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PROCESSING AND CHARACTERIZATION OF TRANSFORMATION-TOUGHENED CERAMICS WITH STRENGTH RETENTION TO ELEVATED TEMPERATURES

R. A. Cutler and C. B. Brinkpeter
Ceramatec, Inc.

and

A. V. Vircar and D. K. Shetty
University of Utah

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Prepared by Ceramatec, Inc.
2425 South 900 West
Salt Lake City, Utah 84119

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DISCLAIMER

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PROCESSING AND CHARACTERIZATION OF TRANSFORMATION-TOUGHENED CERAMICS WITH STRENGTH RETENTION TO ELEVATED TEMPERATURES

R. A. Cutler, C. B. Brinkpeter, A. V. Virkar and D. K. Shetty

ABSTRACT

Monolithic and three-layered Al_2O_3-15 vol. % ZrO_2 composites were fabricated by slip casting aqueous slurries. The outer and inner layers of three-layer composites contained unstabilized and partially stabilized ZrO_2, respectively. Transformation of part of the unstabilized ZrO_2 led to surface compressive stresses in the outer layers. Strain gage, x-ray, indentation crack length, and strength measurements were used to determine the magnitude of residual stresses in the composites. The strength of the three-layer composites (≈1200 MPa) was 500-700 MPa higher than that of the monolithic outer layer composites at room temperature and 350 MPa higher at 750°C. The strength differential decreased rapidly above the m-->t transformation temperature. Three-layered composites showed excellent damage resistance and improved reliability. Cam follower rollers were fabricated to demonstrate the applicability of this technique for making automotive components.

INTRODUCTION

Transformation of ZrO_2 from the partially stabilized tetragonal polymorph to the equilibrium monoclinic form has been successfully used to toughen a variety of ceramic matrices[1]. Transformation of the metastable tetragonal ZrO_2 in the near surface regions of ceramics has also been used to produce compressive residual stresses and, thus, strengthen ceramics containing ZrO_2[2,3]. The techniques developed to date for this purpose, such as grinding[3] or partial removal of the stabilizer[4], produce only modest thickness (≈30 μm) of the compressive stress zones. These ceramics do not sustain the improved strength under severe damage conditions that may produce a flaw larger than the compressive stress zone. The compressive stress produced by grinding can also be irreversibly lost by a high-temperature exposure and reverse transformation of the monoclinic ZrO_2 to the tetragonal form.

Virkar[5] patented a simple technique for introducing residual stresses in ceramics using transformation-induced stresses. Virkar, et al.[6] showed that significant compressive residual stress can be introduced in Al_2O_3-10 vol. % ZrO_2 with surface compression zones of the order of 300 to 1000 μm. The technique involves fabrication of three-layer composite ceramics consisting of outer layers that contain unstabilized ZrO_2 in an oxide matrix, and an inner layer that contains ZrO_2 (partially stabilized with an oxide additive such as Y_2O_3) dispersed in the same oxide matrix. On cooling from the fabrication temperature, a large fraction of the ZrO_2 in the outer layer transforms to the monoclinic form, while nearly all of the ZrO_2 in the inner layer is retained metastably in the tetragonal form. This selective transformation of the ZrO_2 (with the accompanying volume expansion) in the outer layers and the constraint of the bulk inner material leads to significant compressive stress in the outer layers and balancing tensile stress in the bulk. The residual stress will not decrease with temperature until the monoclinic to tetragonal transformation temperature is reached, since monoclinic and tetragonal ZrO_2 polymorphs have nearly the same coefficients of thermal expansion.

Cutler et al.[7,8] have successfully applied the three-layer technique to Al_2O_3-ZrO_2 composites. Using dry pressing to form the sandwich composites, a compressive stress of 400 MPa was produced in the outer layers of Al_2O_3-15 vol. % ZrO_2 composites with an outer layer thickness of 375 μm. During the first two years of ORNL funding[9] it was demonstrated that: 1) three-layer composites could be made with retention of a significant fraction of this residual stress (≈200 MPa) to temperatures of 750°C[7,8], 2) residual stresses could be detected by strength testing[7,8], strain gage measurements[10], characterization of monoclinic content by x-ray diffraction[7], or indentation/strength measurements[11], 3) the three-layer composites have excellent damage resistance[11], and 4) significant (300-400 MPa) residual compressive stress which is not transformation-induced can be introduced by grinding monolithic Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3).

This report summarizes highlights of a two year subcontract extension with objectives to: 1) increase the use temperature of three-layer composites by substituting HfO_2 for ZrO_2, 2) develop aqueous slip casting techniques for three-layer composites in order to obtain better layer uniformity and to maximize residual compressive stress by optimizing the outer layer thickness, 3) superimpose temperature stresses on transformation-induced stresses in three-layer composites, and 4) demonstrate improved thermal shock resistance and damage resistance in optimized composites.

EXPERIMENTAL PROCEDURES

Al_2O_3-15 vol. % ZrO_2 composites were prepared by dry pressing using techniques discussed previously[7-11] and by slip casting[12,13] using Al_2O_3 (ERC-DBM, Reynolds Metal Co., Bauxite, AR.) and ZrO_2 (DK-1, Daiichi Kaguku Kogyo Co. Ltd, Osaka, Japan) as starting materials for the outer layer monolithic material. The inner layer monolithic material used the same source of Al_2O_3 but partially stabilized (3.0 mol. % ZrO_2) ZrO_2 (HSY-3.0, Daiichi) was used in place of unstabilized zirconia. The two slips were dispersed using 0.5 wt. % citric acid and 2.0 wt. % Darvan C (R. T. Vanderbilt, Norwalk, CT.) in an aqueous slip at 70 wt. % (35 vol. %) solids. The slips were vibratory milled 16 hours with ZrO_2(3.0 mol. % Y_2O_3) media and degassed prior to slip casting. Three-layer composites were slip cast in plaster molds by first casting the outer layer slip for a given time period and then pouring out the outer layer slip and quickly pouring in the inner layer slip. The inner layer slip was allowed to remain in the mold when making solid plates or cylinders. When hollow tubes for cam followers were fabricated, the inner layer slip was drained followed by introduction of the outer layer slip for a second time. The thickness of the layers was controlled by the slip casting times of the outer layers. The slip cast parts were dried under controlled conditions and sintered at 1587°C for 30 minutes. The sintered parts were subsequently HIPed in 200 MPa Ar at 1550°C for 30 minutes.

Unstabilized HfO_2 and coprecipitated HfO_2(4 mol. % Y_2O_3) powders were supplied at no cost to the program by Teledyne Wah Chang Albany. Solid solution HfO_2·50 mol. % ZrO_2 powder was prepared by milling the Teledyne HfO_2 powder with Daiichi DK-1 powder, calcining at 1700°C for 1 hour, and remilling the calcined solid solution.

Characterization for slip cast materials[13] was similar to that used previously for dry-pressed samples[6-11]. Strength testing was generally performed on bars in four-point bending. Thermal shock testing was performed on 6.5 mm diameter by 50 mm long rods. The samples were heated to various temperatures prior to quenching in ice water (0°C). Strength was measured in three-point bending at room temperature for thermally shocked rods.

Ceramic cam followers were ground (Advanced Materials Technology, Inc.(AMATEC), Georgetown, S.C.) to Chrysler Motor Co. drawing SK-783-50201 (revision D). Impact testing at Chrysler consisted of running the rollers on a motorized 2.2 liter cylinder head at 2750 rpm for five minutes with a 0.635-0.762 mm lash between the roller and the base circle of the cam.

RESULTS AND DISCUSSION

CHARACTERIZATION OF RESIDUAL STRESS

A schematic of the three-layer composites is shown in Figure 1. Both the outer and inner layers consist of Al_2O_3-15 vol. % ZrO_2 so that thermal expansion coefficients are similar. The main difference is that the ZrO_2 in the outer layers is unstabilized and the ZrO_2 in the inner layer has been coprecipitated with 3 mol. % ZrO_2. Upon cooling from sintering temperatures (≈1600°C) where ZrO_2 in both outer and inner layers is tetragonal, most of the unstabilized ZrO_2 in the outer layers transforms to monoclinic with an accompanying volume expansion. The constraint of the inner layer puts the outer layers under compression and the inner layer in tension (see Figure 2). Assuming a square wave stress distribution the accompanying residual compressive stress, σ_1, in the outer layers is

$$\sigma_1 = -\Delta\varepsilon_o E d_2/[(1-\upsilon)d] \qquad (1)$$

where $\Delta\varepsilon_o$ is the unconstrained strain in the outer layers from the transformation of ZrO_2, E is Young's modulus, d is thickness, υ is Poisson's ratio and the subscripts 1 and 2 refer to the outer and inner layers, respectively. Correspondingly, the residual tensile stress, σ_2, in the

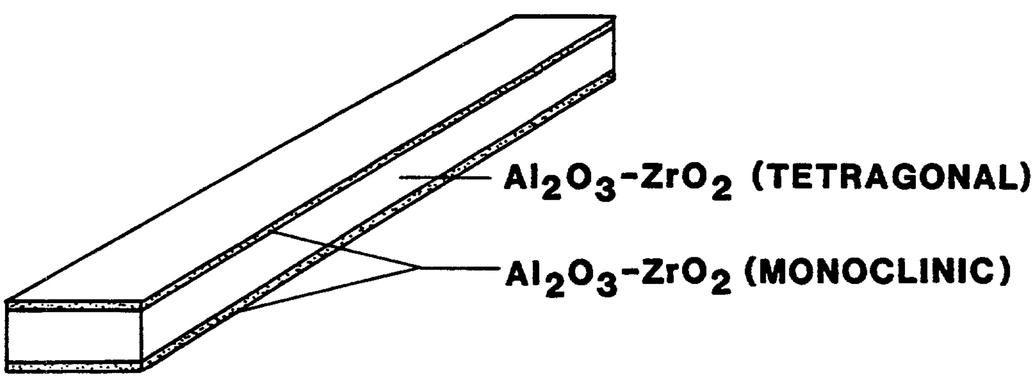


Figure 1. Schematic of three-layer Al_2O_3-15 vol. % ZrO_2 composites with unstabilized ZrO_2 in outer layers and partially stabilized ZrO_2 in inner layer.

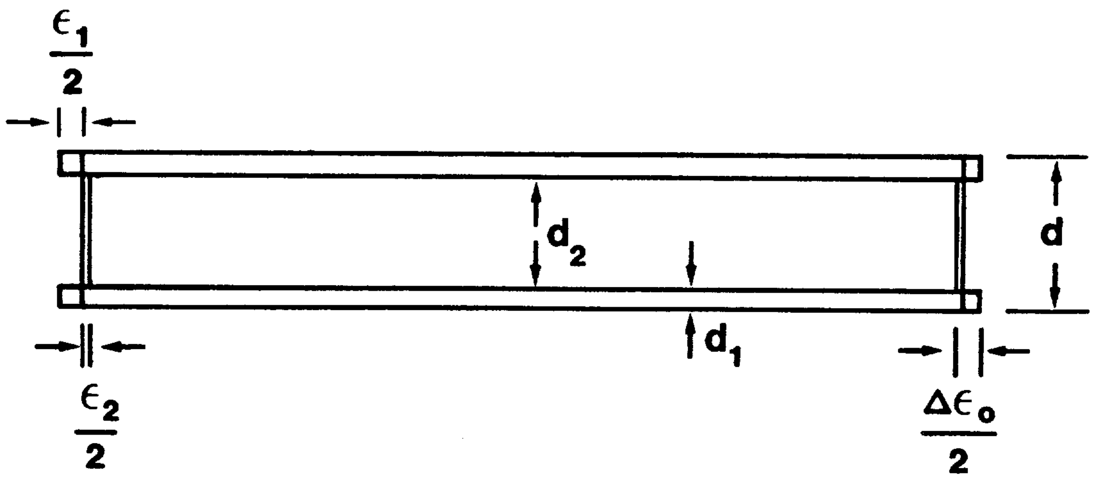


Figure 2. Schematic of three-layer sample showing unconstrained outer and inner layers as well as constrained length of composite.

inner layer is

$$\sigma_2 = 2\Delta\varepsilon_o E d_1/[(1-\nu)d] \quad (2)$$

Based on fracture of three-layer composites from within the outer layers, as would be expected in flexure, the failure strength, σ_f, is

$$\sigma_f = \sigma_o + \Delta\varepsilon_o E d_2/[(1-\nu)d] \quad (3)$$

where σ_o is the failure strength of the outer layers in the absence of residual stress. A plot of strength as a function of normalized inner layer thickness (d_2/d) would be expected to follow a linear relationship with slope of $\Delta\varepsilon_o E/(1-\nu)$ and intercept equal to σ_o. Experimental verification of Equation (3) has been demonstrated for samples with flaw populations typical of "as-sintered" samples[7], as well as for samples with well characterized indentation flaws[8,11]. The value of $\Delta\varepsilon_o$ determined from these measurements agree with estimates from x-ray measurements[7].

Virkar[14] has developed a technique for determining the residual stress profile of sintered ceramics using inexpensive strain gages. A strain gage is attached to one side of a three-layer ceramic which initially has outer layers of equal thickness. One side is then incrementally ground off (see

Figure 3) and the strain (ε) is measured as a function of thickness removed (δ). Using simple beam theory it is possible to predict the shape of the strain vs thickness removed[14] for a three-layer composite (see Figure 4). Considering a symmetric stress profile ($\sigma_{xx}=\sigma_{yy}$) so that residual stress is a function of z (thickness direction of a three-layer composite) only, the measured strain, $\varepsilon_M(\delta)$, vs δ data can be used to determine the residual stress profile. For $0\leq\delta\leq d_1$

$$\varepsilon_M(\delta) = \{\Delta\varepsilon_o d_2 \delta(2d+\delta)/(d-\delta)^2 d\} \quad (4)$$

so that the residual stress can be calculated as

$$\sigma_1 = -\Delta\varepsilon_o d_2 E_1 E_2/\{(2E_1 d_1)(1-\upsilon_1)+(E_2 d_2)(1-\upsilon_2)\} \quad (5)$$

The expected tensile strain in the inner layer, for $d_1\leq\delta\leq d_1+d_2$, is given by

$$\varepsilon_M(\delta) = \Delta\varepsilon_o\{[(d_1^2-(d_2-\delta)^2)/(d-\delta)^2] + (d_2/d)\} \quad (6)$$

Verification of this predicted response is shown for experimentally determined curves in Figure 5. As expected, the monolithic outer and inner layer composites show no change in strain as a function of thickness of material removed, while the three-layer composites show significant residual compressive stress in the outer layers. The bend in $\varepsilon_M(\delta)$ vs δ occurs at $\delta=d_1$ (the interface between outer and inner layers). The magnitude of $\Delta\varepsilon_o$ was measured to be 1.2×10^{-3} giving a calculated compressive residual stress in the outer layers of ≈520 MPa[10].

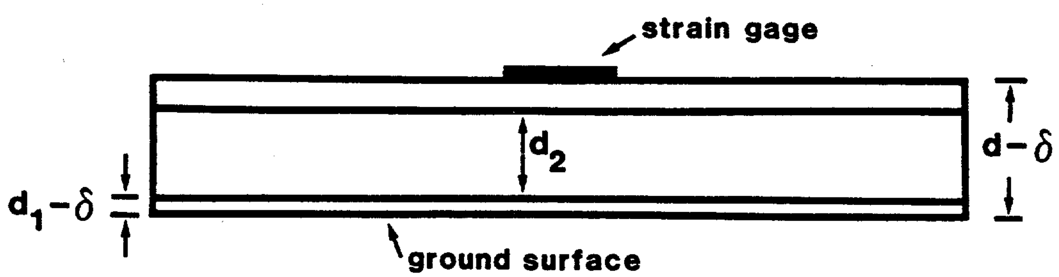


Figure 3. Schematic showing the three-layer sample with a strain gage mounted on one face. The other face is incrementally ground off and the strain is recorded as a function of the thickness ground off.

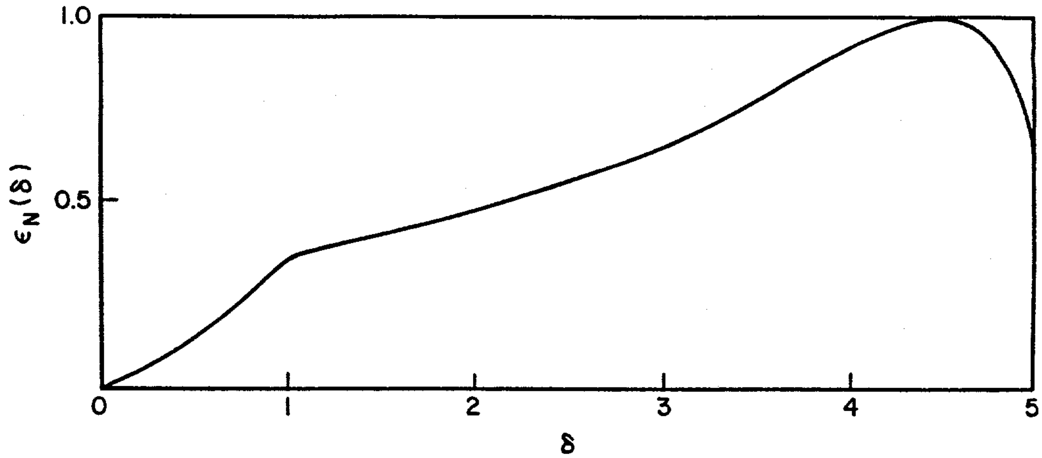


Figure 4. Analytically determined normalized strain for three-layer composite with $d_1=1$ and $d_2=4$ as a function of thickness removed[10].

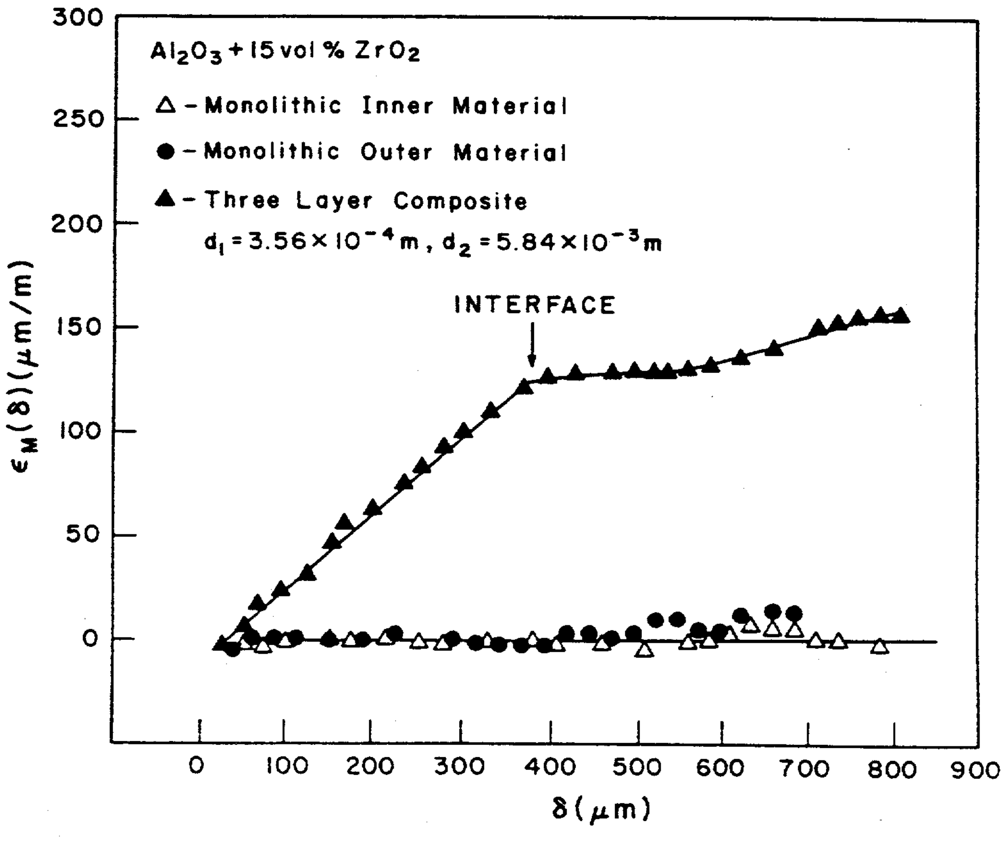


Figure 5. Measured strain vs thickness removed for monolithic and three-layer Al_2O_3-15 vol. % ZrO_2 samples. Monolithic samples show no residual stress while three-layer sample shows compressive stress in outer layer[10].

In the discussion of residual stress above, it was assumed that an equibiaxial state of stress and strain exists in the three-layer composites. In order to verify this assumption, longitudinal and transverse strains were measured on both bend bars (≈4.5 mm in width and thickness) and plates (20 mm square-shaped samples) as a function of thickness removed. As shown in Figure 6, the strain in the transverse direction was lower by about 20% than the strain in the longitudinal direction for bar shaped samples. As expected, the longitudinal and transverse strains were similar for the square-shaped sample (see Figure 7). These data suggest that the assumption of equibiaxiality is reasonable.

The influence of residual stresses present in the three-layer composites can also be shown by measuring indentation cracks. Assuming a simplistic analysis for indentation behavior, the indentation crack length, c_o, in the absence of residual stress is given by

$$c_o = [0.016(E/H)^{1/2}(P/K_{Ic})]^{2/3} \qquad (7)$$

In the presence of residual stress, σ_R, the half-penny crack after indentation is shorter in the outer layers which are under uniform compressive stress or longer in the inner layer which is under a lesser tensile stress. The crack length, c, in the presence of residual stress may be calculated by

$$(c_o/c)^2 - (c_o/c)^{1/2} + \gamma = 0 \qquad (8)$$

where $\gamma = A\sigma_R[(P/K_{Ic}^4)(E/H)^{1/2}]^{1/3}$ and A is a nondimensional constant[11]. Previous work has confirmed the validity of crack lengths which occur in the outer layers of three-layer composites by comparing indentation cracks on the outer surfaces of three-layer composites in relation to the indentation cracks on the outer surfaces of monolithic bars.

Indenting cross-sections shows the effect of strong residual stresses more dramatically (see Figure 8). Assuming typical values[11] of toughness (4.3 MPa·$m^{1/2}$), Young's modulus (340 GPa),hardness (17 GPa), compressive stress (-500 MPa) in the outer layers and balancing tensile stress (100 MPa) in the inner layer (1/12-5/6-1/12 bars), Equations (7) and (8) give c_o=115 μm and c=73.7 μm in the outer layers and c=140.8 μm in the inner layer. By aligning cracks parallel and perpendicular to the surface (and interface), it is possible to see shorter cracks in the outer layers perpendicular to the interface/surface (i.e., in the direction where the residual compressive stress is closing the crack tip) and longer cracks in the inner layer perpendicular to the interface/surface (i.e., in the direction where the residual tensile stress is opening the

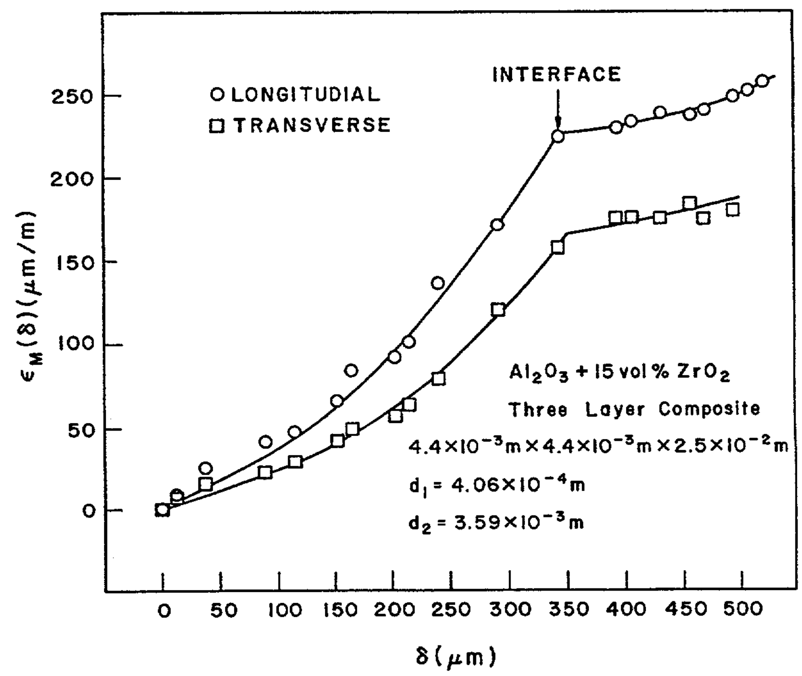


Figure 6. Measured strain as a function of thickness removed for bar-shaped three-layer composites[10].

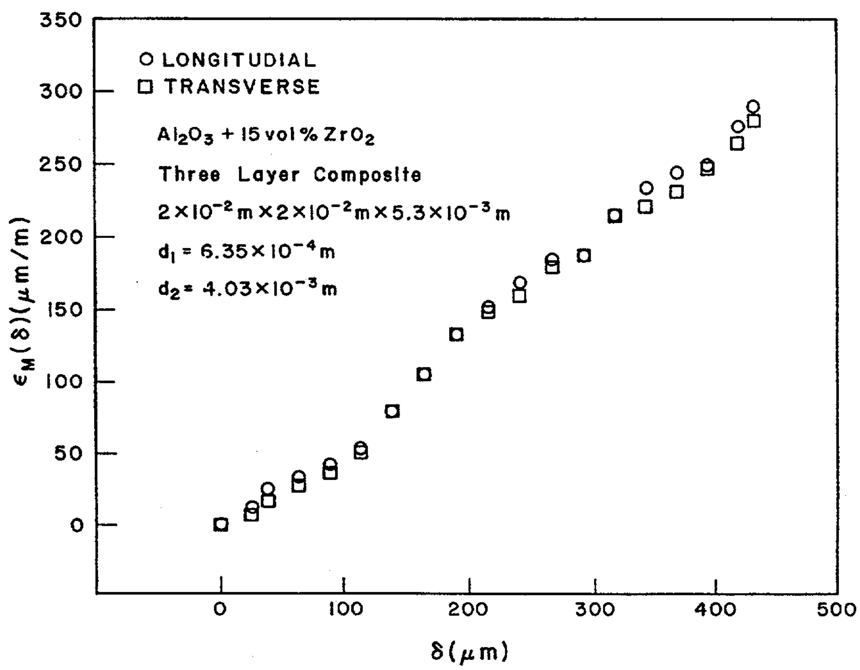
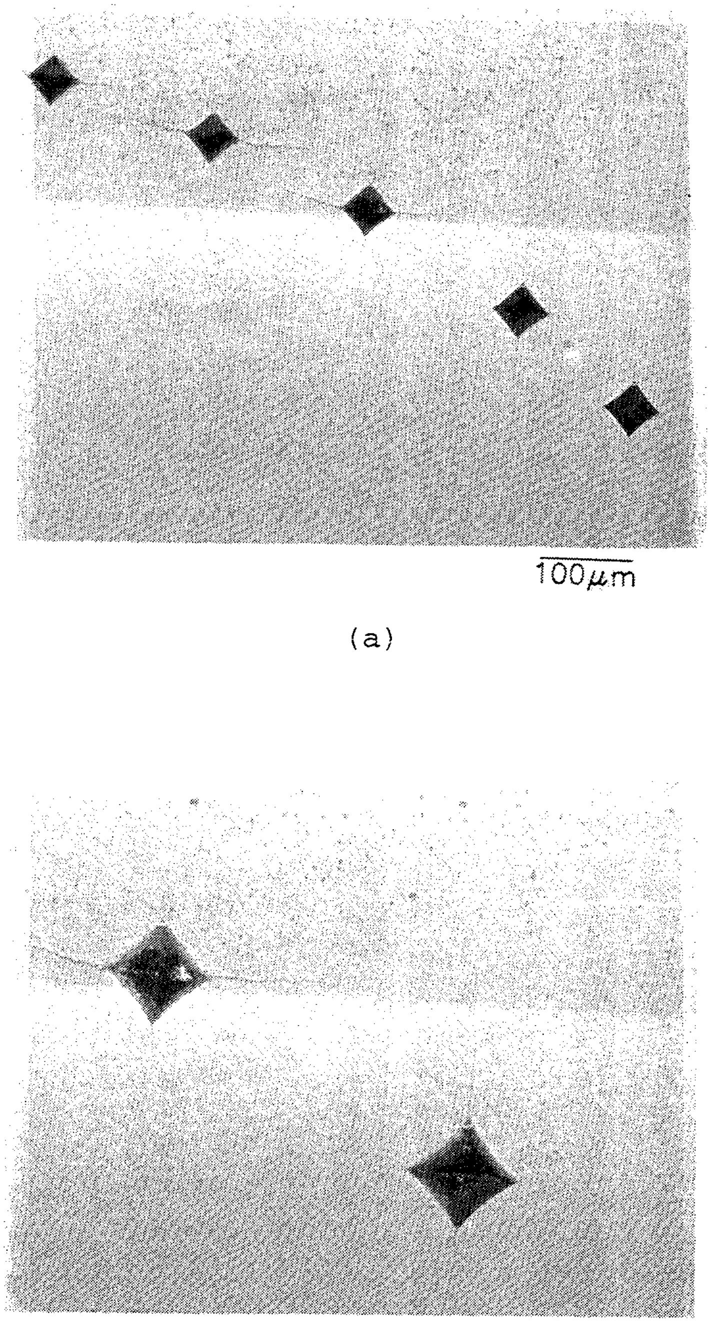


Figure 7. Measured strain as a function of thickness removed for square-shaped three-layer composites[10].



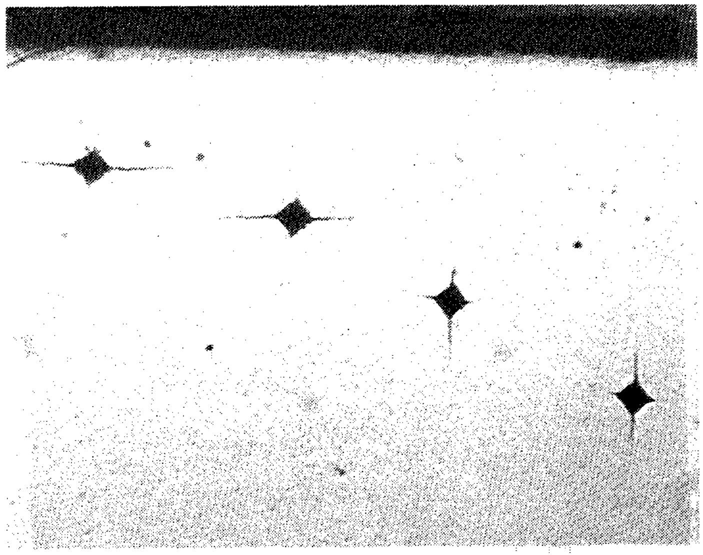
100μm

(a)

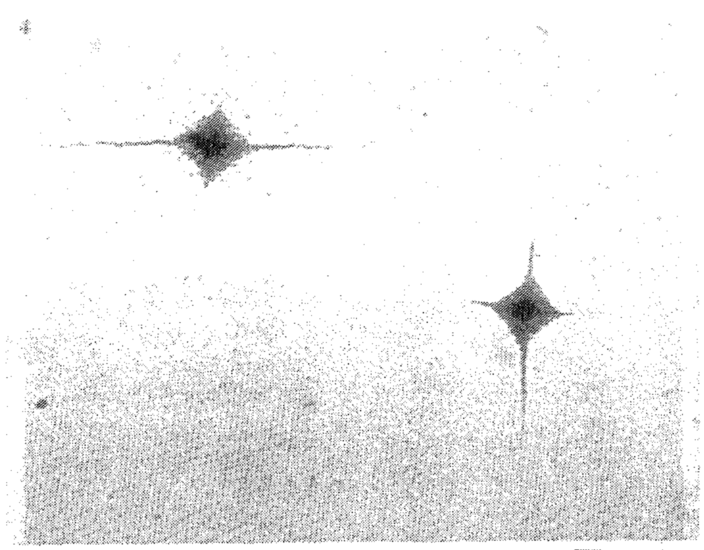
25 μm

(b)

Figure 8. Optical micrographs of cross-sections of three-layer (Outer layers of Al_2O_3-15 vol. % ZrO_2 and inner layer of ZrO_2(3 mol. % Y_2O_3)-40 vol. % Al_2O_3) composites with indents oriented with cracks parallel and perpendicular to the interface/surface. Note change in crack lengths between outer (top) and inner (bottom) layers.



(c)



(d)

Figure 8 (cont). (c,d). Cracks in three-layer Al_2O_3-15 vol. % ZrO_2 after removing surface of indentation cracks to make cracks more visible. Crack lengths are strongly influenced by residual compression (outer layers) and residual tension (inner layer).

crack tip). These cracks are shorter (outer layers) and longer (inner layer) than the cracks in the absence of residual stresses, as predicted by the simple analysis above.

It is interesting to note, however, the resulting difference in crack length parallel to the interface for indentation cracks in the outer and inner layers. The cracks parallel to the interface are longer in the outer layers and shorter in the inner layer. The longer crack length in the outer layers is due to the spontaneous energy release when the crack parallel to the interface extends.

The integrity of the smooth interface obtained by slip casting is also seen by aligning indentation cracks with the interface. There is no tendency for the cracks to run along the interface (see Figure 8).

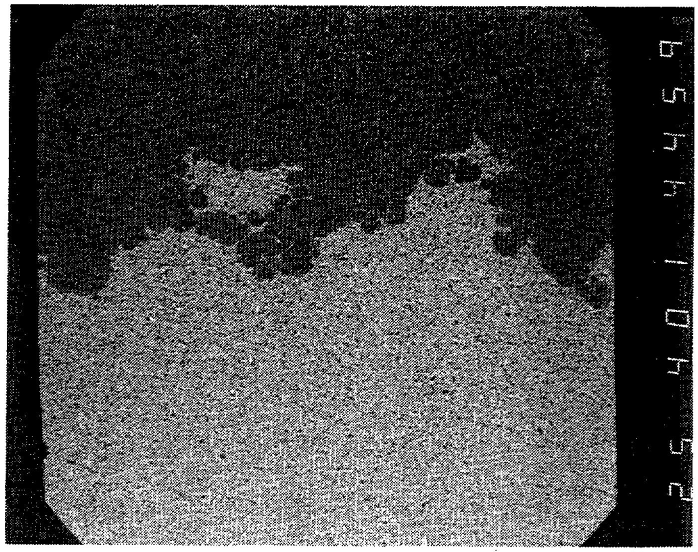
The above observations with indentation cracks further explain why strength is high when failure occurs within the outer region and may be low when defects initiate failure within the inner layer. These results give another method for identifying materials with substantial residual stresses and also provide a means for identifying interfaces in three-layer materials without phase contrast.

COMPARISON OF SLIP CAST AND DRY PRESSED COMPOSITES

The advantages of slip casting three-layer Al_2O_3-ZrO_2 composites compared to dry pressing the same layered composition are improved uniformity in outer layer thickness, and better particle packing prior to sintering resulting in reduced flaw populations after densification. The disadvantages of slip casting are that the thickness of components is limited and the cycle times are increased due to drying constraints.

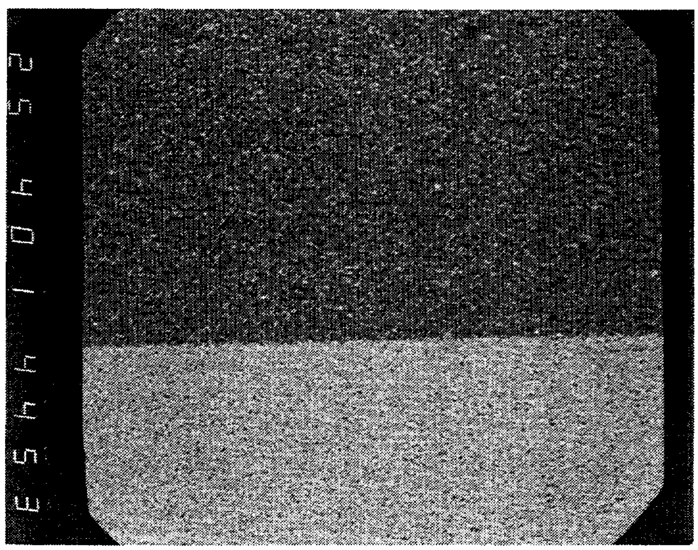
Figure 9 shows a comparison of cross-sections of a three-layer composite where the inner layer has been made with higher ZrO_2 content such that the interface is easily seen in the SEM. Figure 9(a) shows the interface of dry pressed composites where spray dried agglomerates are readily observed. Note how much more uniform the interface is for the same composition when slip cast (Figure 9(b)). Figure 10 shows a strength comparison between monolithic and three-layer Al_2O_3-15 vol. % ZrO_2 composites made by dry pressing (followed by isostatic pressing) and slip casting. The 10 to 20% strength improvement is due to the advantages of slip casting outlined above.

Figure 11(a) shows the strength improvement at room temperature for monolithic and layered composites which occurred during the contract. The higher strengths were the result of improved processing including limiting defects (i.e., agglomerates, pores, inclusions), improved interface control, and optimization of drying/sintering schedules.



25 μm

(a)



25 μm

(b)

Figure 9. SEM micrographs of cross-sections of (a) dry pressed and (b) slip cast three-layer (outer layers of Al_2O_3-15 vol. % ZrO_2 and inner layer of ZrO_2(3 mol. % Y_2O_3)-40 vol. % Al_2O_3) composites. Note smooth interface in slip cast composites and remnants of spray dried agglomerates in dry pressed outer layer.

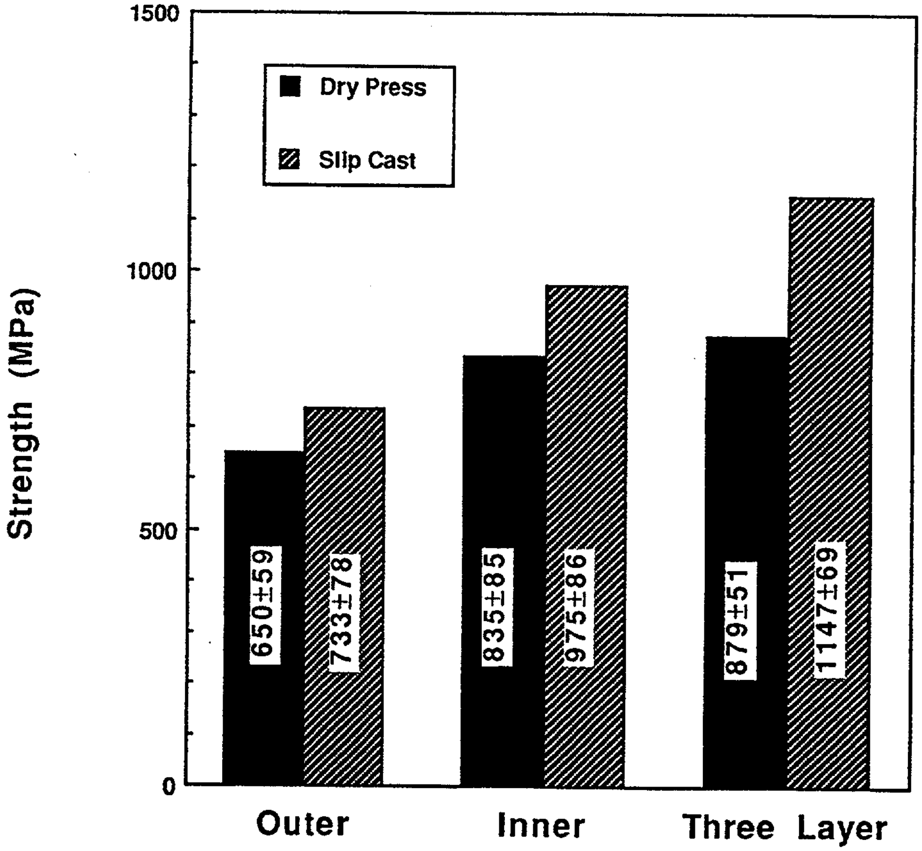


Figure 10. Room temperature flexural strength comparison of Al_2O_3-15 vol. % ZrO_2 fabricated by dry pressing (followed by isostatic pressing) and slip casting.

Improved strength was also achieved by controlling the grinding process as described below. High temperature (1000°C) strength also improved, as shown in Figure 11(b), during the same time period due to improved processing. The strength, however, decreases dramatically at elevated temperature for both the monolithic and three-layer composites due to the absence of transformation toughening. The key to increasing the use temperature for transformation-induced stress is the successful substitution of HfO_2 for ZrO_2 (see section below on elevated temperature testing).

EFFECT OF GRINDING ON STRENGTH

The strength of the monolithic inner layer material (Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3)) nearly doubled upon grinding as shown in Table 1. The strength degraded slightly with temperature (see Table 1) but testing at 750°C (after

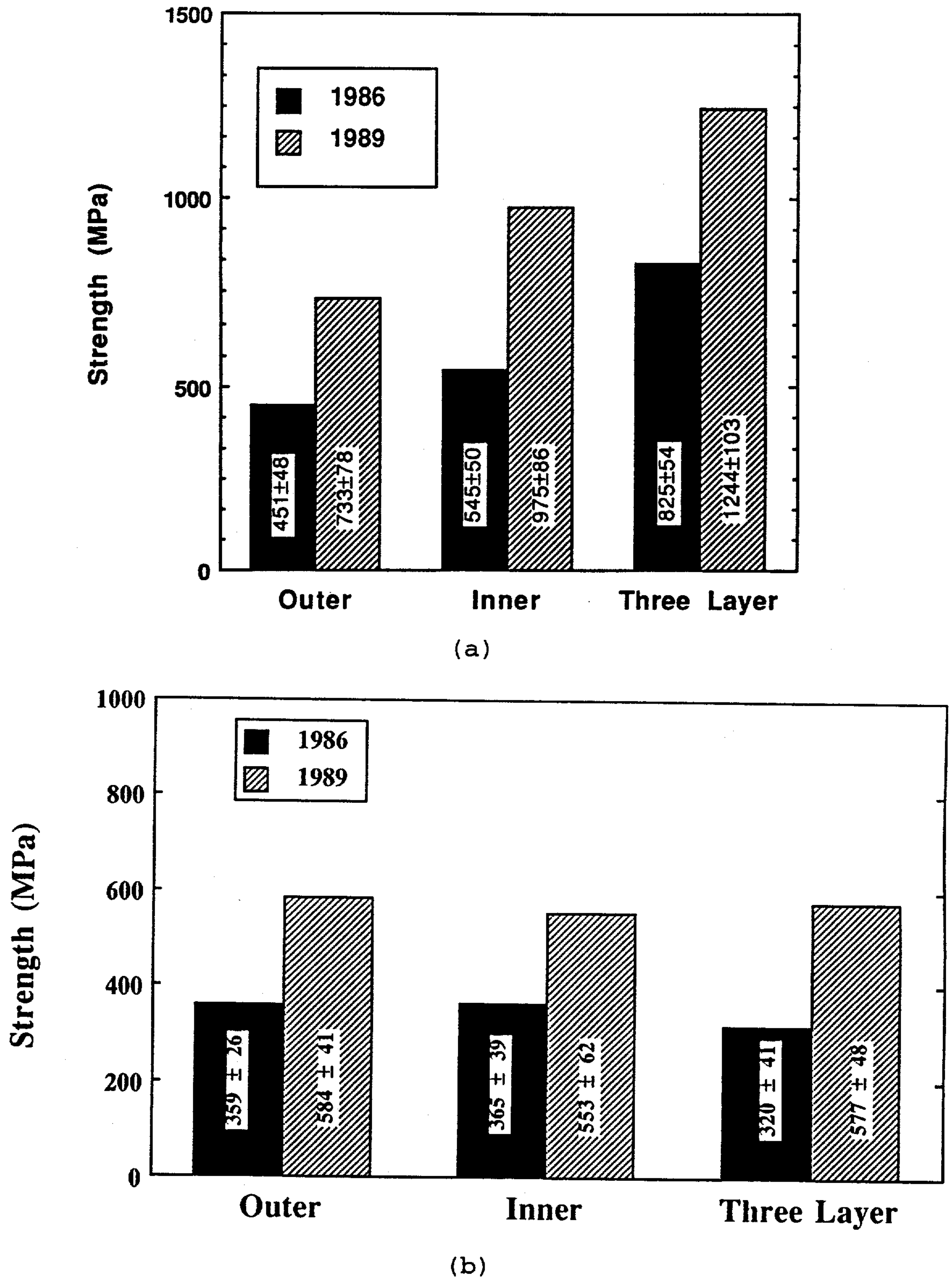


Figure 11. Strength improvement between 1986 and 1989 for monolithic and three-layer Al_2O_3-15 vol. % ZrO_2 composites . (a) Room temperature, (b) 1000°C.

cooling from 1200°C) showed that transformation-induced stresses are not the primary strengthening mechanism since strength was identical to that measured upon heating to 750°C. This is consistent with x-ray diffraction data which showed no transformation to monoclinic zirconia upon grinding (see Table 2). Specimens polished to a one µm finish without grinding showed only a moderate strength improvement (strength of 551±49 MPa) despite a significant improvement in surface finish, demonstrating that grinding does more than improve surface finish. In order to see if it is possible that grinding introduces compressive stresses due to plastic deformation, Al_2O_3, ZrO_2, monolithic outer layer material (Al_2O_3-15 vol. % ZrO_2) and three-layer composites were all tested (see Table 2) in the unground (edges chamfered before HIPing) and ground state (220 grit wheel at down feed of 2.5 microns/pass before sparking out). The strength increased (by 30-67%) for all specimens, although the magnitude of the strength increase was greatest for ZrO_2 and the monolithic inner layer specimen. Fractography showed alumina, zirconia, and monolithic outer layer bars all failed from the tensile surface, whereas monolithic inner layer and three-layer bars showed a large percentage of bars failing from near the chamfers.

A number of factors affect the strength increase including improved surface finish and transformation-induced stresses for the ZrO_2, monolithic outer, and three-layer specimens. The inner layer monolithic ZrO_2 is unique in that no measurable monoclinic ZrO_2 was observed on the ground or fractured surface (see Table 2). While others[16-19] have seen a strengthening via grinding for transformation toughened ceramics, it has always been accompanied by an

Table 1
Strengthening of Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3) Due to Grinding

Specimen Preparation	Test Temperature (°C)	Samples Tested	Strength (MPa)
Unground	25	6	452±11
Ground (220 Grit)	25	6	835±85
Ground, 1200°C Anneal	25	5	731±168
Ground, 1600°C Anneal	25	6	666±68
Ground, 1200°C Anneal	750	3	621±47
Ground, 1200°C Anneal	850	3	606±5
Ground, 1200°C Anneal	950	3	528±21
Ground, 1200°C Anneal	1200->750	3	627±95

Table 2
Strength Comparison of Ground and Unground Bars

Material	Density (g/cc)	Strength (MPa) Unground[b] #[e]	x^f	s^g	Ground[c] #	x	s	m-ZrO_2 Content[a] (%) U[b]	G[c]	F.S.[d]
Al_2O_3[h]	3.98	8	308±30		8	434±64		---	---	---
ZrO_2[i]	6.04	8	677±63		5	998±73		<1	1.9	24.8
Outer[j]	4.29	8	389±23		5	650±59		83.1	85.6	88.5
Inner[k]	4.32	8	485±34		8	806±52		<1	<1	<1
3-layer[l]	4.32	7	679±56		4	879±51		---[m]	---[m]	---[m]

a. Monoclinic content of ZrO_2 in specimen[15].
b. Chamfered before HIPing with no other grinding.
c. Ground with a 220 grit wheel at a down feed of 2.5 µm/pass before spark out.
e. Number of specimens tested in four point bending.
f. Mean value.
g. Standard deviation.
h. Reynold's ERC-DBM alumina.
i. Daiichi's HSY-3.0 zirconia.
j. Outer layer composition.
k. Inner layer composition.
l. Three-layer composition with outer layers 1/12 total thickness.
m. Not measured but has substantial monoclinic ZrO_2[7,8].

increase in monoclinic content. Stresses introduced during grinding of the inner layer material, however, do not show strength hysteresis dependent on the m<-->t transformation temperatures and ground monolithic inner layer specimens have higher strength than three-layer composites at temperatures in excess of 950°C. The lack of monoclinic zirconia on ground and fracture surfaces, and prior work showing that the effect is not solely due to improved surface finish, suggests that plastic deformation during grinding may account for part of the improved strength of the inner layer composition.

In order to optimize the stresses introduced into the monolithic inner material during grinding, the rate of down feed was varied. Table 3 shows that strengths over 900 MPa were observed when the down feed was increased to 10.2 microns/pass. Furthermore, grinding on the sides of the bar decreased the tendency for the bars to fail from chamfers and increased the strength.

Monolithic Al_2O_3, ZrO_2(3 mol. % Y_2O_3), Al_2O_3-15 vol. % ZrO_2 and three layer Al_2O_3-15 vol. % ZrO_2 (with and without post HIP grinding) specimens were sent to Rockwell International for determination of residual stresses by x-ray

Table 3
Effect of Grinding Parameters on the Room Temperature Strength of Al_2O_3-15 vol. % $ZrO_2(Y_2O_3)$ Composites

Sides Ground[a]	Down feed[b] (μm/pass)	Strength (MPa) #[c]	$\bar{x}$[d]	s[e]
0	2.5	8	485	34
2	2.5	8	806	52
4	2.5	6	876	100
2	5.1	6	749	105
4	5.1	6	862	119
1	7.6	8	806	52
4	10.2	6	926	78
4	15.2	6	849	102

a. Sides of samples ground with a 220 grit diamond wheel. Tensile surface was ground for all samples except for unground bars.
b. Down feed of 220 grit wheel prior to spark out.
c. Number of bars tested.
e. Mean strength.
f. Standard deviation.

diffraction. Dr. Michael R. James performed the Cu K_α x-ray analysis using (416) and (620) planes for Al_2O_3 and ZrO_2, respectively. When using Cr K_α radiation, (1.1.10) and (331) planes were used for Al_2O_3 and ZrO_2, respectively. In Al_2O_3, the respective penetration depths for Cu and Cr radiation are 32 and 10 μm (i.e., 67% of the diffracted radiation comes from a depth less than these values (87 and 27 μm for 95% return)). Thus, the difference in residual stress between the two radiations represents the gradient of residual stress with depth. The penetration depths are slightly less for ZrO_2 since Zr is approximately three times more absorbent than Al.

The measured compressive residual stresses are given in Table 4. Surprisingly, the inner monolithic material which showed a large strength increase with grinding, despite no observable transformation (see Table 2), also showed low residual stress and low dependence of residual stress on depth of penetration. Further work is needed to explain the significant strengthening which occurs upon grinding the inner layer material. The outer layer and three-layer (the outer layer of the three-layer sample was the surface exposed to radiation) materials showed significantly higher residual stresses at shallower penetration and were strongly influenced by grinding. The 98 MPa incremental change in compressive stress for Al_2O_3 using Cr K_α radiation compares with 125-145 MPa as reported by Lange, et al.[18]. ZrO_2(3

Table 4
Residual Stress Determined at Rockwell International by X-ray Analysis

Composition	Residual Stress (MPa) Cu K_α Radiation unground	ground	Cr K_α Radiation unground	ground
Al_2O_3	-136	-135	-101	-199
ZrO_2(3 mol. % Y_2O_3)	-44	-15	-81	-271
Al_2O_3-15 $^v/_o$ ZrO_2	-131	-256	-267	-822
Al_2O_3-15 $^v/_o$ ZrO_2(3 mol.% Y_2O_3)	-131	-203	-173	-261
Three-layer	-486	-711	-570	-1106

mol. % Y_2O_3) showed a strong dependence of residual stress on depth, with residual stress which correlated well with the observed strength increase. There was no overall correlation of compressive stress with strength increase upon grinding for the materials tested. However, the residual stress difference between three-layer and outer layer Al_2O_3-ZrO_2 as determined by x-ray diffraction and strength measurements were both on the order of 300-400 MPa, verifying the substantial residual stress created by the stress-induced transformation in the three-layer composites.

In an effort to determine the depth of compressive stresses introduced by grinding slip cast Al_2O_3-15 vol. % ZrO_2, specimens were prepared for strength/indentation measurements. The strength of ground monolithic inner-layer bars at indentation loads of 16, 37, 64, 125, and 296 N agreed with data for unground samples (see Figure 12), indicating that residual stresses introduced by grinding are very shallow, as expected. The fact that the stresses are shallow, as compared to stresses in three-layer specimens, further supports the need for a protective compressive stress layer of at least 100 μm[6].

In order to investigate the strengthening which occurs upon grinding Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3) (i.e., monolithic inner layer material), slip cast bars in the unground and chamfered state were compared with machine ground and chamfered bars. The difference in strength at room temperature was 500 MPa, decreasing to 270 MPa at 750°C, and 180 MPa at 1250°C (see Figure 13). Ground samples heated to 1250°C, well above the m--->t transformation temperature, and cooled to 750°C, had strength of 762±129 MPa (6 bars) in comparison to 658±107 MPa measured for the same material tested at 750°C without the high temperature anneal. These data indicate that residual stresses due to grinding are not completely removed by heating to 1250°C and holding for 30

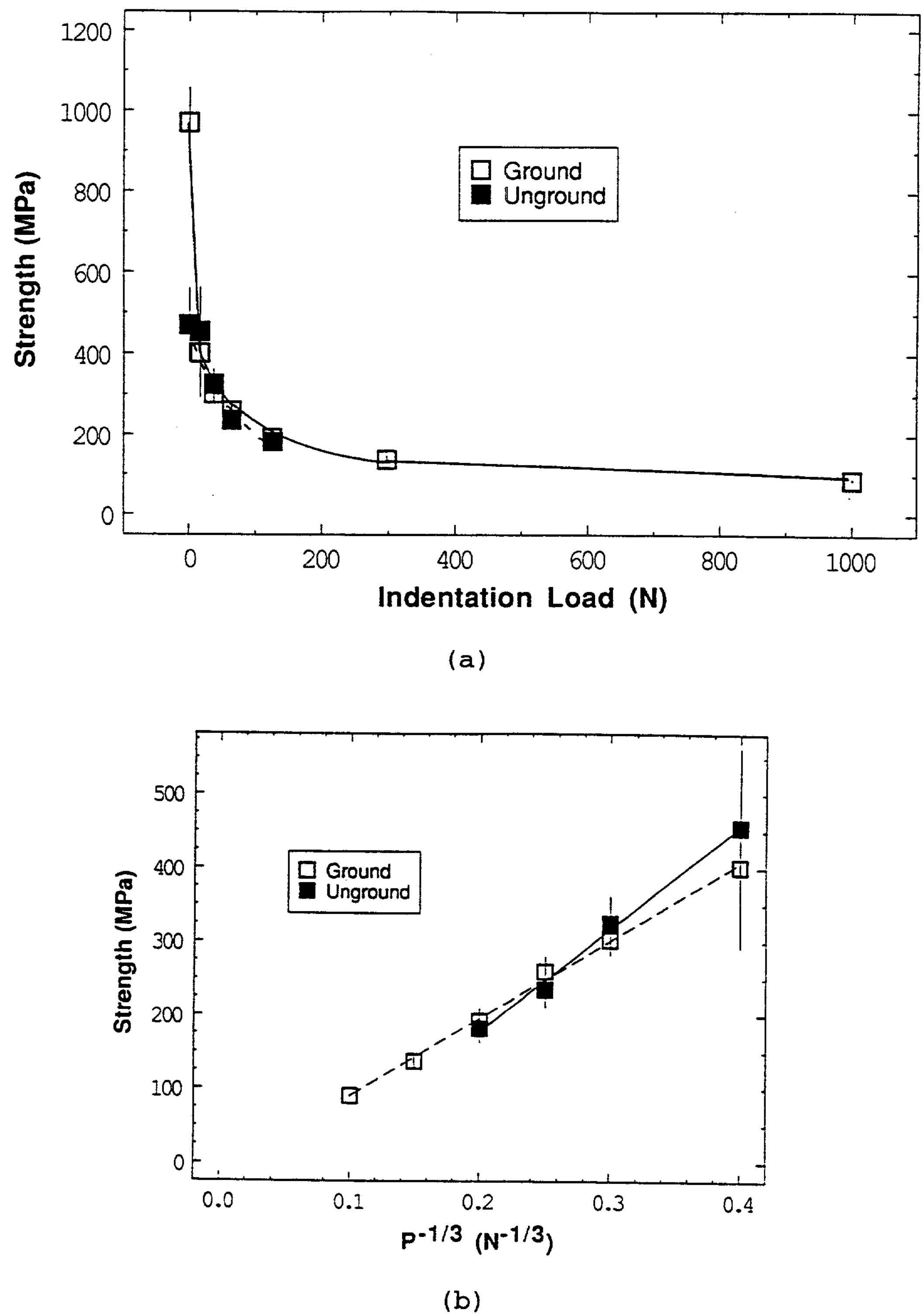


Figure 12. Indentation/strength measurements on ground and unground inner layer material. Strength as a function of (a) indentation load, and (b) inverse cube root indentation load. Note that strength is similar at loads of 16 N.

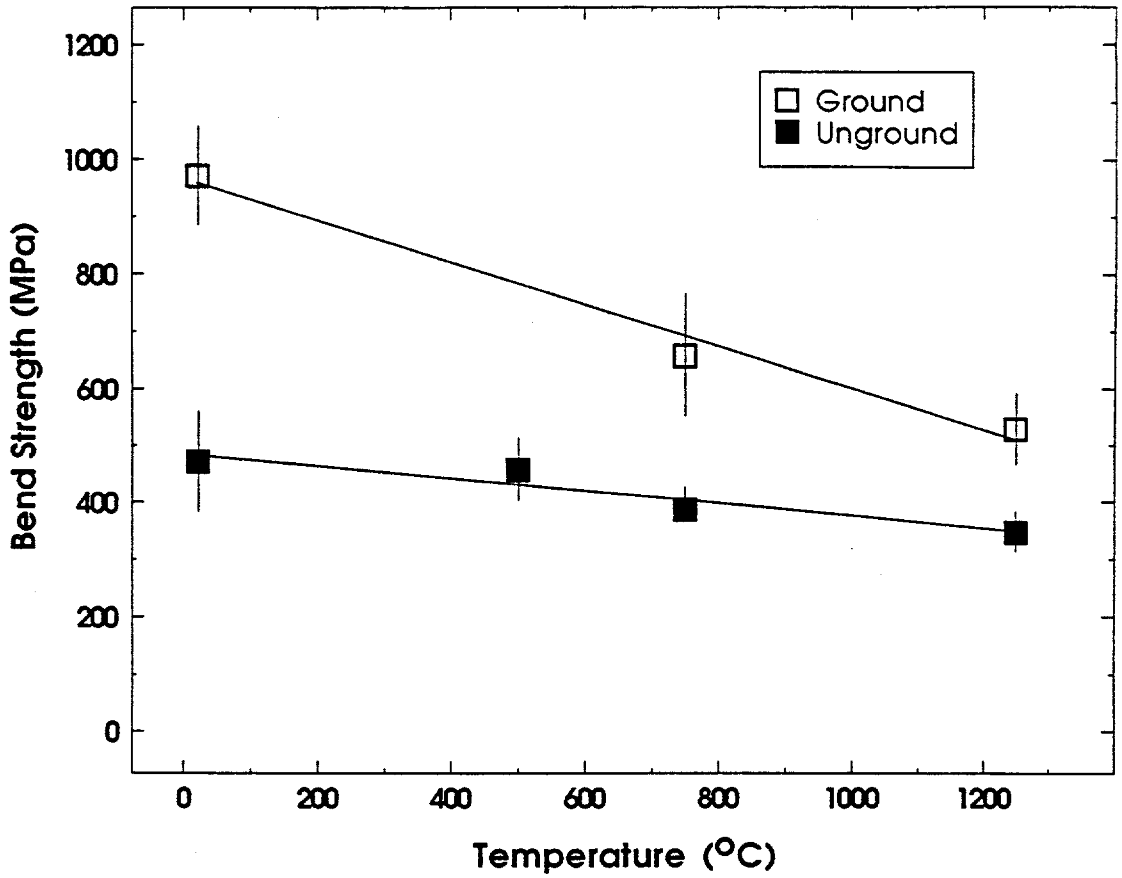


Figure 13. Strength of ground (open) and unground (solid) inner layer material (Al_2O_3-15 vol. % ZrO_2(3 mol.% Y_2O_3)) as a function of temperature.

minutes. Since the inner layer material does not contain monoclinic ZrO_2, it does not show any strength hysteresis.

The difference in strength between ground and unground inner layer material is not due to transformation-induced stress as in the three-layer material. The larger surface flaws on the unground surfaces account for part of the difference in strength, but the experiments described above showed that polishing unground samples to a high surface finish did not result in comparable strengths to the ground bars.

The high strength ground inner material, however, is susceptible to surface damage, showing similar strength to unground inner material at indentation loads as low as 16 N (see Figure 12). These data indicate that the depth of compressive stress, due to surface grinding, is shallow and does not provide protection against surface flaws. This is consistent with shallow surface compression measured in Si_3N_4-based ceramics after grinding[20]. As shown below, a primary advantage of layered composites is their greatly enhanced damage resistance.

DAMAGE RESISTANCE

Indentation/strength testing of slip cast monolithic and three-layer Al_2O_3-15 vol. % ZrO_2 was performed in order to determine their response in comparison to the dry pressed bars tested previously[11]. The main differences between the dry pressed and the slip cast bars were the improved strength of all three slip cast materials and the increased uniformity of the outer layer thickness in the three-layer slip cast bars (see Figure 9). In addition, the thickness of the outer layers of slip cast three-layer bars was ≈250 μm in comparison to dry pressed bars with outer layer thickness of ≈375 μm. The indentation/strength response of three-layer composites was compared with theoretical expectation based on superposition of stress. It can easily be shown[11] that the strength expected for three-layer composites which have been indented with a Vickers indenter of load P is given by

$$\sigma_f = CK_{Ic}{}^{4/3}/((E/H)^{1/6}P^{1/3}) + \Delta\varepsilon_o E d_2/(1-\upsilon)d \qquad (9)$$

where C is a constant equal to 2.02[20,21], K_{Ic} is fracture toughness and H is hardness. According to Equation (9), a plot of σ_f vs $P^{-1/3}$, as shown in Figure 14, should yield a straight line with a slope related to K_{Ic} and an intercept giving the compressive residual stress, σ_1. Taking values of E=340 GPa, H=17 GPa, linear regression of the data for monolithic specimens gave slopes corresponding to K_{Ic} values of 5.35 and 5.03 $MPa\cdot m^{1/2}$ for outer and inner materials, respectively. Both materials had intercepts near zero, showing that they were free of residual stress (see Figure 14). A linear regression of the data for the three-layer composites gave a slope corresponding to a fracture toughness of 5.75 $MPa\cdot m^{1/2}$, similar to the monolithic materials, as predicted previously[11]. It is interesting to note, however, that there was very little decrease in strength at high indentation loads (greater than 125 N) suggesting that the three-layer material has even better damage resistance than predicted by Equation (9). The intercept from the linear regression gave a value of -588 MPa for the compressive stress. This value of σ_1 is higher than the difference in strength of -497 MPa when comparing the unindented bars. These data confirm the superior damage resistance of

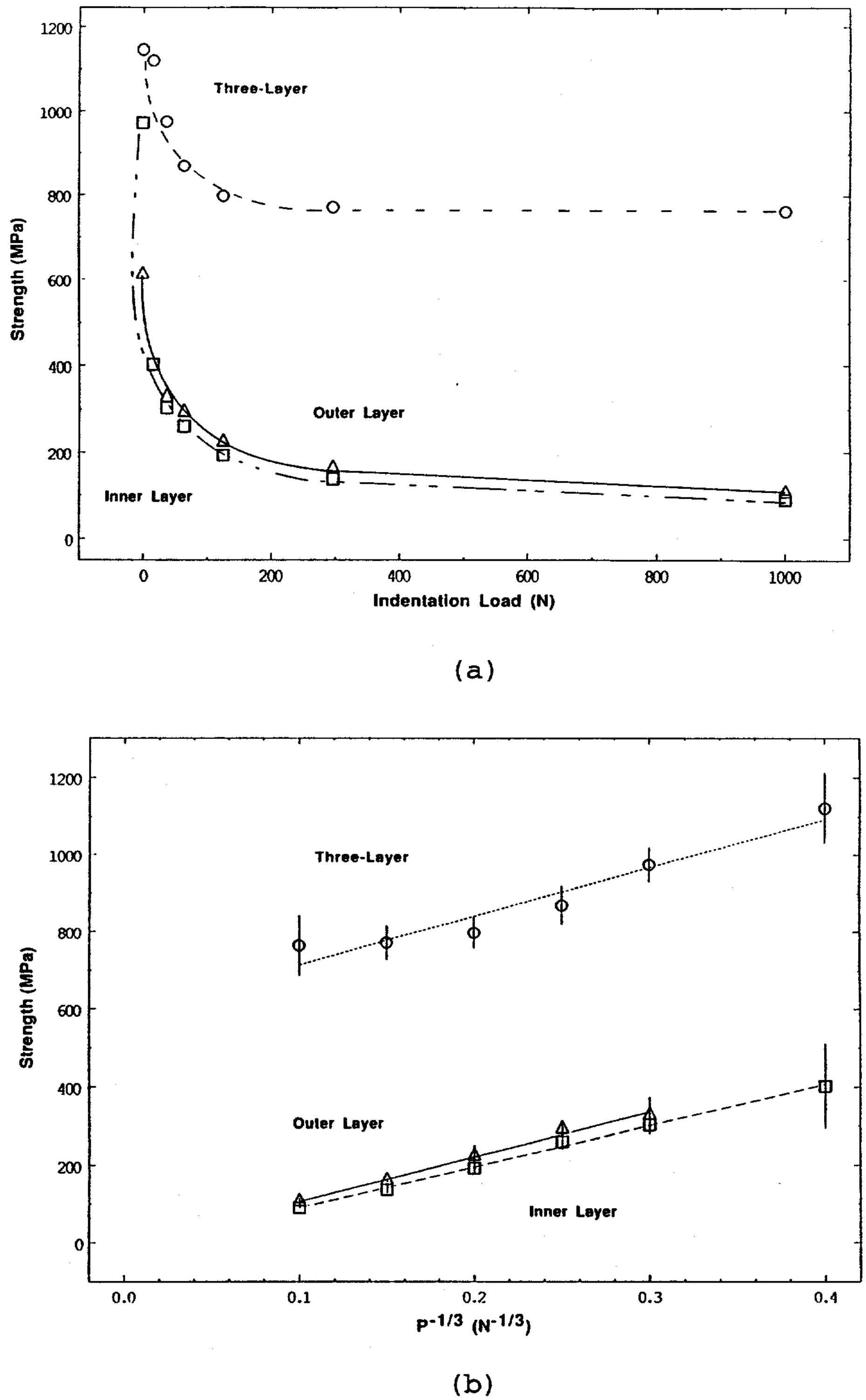


Figure 14. Fracture stress (σ_f) versus (a) load (P) and (b) inverse cube root load ($P^{-1/3}$) plots for the slip cast three-layer and monolithic Al_2O_3-15 vol. % ZrO_2 ceramics.

materials made using the three-layer concept and show that improved resistance to contact damage can be expected for ceramic components made using this technique.

In order to show the practical extension of this technology, totally encapsulated ≈5 mm diameter rods were fabricated by slip casting such that pits on the order of 50-250 μm were prevalent on the surface but were rarely present in the bulk. The outer layer of the rods was ≈425 μm thick and surface compressive stress was on the order of 400 MPa. The strength (5 rods broken in 4-point bending using an inner span of 20 mm and an outer span of 40 mm) of the "as-HIPed" material was 908±116 MPa, as compared to strength of 1,211±123 MPa for three-layer rods which were ground to a 30 μm surface finish. In contrast, monolithic outer layer rods had strength of 476±84 MPa in the "as-HIPed" state and 830±27 MPa in the ground state. The change in strength between the three-layer and monolithic outer layer materials was improved at larger flaw sizes. This means that as long as the outer layer thickness of the three-layer composite is sufficiently larger than the surface flaws, that strength improvement consistent with the indentation/strength data can be expected in components.

In order to further explore the use of three-layer composites, monolithic and three-layer (outer layer ≈1/12th the total thickness of the ceramic) composite cam followers were fabricated and tested by Chrysler Motor Corp. The o.d. of the roller was ≈17.8 mm, the i.d. was ≈7.59 mm, and the length ≈12.7 mm. Ceramatec had previously tested Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3) cam followers of similar geometry which failed the lash test where high impact loads are applied. The test consisted of running the rollers on a motorized 2.2 L cylinder head at 2750 rpm for five minutes with a 0.635-0.762 mm (0.025-0.030") lash between the roller and the base circle of the cam. Previous tests were run at 3000 rpm, but due to limitations of the test fixture used at Chrysler only 2750 rpm were applied.

Fifteen rollers were supplied and eight were selected for testing. One monolithic outer roller broke during setup, most likely due to expansion of the pin during welding. Of the seven rollers tested, six of them passed the lash test. This is in stark contrast to the previous testing of the inner layer material made by dry pressing where three out of three rollers failed the lash test. Two of each of the three types of materials (i.e., monolithic outer, monolithic inner, and three-layer) tested survived five minutes of the lash test. The only material to fail was a three-layer roller which was terminated after 18 seconds.

Due to the fact that the monolithic materials survived the lash test, it was requested that a larger lash be applied to see if the compressive stress in the outer layer of the three-layer material allowed improved damage resistance. Additional testing was not performed by Chrysler for two reasons: 1) the coefficient of friction for Si_3N_4 is lower than Al_2O_3-ZrO_2 and wear is consequently improved for silicon nitride rollers compared to oxide-based rollers (no wear measurements were done on the slip cast rollers sent), and 2) the cost per ground roller was $0.50/roller and Chrysler was only interested in testing rollers from suppliers who were serious about getting into this business (At the time the rollers were tested, Si_3N_4 supplied by Sullivan Mining Co. looked very promising and Ceramatec was not committed to competing in this market). To the best of the authors' knowledge, these are the only tests performed where aluminum oxide-based ceramics have passed the impact test.

THERMAL SHOCK TESTING

Fracture strengths of monoliths and three-layer rods after thermal shocking over different temperature ranges are displayed in Figure 15. The as-HIPed strengths were 830±27 MPa, 1185±109 MPa, and 1206±36 MPa for outer, inner and layered composite rods, respectively. These strengths are similar to those reported above for bars in four-point bending. The inner layer rods had a ΔT of slightly less than 300°C, the outer layer rods had a ΔT of ≈325°C and the layered rods had a ΔT of ≈425°C. The exposed ends of the layered rods were the regions most susceptible to thermal shock and totally encapsulated rods would likely have resulted in a higher ΔT for this material. Individual layered composite rods had strengths greater than 1200 MPa at temperatures up to 425°C. Since thermal shock under severe cooling conditions generally initiates from the outer surface of monolithic components, the increase in thermal shock resistance of the three-layer rods over the outer layer rods is of interest. This increase of 100°C can be compared with what would be expected for the strength improvement due to the surface compressive layer. The compressive stress, σ_1, in the outer case and the balancing tensile stress, σ_2, in the inner core can be easily calculated assuming a square-wave stress distribution. These stresses are approximately given by

$$\sigma_1 = -(A_2/A)E\Delta\varepsilon_o/(1-\upsilon) \tag{10}$$

and

$$\sigma_2 = (A_1/A)E\Delta\varepsilon_o/(1-\upsilon) \tag{11}$$

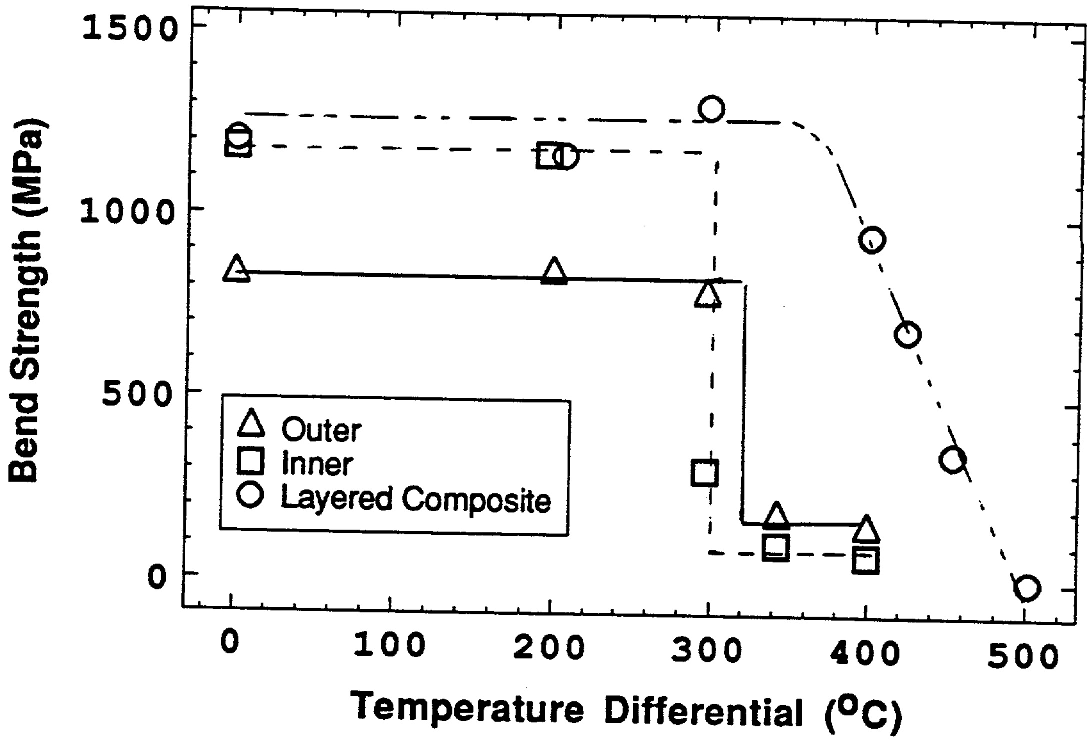


Figure 15. Thermal shock behavior of monolithic and "three-layer" Al_2O_3-15 vol. % ZrO_2 quenched in ice water. Layered rods with surface compressive stress of ≈400 MPa have critical ΔT 100°C higher than monolithic rods.

where A is the cross-sectional area of the rods. The outer case thickness was approximately 0.375 mm or 1/12th the diameter of the rods. Taking values of 4.86×10^{-6} m^2, 1.10×10^{-5} m^2, and 1.59×10^{-5} m^2 for A_1, A_2, and A, respectively, E of 340 GPa, $\Delta\varepsilon_o$ of 1.3×10^{-3} and Poisson's ratio of 0.25, results in a compressive residual stress of 409 MPa in the case and a residual tensile stress of 180 MPa in the core. The calculated compressive stress is in good agreement with the increase in room temperature strength between the outer and the layered composite rods of 376 MPa.

Based on the early work of Hasselman[23,24] it is possible to explain the improved thermal shock resistance of the Al_2O_3-15 vol. % ZrO_2 ceramics having substantial compressive surface stresses as compared to the monolithic ceramics of similar composition, modulus and thermal expansion. The expected increase in ΔT_c, the critical temperature difference to which the rods are subjected in order to initiate crack growth and decrease strength, for a material with compressive residual stress is given by

$$\Delta T_c = \Delta T_c^{\circ} + [\sigma_c(1-\upsilon)/\alpha E]f(k/ha) \quad (12)$$

where ΔT_c° is the temperature differential in the absence of residual stress given by $[\sigma_f(1-\upsilon)/aE]f(k/ah)$, where σ_f is the unquenched strength, α is linear coefficient of thermal expansion, k is the thermal conductivity, h is the surface heat-transfer coefficient and a is the characteristic heat transfer length. The work of Becher et al.[25] investigating the effect of sample size on the thermal shock resistance of ceramics shows the need to take the Biot modulus (ah/k) into account in calculating ΔT_c. For a conservative prediction, a relatively high heat transfer coefficient, h=10 W/cm^2°C was assumed. With a=0.225 cm and k=0.25 W/cm°C, the Biot modulus equals 9. For this value of ß, f(k/ah)=0.433. From Equation (12), the predicted increase in thermal shock resistance for three-layer rods is 243°C for σ_1=-376 MPa. The increase obtained in experiments was ≈100°C. The reason for this discrepancy is likely related to the fact that the ends of the rods were not encapsulated. The reason for the low values for ΔT_c (300-425°C) measured in the present study in contrast to the high values (ΔT_c > 800°C) reported by Becher[26] for monolithic Al_2O_3-ZrO_2 composites is believed to be due to the more vigorous quench of the ice water as compared to boiling water[27] and to the difference in sample thickness between the two studies.

Improved thermal shock resistance of the three-layer composites is expected due to the presence of residual compressive stresses. These results show that residual compressive stress of substantial depth in layered composites not only increases strength, apparent toughness[7], and damage resistance, but also makes the materials more resistant to thermal shock.

IMPROVED RELIABILITY

Superposition of temperature stress (due to difference in thermal expansion) on transformation-induced stress (due to volume expansion differences between monoclinic and tetragonal ZrO_2) was demonstrated using ZrO_2(3 mol. % Y_2O_3)-40 vol. % Al_2O_3 as the inner layer material in three-layer slip cast composites. The temperature-induced stress enhance the strength of three-layer composites at low temperatures.

The expected compressive residual stress in the outer layer of the bars is the combination of transformation-induced and temperature-induced stresses. The transformation-induced compressive stress, σ_1, in the outer layer, assuming a square wave stress distribution, is

$$\sigma_1 = \frac{-(E_1E_2d_2\Delta\varepsilon_o)}{[(1-\upsilon)(2E_1d_1+E_2d_2)]} \tag{13}$$

in an analogous manner to Equation (1). The temperature induced stress, σ_1, in the outer layer, also assuming a square wave distribution, is given as

$$\sigma_1 = \frac{-[(E_1E_2d_2\Delta T)(\alpha_2-\alpha_1)]}{[(1-\upsilon)(2E_1d_1+E_2d_2)]} \tag{14}$$

where ΔT is the temperature difference over which stress builds up and α is the coefficient of linear thermal expansion. By superposition, the expected residual stress in the outer layer is

$$\sigma_1 = \frac{-(E_1E_2d_2)(\Delta\varepsilon_o+\Delta T(\alpha_2-\alpha_1))}{[(1-\upsilon)(2E_1d_1+E_2d_2)]} \tag{15}$$

Taking E_1 as 340 GPa, E_2 as 275 GPa, $\Delta\varepsilon_o$ as 1.3×10^{-3}, ΔT as 1000°C, $\alpha_2-\alpha_1$ as 1×10^{-6}/°C, υ as 0.25, d_1 as 500 µm, and d_2 as 5 mm, the expected compressive residual stress in the outer layer is slightly over 1,000 MPa. The corresponding residual tensile stress in the inner layer is approximately 200 MPa. Strain gage measurements resulted in a measured compressive stress of 1,100 MPa, in excellent agreement with prediction.

The room temperature fracture strength of three-layer composites was 1,275 MPa as compared to a strength of 549 MPa for the outer layer bars. The compressive stress of -725 (difference between inner and three-layer bars) is 70% of the predicted value. More importantly, it shows that high strengths can be achieved in layered ceramic composites using a combination of transformation and temperature-induced stresses, as expected.

Figure 16 shows linearized Weibull plots of fracture stresses of monolithic outer, monolithic inner and three-layer composites with transformation-induced stresses,

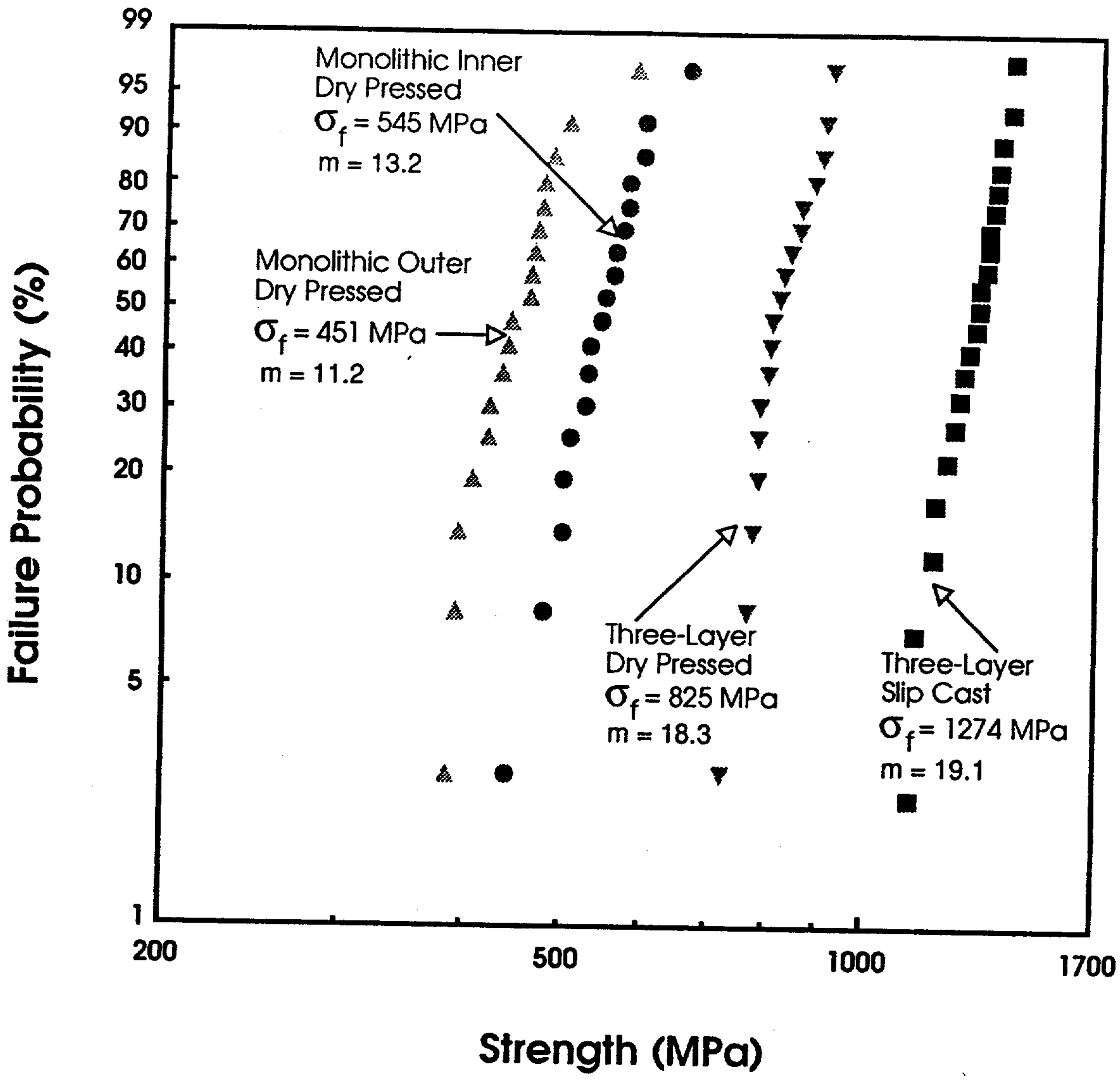


Figure 16. Linearized Weibull plots of fracture stress of monolithic and three-layer composites. Slip cast composites have both transformation-induced and temperature-induced residual stress. Note higher modulus of three-layer composites compared to monoliths, indicating enhanced reliability.

fabricated by dry pressing, as well as slip cast three-layer composites with both transformation-induced and temperature-induced stresse. It is noted that the three-layer composites exhibit both increased strengths as well as improved Weibull moduli relative to the monolithic ceramics. Improvements in uniformity in the thickness of the layers by slip casting is the main reason for the improved strength of the slip cast composites. The improvement in Weibull modulus is due to superposition of stress[7]. Residual compression deliberately introduced into the near surface regions of structural ceramics can be a viable approach to increase Weibull modulus and reliability.

ELEVATED TEMPERATURE TESTING

Strength measurements as a function of temperature are shown in Figure 17 for monolithic outer, inner and three-layer samples. At room temperature the three-layer composites have a strength of 1150 MPa as compared to a strength of 660 MPa for the outer layer monolithic bars. The strength differential between the three-layer and outer layer bars, as shown in Figure 18, is due to the compressive

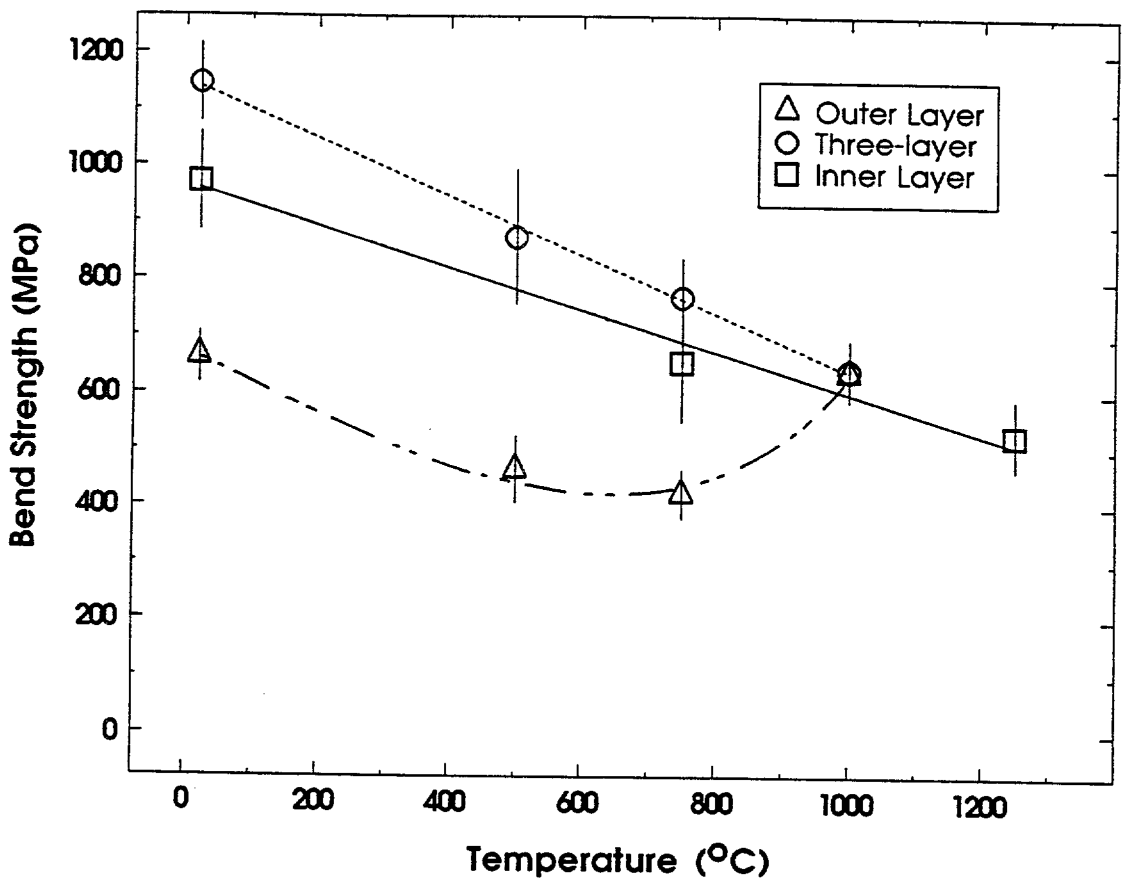


Figure 17. Strength of three-layer Al_2O_3-15 vol. % ZrO_2 composites as a function of temperature, in comparison to monolithic inner (Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3)) and outer (Al_2O_3-15 vol. % ZrO_2) layer materials.

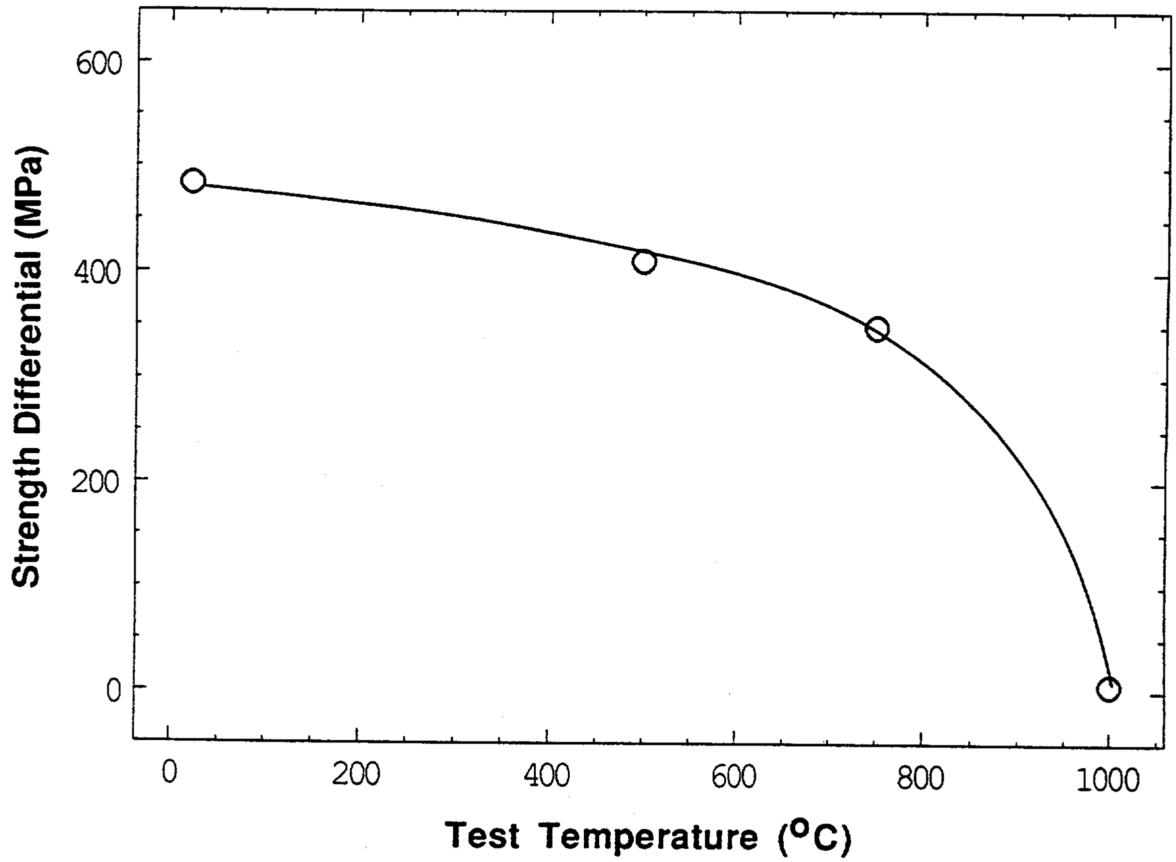


Figure 18. Strength differential between three-layer and outer layer materials. Strength differential is due to compressive residual stress in the outer layers of the three-layer composite.

residual stress in the outer layers of the three-layer bars. The experimentally observed value of -490 MPa from strength testing agrees well with the value of -520 MPa from strain gage measurements.

The residual stresses are effective in strengthening the three-layer composites until the monoclinic to tetragonal (m-->t) transformation is completed at a temperature above 750°C (see Figure 18). Dilatometric studies of the outer layer material show the A_s temperature as ≈900°C and the A_f temperature as ≈1000°C. The M_s temperature was ≈600°C and the M_f temperature was ≈500°C. Since these temperatures are dependent on the constraint of the ZrO_2 in the outer layers, these temperatures may be shifted slightly lower in the three-layer composite. Figure 19 shows the strength

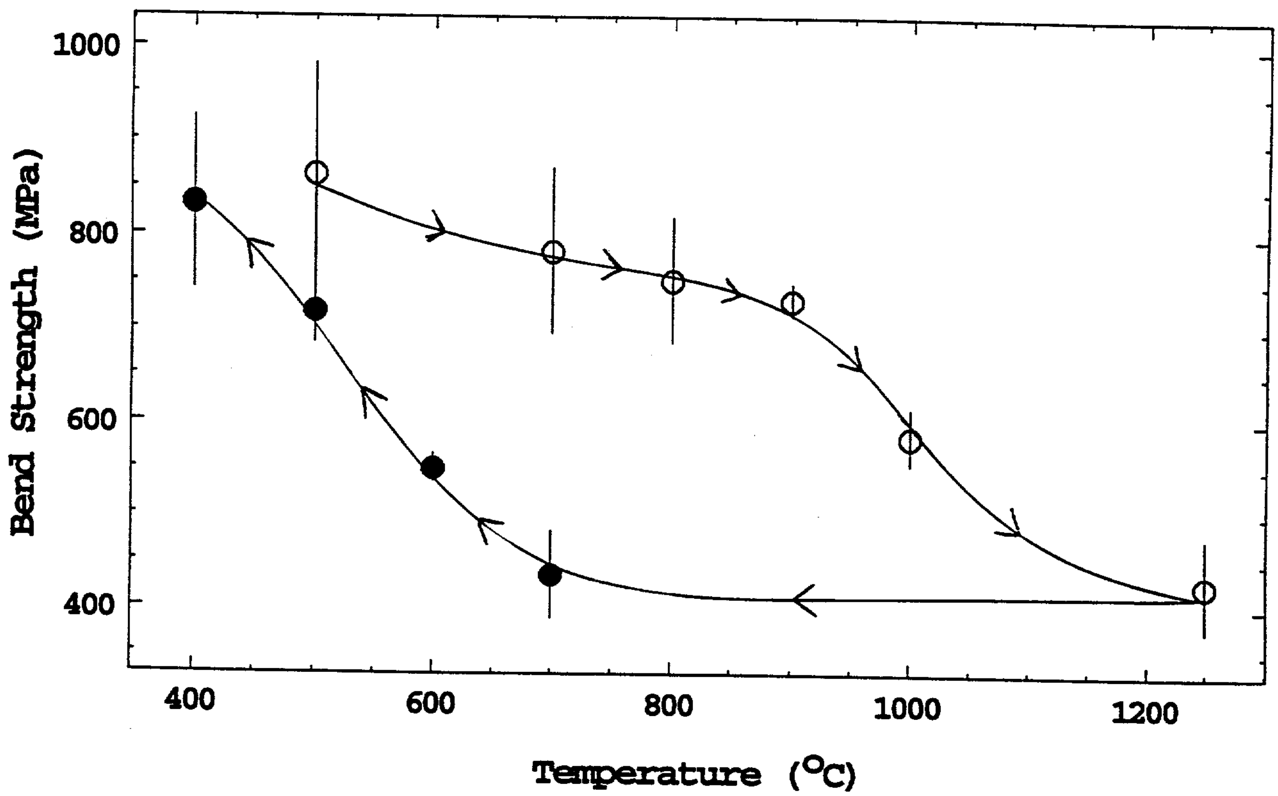


Figure 19. Strength hysteresis for three-layer Al_2O_3-15 vol. % ZrO_2 composites as a function of temperature cycling. Open symbols represent samples heated to the test temperature. Solid symbols represent samples heated to 1250°C and then cooled to the test temperature. Note that when layered composites are heated above A_s, the martensitic start temperature during heating, that strength is not fully recovered until the samples are cooled below the M_f, the martensitic finish temperature during cooling.

hysteresis measured for three-layer composites cycled above the A_f temperature and tested on cooling above the M_s temperature. These results are in excellent agreement with expectation and show that layered composites cycled above the A_s temperature will have decreased strength during cooling until they are cooled below the M_f temperature. This limits the use of layered Al_2O_3-ZrO_2 ceramics with high damage resistance to temperatures not exceeding ≈800°C (see Figures 18 and 19).

Substituting HfO_2 for ZrO_2 is the most straightforward approach for increasing the use temperature of layered composites. Al_2O_3-15 vol. % HfO_2, Al_2O_3-15 vol. % HfO_2(4 mol. % Y_2O_3), and HfO_2(4 mol. % Y_2O_3) compositions were dry pressed, sintered and HIPed using hafnia powders received from Teledyne Wah Chang Albany. X-ray diffraction showed that the sintering temperature of 1600°C was not high enough to fully convert the monoclinic $HfO_2(Y_2O_3)$ to tetragonal. Approximately 60 % of the $HfO_2(Y_2O_3)$ was monoclinic after holding for two hours at 1600°C. The $HfO_2(Y_2O_3)$ with no Al_2O_3 added had a strength of 329±28 MPa. Monolithic outer (Al_2O_3-15 vol. % HfO_2), monolithic inner (Al_2O_3-15 vol. % HfO_2(4 mol. % Y_2O_3)) and three-layer composites had strengths of 458±30 MPa, 437±22 MPa, and 492±31 MPa, respectively. The thermal expansion of the monolithics materials were both ≈8.4×10^{-6}/°C over the temperature range 25-1000°C. The slightly higher strength of the three-layer composites is not likely due to transformation-induced stresses since sintering occurred in the monoclinic stability range for the unstabilized HfO_2.

In an effort to lower the m-->t transformation temperature, $HfO_2 \cdot ZrO_2$ powder was made and x-rayed to verify that a solid solution had formed. Al_2O_3-15 vol. % ($HfO_2 \cdot$50 mol. % ZrO_2) was used as the outer layer and Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3)) was the inner layer in three-layer composites. The room temperature strength of the three-layer composite was 947±164 MPa and the strength at 1000°C was 523±23 MPa. The strength at 1000°C is not improved over the strength of 644±51 MPa measured at the same temperature for three-layer Al_2O_3-15 vol. % ZrO_2 composites. While the concept of improving the high temperature strength of three-layer composites by substituting HfO_2 for ZrO_2 is sound, improved HfO_2 or $HfO_2 \cdot ZrO_2$ powders will be required before this concept is realized in practice.

SUMMARY AND CONCLUSIONS

Three-layer oxide ceramics with compressive residual stress ranging between 300 and 600 MPa in the outer layers were fabricated using dry pressing and slip casting. The outer layer thickness was controlled in the green state. The outer layer protected against damage due to surface flaws or sliding contact.

Transformation-induced stresses were present at temperatures in excess of 750°C. Stress due to the thermal expansion mismatch between inner and outer layers were superimposed to give strength increases greater than 500 MPa at room temperature.

Slip casting was used to improve the uniformity of the interface between layers to allow composites with outer layer thicknesses of 200-300 μm to be fabricated. The room temperature strength of Al_2O_3-15 vol. % ZrO_2 composites increased from 825 MPa to 1150 MPa and the strength at 1000°C increased from 320 MPa to 640 MPa. Strength in excess of 1200 MPa at room temperature was achieved by superimposing temperature stress on transformation-induced stress. Both monolithic and layered cam rollers fabricated by slip casting survived the impact testing required for cam followers in automobiles.

In addition to strength measurements, residual stresses were detected by strain gage measurements, characterization of monoclinic content as a function of temperature by x-ray diffraction, strength and indentation/strength measurements, indentation crack length measurements, and thermal shock testing.

Grinding dramatically improves the strength of Al_2O_3-15 vol. % ZrO_2(3 mol. % Y_2O_3) without creating substantial transformation-induced stresses detectable by standard x-ray techniques. These monolithic materials do have surface induced residual stresses which do not protect against surface damage. In contrast, three-layer composites, with outer layers exceeding 100 μm, have excellent damage resistance. Further work is needed to identify applications where layered composites are cost effective in replacing monolithic ceramics due to their improved damage resistance.

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Short Hills NJ 07078

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22 Commerce Road
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21000 Brookpark Road, MS:49-3
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Argonne National Laboratory
9700 S. Cass Avenue, Bldg. 900
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Shanghai Institute of Ceramics
1295 Ding-xi Road
Shanghai 200050
CHINA

J. P. Gallagher
University of Dayton Research Institute
300 College Park, JPC-250
Dayton OH 45469-0120

Garry Garvey
Golden Technologies Company Inc.
4545 McIntyre Street
Golden CO 80403

Richard Gates
NIST
Materials Bldg., A-256
Gaithersburg MD 20899

L. J. Gauckler
ETH-Zurich
Sonneggstrasse 5
CH-8092 Zurich 8092
SWITZERLAND

George E. Gazza
U.S. Army Materials Technology
Ceramics Research Division
405 Arsenal Street
Watertown MA 02172-0001

D. Gerster
CEA-DCOM
33 Rue De La Federation
Paris 75015
FRANCE

John Ghinazzi
Coors Technical Ceramics Company
1100 Commerce Park Drive
Oak Ridge TN 37830

Robert Giddings
General Electric Company
P.O. Box 8
Schenectady NY 12301

A. M. Glaeser
University of California
Lawrence Berkeley Laboratory
Hearst Mining Building
Berkeley CA 94720

Joseph W. Glatz
Naval Air Propulsion Center
Systems Engineering Division
510 Rocksville Road
Holland PA 18966

W. M. Goldberger
Superior Graphite Company
R&D
2175 E. Broad Street
Columbus OH 43209

Allan E. Goldman
U.S. Graphite, Inc.
907 W. Outer Drive
Oak Ridge TN 37830

Stephen T. Gonczy
Allied Signal Research
P.O. Box 5016
Des Plaines IL 60017

Jeffrey M. Gonzales
GTE Products Corporation
Hawes Street
Towanda PA 18848-0504

Robert J. Gottschall
U.S. Department of Energy
ER-131, MS:G-236
Washington DC 20585

Earl Graham
Cleveland State University
Dept. of Chemical Engineering
Euclid Avenue at East 24th Street
Cleveland OH 44115

John W. Graham
Astro Met, Inc.
9974 Springfield Pike
Cincinnati OH 45215

G. A. Graves
U. of Dayton Research Institute
300 College Park
Dayton OH 45469-0001

Robert E. Green, Jr.
Johns Hopkins University
Materials Science and Engineering
Baltimore MD 21218

Alex A. Greiner
Plint & Partners
Oaklands Park
Wokingham Berkshire RG11 2FD
UNITED KINGDOM

Lance Groseclose
General Motors Corporation
Allison Gas Turbine Division
P.O. Box 420, MS:W-5
Indianapolis IN 46206

Thomas J. Gross
U.S. Department of Energy
Transportation Technologies
CE-30, Forrestal Building
Washington DC 20585

Mark F. Gruninger
Union Carbide Corporation
Specialty Powder Business
1555 Main Street
Indianapolis IN 46224

Ernst Gugel
Cremer Forschungsinstitut
GmbH&Co.KG
Oeslauer Strasse 35
D-8633 Roedental 8633
GERMANY

John P. Gyekenyesi
NASA Lewis Research Center
21000 Brookpark Road, MS:6-1
Cleveland OH 44135

Nabil S. Hakim
Detroit Diesel Corporation
13400 Outer Drive West
Detroit MI 48239

Philip J. Haley
General Motors Corporation
P.O. Box 420, MS:T12A
Indianapolis IN 46236

Judith Hall
Fiber Materials, Inc.
Biddeford Industrial Park
5 Morin Street
Biddeford ME 04005

Y. Hamano
Kyocera Industrial Ceramics Corp.
5713 E. Fourth Plain Blvd.
Vancouver WA 98661-6857

Y. Harada
IIT Research Institute
10 West 35th Street
Chicago IL 60616

R. A. Harmon
25 Schalren Drive
Latham NY 12110

Norman H. Harris
Hughes Aircraft Company
P.O. Box 800520
Saugus CA 91380-0520

Alan M. Hart
Dow Chemical Company
1776 Building
Midland MI 48674

Pat E. Hart
Battelle Pacific Northwest Labs
Ceramics and Polymers Development
P.O. Box 999
Richland WA 99352

Michael H. Haselkorn
Caterpillar In.
Technical Center, Building E
P.O. Box 1875
Peoria IL 61656-1875

Debbie Haught
U.S. Department of Energy
Off. of Transportation Materials
EE-34, Forrestal Bldg.
Washington DC 20585

N. B. Havewala
Corning Inc.
SP-PR-11
Corning NY 14831

John Haygarth
Teledyne WAA Chang Albany
P.O. Box 460
Albany OR 97321

Norman L. Hecht
U. of Dayton Research Institute
300 College Park
Dayton OH 45469-0172

Peter W. Heitman
General Motors Corporation
P.O. Box 420, MS:W-5
Indianapolis IN 46206-0420

Robert W. Hendricks
VPI & SU
210 Holden Hall
Blacksburg VA 24061-0237

Thomas L. Henson
GTE Products Corporation
Chemical & Metallurgical Division
Hawes Street
Towanda PA 18848

Thomas P. Herbell
NASA Lewis Research Center
21000 Brookpark Road, MS:49-3
Cleveland OH 44135

Marlene Heroux
Rolls-Royce, Inc.
2849 Paces Ferry Road, Suite 450
Atlanta GA 30339-3769

Robert L. Hershey
Science Management Corporation
1255 New Hampshire Ave., N.W.
Suite 1033
Washington DC 20036

Hendrik Heystek
Bureau of Mines
Tuscaloosa Research Center
P.O. Box L
University AL 35486

Robert V. Hillery
GE Aircraft Engines
One Neumann Way, M.D. H85
Cincinnati OH 45215

Arthur Hindman
Instron Corporation
100 Royall Street
Canton MA 02021

Hans Erich Hintermann
CSEM
Rue Breguet 2
Neuchatel 2000
SWITZERLAND

Shinichi Hirano
Mazda R&D of North America, Inc.
1203 Woodridge Avenue
Ann Arbor MI 48105

Tommy Hiraoka
NGK Locke, Inc.
1000 Town Center
Southfield MI 48075

Fu H. Ho
General Atomics
P.O. Box 85608
San Diego CA 92186-9784

John M. Hobday
U.S. Department of Energy
Morgantown Energy Technology Ctr
P.O. Box 880
Morgantown WV 26507

Clarence Hoenig
Lawrence Livermore National Lab
P.O. Box 808, Mail Code L-369
Livermore CA 94550

Thomas Hollstein
Fraunhofer-Institut fur
Werkstoffmechanik
Wohlerstrasse 11
79108 Freiburg
GERMANY

Richard Holt
National Research Council Canada
Structures and Materials Lab
Ottawa Ontario K1A OR6
CANADA

Woodie Howe
Coors Technical Ceramics Company
1100 Commerce Park Drive
Oak Ridge TN 37830

Stephen M. Hsu
NIST
Gaithersburg MD 20899

Hann S. Huang
Argonne National Laboratory
9700 S. Cass Avenue
Argonne IL 60439-4815

Gene Huber
Precision Ferrites & Ceramics
5576 Corporate Drive
Cypress CA 90630

Harold A. Huckins
Princeton Advanced Technology
4 Bertram Place
Hilton Head SC 29928

Fred R. Huettic
Advanced Magnetics Inc.
45 Corey Lane
Mendham NJ 07945

Brian K. Humphrey
Lubrizol Petroleum Chemicals Co.
3000 Town Center, Suite 1340
Southfield MI 48075-1201

Robert M. Humrick
Dylon Ceramic Technologies
3100 Edgehill Road
Cleveland Heights OH 44118

Lorretta Inglehart
National Science Foundation
Division of Materials Research
1800 "G" Street, N.W., Room 408
Washington DC 20550

Michael S. Inoue
Kyocera International, Inc.
8611 Balboa Avenue
San Diego CA 92123-1580

Joseph C. Jackson
U.S. Advanced Ceramics Assoc.
1600 Wilson Blvd., Suite 1008
Arlington VA 22209

Osama Jadaan
U. of Wisconsin-Platteville
1 University Plaza
Platteville WI 53818

Said Jahanmir
NIST
Materials Bldg., Room A-237
Gaithersburg MD 20899

Curtis A. Johnson
General Electric Company
P.O. Box 8
Schenectady NY 12301

Sylvia Johnson
SRI International
333 Ravenswood Avenue
Menlo Park CA 94025

Thomas A. Johnson
Lanxide Corporation
P.O. Box 6077
Newark DE 19714-6077

W. S. Johnson
Indiana University
One City Centre, Suite 200
Bloomington IN 47405

Walter F. Jones
AFOSR/NA
110 Duncan Ave., Ste. B115
Washington DC 20332-0001

Jill E. Jonkouski
U.S. Department of Energy
9800 S. Cass Avenue
Argonne IL 60439-4899

L. A. Joo
Great Lakes Research Corporation
P.O. Box 1031
Elizabethton TN 37643

A. David Joseph
SPX Corporation
700 Terrace Point
Muskegon MI 49443

Adam Jostsons
Australian Nuclear Science & Technology
New Illawarra Road
Lucas Heightss New South Wales
AUSTRALIA

Matthew K. Juneau
Ethyl Corporation
451 Florida Street
Baton Rouge LA 70801

Tom Kalamasz
Norton/TRW Ceramics
7A-4 Raymond Avenue
Salem NH 03079

Lyle R. Kallenbach
Phillips Petroleum
Mail Drop:123AL
Bartlesville OK 74004

Nick Kamiya
Kyocera Industrial Ceramics Corp.
25 Northwest Point Blvd., #450
Elk Grove Village IL 60007

Roy Kamo
Adiabatics, Inc.
3385 Commerce Park Drive
Columbus IN 47201

Chih-Chun Kao
Industrial Technology Research Institute
195 Chung-Hsing Road, Sec. 4
Chutung Hsinchu 31015 R.O.C.
TAIWAN

Keith R. Karasek
AlliedSignal Aerospace Company
50 E. Algonquin Road
Des Plaines IL 60017-5016

Martha R. Kass
U.S. Department of Energy
Oak Ridge Operations
Building 4500N, MS:6269
Oak Ridge TN 37831-6269

Robert E. Kassel
Ceradyne, Inc.
3169 Redhill Avenue
Costa Mesa CA 92626

Allan Katz
Wright Laboratory
Metals and Ceramics Division
Wright-Patterson AFB OH 45433

R. Nathan Katz
Worcester Polytechnic Institute
100 Institute Road
Worcester MA 01609

Tony Kaushal
Detroit Diesel Corporation
13400 Outer Drive, West
Detroit MI 48239-4001

Ted Kawaguchi
Tokai Carbon America, Inc.
375 Park Avenue, Suite 3802
New York NY 10152

Noritsugu Kawashima
TOSHIBA Corporation
4-1 Ukishima-Cho
Kawasaki-Ku Kawasaki 210
JAPAN

Lisa Kempfer
Penton Publishing
1100 Superior Avenue
Cleveland OH 44114-2543

Frederick L. Kennard, III
AC Rochester
1300 N. Dort Highway
Flint MI 48556

David O. Kennedy
Lester B. Knight Cast Metals Inc.
549 W. Randolph Street
Chicago IL 60661

George Keros
Photon Physics
3175 Penobscot Building
Detroit MI 48226

Thomas Ketcham
Corning, Inc.
SP-DV-1-9
Corning NY 14831

Pramod K. Khandelwal
General Motors Corporation
Allison Gas Turbine Division
P.O. Box 420, MS:W05
Indianapolis IN 46206

Jim R. Kidwell
AlliedSignal Engines
P.O. Box 52180
Phoenix AZ 85072-2180

Shin Kim
The E-Land Group
19-8 ChangJeon-dong
Mapo-gu, Seoul 121-190
KOREA

W. C. King
Mack Truck, Z-41
1999 Pennsylvania Avneue
Hagerstown MD 21740

Carol Kirkpatrick
MSE, Inc.
P.O. Box 3767
Butte MT 59702

Tony Kirn
Caterpillar Inc.
Defense Products Department, JB7
Peoria IL 61629

James D. Kiser
NASA Lewis Research Center
21000 Brookpark Road, MS:49-3
Cleveland OH 44135

Max Klein
900 24th Street, N.W., Unit G
Washington DC 20037

Richard N. Kleiner
Golden Technologies Company
4545 McIntyre Street
Golden CO 80403

Stanley J. Klima
NASA Lewis Research Center
21000 Brookpark Road, MS:6-1
Cleveland OH 44135

Albert S. Kobayashi
University of Washington
Mechanical Engineering Department
Mail Stop:FU10
Seattle WA 98195

Shigeki Kobayashi
Toyota Central Research Labs
Nagakute Aichi 480-11
JAPAN

Richard A. Kole
Z-Tech Corporation
8 Dow Road
Bow NH 03304

Joseph A. Kovach
Eaton Corporation
32500 Chardon Road
Willoughby Hills OH 44094

Kenneth A. Kovaly
Technical Insights Inc.
P.O. Box 1304
Fort Lee NJ 07024-9967

Ralph G. Kraft
Spraying Systems Company
North Avenue at Schmale Road
Wheaton IL 60189-7900

Arthur Kranish
Trends Publishing Inc.
1079 National Press Building
Washington DC 20045

A. S. Krieger
Radiation Science, Inc.
P.O. Box 293
Belmont MA 02178

Pieter Krijgsman
Ceramic Design International
Holding B.V.
P.O. Box 68
Hattem 8050-AB
THE NETHERLANDS

Waltraud M. Kriven
University of Illinois
105 S. Goodwin Avenue
Urbana IL 61801

Edward J. Kubel, Jr.
ASM International
Advanced Materials & Processes
Materials Park OH 44073

Dave Kupperman
Argonne National Laboratory
9700 S. Cass Avenue
Argonne IL 60439

Oh-Hun Kwon
North Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

W. J. Lackey
GTRI
Materials Science and Tech. Lab
Atlanta GA 30332

Jai Lala
Tenmat Ltd.
40 Somers Road
Rugby Warwickshire CV22 7DH
ENGLAND

Hari S. Lamba
General Motors Corporation
9301 West 55th Street
LaGrange IL 60525

Richard L. Landingham
Lawrence Livermore National Lab
P.O. Box 808, L-369
Livermore CA 94550

James Lankford
Southwest Research Institute
6220 Culebra Road
San Antonio TX 78228-0510

Stanley B. Lasday
Business News Publishing Co.
1910 Cochran Road, Suite 630
Pittsburgh PA 15220

S. K. Lau
Carborundum Company
Technology Division
P.O. Box 832, B-100
Niagara Falls NY 14302

J. Lawrence Lauderdale
Babcock & Wilcox
1850 "K" Street, Suite 950
Washington DC 20006

Jean F. LeCostaouec
Textron Specialty Materials
2 Industrial Avenue
Lowell MA 01851

Benson P. Lee
Technology Management, Inc.
4440 Warrensville Rd., Suite A
Cleveland OH 44128

Burtrand I. Lee
Clemson University
Olin Hall
Clemson SC 29634-0907

June-Gunn Lee
KIST
P.O. Box 131, Cheong-Ryang
Seoul 130-650
KOREA

Ran-Rong Lee
Ceramics Process Systems
Corporation
155 Fortune Boulevard
Mildford MA 01757

Stan Levine
NASA Lewis Research Center
21000 Brookpark Road, MS:49-3
Cleveland OH 44135

David Lewis, III
Naval Research Laboratory
Code 6370
Washington DC 20375-5343

Ai-Kang Li
Materials Research Labs., ITRI
195-5 Chung-Hsing Road, Sec. 4
Chutung Hsinchu 31015 R.O.C.
TAIWAN

Winston W. Liang
Hong Kong Industrial Technology
Centre
78 Tat Chee Avenue
4/F, HKPC Building -- Kowloon
HONG KONG

Robert Licht
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

E. Lilley
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

Chih-Kuang Lin
National Central University
Dept. of Mechanical Engineering
Chung-Li 32054
TAIWAN

Laura J. Lindberg
AlliedSignal Aerospace Company
Garrett Fluid Systems Division
P.O. Box 22200
Tempe AZ 85284-2200

Hans A. Lindner
Cremer Forschungsinstitut
GmbH&Co.KG
Oeslauer Strasse 35
D-8633 Rodental 8866
GERMANY

Ronald E. Loehman
Sandia National Laboratories
Chemistry & Ceramics Dept. 1840
P.O. Box 5800
Albuquerque NM 87185

Jeffrey C. Logas
Winona State University
115 Pasteur Hall
Winona MN 55987

Bill Long
Babcock & Wilcox
P.O. Box 11165
Lynchburg VA 24506

L. A. Lott
EG&G Idaho, Inc.
Idaho National Engineering Lab
P.O. Box 1625
Idaho Falls ID 83415-2209

Raouf O. Loutfy
MER Corporation
7960 S. Kolb Road
Tucson AZ 85706

Gordon R. Love
Aluminum Company of America
Alcoa Technical Center
Alcoa Center PA 15960

Lydia Luckevich
Ortech International
2395 Speakman Drive
Mississauga Ontario L5K 1B3
CANADA

James W. MacBeth
Carborundum Company
Structural Ceramics Division
P.O. Box 1054
Niagara Falls NY 14302

George Maczura
Aluminum Company of America
3450 Park Lane Drive
Pittsburgh PA 15275-1119

David Maginnis
Tinker AFB
OC-ALC/LIIRE
Tinker AFB OK 73145-5989

Frank Maginnis
Aspen Research, Inc.
220 Industrial Boulevard
Moore OK 73160

Tai-il Mah
Universal Energy Systems, Inc.
4401 Dayton-Xenia Road
Dayton OH 45432

Kenneth M. Maillar
Barbour Stockwell Company
83 Linskey Way
Cambridge MA 02142

S. G. Malghan
NIST
I-270 & Clopper Road
Gaithersburg MD 20899

Lars Malmrup
United Turbine AB
Box 13027
Malmo S-200 44
SWEDEN

John Mangels
Ceradyne, Inc.
3169 Redhill Avenue
Costa Mesa CA 92626

Murli Manghnani
University of Hawaii
2525 Correa Road
Honolulu HI 96822

Russell V. Mann
Matec Applied Sciences, Inc.
75 South Street
Hopkinton MA 01748

William R. Manning
Champion Aviation Products Div
P.O. Box 686
Liberty SC 29657

Ken Marnoch
Amercom, Inc.
8928 Fullbright Avenue
Chatsworth CA 91311

Robert A. Marra
Aluminum Company of America
Alcoa Technical Center
Alcoa Center PA 15069

Chauncey L. Martin
3M Company
3M Center, Building 60-1N-01
St. Paul MN 55144

Steve C. Martin
Advanced Refractory Technologies
699 Hertel Avenue
Buffalo NY 14207

Kelly J. Mather
William International Corporation
2280 W. Maple Road
Walled Lake MI 48088

James P. Mathers
3M Company
3M Center, Bldg. 201-3N-06
St. Paul MN 55144

Ron Mayville
Arthur D. Little, Inc.
15-163 Acorn Park
Cambridge MA 02140

F. N. Mazadarany
General Electric Company
Bldg. K-1, Room MB-159
P.O. Box 8
Schenectady NY 12301

James W. McCauley
Alfred University
Binns-Merrill Hall
Alfred NY 14802

Louis R. McCreight
2763 San Ramon Drive
Rancho Palos Verdes CA 90274

Colin F. McDonald
McDonald Thermal Engineering
1730 Castellana Road
La Jolla CA 92037

B. J. McEntire
Norton Company
10 Airport Park Road
East Granby CT 06026

Chuck McFadden
Coors Ceramics Company
600 9th Street
Golden CO 80401

Thomas D. McGee
Iowa State University
110 Engineering Annex
Ames IA 50011

Carol McGill
Corning Inc.
Sullivan Park, FR-02-08
Corning NY 14831

James McLaughlin
Sundstrand Power Systems
4400 Ruffin Road
P.O. Box 85757
San Diego CA 92186-5757

Matt McMonigle
U.S. Department of Energy
Improved Energy Productivity
CE-231, Forrestal Building
Washington DC 20585

J. C. McVickers
AlliedSignal Engines
P.O. Box 52180, MS:9317-2
Phoenix AZ 85072-2180

D. B. Meadowcroft
"Jura," The Ridgeway
Oxshott
Leatherhead Surrey KT22 OLG
UNITED KINGDOM

Joseph J. Meindl
Reynolds International, Inc.
6603 W. Broad Street
P.O. Box 27002
Richmond VA 23261-7003

Michael D. Meiser
AlliedSignal, Inc.
Ceramic Components
P.O. Box 2960, MS:T21
Torrance CA 90509-2960

George Messenger
National Research Council of Canada
Building M-7
Ottawa Ontario K1A OR6
CANADA

D. Messier
U.S. Army Materials Technology
SLCMT-EMC
405 Arsenal Street
Watertown MA 02172-0001

Arthur G. Metcalfe
Arthur G. Metcalfe and Associates, Inc.
2108 East 24th Street
National City CA 91950

R. Metselaar
Eindhoven University
P.O. Box 513
Endhoven 5600 MB
THE NETHERLANDS

David J. Michael
Harbison-Walker Refractories Co.
P.O. Box 98037
Pittsburgh PA 15227

Ken Michaels
Chrysler Motors Corporation
P.O. Box 1118, CIMS:418-17-09
Detroit MI 48288

Bernd Michel
Institute of Mechanics
P.O. Box 408
D-9010 Chemnitz
GERMANY

D. E. Miles
Commission of the European Comm.
rue de la Loi 200
B-1049 Brussels
BELGIUM

Carl E. Miller
AC Rochester
1300 N. Dort Highway, MS:32-31
Flint MI 48556

Charles W. Miller, Jr.
Centorr Furnaces/Vacuum Industries
542 Amherst Street
Nashua NH 03063

R. Minimmi
Enichem America
2000 Cornwall Road
Monmouth Junction NJ 08852

Michele V. Mitchell
AlliedSignal, Inc.
Ceramic Components
P.O. Box 2960, MS:T21
Torrance CA 90509-2960

Howard Mizuhara
WESGO
477 Harbor Boulevard
Belmont CA 94002

Helen Moeller
Babcock & Wilcox
P.O. Box 11165
Lynchburg VA 24506-1165

Francois R. Mollard
Concurrent Technologies Corp.
1450 Scalp Avenue
Johnstown PA 15904-3374

Phil Mooney
Panametrics
221 Crescent Street
Waltham MA 02254

Geoffrey P. Morris
3M Company
3M Traffic Control Materials
Bldg. 209-BW-10, 3M Center
St. Paul MN 55144-1000

Jay A. Morrison
Rolls-Royce, Inc.
2849 Paces Ferry Road, Suite 450
Atlanta GA 30339-3769

Joel P. Moskowitz
Ceradyne, Inc.
3169 Redhill Avenue
Costa Mesa CA 92626

Brij Moudgil
University of Florida
Material Science & Engineering
Gainesville FL 32611

Christoph J. Mueller
Sprechsaal Publishing Group
P.O. Box 2962, Mauer 2
D-8630 Coburg
GERMANY

Thomas W. Mullan
Vapor Technologies Inc.
345 Route 17 South
Upper Saddle River NJ 07458

Theresa A. Mursick-Meyer
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

M. K. Murthy
MkM Consultants International
10 Avoca Avenue, Unit 1906
Toronto Onntario M4T 2B7
CANADA

David L. Mustoe
Custom Technical Ceramics
8041 West I-70 Service Rd. Unit 6
Arvada CO 80002

Curtis V. Nakaishi
U.S. Department of Energy
Morgantown Energy Technology Ctr.
P.O. Box 880
Morgantown WV 26507-0880

Yoshio Nakamura
Faicera Research Institute
3-11-12 Misono
Sagamihara, Tokyo
JAPAN

Stefan Nann
Roland Berger & Partner GmbH
Georg-Glock-Str. 3
40474 Dusseldorf
GERMANY

K. S. Narasimhan
Hoeganaes Corporation
River Road
Riverton NJ 08077

Robert Naum
Applied Resources, Inc.
P.O. Box 241
Pittsford NY 14534

Malcolm Naylor
Cummins Engine Company, Inc.
P.O. Box 3005, Mail Code 50183
Columbus IN 47202-3005

Fred A. Nichols
Argonne National Laboratory
9700 S. Cass Avenue
Argonne IL 60439

H. Nickel
Forschungszentrum Juelich (KFA)
Postfach 1913
D-52425 Juelich
GERMANY

Dale E. Niesz
Rutgers University
Center for Ceramic Research
P.O. Box 909
Piscataway NJ 08855-0909

Paul W. Niskanen
Lanxide Corporation
P.O. Box 6077
Newark DE 19714-6077

David M. Nissley
United Technologies Corporation
Pratt & Whitney Aircraft
400 Main Street, MS:163-10
East Hartford CT 06108

Bruce E. Novich
Ceramics Process Systems Corp.
155 Fortune Boulevard
Milford MA 01757

Daniel Oblas
50 Meadowbrook Drive
Bedford MA 01730

Don Ohanehi
Magnetic Bearings, Inc.
1908 Sussex Road
Blacksburg VA 24060

Hitoshi Ohmori
ELID Team
Itabashi Branch
1-7 13 Kaga Itabashi
Tokyo 173
JAPAN

Robert Orenstein
General Electric Company
55-112, River Road
Schenectady NY 12345

Norb Osborn
Aerodyne Dallas
151 Regal Row, Suite 120
Dallas TX 75247

Richard Palicka
Cercom, Inc.
1960 Watson Way
Vista CA 92083

Muktesh Paliwal
GTE Products Corporation
Hawes Street
Towanda PA 18848

Joseph N. Panzarino
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

Pellegrino Papa
Corning Inc.
MP-WX-02-1
Corning NY 14831

Terry Paquet
Boride Products Inc.
2879 Aero Park Drive
Traverse City MI 49684

E. Beth Pardue
MPC
8297 Williams Ferry Road
Lenior City TN 37771

Soon C. Park
3M Company
Building 142-4N-02
P.O. Box 2963
St. Paul MN 55144

Vijay M. Parthasarathy
Caterpillar/Solar Turbines
2200 Pacific Highway
P.O. Box 85376
San Diego CA 92186-5376

Harmut Paschke
Schott Glaswerke
Christoph-Dorner-Strasse 29
D-8300 Landshut
GERMANY

James W. Patten
Cummins Engine Company, Inc.
P.O. Box 3005, Mail Code 50183
Columbus IN 47202-3005

Robert A. Penty
Eastman Kodak Company
Kodak Park
Bldg., 326, 3rd Floor
Rochester NY 14652-5120

Robert W. Pepper
Textron Specialty Materials
2 Industrial Avenue
Lowell MA 01851

Peter Perdue
Detroit Diesel Corporation
13400 Outer Drive West,
Speed Code L-04
Detroit MI 48239-4001

John J. Petrovic
Los Alamos National Laboratory
Group MST-4, MS:G771
Los Alamos NM 87545

Frederick S. Pettit
University of Pittsburgh
Pittsburgh PA 15261

Ben A. Phillips
Phillips Engineering Company
721 Pleasant Street
St. Joseph MI 49085

Richard C. Phoenix
Ohmtek, Inc.
2160 Liberty Drive
Niagara Falls NY 14302

Bruce J. Pletka
Michigan Technological University
Metallurgical & Materials Engr.
Houghton MI 49931

John P. Pollinger
AlliedSignal, Inc.
Ceramic Components
P.O. Box 2960, MS:T21
Torrance CA 90509-2960

P. Popper
High Tech Ceramics International
Journal
22 Pembroke Drive - Westlands
Newcastle-under-Lyme
Staffs ST5 2JN
ENGLAND

F. Porz
Universitat Karlsruhe
Institut fur Keramik Im
Maschinendau
Postfach 6980
D-76128 Karlsruhe
GERMANY

Harry L. Potma
Royal Netherlands Embassy
Science and Technology
4200 Linnean Avenue, N.W.
Washington DC 20008

Bob R. Powell
North American Operations
Metallurgy Department
Box 9055
Warren MI 48090-9055

Stephen C. Pred
ICD Group, Inc.
1100 Valley Brook Avenue
Lyndhurst NJ 07071

Karl M. Prewo
United Technologies Research Ctr.
411 Silver Lane, MS:24
East Hartford CT 06108

Vimal K. Pujari
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

George Quinn
NIST
Ceramics Division, Bldg. 223
Gaithersburg MD 20899

Ramas V. Raman
Ceracon, Inc.
1101 N. Market Boulevard, Suite 9
Sacramento CA 95834

Charles F. Rapp
Owens Corning Fiberglass
2790 Columbus Road
Granville OH 43023-1200

Dennis W. Readey
Colorado School of Mines
Metallurgy and Materials Engr.
Golden CO 80401

Wilfred J. Rebello
PAR Enterprises, Inc.
12601 Clifton Hunt Lane
Clifton VA 22024

Harold Rechter
Chicago Fire Brick Company
7531 S. Ashland Avenue
Chicago IL 60620

Robert R. Reeber
U.S. Army Research Office
P.O. Box 12211
Research Triangle Park NC
27709-2211

K. L. Reifsnider
VPI & SU
Engineering Science and Mechanics
Blacksburg VA 24061

Paul E. Rempes
McDonnell Douglass Aircraft Co.
P.O. Box 516, Mail Code:0642263
St. Louis MO 63166-0516

Gopal S. Revankar
John Deere Company
3300 River Drive
Moline IL 61265

K. Y. Rhee
Rutgers University
P.O. Box 909
Piscataway NJ 08854

James Rhodes
Advanced Composite Materials Corp
1525 S. Buncombe Road
Greer SC 29651

Roy W. Rice
W. R. Grace and Company
7379 Route 32
Columbia MD 21044

David W. Richerson
2093 E. Delmont Drive
Salt Lake City UT 84117

Tomas Richter
J. H. France Refractories
1944 Clarence Road
Snow Shoe PA 16874

Michel Rigaud
Ecole Polytechnique
Campus Universite De Montreal
P.O. Box 6079, Station A
Montreal, P.Q. Quebec H3C 3A7
CANADA

John E. Ritter
University of Massachusetts
Mechanical Engineering Department
Amherst MA 01003

Frank L. Roberge
AlliedSignal Engines
P.O. Box 52180
Phoenix AZ 85072-2180

W. Eric Roberts
Advanced Ceramic Technology, Inc.
990 "F" Enterprise Street
Orange CA 92667

Y. G. Roman
TNO TPD Keramick
P.O. Box 595
Einhoven 5600 AN
HOLLAND

Michael Rossetti
Arthur D. Little, Inc.
15 Acorn Park
Cambridge MA 01240

Barry Rossing
Lanxide Corporation
P.O. Box 6077
Newark DE 19714-6077

Steven L. Rotz
Lubrizol Corporation
29400 Lakeland Boulevard
Wickliffe OH 44092

Robert Ruh
Wright Laboratory
WL/MLLM
Wright-Patterson AFB OH 45433

Robert J. Russell
17 Highgate Road
Framingham MA 01701

Jon A. Salem
NASA Lewis Research Center
21000 Brookpark Road
Cleveland OH 44135

W. A. Sanders
NASA Lewis Research Center
21000 Brookpark Road, MS:49-3
Cleveland OH 44135

J. Sankar
North Carolina A&T State Univ.
Dept. of Mechanical Engineering
Greensboro NC 27406

Yasushi Sato
NGK Spark Plugs (U.S.A.), Inc.
1200 Business Center Drive, #300
Mt. Prospect IL 60056

Maxine L. Savitz
AlliedSignal, Inc.
Ceramic Components
P.O. Box 2960, MS:T21
Torrance CA 90509-2960

Ashok Saxena
GTRI
Materials Engineering
Atlanta GA 30332-0245

David W. Scanlon
Instron Corporation
100 Royall Street
Canton MA 02021

Charles A. Schacht
Schacht Consulting Services
12 Holland Road
Pittsburgh PA 15235

Robert E. Schafrik
National Materials Advisory Board
2101 Constitution Ave., N.W.
Washington DC 20418

James Schienle
AlliedSignal Engines
P.O. Box 52180, MS:1302-2P
Phoenix AZ 85072-2180

John C. Schneider
San Juan Technologies, Inc.
3210 Arena Road
Colorado Springs CO 80921-1503

Gary Schnittgrund
Rocketdyne, BA05
6633 Canoga Avenue
Canoga Park CA 91303

Mark Schomp
Lonza, Inc.
17-17 Route 208
Fair Lann NJ 07410

Joop Schoonman
Delft University of Technology
P.O. Box 5045
2600 GA Delft
THE NETHERLANDS

Robert B. Schulz
U.S. Department of Energy
Office of Transportation Matrls.
CE-34, Forrestal Building
Washington DC 20585

Murray A. Schwartz
Materials Technology Consulting
30 Orchard Way, North
Potomac MD 20854

Peter Schwarzkopf
SRI International
333 Ravenswood Avenue
Menlo Park CA 94025

William T. Schwessinger
Multi-Arc Scientific Coatings
1064 Chicago Road
Troy MI 48083-4297

W. D. Scott
University of Washington
Materials Science Department
Mail Stop:FB10
Seattle WA 98195

Nancy Scoville
Thermo Electron Technologies
P.O. Box 9046
Waltham MA 02254-9046

Thomas M. Sebestyen
U.S. Department of Energy
Advanced Propulsion Division
CE-322, Forrestal Building
Washington DC 20585

Brian Seegmiller
Coors Ceramics Company
600 9th Street
Golden CO 80401

T. B. Selover
AICRE/DIPPR
3575 Traver Road
Shaker Heights OH 44122

Charles E. Semler
Semler Materials Services
4160 Mumford Court
Columbus OH 43220

Thomas Service
Service Engineering Laboratory
324 Wells Street
Greenfield MA 01301

Kish Seth
Ethyl Corporation
P.O. Box 341
Baton Rouge LA 70821

William J. Shack
Argonne National Laboratory
9700 S. Cass Avenue, Bldg. 212
Argonne IL 60439

Peter T.B. Shaffer
Technical Ceramics Laboratories,
4045 Nine/McFarland Drive
Alpharetta GA 30201

Richard K. Shaltens
NASA Lewis Research Center
21000 Brookpark Road, MS:302-2
Cleveland OH 44135

Robert S. Shane
1904 NW 22nd Street
Stuart FL 34994-9270

Ravi Shankar
Chromalloy
Research and Technology Division
Blaisdell Road
Orangeburg NY 10962

Terence Sheehan
Alpex Wheel Company
727 Berkley Street
New Milford NJ 07646

Dinesh K. Shetty
University of Utah
Materials Science and Engineering
Salt Lake City UT 84112

Masahide Shimizu
New Ceramics Association
Shirasagi 2-13-1-208, Nakano-ku
Tokyo 165
JAPAN

Thomas Shreves
American Ceramic Society, Inc.
735 Ceramic Place
Westerville OH 43081-8720

Jack D. Sibold
Coors Ceramics Company
4545 McIntyre Street
Golden CO 80403

Johann Siebels
Volkswagen AG
Werkstofftechnologie
Postfach 3180
Wolfsburg 1
GERMANY

George H. Siegel
Point North Associates, Inc.
P.O. Box 907
Madison NJ 07940

Richard Silberglitt
FM Technologies, Inc.
10529-B Braddock Road
Fairfax VA 22032

Mary Silverberg
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

Gurpreet Singh
Department of the Navy
Code 56X31
Washington DC 20362-5101

Maurice J. Sinnott
University of Michigan
5106 IST Building
Ann Arbor MI 48109-2099

John Skildum
3M Company
3M Center
Building 224-2S-25
St. Paul MN 55144

Richard H. Smoak
Smoak & Associates
3554 Hollyslope Road
Altadena CA 91001-3923

Jay R. Smyth
AlliedSignal Engines
111 S. 34th Street, MS:503-412
Phoenix AZ 85034

Rafal A. Sobotowski
British Petroleum Company
Technical Center, Broadway
3092 Broadway Avenue
Cleveland OH 44115

S. Somiya
Nishi Tokyo University
3-7-19 Seijo, Setagaya
Tokyo 157
JAPAN

Boyd W. Sorenson
DuPont Lanxide Composites
1300 Marrows Road
Newark DE 19711

Charles A. Sorrell
U.S. Department of Energy
Advanced Industrial Concepts
CE-232, Forrestal Building
Washington DC 20585

C. Spencer
EA Technology
Capenhurst Chester CH1 6ES
UNITED KINGDOM

Allen Spizzo
Hercules Inc.
Hercules Plaza
Wilmington DE 19894

Richard M. Spriggs
Alfred University
Center for Advanced Ceramic
Technology
Alfred NY 14802

Charles Spuckler
NASA Lewis Research Center
21000 Brookpark Road, MS:5-11
Cleveland OH 44135-3191

M. Srinivasan
Material Solutions
P.O. Box 663
Grand Island NY 14702-0663

Gordon L. Starr
Cummins Engine Company, Inc.
P.O. Box 3005, Mail Code:50182
Columbus IN 47202-3005

Tom Stillwagon
AlliedSignal, Inc.
Ceramic Components
P.O. Box 2960, MS:T21
Torrance CA 90509-2960

H. M. Stoller
TPL Inc.
3754 Hawkins, N.E.
Albuquerque NM 87109

Paul D. Stone
Dow Chemical USA
1776 "Eye" Street, N.W., #575
Washington DC 20006

F. W. Stringer
Aero & Industrial Technology Ltd.
P.O. Box 46, Wood Top
Burnley Lancashire BB11 4BX
UNITED KINGDOM

Thomas N. Strom
NASA Lewis Research Center
21000 Brookpark Road, MS:86-6
Cleveland OH 44135

M. F. Stroosnijder
Institute for Advanced Materials
Joint Research Centre
21020 Ispra (VA)
ITALY

Karsten Styhr
30604 Ganado Drive
Rancho Palos Verdes CA 90274

T. S. Sudarshan
Materials Modification, Inc.
2929-P1 Eskridge Center
Fairfax VA 22031

M. J. Sundaresan
University of Miami
P.O. Box 248294
Coral Gables FL 33124

Patrick L. Sutton
U.S. Department of Energy
Office of Propulsion Systems
CE-322, Forrestal Building
Washington DC 20585

Willard H. Sutton
United Technologies Corporation
Silver Lane, MS:24
East Hartford CT 06108

J. J. Swab
U.S. Army Materials Technology
Ceramics Research Division,
SLCMT-EMC
405 Arsenal Street
Watertown MA 02172

Robert E. Swanson
Metalworking Technology, Inc.
1450 Scalp Avenue
Johnstown PA 15904

Steve Szaruga
Air Force Wright Aeronautical Lab
WL/MLBC
Wright-Patterson AFB OH
45433-6533

Yo Tajima
NGK Spark Plug Company
2808 Iwasaki
Komaki-shi Aichi-ken 485
JAPAN

Fred Teeter
5 Tralee Terrace
East Amherst NY 14051

Monika O. Ten Eyck
Carborundum Microelectronics
P.O. Box 2467
Niagara Falls NY 14302-2467

David F. Thompson
Corning Glass Works
SP-DV-02-1
Corning NY 14831

Merle L. Thorpe
Hobart Tafa Technologies, Inc.
20 Ridge Road
Concord NH 03301-3010

T. Y. Tien
University of Michigan
Materials Science and Engineering
Dow Building
Ann Arbor MI 48103

D. M. Tracey
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

L. J. Trostel, Jr.
Box 199
Princeton MA 01541

W. T. Tucker
General Electric Company
P.O. Box 8, Bldg. K1-4C35
Schenectady NY 12301

Masanori Ueki
Nippon Steel Corporation
1618 Ida
Nakahara-Ku Kawasaki 211
JAPAN

Filippo M. Ugolini
ATA Studio
Via Degli Scipioni, 268A
ROMA, 00192
ITALY

Donald L. Vaccari
General Motors Corporation
Allison Gas Turbines
P.O. Box 420, Speed Code S49
Indianapolis IN 46206-0420

Carl F. Van Conant
Boride Products, Inc.
2879 Aero Park Drive
Traverse City MI 49684

Marcel H. Van De Voorde
Commission of the European Comm.
P.O. Box 2
1755 ZG Petten
THE NETHERLANDS

O. Van Der Biest
Katholieke Universiteit Leuven
Dept. Metaalkunde en Toegepaste
de Croylaan 2
B-3030 Leuven
BELGIUM

Michael Vannier
Washington University, St. Louis
510 S. Kings Highway
St. Louis MO 63110

Stan Venkatesan
Southern Coke & Coal Corporation
P.O. Box 52383
Knoxville TN 37950

V. Venkateswaran
Carborundum Company
Niagara Falls R&D Center
P.O. Box 832
Niagara Falls NY 14302

Dennis Viechnicki
U.S. Army Materials Technology
405 Arsenal Street
Watertown MA 02172-0001

A. V. Vircar
University of Utah
Materials Science and Engineering
Salt Lake City, UT 84112

Ted Vojnovich
U.S. Department of Energy, ST-311
Office of Energy Research, 3F077P
Washington DC 20585

John D. Volt
E.I. Dupont de Nemours & Co. Inc.
P.O. Box 80262
Wilmington DE 19880

John B. Wachtman
Rutgers University
P.O. Box 909
Piscataway NJ 08855

Shigetaka Wada
Toyota Central Research Labs
Nagakute Aichi 480-11
JAPAN

Janet Wade
AlliedSignal Engines
P.O. Box 52180, MS:1303-2
Phoenix AZ 85072-2180

Richard L. Wagner
Ceramic Technologies, Inc.
537 Turtle Creek South Dr., #24D
Indianapolis IN 46227

J. Bruce Wagner, Jr.
Arizona State University
Center for Solid State Science
Tempe AZ 85287-1704

Daniel J. Wahlen
Kohler, Co.
444 Highland Drive
Kohler WI 53044

Ingrid Wahlgren
Royal Institute of Technology
Studsvik Library
S-611 82 Nykoping
SWEDEN

Ron H. Walecki
AlliedSignal, Inc.
Ceramic Components
P.O. Box 2960, MS:T21
Torrance CA 90509-2960

Michael S. Walsh
Vapor Technologies Inc.
6300 Gunpark Drive
Boulder CO 80301

Chien-Min Wang
Industrial Technology Research Institute
195 Chung-Hsing Road, Sec. 4
Chutung Hsinchu 31015 R.O.C.
TAIWAN

Robert M. Washburn
ASMT
11203 Colima Road
Whittier CA 90604

Gerald Q. Weaver
Carborundum Specialty Products
42 Linus Allain Avenue
Gardner MA 01440-2478

Kevin Webber
Toyota Technical Center, U.S.A.
1410 Woodridge, RR7
Ann Arbor MI 48105

Karen E. Weber
Detroit Diesel Corporation
13400 Outer Drive West
Detroit MI 48239-4001

James K. Weddell
Du Pont Lanxide Composites Inc.
P.O. Box 6100
Newark DE 19714-6100

R. W. Weeks
Argonne National Laboratory
MCT-212
9700 S. Cass Avenue
Argonne IL 60439

Ludwig Weiler
ASEA Brown Boveri AG
Eppelheimer Str. 82
D-6900 Heidelberg
GERMANY

James Wessel
Dow Corning Corporation
1800 "M" Street, N.W., #325 South
Washington DC 20036

Robert D. West
Therm Advanced Ceramics
P.O. Box 220
Ithaca NY 14851

Thomas J. Whalen
Ford Motor Company
SRL Bldg., Mail Drop 2313
P.O. Box 2053
Dearborn MI 48121-2053

Ian A. White
Hoeganaes Corporation
River Road
Riverton NJ 08077

Sheldon M. Wiederhorn
NIST
Building 223, Room A329
Gaithersburg MD 20899

John F. Wight
Alfred University
McMahon Building
Alfred NY 14802

D. S. Wilkinson
McMaster University
1280 Main Street, West
Hamilton Ontario L8S 4L7
CANADA

James C. Williams
General Electric Company
Engineering Materials Technology
One Neumann Way, Mail Drop:H85
Cincinnati OH 45215-6301

Steve J. Williams
RCG Hagler Bailly, Inc.
1530 Wilson Boulevard, Suite 900
Arlington VA 22209-2406

Thomas A. Williams
National Renewable Energy Lab
1617 Cole Boulevard
Golden CO 80401

Craig A. Willkens
Norton Company
SGNICC/NRDC
Goddard Road
Northboro MA 01532-1545

Roger R. Wills
TRW, Inc.
Valve Division
1455 East 185th Street
Cleveland OH 44110

David Gordon Wilson
Massachusetts Institute of
Technology
77 Massachusetts Ave., Room 3-455
Cambridge MA 02139

Matthew F. Winkler
Seaworthy Systems, Inc.
P.O. Box 965
Essex CT 06426

Gerhard Winter
Hermann C. Starck Berlin GmbH
P.O. Box 25 40
D-3380 Goslar 3380
GERMANY

William T. Wintucky
NASA Lewis Research Center
Terrestrial Propulsion Office
21000 Brookpark Road, MS:86-6
Cleveland OH 44135

Thomas J. Wissing
Eaton Corporation
Engineering and Research Center
P.O. Box 766
Southfield MI 48037

James C. Withers
MER Corporation
7960 S. Kolb Road
Building F
Tucson AZ 85706

Dale E. Wittmer
Southern Illinois University
Mechanical Engineering Department
Carbondale IL 62901

Warren W. Wolf
Owens Corning Fiberglass
2790 Columbus Road, Route 16
Granville OH 43023

Egon E. Wolff
Caterpillar Inc.
Technical Center
P.O. Box 1875
Peoria IL 61656-1875

George W. Wolter
Howmet Turbine Components Corp.
Technical Center
699 Benston Road
Whitehall MI 49461

James C. Wood
NASA Lewis Research Center
21000 Brookpark Road, MS:86-6
Cleveland OH 44135

Marrill Wood
LECO Corporation
P.O. Box 211688
Augusta GA 30917-1688

Wayne L. Worrell
University of Pennsylvania
3231 Walnut Street
Philadelphia PA 19104

John F. Wosinski
Corning Inc.
ME-2 E-5 H8
Corning NY 14830

Ian G. Wright
BCL
505 King Avenue
Columbus OH 43201

Ruth Wroe
ERDC
Capenhurst Chester CH1 6ES
ENGLAND

Bernard J. Wrona
Advanced Composite Materials Corp
1525 S. Buncombe Road
Greer SC 29651

Carl C. M. Wu
Naval Research Laboratory
Ceramic Branch, Code 6373
Washington DC 20375

John C. Wurst
U. of Dayton Research Institute
300 College Park
Dayton OH 45469-0101

Neil Wyant
ARCH Development Corp.
9700 S. Cass Avenue, Bldg. 202
Argonne IL 60439

Roy Yamamoto
Texaco Inc.
P.O. Box 509
Beacon NY 12508-0509

John Yamanis
AlliedSignal Aerospace Company
P.O. Box 1021
Morristown NJ 07962-1021

Harry C. Yeh
AlliedSignal, Inc.
Ceramic Components
P.O. Box 2960, MS:T21
Torrance CA 90509-2960

Hiroshi Yokoyama
Hitachi Research Lab
4026 Kuji-Cho
Hitachi-shi Ibaraki 319-12
JAPAN

Thomas M. Yonushonis
Cummins Engine Company, Inc.
P.O. Box 3005, Mail Code 50183
Columbus IN 47202-3005

Thomas J. Yost
Corning Inc.
Technical Products Div., 21-1-2
Corning NY 14831

Jong Yung
Sundstrand Aviation Operations
4747 Harrison Avenue
Rockford IL 61125

A. L. Zadoks
Caterpillar Inc.
Technical Center, Building L
P.O. Box 1875
Peoria IL 61656-1875

Avi Zangvil
University of Illinois
104 S. Goodwin Avenue
Urbana IL 61801

Charles H. Zenuk
Transtech
6662 E. Paseo San Andres
Tucson AZ 85710-2106

Carl Zweben
General Electric Company
P.O. Box 8555, VFSC/V4019
Philadelphia PA 19101

Department of Energy
Oak Ridge Operations Office
Assistant Manager for Energy
Research and Development
P.O. Box 2001
Oak Ridge, TN 37831-8600

Department of Energy (2)
Office of Scientific and
Technical
Information
Office of Information Services
P.O. Box 62
Oak Ridge, TN 37831

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