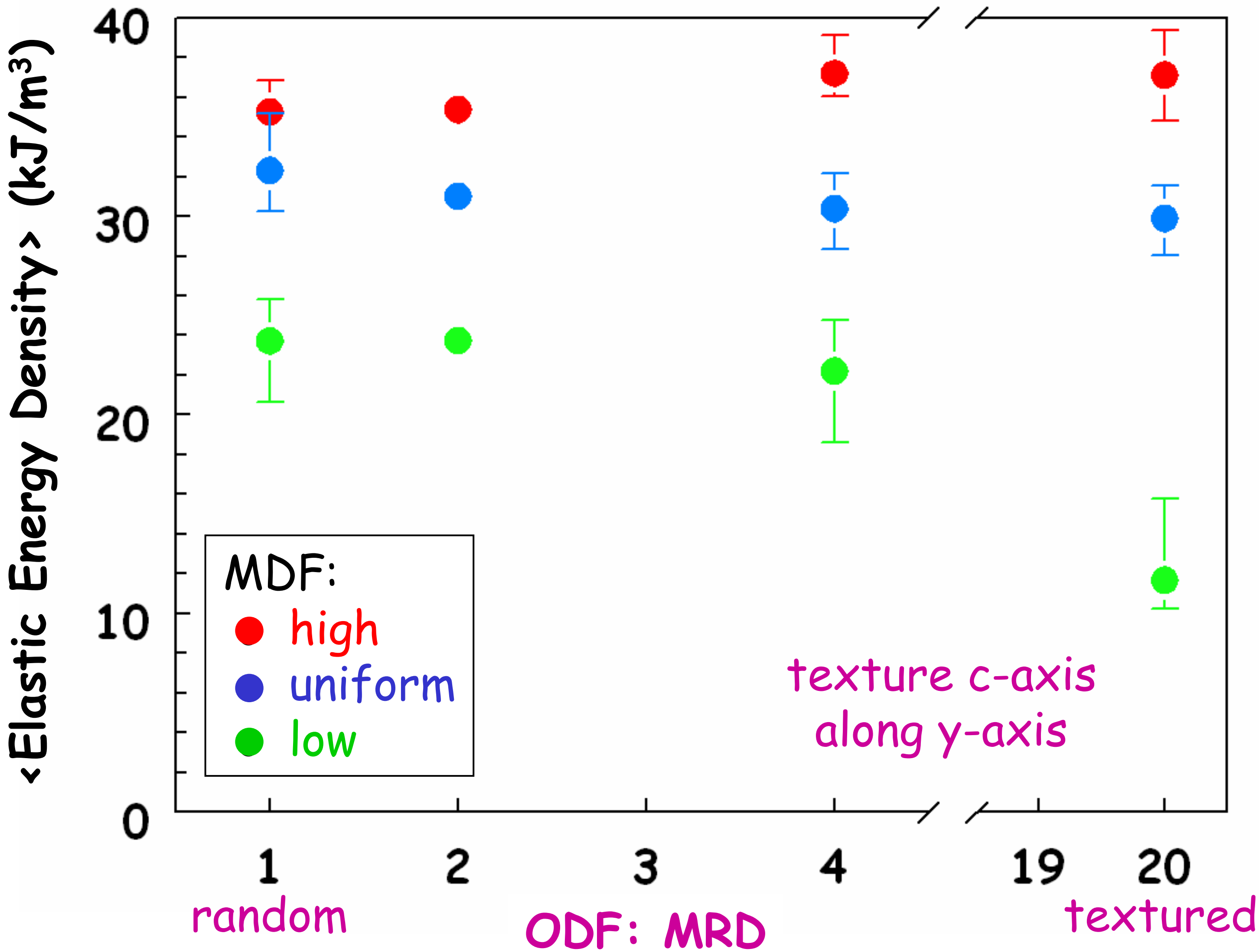
Venkata R. Vedula, Edwin R. Fuller, Jr. , and S. Jill Glass

National Institute of Standards and Technology

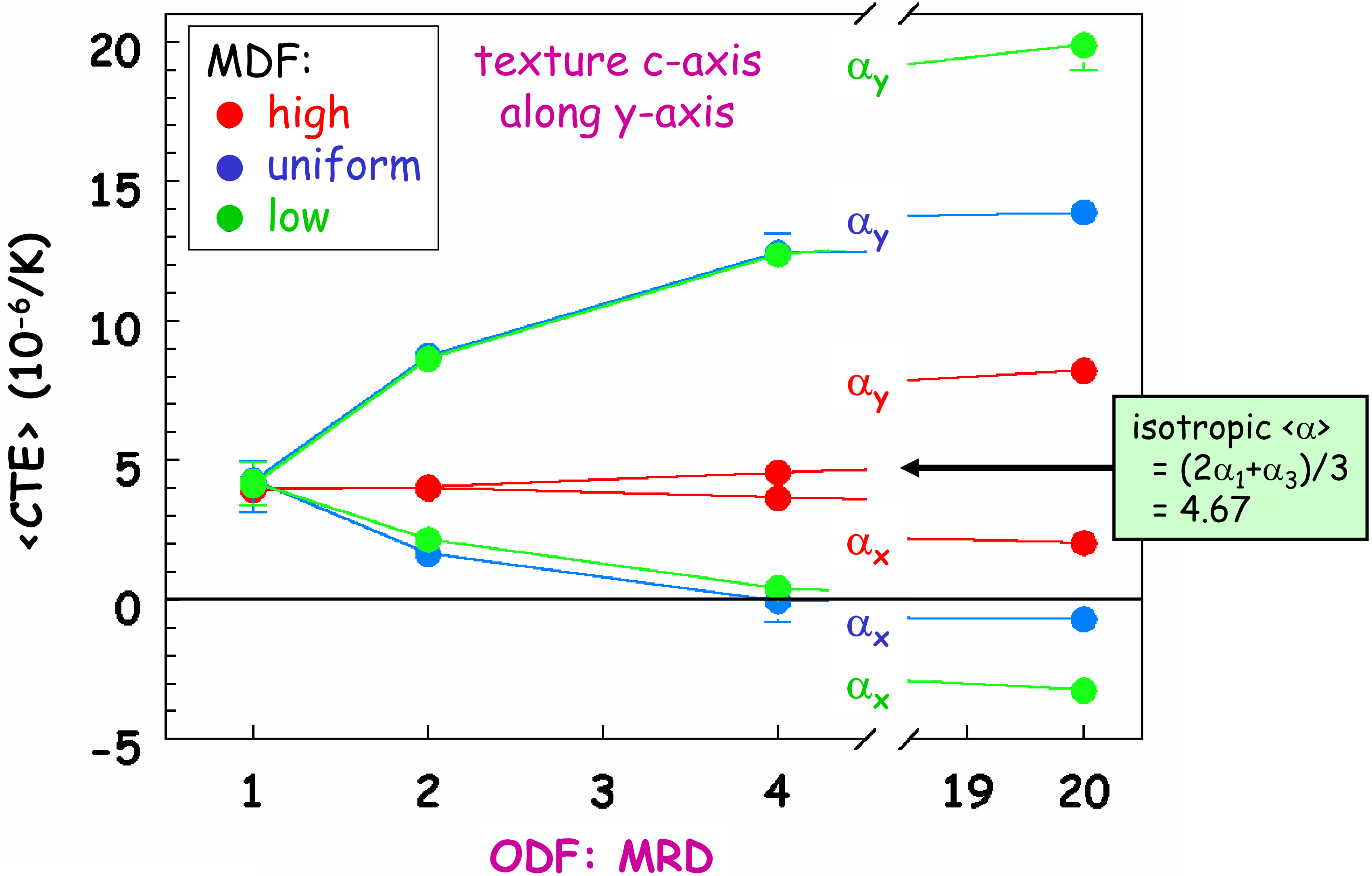
Ceramics Division

Elastic Strain Energy Density

for textured calcite heated +100°C



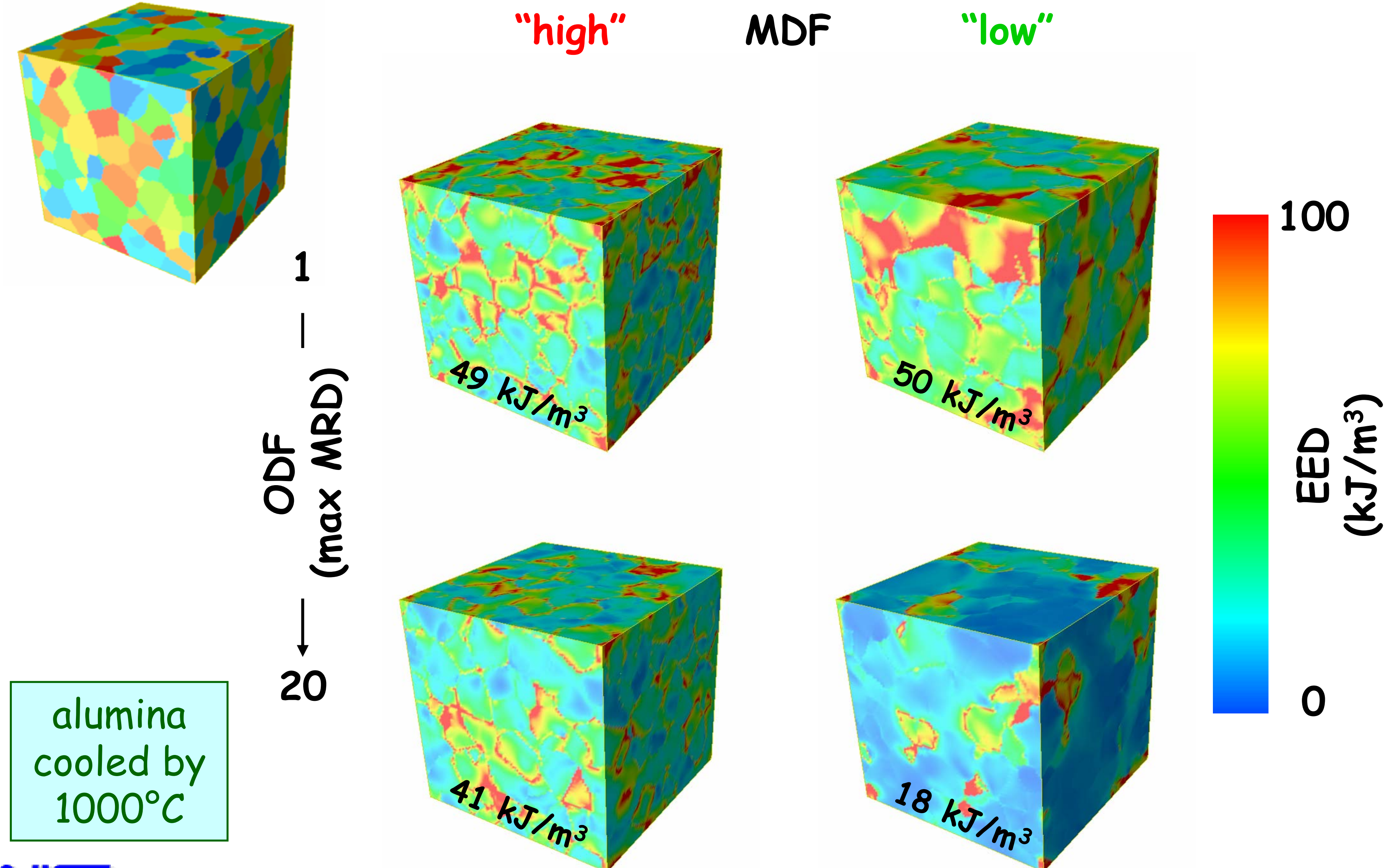
Bulk Thermal Expansion Anisotropy
for textured calcite heated +100°C



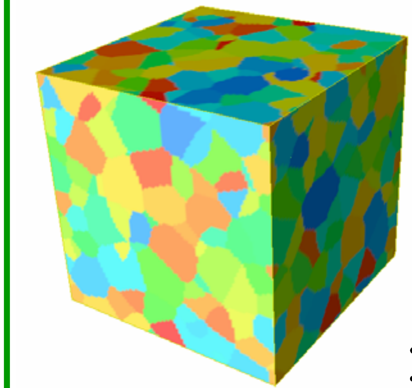
NIST National Institute of Standards and Technology

Ceramics Division

3-D Simulations: Elastic Energy Density



3-D Simulations: Elastic Energy Density

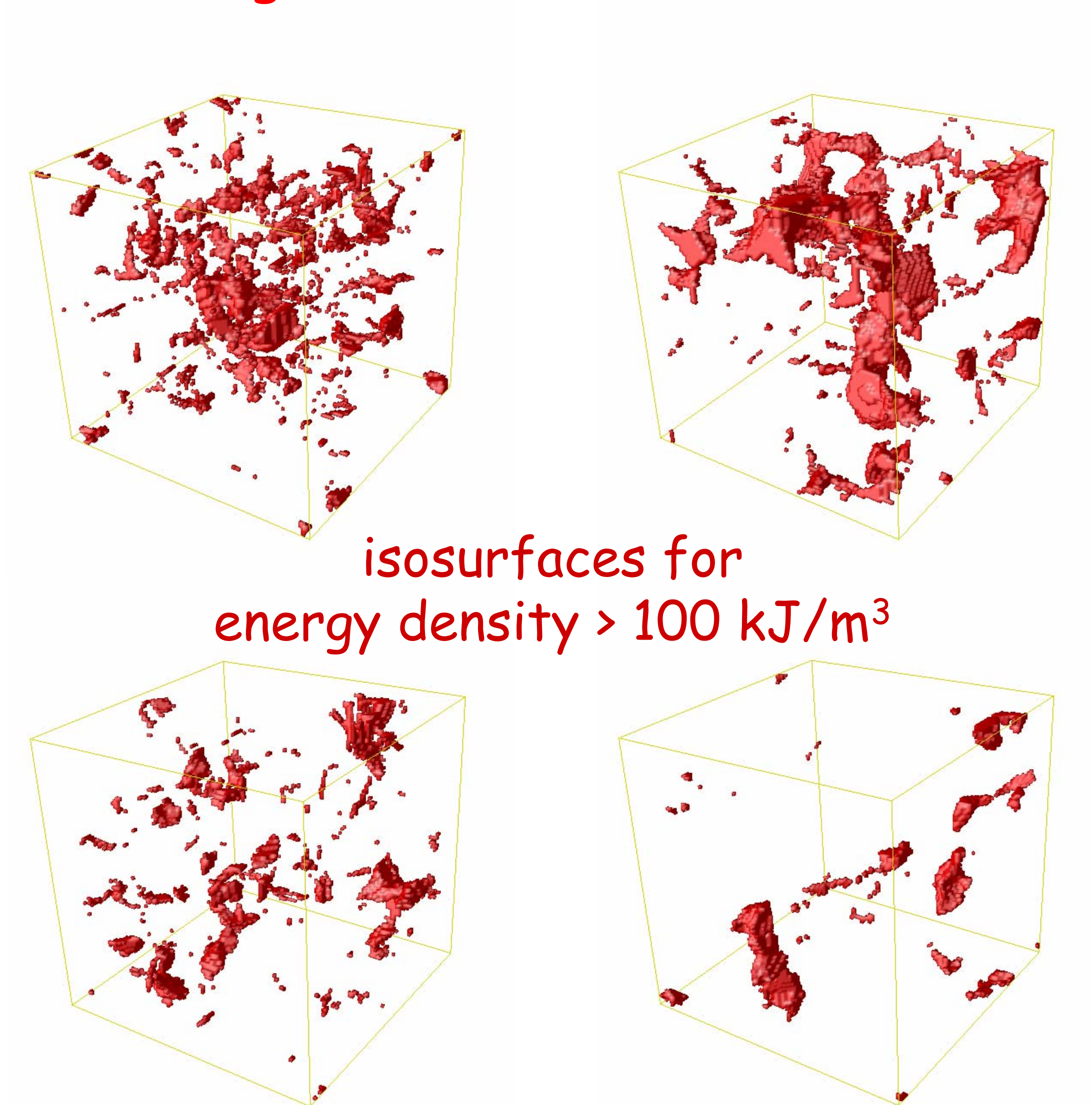


"high" MDF "low"

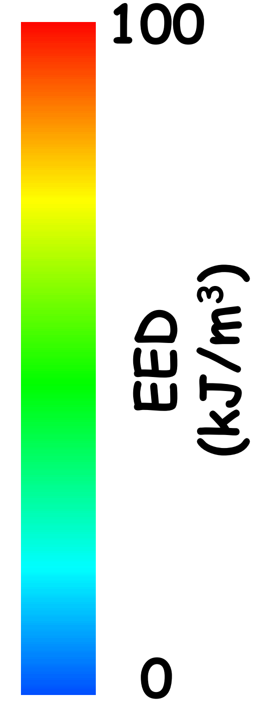
1

ODF (max MRD)

20



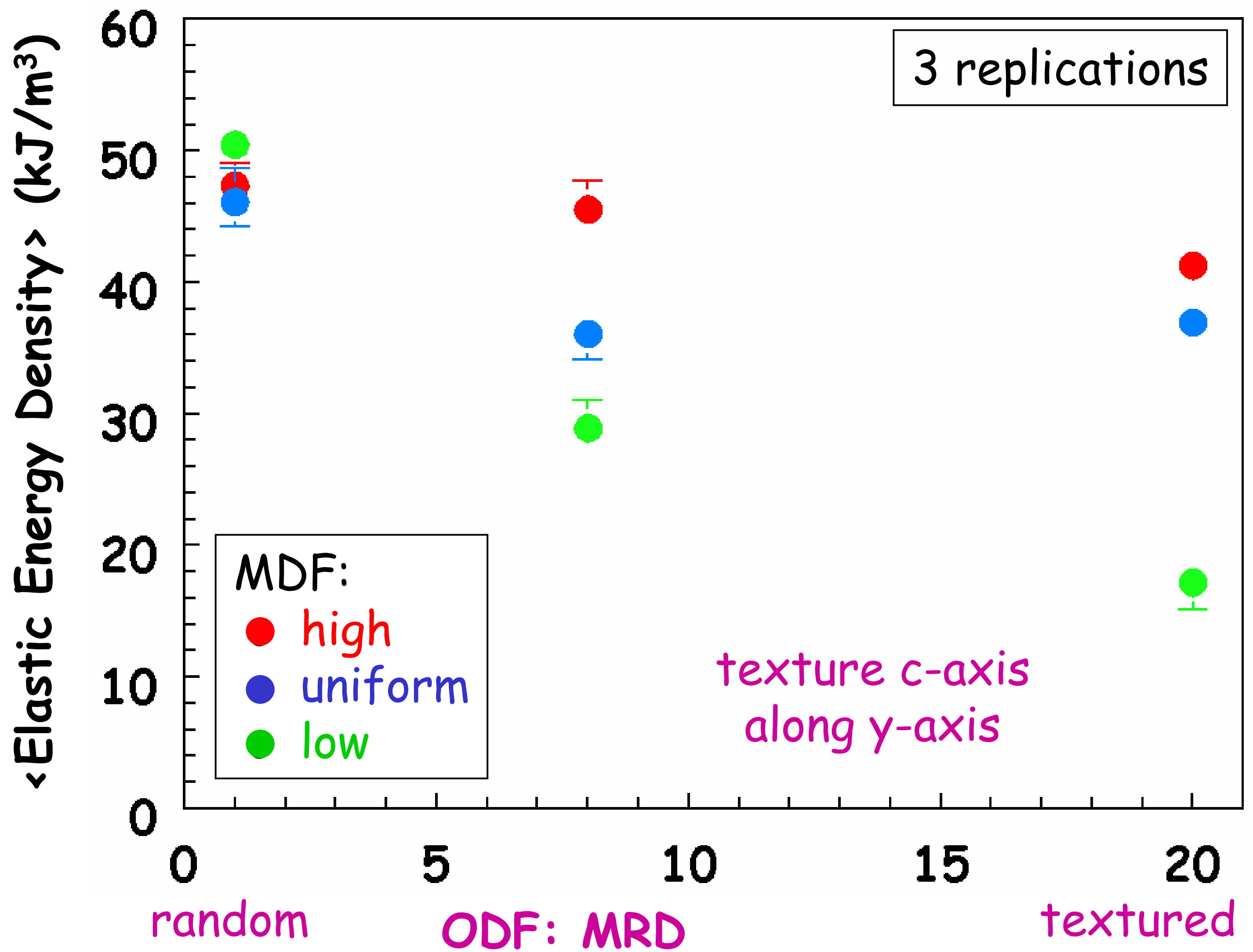
isosurfaces for energy density > 100 kJ/m^3



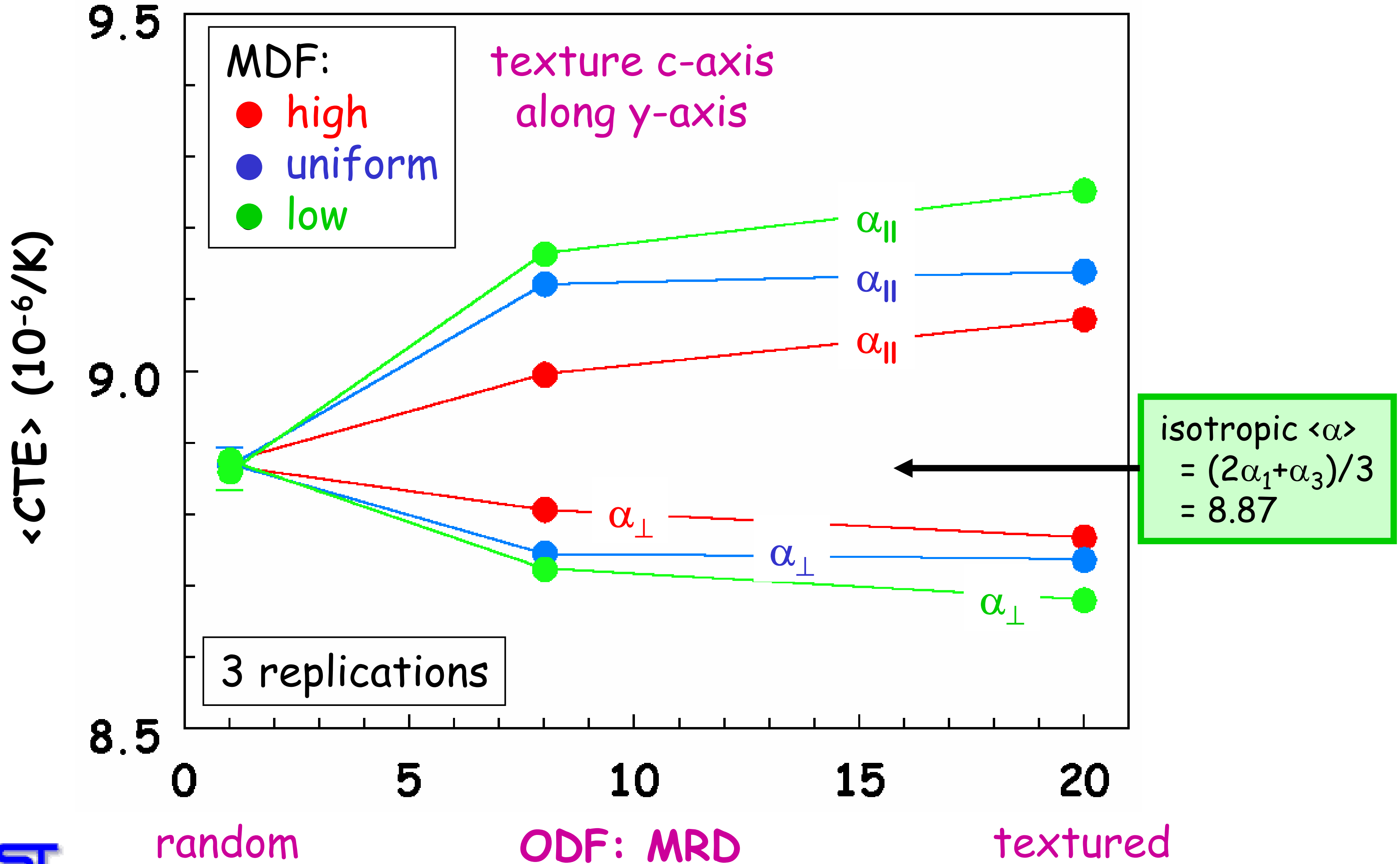
alumina cooled by 1000°C

Elastic Strain Energy Density

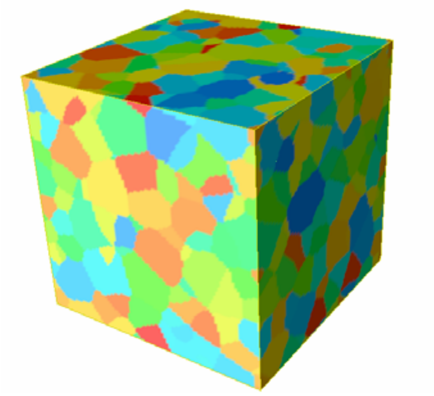
for textured alumina cooled 1000°C



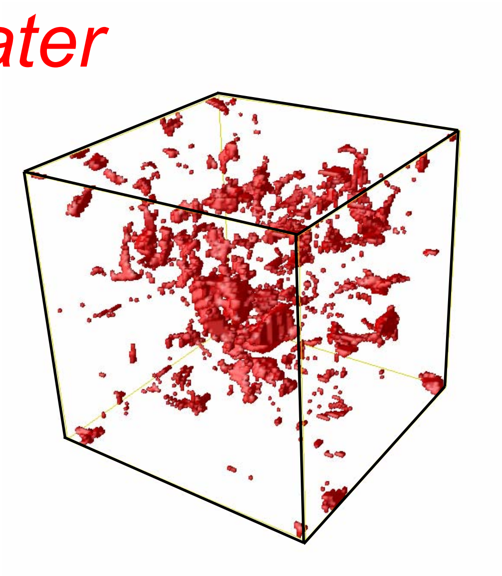
Bulk Thermal Expansion Anisotropy
for textured alumina cooled 1000°C



Residual-Stress Isosurfaces

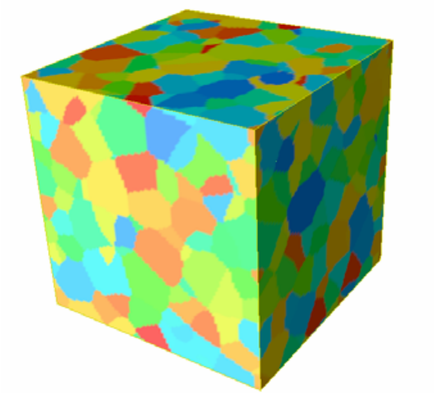


e.g., isosurface of elastic energy densities greater than 100 kJ/m^3



What is a good metric for characterizing residual-stress isosurfaces?

Residual-Stress Isosurfaces



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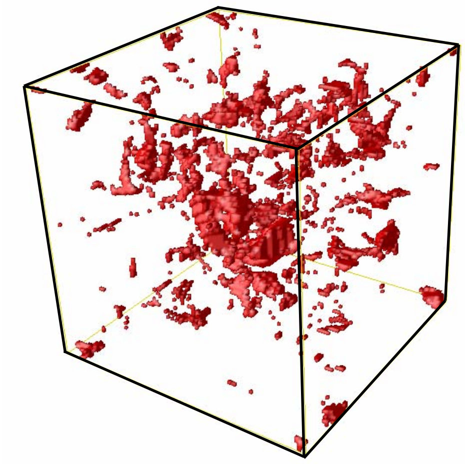


Thomas Wanner, *George Mason University, proposed the use of:*

homology groups *and* ***topological invariants*** — measure the topological complexity of objects in any dimension.

Computational Algebraic Topology

Algebraic Topology:
a branch of mathematics, in which tools from abstract algebra are used to study topological spaces.

Challenge in Computational Algebraic Topology:
Theoretically, determination of homology groups and topological invariants is straightforward. Computationally, such determination may be intractable or infeasible for large data sets.

CHomP (Computational Homology Program): pixel/voxel oriented public-domain software for computing algebraic topological invariants.

Associated New Textbook: ***Computational Homology*** by Tomasz Kaczynski, Konstantin Mischaikow , and Marian Mrozek, (Applied Mathematical Sciences , Vol. **157**, Springer-Verlag, 2004).

Topological Invariants of Space

… ***invariant under transformations*** that do not require cutting or gluing of the object.

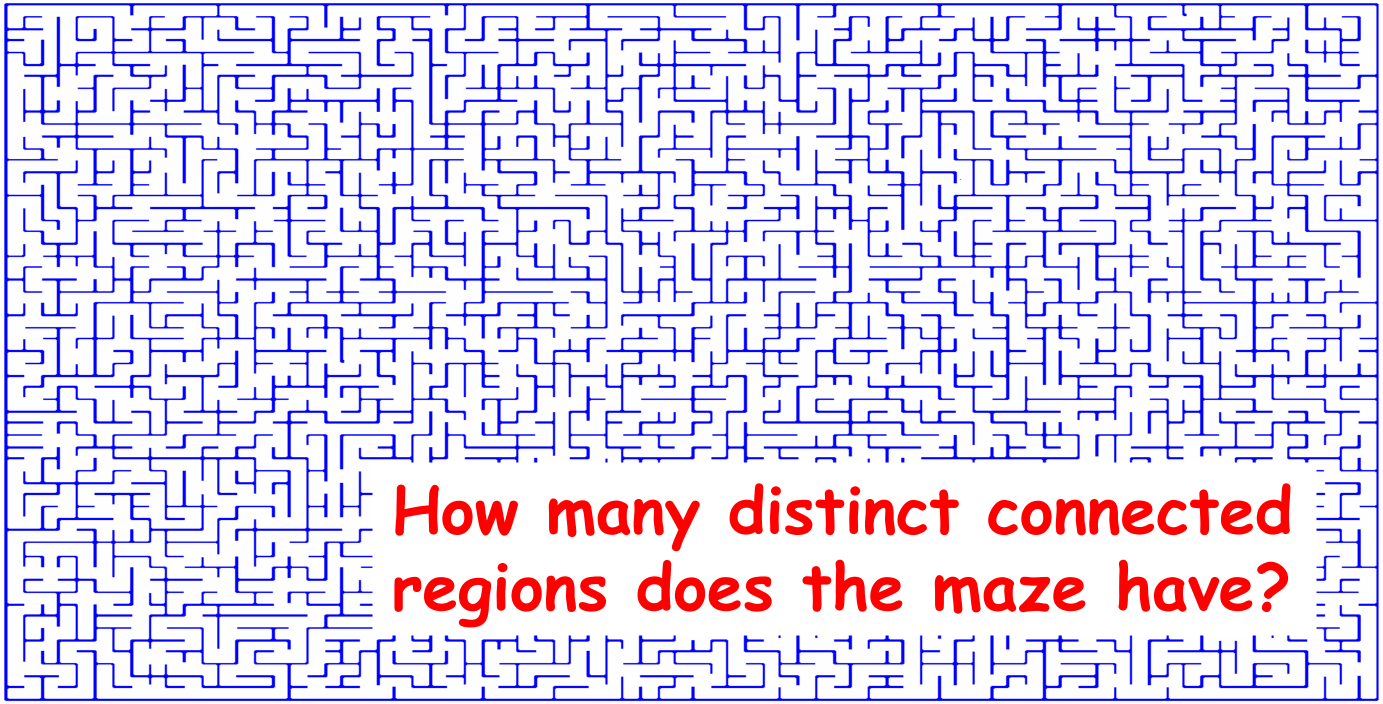
Topological Invariants of a Complex Object

- ***Betti numbers***, named for Enrico Betti, are a sequence β_0, β_1, ... of topological invariants, which are natural numbers, or infinity.
- Other invariants include: ***torsion coefficients*** and the ***Euler characteristic***.

Homology groups: a more general measure of the complexity of the object in any dimension.

Zeroth Betti Numbers

Betti number β_0 counts the ***number of connected components*** of the structure



Computational Homology, Applied Mathematical Sciences , Vol. 157, Springer-Verlag, 2004).

Zeroth Betti Numbers

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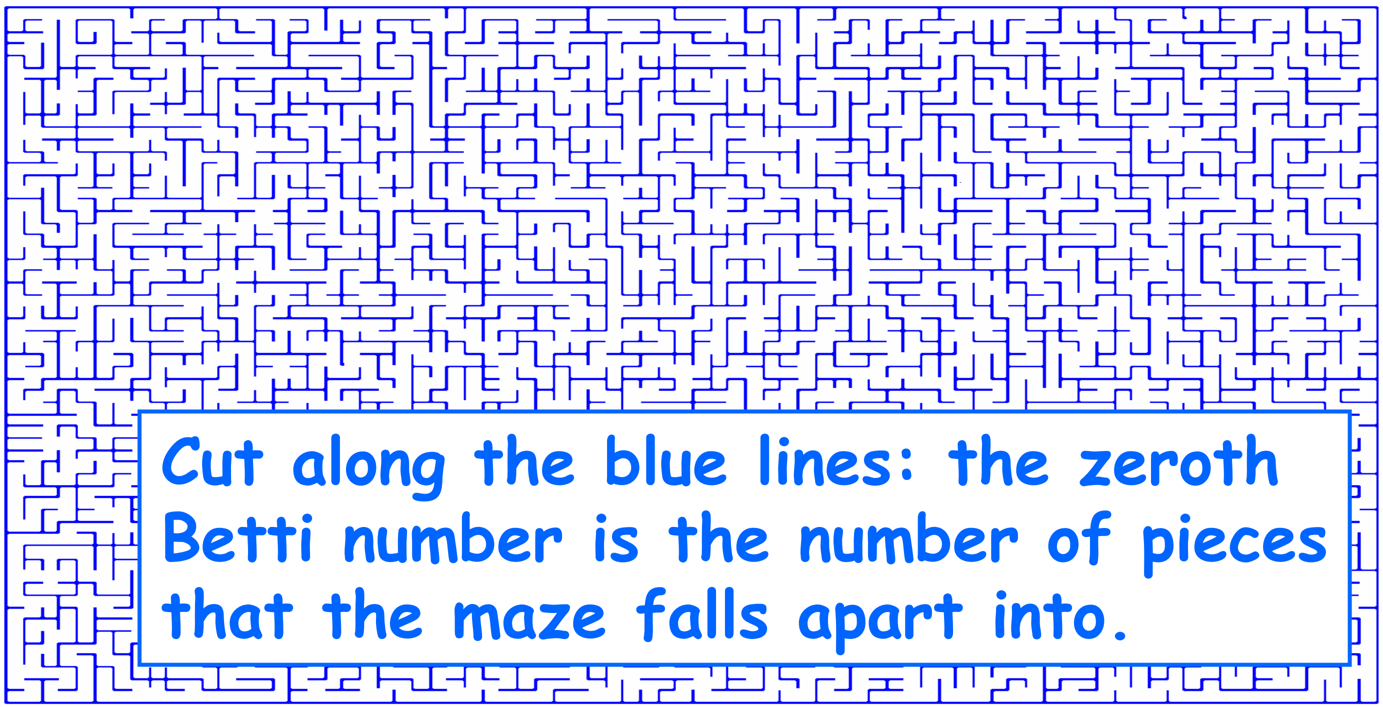


Computational Homology, Applied Mathematical Sciences , Vol. 157, Springer-Verlag, 2004).

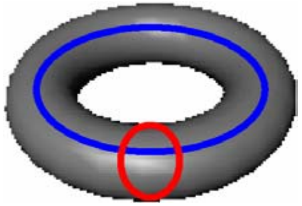
First Betti Number

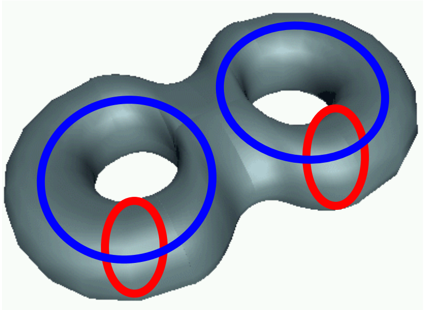
Betti number β_1 counts the ***number of independent tunnels*** created by the structure: the ***number of loops*** in the structure that cannot be

- shrunk to a point, or
- morphed into each other.

$\beta_1 = 0$

$\beta_1 = 2$

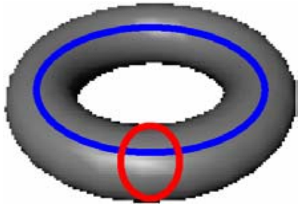




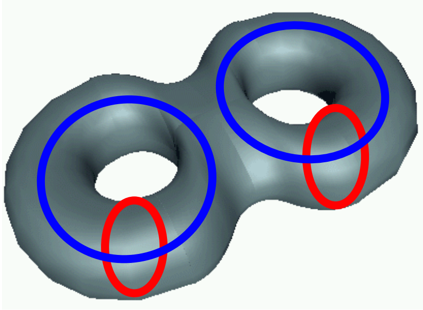
$\beta_1 = 4$

Second Betti Number

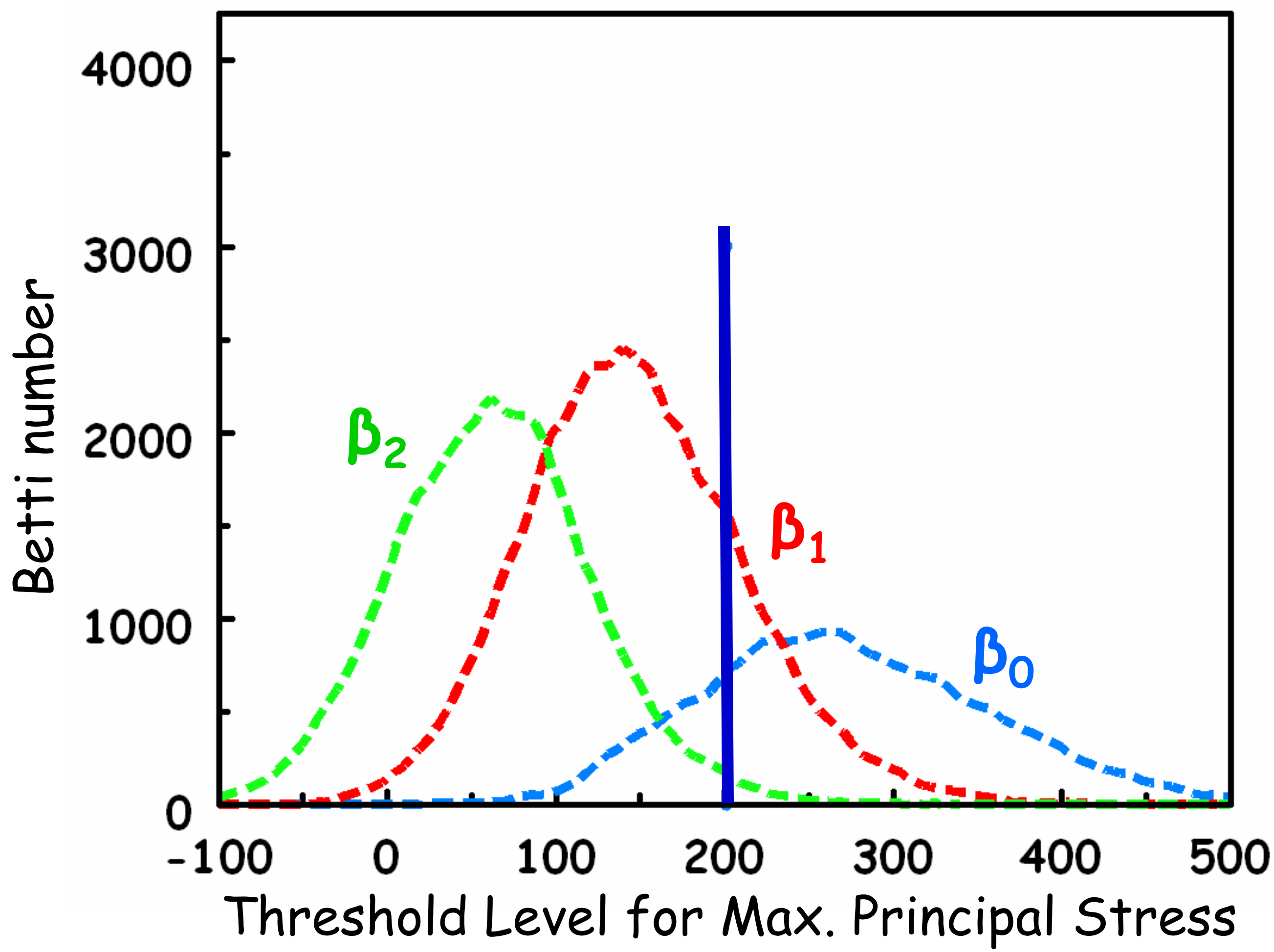
Betti number β_2 counts the ***number of closed regions*** created by the structure.

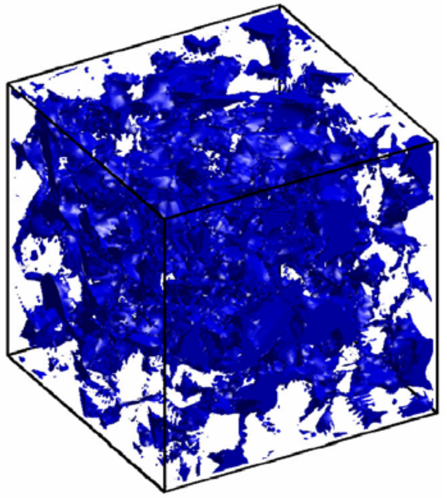


all surfaces have $\beta_2 = 1$



Max. Principal Stress Networks



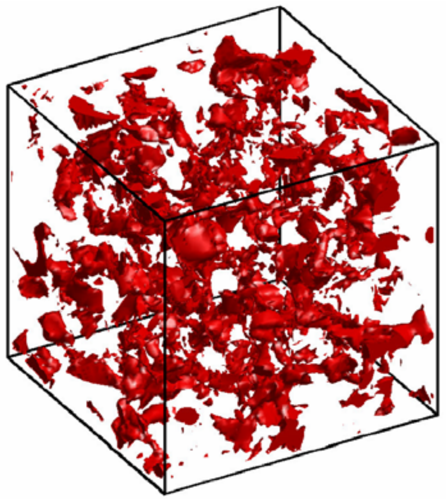


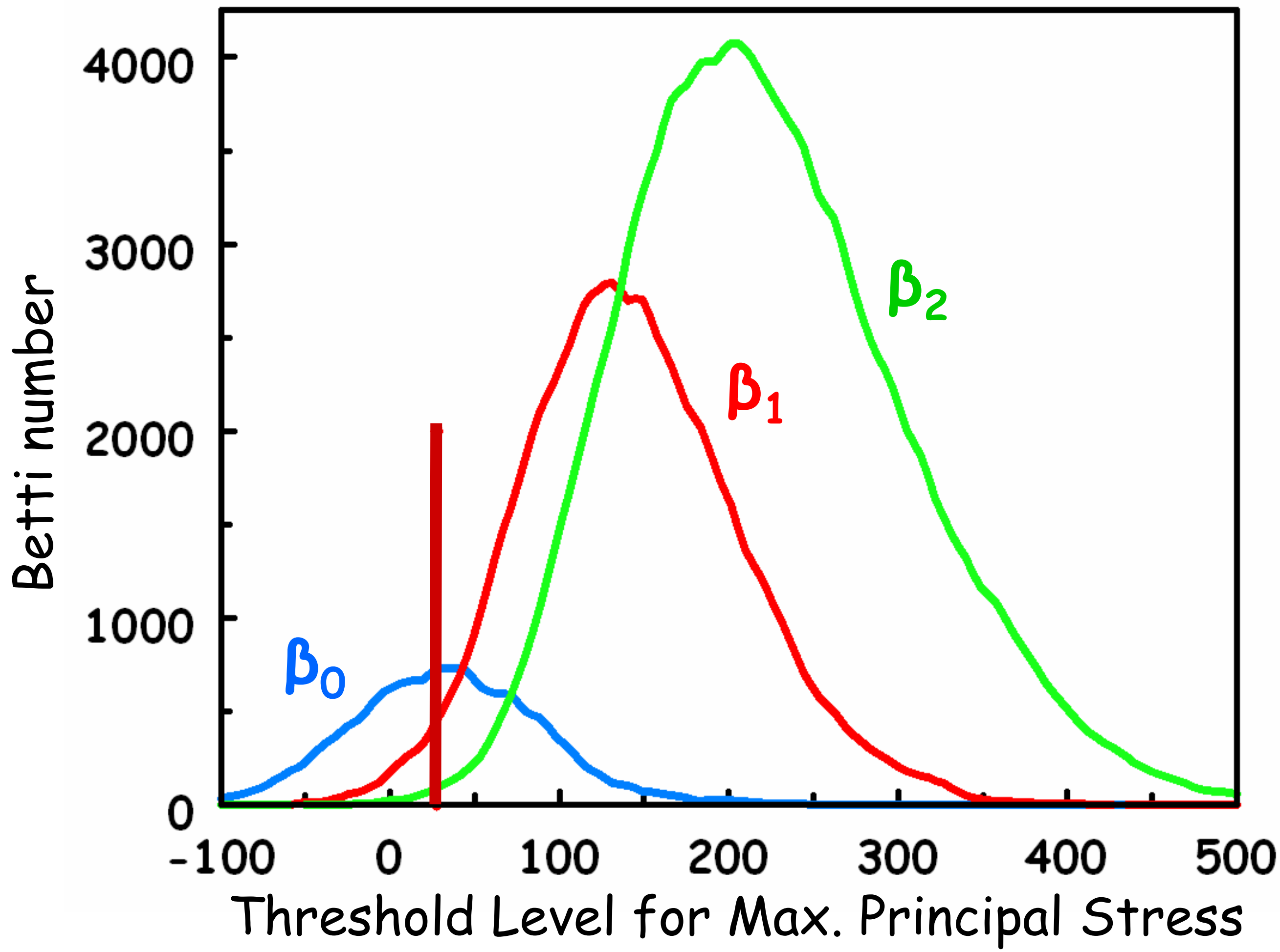
isosurface for σ_1 > 201 MPa

Thomas Wanner, *George Mason University*
Konstantin Mischaikow, *Georgia Institute of Technology*

Max. Principal Stress Networks

isosurface for
$\sigma_1 < 27.5$ MPa





Thomas Wanner, *George Mason University*
Konstantin Mischaikow, *Georgia Institute of Technology*

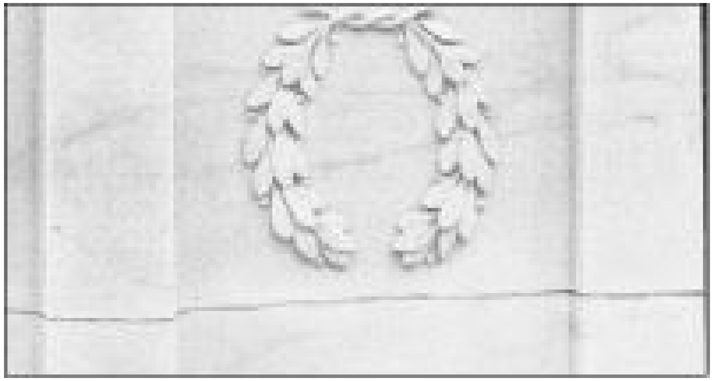
Microstructural Stresses

Contents:

- Residual-Stress Networks
  in Polycrystalline Ceramics
- Microstructural Finite-Element Analysis
- Statistical Description
  of Microstructure and Texture
- Residual Stress and
  Physical Property Simulations
- The *Old* and the *New*

The Tomb of the Unknowns

Made from a 55-ton block of white marble from the Yule Marble Quarry located near Marble, Colorado, the monument was dedicated in 1932.



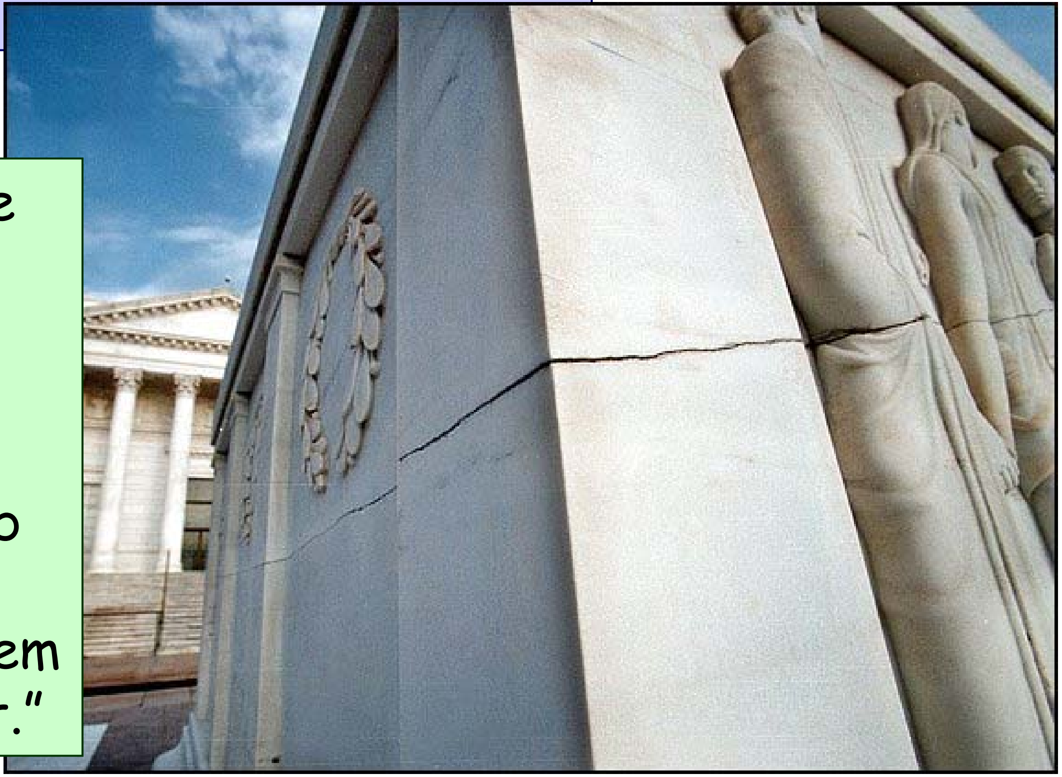
A crack, first discovered in the 1940's during the Truman administration, has continued to grow.

Unknowns Monument Will Be Replaced

By Annie Gowen, The Washington Post, Monday, May 26, 2003, B01

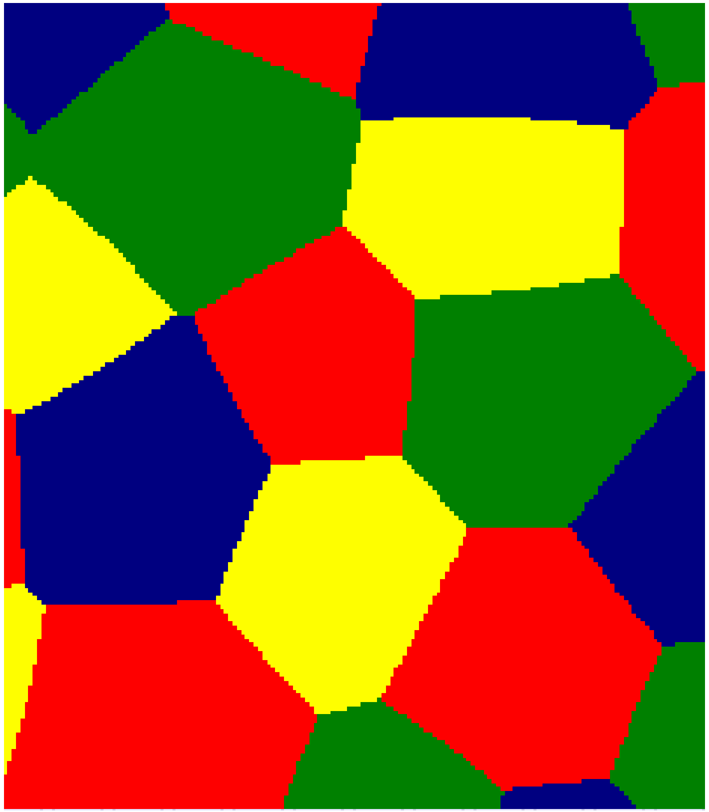
"The growing crack that now circles the marble monument has split the three figures representing Peace, Victory and Valor."

"Officials decided to replace the stone after concluding that a 1989 cosmetic repair job — which cemetery historian Thomas Sherlock compared to fixing a bathtub with tile grout — had done nothing to conceal the problem and may have exacerbated it."



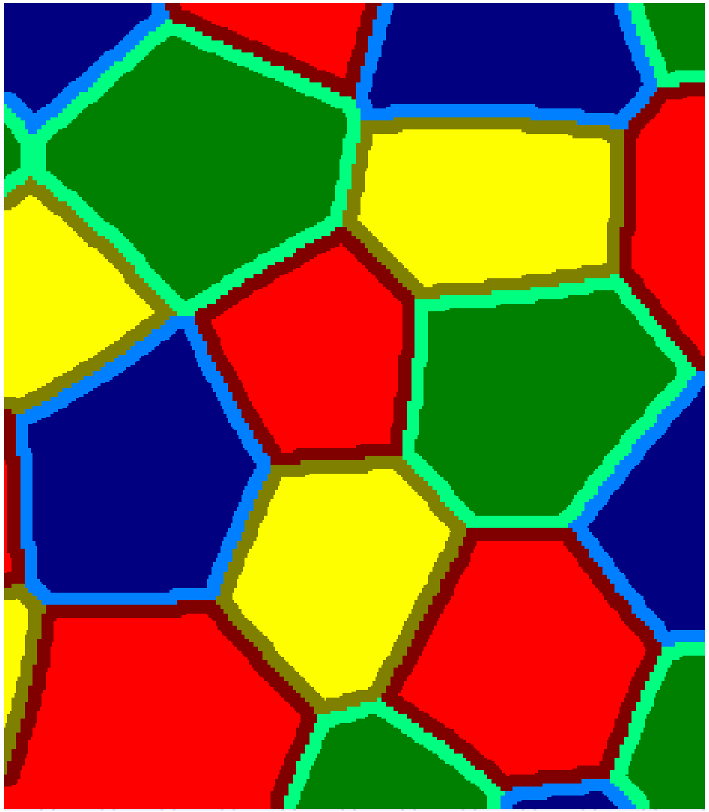
(James A. Parcell - The Washington Post)

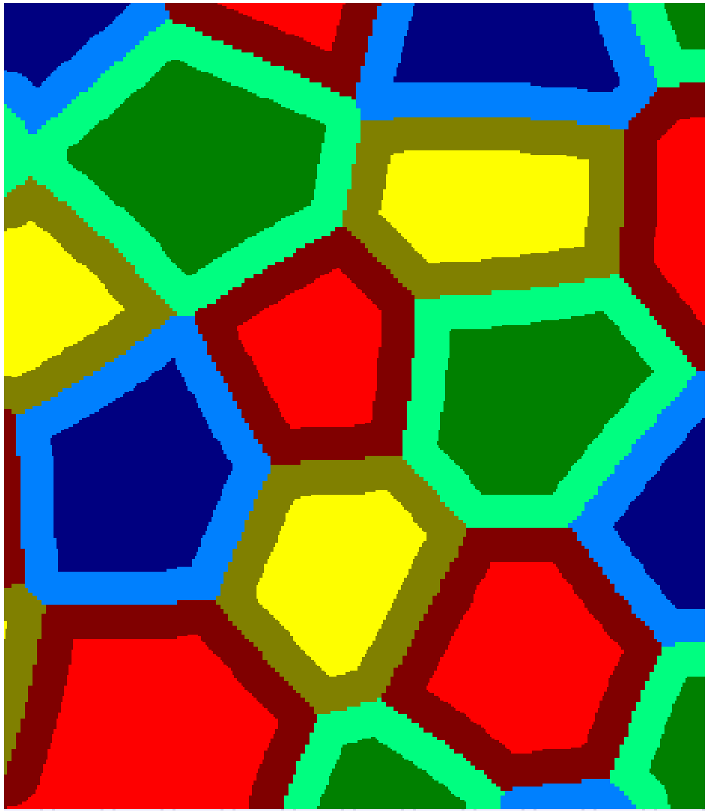
Residual Stresses in Nanostructured Ceramics



Microstresses are independent of grain size

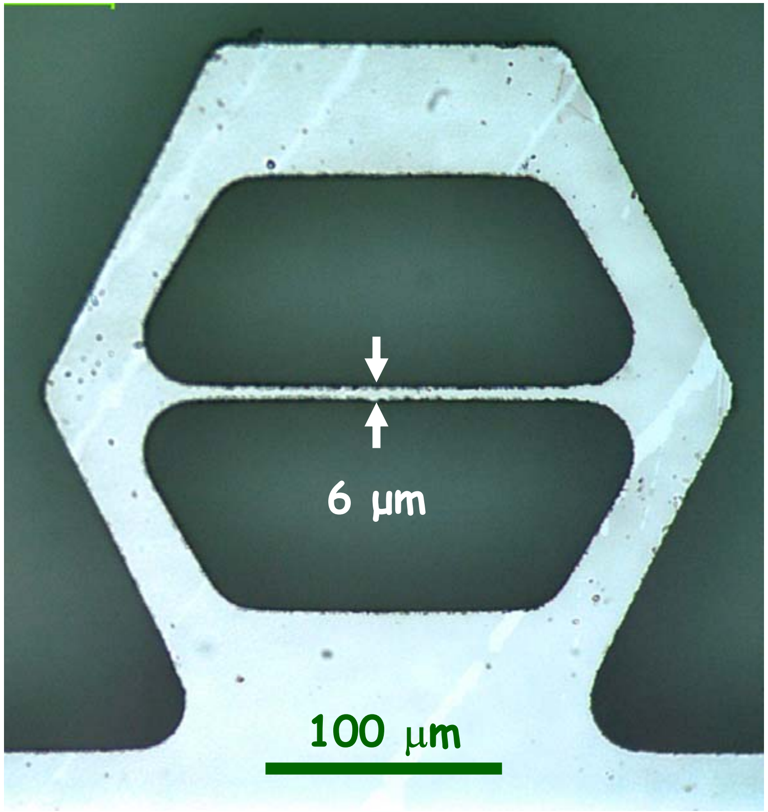
Residual Stresses in Nanostructured Ceramics

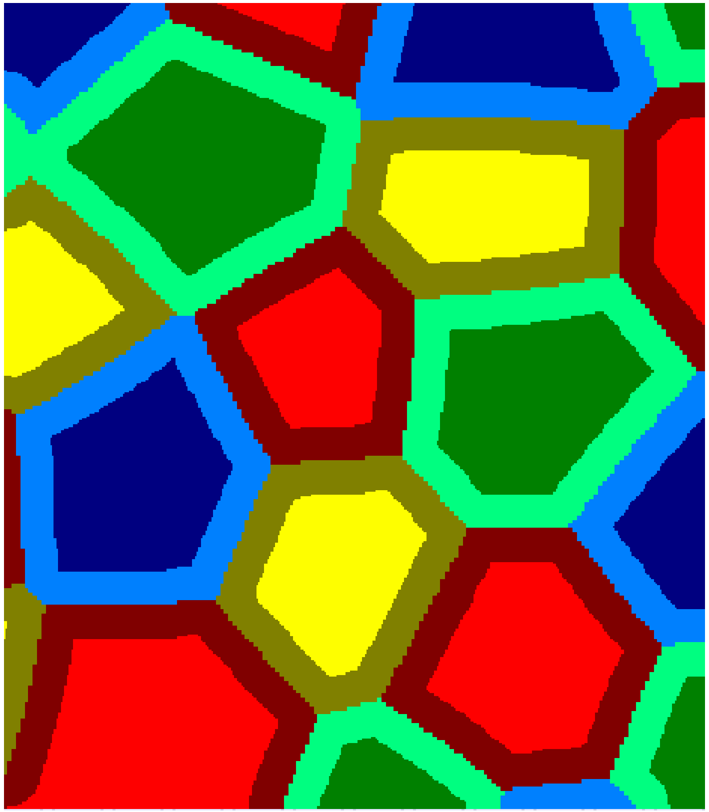




new phenomena (physics) at the small-scale?
e.g., surface stresses: $\sigma_{ij} = \gamma\,\delta_{ij} + (\partial\gamma/\partial\varepsilon_{ij})$

Residual Stresses in Nanostructured Ceramics





new phenomena (physics) at the small-scale?
e.g., surface stresses: $\sigma_{ij} = \gamma\, \delta_{ij} + (\partial\gamma/\partial\varepsilon_{ij})$

Collaborators & Acknowledgments

- Stephen A. Langer, Information Tech. Lab, NIST
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- Edward Lin, Montgomery Blair High School
- Thomas Weiß & Siegfried Siegesmund, Geowissenschaftliches Zentrum der Universität Göttingen, Germany
- Thomas Wanner, George Mason University
- André Zimmermann, Bosch GmbH, Germany
- Susan Galal Yousef & Jürgen Rödel, Technische Universität Darmstadt, Germany
- Venkata R. Vedula, Shekhar Kamat, Raj Tandon, Saundra Monroe, S. Jill Glass & Clay Newton, Sandia Natl. Labs
- Chang-Soo Kim & Gregory S. Rohrer, Carnegie Mellon University
- Albert Paul and Grady White, MSEL, NIST
- Barbara Fuller

Abstract

Microstructural Stresses: Networks, Structural Reliability, & Antiquity Preservation

Edwin R. Fuller, Jr.
National Institute of Standards and Technology,
Gaithersburg, Maryland 20899-8522, U.S.A.

Macroscopic behavior and ensemble physical properties of heterogeneous materials, such as polycrystalline ceramics or stone, depend on the thermoelastic properties of the constituent phases, their morphology, and their topology. Or simply, on the crystallites, their shape, and how they are arranged. Because crystalline properties are typically anisotropic, this dependence encompasses the distribution of crystallite orientations (crystallographic texture) as well as distributional aspects of crystallite shape and spatial arrangements. A property profoundly affected by these heterogeneous and stochastic features is the internal, or microstructural stresses. Their influence on behavior can be either deleterious, as in performance degradation of ceramic components or physical deterioration of stone sculptures and monuments, or advantageous, as in transformation and grain-bridging toughening phenomena. Microstructural stresses occur from many causes: temperature changes in conjunction with thermal expansion differences between features, phase transformations, or crystallization of fluids or salts in pores (e.g., freeze/thaw cycles).

Many materials science tools are available to elucidate these phenomena. Computational materials science, however, provides a particularly facile means for examining material response to a wide variety of physical conditions. A recently observed phenomenon in polycrystalline materials with crystalline thermal expansion anisotropy is the development of residual-stress networks upon cooling or heating. Moreover, the length-scale of these networks encompasses many grains. To study this and related phenomena, two- and three-dimensional model microstructures were generated with a pixel/voxel-based tessellation technique that constructs grains whose morphologies conform to a predefined statistical distribution. Crystal orientations were overlaid on the grain structure such that grain orientation and misorientation distribution functions also match predefined statistical distributions. Microstructure-based finite-element simulations were then used to elucidate the origin of the residual stress networks, and to characterize the influence of texture on network size and associated polycrystalline physical properties, such as bulk thermal expansion and microcracking propensity.