TEXTURE AND ANISOTROPY CORRELATIONS IN CERAMIC COMPOSITES

Keith J. Bowman[+], Peter Miller[#],
Zhen Guo[*], and Ryan Roeder[+] Kevin P. Trumble[+]

[+]School of Materials Engineering, Purdue University,
West Lafayette, IN 47907

[#]Hewlett-Packard, 1000 NE Circle Blvd., Mail Stop 3Up4,
Corvallis, Oregon 97330

[*]Department of Materials Science and Engineering, The University of
Michigan, Ann Arbor, MI 48109

Abstract

Current strategies for ceramic composites that include heterogeneous phase and orientation distributions place even stronger demands on characterization approaches. Correlations of microstructure and orientation with property anisotropy will require enhanced descriptions of materials incorporating spatial variation. Unfortunately, few complete examples of microstructure and orientation correlated with properties exist for bulk materials, and particularly so for ceramics. This paper provides an assessment of the challenges for quantitative evaluations of layered and gradient ceramic composites by comparison to silicon nitride, the most advanced example of texture and anisotropy correlation in ceramic materials.

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Introduction

For homogeneous, bulk materials correlation of property anisotropy with texture and microstructure relies upon sound models for the relationship between the specific property and orientation and microstructure information. For properties that are easily expressed in a tensorial framework (e.g. conductivity, thermal expansion, piezoelectricity and elasticity) the correlation of single crystal properties with bulk properties can be accomplished readily if the single crystal properties are known, but the reverse process is not straightforward [1]. Differences between calculations and experimental results can be attributed to unknown defects, measurement uncertainties and the constraints of the model (e.g. upper bound, lower bound). For layered or gradient materials, specification of orientation and microstructure information that varies spatially adds to the complexity of any evaluation. For materials described as hierarchical [2], local relative orientations (misorientations) may be the critical element for understanding properties. When properties of such heterogeneous materials are evaluated the uncertainty in the bulk measurements includes assumptions at every point within the material that are as complex as those in an individual bulk material. Thus, heterogeneous composites superpose inherent anisotropy on a size scale between that of the individual crystals and the macroscopic specimen.

Property Anisotropy in Ceramics and Ceramic Composites

One important property for structural applications that is not explicitly tensorial in nature is fracture toughness. Fracture toughness in brittle materials includes a relationship to elasticity, so it is apparent that elastic anisotropy influences fracture anisotropy. At least this portion of the fracture toughness will have the symmetry associated with the elasticity tensor. Fracture toughness is also related to the energy release rate, which for a brittle material is correlated with the energy to create new surfaces. Preferential orientations can then lead to an anisotropy in the surface energy and thereby fracture anisotropy even if the elastic constants are isotropic. Mismatch in elastic and thermal properties can also contribute to anisotropy. However, fracture is a complicated process that includes definition of a direction and a plane. Unlike dislocation glide, which takes place by shear across the plane in a given direction, a crack propagates from either normal stresses (Mode I) or two shear geometries (Mode II and Mode III). For a ductile material the crack tip plasticity can also be influenced by plastic anisotropy. Another case wherein anisotropy and other tensorial properties interpose is with electric field effects on fracture in piezoelectric materials. In these inherently anisotropic materials Park and Sun have compared the relative utilities of stress intensity factor, the total energy release rate and mechanical energy release rate approaches [3]. The interaction of the vector electric field with elastic and piezoelectric anisotropy can be superposed over any anisotropy in surface energy.

In layered or gradient materials the size scale of the layers or the steepness of the gradient can play a role in anisotropy, depending on the initial position of the crack. For materials with distinct interfaces, interface fracture criteria must be applied to distinguish interface fracture. Figure 1 shows a section of a Al_2O_3 powder - 10 v/o Al_2O_3 platelet composite tube centrifugally slip cast into a plaster mold at approximately twenty-five times earth's gravitational acceleration [4]. During sintering at 1500°C for 2 hours the initial platelets undergo grain growth consuming a large fraction of the alumina powder matrix as the tubes sinter to a density greater than ninety percent of theoretical. The tube consisted of four injections of slurry resulting in a wall thickness of approximately two millimeters for a tube with a final diameter of 13.2 mm. The transition regions between the layers contained a region approximately 40 μm in thickness with a somewhat higher concentration of platelets and thereby a larger porosity content. The combination of oriented alumina grains and the oriented porosity lead to a material with strong fracture anisotropy as demonstrated by the Vickers indent placed just within the higher density region of the tube. Quantitative evaluation of such a fracture is hampered unless information on the local elastic constants can be assessed.

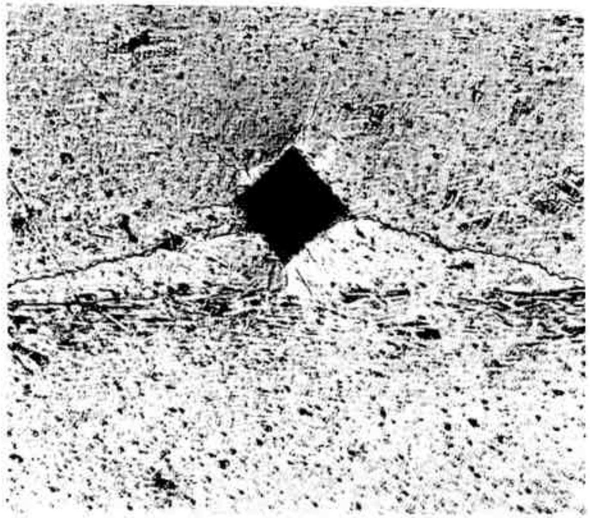

Figure 1 - Nomarski interference contrast of 5 kg Vickers indent placed adjacent to the interface between two separate layers in a centrifugally slip cast alumina-alumina platelet composite showing fracture anisotropy due to both the oriented platelets and the local increase in porosity. Uplift of the surface layer at the top and bottom of the microhardness indent is apparent and coincides with much less extensive cracking from these points on the indent. The inner surface of the tube lies below this micrograph and the indent axes in this micrograph are approximately 75μm.

Texture and Anisotropy in Silicon Nitride

For the last several years our research group has utilized silicon nitride ceramics to serve as a model material for relating texture to anisotropy resulting from hot working and hot pressing [5-10]. Fracture toughness anisotropy can be very difficult to evaluate from measurements of macroscopic propagation. Since sample size is often important in fracture assessments, measuring transverse or through thickness crack propagation is often impossible for materials with strongly anisotropic microstructures derived from processing. The majority of our fracture toughness measurements on textured materials have been produced using the Vickers indent technique shown in Figure 1.

For materials with axisymmetric textures the measurements of fracture toughness reported in Tables I and II have been carried out using the geometrical relationships shown in Figure 2. Table I gives indentation fracture toughness values for hot-pressed materials. Hot-pressing textures vary in intensity from 1.5 to 2 multiples of a random distribution (MRD) for the basal normal lying in the hot-pressing plane. The amount and type of sintering aid as well as firing conditions may be important; however, the extent to which shear deformation of the billet is possible during hot-pressing is probably the most significant factor. Samples loaded before sintering can pull the billet away from the die walls inhibiting the degree of shear deformation occurring from 'forging' [5].

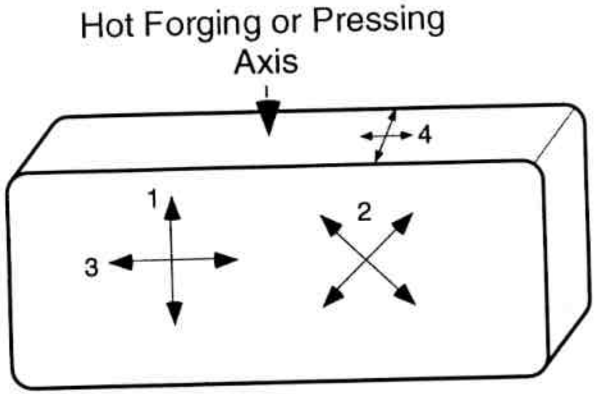

Figure 2 - Definition of the crack propagation directions assessed in textured silicon nitrides.

TABLE I
Fracture Toughness Anisotropy in Hot-Pressed Silicon Nitrides

MATERIAL	K_{IC} Fracture Type (MPa $\sqrt{m}$) 1	2	3	4
10w/o YAG (6:4)^{5,6}	5.7	4.7	3.9	6.7
7.5w/o YAG (3:5)$^{9-11}$	3.0	---	2.9	---
7.5w/o YAG (3:5)$^{9-11,*}$	4.7	---	3.9	---
7.5w/o YAG (2:1)$^{9-11*}$	4.9	---	3.8	---
7.5w/o YAG (4:1)$^{9-11*}$	4.1	---	3.8	---
15w/o YAG (3:5)$^{9-11,*}$	4.8	---	3.5	---

TABLE II
Fracture Toughness Anisotropy in Hot-Forged Silicon Nitrides

MATERIAL	Height Reduction	MRD Max	K_{IC} Fracture Type (MPa $\sqrt{m}$) 1	2	3	4
Axisymmetric						
10w/o YAG (6:4)^{5,6}	0.37	2.5	8.4	7.0	5.9	6.8
10w/o YAG (6:4)^{5,6}	0.57	4.0	6.9	---	4.0	---
Orthotropic						
20w/o YAG (6:4)^{5,6}	0.50	6.1	8.1	5.7	3.9	---
15w/o YAG (3:5)$^{9-11}$	0.50	5.2	6.4	---	1.7	---
15w/o YAG (3:5)$^{9-11**}$	0.50	5.7	6.2	---	3.6	---
15w/o YAG (3:5)$^{9-11***}$	0.50	5.7	5.6	---	2.0	---

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*1200°C 200 hours crystallization treatment.

** Miller - Sintered at 1500°C and forged.

*** Miller - Hot-pressed and then forged, KIC measured by SEPB method.

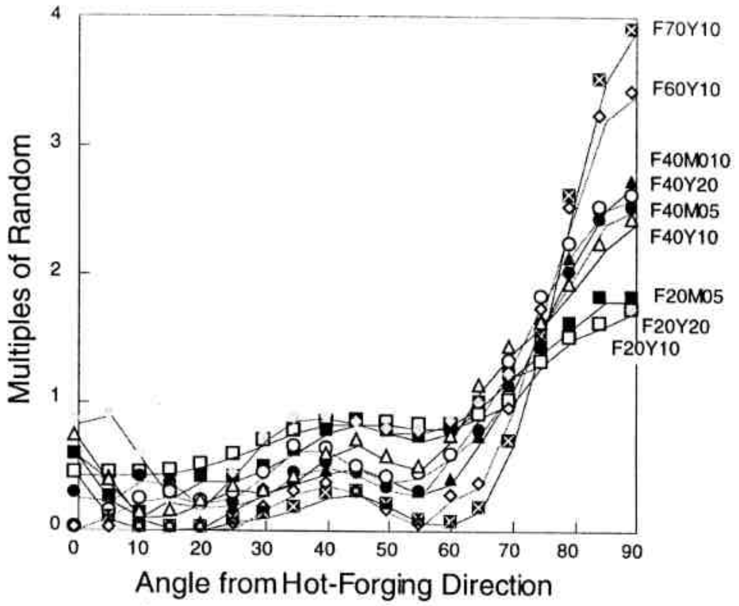

Figure 3 - Pole figure sections for hot-forged silicon nitrides with axisymmetric textures. The designations indicate F-forged followed by the height reduction in percent, the sintering aid (Y for YAG and M for MgO) and the amount of the sintering aid in weight percent. See Lee and Bowman [5-8] for additional processing information on axisymmetric forged materials.

Textures from forging, both axisymmetric (compression) and orthotropic (plane strain) are much stronger than those from hot pressing. Correspondingly, the fracture anisotropy in the materials is much greater as shown in Table II. The pole figures for the axisymmetric samples in Table II are shown in Figure 3 as sections of the axisymmetric pole figures. Figure 4 shows the modest degree of elastic anisotropy for the materials with pole figures shown in Figure 3. From these results it is not clear whether the anisotropy is due to crystal anisotropy or non-uniformities in hot-forged billets. Morphological texture influences may also determine the anisotropy. For very oriented tape-cast silicon nitrides Hirao et al. have shown strong thermal conductivity anisotropy that may occur more from grain morphology rather than crystal anisotropy [12].

The pole figures of the orthotropic materials produced in these investigations all have textures of the type shown in Figure 5. These materials also show only modest elastic anisotropy. The elastic constants for the first orthotropic material with 15 w/o YAG given in Table II are

$$C = \left|\begin{matrix} 380 & 165 & 131 & 0 & 0 & 0 \\ 165 & 396 & 157 & 0 & 0 & 0 \\ 131 & 157 & 422 & 0 & 0 & 0 \\ 0 & 0 & 0 & 128 & 0 & 0 \\ 0 & 0 & 0 & 0 & 114 & 0 \\ 0 & 0 & 0 & 0 & 0 & 111 \end{matrix}\right| \text{(GPa)}$$

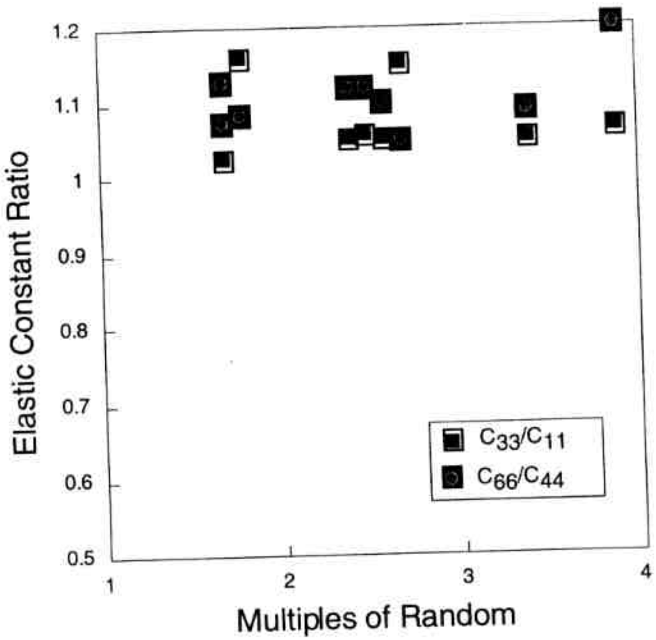

Figure 4 - Stiffness ratios for textured silicon nitrides in Figure 3 demonstrating modest elastic anistropy of these materials with little correlation of the anisotropy with the degree of preferred orientation.

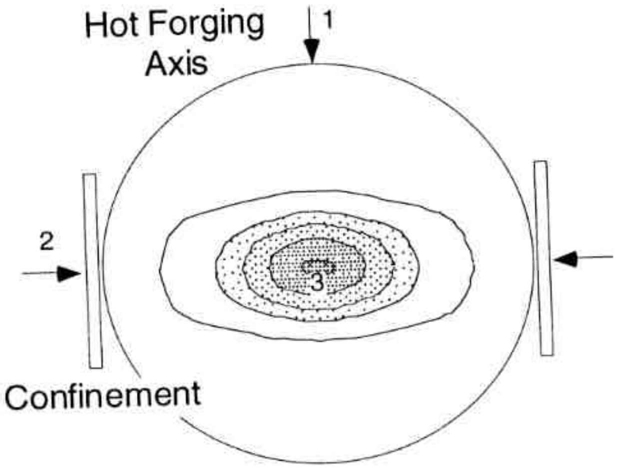

Figure 5 - Pole figure of the basal normal for textured silicon nitride oriented using plane strain deformation and a height reduction of 0.50.

Summary

The degree of a morphological effect versus a crystal orientation effect is not known for texture measurements on silicon nitride, and any other similar ceramic in which the single crystal elastic constants cannot be independently determined. In fact, without information on the single crystal properties quantitatively discerning and predicting properties for particular microstructures seems beyond our current capabilities. Similar evaluations on the heterogeneous layered and gradient materials discussed earlier in this paper are even more challenging.

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