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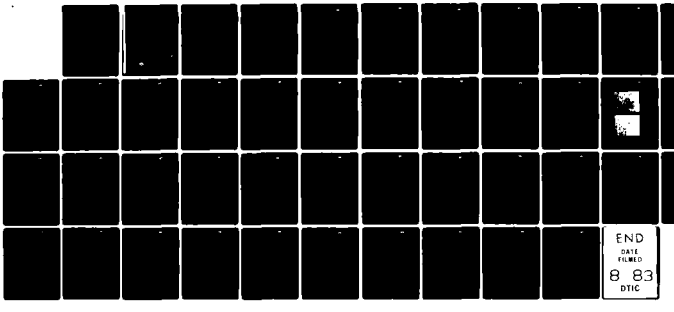

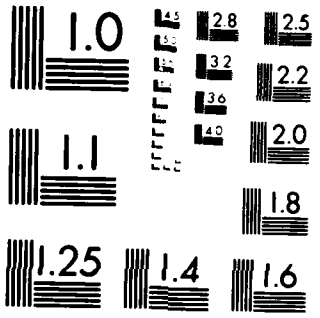

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RESEARCH ON MICROSTRUCTURALLY DEVELOPED TOUGHENING MECHANISMS IN CERAMICS

FINAL REPORT FOR THE PERIOD
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D.J. Green
Principal Investigator

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

An approach is presented for predicting the strengthening of brittle materials by compressive surface stresses. The approach incorporates the effect of crack closure, which can significantly influence the stress intensity factor. The analysis is used for the example of tempering and it is found that the results give a better understanding of crack growth but also indicate approaches for optimization of the strengthening.

Compressive surface stresses can be introduced into transformation-toughened

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ZrO_2 ceramics by grinding or impact. These techniques usually have associated damage effects. A new approach is introduced whereby surface compression is introduced by a heat treatment, which involves the removal of the oxide additives, that were originally introduced to retain the ZrO_2 in its tetragonal phase, from the surface region. The presence of the compressive surface stresses is confirmed and the ensuing improved resistance to indentation cracking is described.

Finally, the work performed in the last two years on compressive surface stresses in Al_2O_3/ZrO_2 composites is reviewed.

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RESEARCH OF MICROSTRUCTURALLY DEVELOPED TOUGHENING MECHANISMS IN CERAMICS

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Part 1: Compressive Surface Strengthening of Brittle Materials by a General Residual Stress Distribution

Part 2: A Technique for Introducing Surface Compression Into Zirconia Ceramics

Part 3: Residual Surface Stresses in Al_2O_3-ZrO_2 Composites

Prepared for:

Office of Naval Research
Arlington, VA 22215

Prepared by:

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INTRODUCTION

The introduction of surface compressive stresses into transformation-toughened ZrO_2 ceramics has the potential to strengthen and to improve the wear resistance of these materials. For this final report, three pre-prints of papers are included that represent the progress in the past year. In the first of these, the prediction of strengthening due to a general residual surface stress distribution is analyzed. In the second, a new technique for introducing surface compression, that appears to have great potential is presented. In the final part, the work on compressive surface stresses is reviewed, which includes some previously unpublished work.

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COMPRESSIVE SURFACE STRENGTHENING OF BRITTLE MATERIALS BY A GENERAL RESIDUAL STRESS DISTRIBUTION

No. 17

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ABSTRACT

A theoretical approach is presented for predicting the strengthening of brittle materials subjected to a general residual stress distribution represented by a polynomial series. In the approach the stress intensity factor for a surface crack is derived incorporating the effect of crack closure. The crack closure distance is then calculated using an approximate approach which allows the strengthening due to the residual stresses to be estimated. Illustrating the approach using residual stresses typical of tempering, it was found the approach agreed well with previous work. The influence of partial crack closure was found to give higher values of the stress intensity factor than would be calculated if the crack is assumed to be open. This effect decreases the amount of strengthening predicted and gives a wide range of conditions for which subcritical crack growth processes can occur. For the example of tempering it was also found that these are conditions when weakening or spontaneous failure of the body can occur.

1.0 INTRODUCTION

The failure of ceramics or glasses often occurs from surface cracks and thus techniques that place the surfaces in compression have the potential for strengthening these materials. For example, in the glass industry tempering and ion exchange have been accepted as viable techniques. In using such approaches it is clearly useful to be able to predict the amount of strengthening that is obtained for a particular residual stress distribution and the stress state at the crack tip. Fracture mechanics can be used to make such predictions and for many situations this is relatively straightforward as many crack loading geometries have been solved. In dealing with compressive residual stresses, however, it is possible to have crack closure and this leads to some complications in the fracture mechanics analysis. In a previous paper,[1] it was shown for a simple residual stress distribution that partial crack closure influences the stress intensity factor of a surface crack and hence the amount of strengthening predicted. Partial crack closure in simple configurations has been analyzed by several authors,[2-6] while for more complex situations numerical approaches have been used.[7-11] In this paper the stress intensity factor for a surface crack is derived for a more general type of residual stress distribution and by using an approximate approach for calculating the closure distance, the amount of strengthening for bodies containing a compressive residual stress distribution is estimated.

2.0 THEORETICAL APPROACH

A. Calculation of Stress Intensity Factor

Consider a surface crack in a semi-infinite body, which is partially closed due to the application of an arbitrary nonuniform stress $P(x)$. This is illustrated in Fig. 1, in which a_o represents the length of the actual crack and 2a is the length of the open portion of the crack. The loading $P(x)$ is the stress present at the location of the crack prior to its introduction. Provided the crack is small compared to the dimensions of the body, the prior residual stresses should not be relaxed by the presence of the crack.

As pointed out previously,[1] the stress intensity factor (K_I) for a partially-closed edge crack is given by

$$K_I = 2\left(\frac{a_o}{\pi}\right)^{1/2}(1 - c_1^2)^{1/2}\int_{c_1}^{1}\frac{P(x_1)f(x_1)x_1 dx_1}{[(1 - x_1^2)(x_1^2 - c_1^2)]^{1/2}}, \qquad (1)$$

where $x_1 = x/a_o$, $c_1 = c/a_o$, c is the closure distance $(a_o - 2a)$ and $f(x_1)$ is given by

$$f(x_1) = 1.294 - 0.6857x_1^2 + 1.1597x_1^4 - 1.7627x_1^6$$

$$+ 1.5036x_1^8 - 0.5094x_1^{10} = \sum_{m=0}^{5} A_{2m}x_1^{2m} \qquad (2)$$

Let us consider that the residual stress distribution can be represented by a polynomial of the type

$$P(x_1) = \sum_{n=0}^{I} \alpha_n x_1^n \quad . \qquad (3)$$

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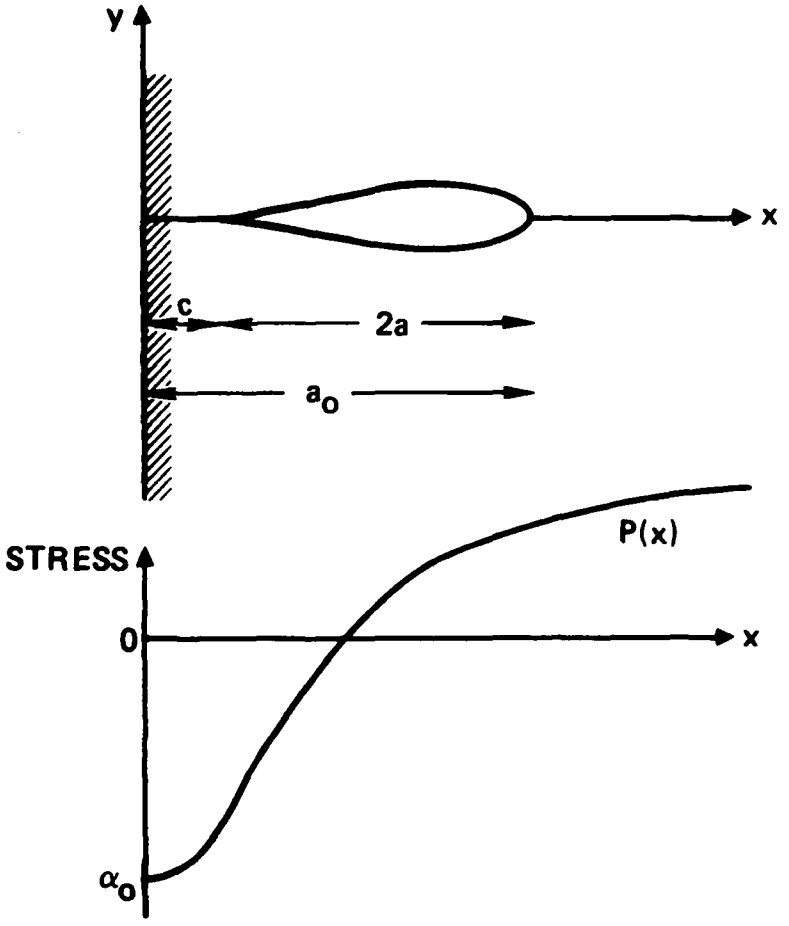

Fig. 1 Partially closed surface crack subjected to a residual stress distribution P(x).

Eq. (1) can therefore be written as

$$K_I = 2\left(\frac{a_o}{\pi}\right)^{1/2}(1 - c_1^2)^{1/2} \int_{c_1}^{1} \frac{\sum_{n=0}^{I} \alpha_n x_1^{n+1}(f(x_1))\, dx_1}{[(1 - x_1^2)(x_1^2 - c_1^2)]^{1/2}} \qquad (4)$$

It has been shown that[12]

$$G_{n+m} = \int_{c_1}^{1} \frac{x_1^{n+m}\, dx_1}{[(1 - x_1^2)(x_1^2 - c_1^2)]^{1/2}} = [-[n+m-3)c^2 G_{n+m-4} + (n+m-2)(1+c^2)G_{n+n-2}] /(n+m-1) \qquad (5)$$

where $G_o = K(\sqrt{(1-c_1^2)})$, $G_1 = \pi/2$, $G_2 = E(\sqrt{(1-c_1^2)})$ and $G_3 = \pi(1+c_1^2)/4$, and where $K(\sqrt{(1-c_1^2)})$ and $E(\sqrt{(1-c_1^2)})$ are the complete elliptical integrals of the first and second kind respectively. Performing the integrals for various values of c_1, one can write the solution in the form

$$K_I = \sqrt{\pi a_o}\,(1-c_1^2)^{1/2}\left(\sum_{n=0}^{I} \alpha_n H_n(c_1)\right) \qquad (6)$$

where $H_n(c_1)$ have been tabulated in Table 1. From this table it can be seen that the solution for a completely open crack is given by

$$\begin{aligned} K_I = \sqrt{(\pi a_o)}(&1.1215\ \alpha_o + 0.6829\alpha_1 + 0.5256\ \alpha_2 + 0.4410\alpha_3 \\ &+ 0.3870\alpha_4 + 0.3485\alpha_5 + 0.3197\alpha_6 \\ &+ 0.2969\alpha_7 + 0.2784\alpha_8 + 0.2628\alpha_9 + 0.2498\alpha_{10} \\ &+ .....) \quad , \end{aligned} \qquad (7)$$

Table 1
Coefficient $H_n(C)$ as a Function of Closure Distance

G	H_0	H_1	H_2	H_3	H_4	H_5	H_6	H_7	H_8	H_9	H_{10}
0	1.1215	0.6829	0.5256	0.4410	0.3870	0.3485	0.3197	0.2969	0.2784	0.2628	0.2498
0.1	1.1193	0.6950	0.5312	0.4446	0.3896	0.3509	0.3217	0.2986	0.2800	0.2644	0.2512
0.2	1.1131	0.7195	0.5478	0.4558	0.3981	0.3579	0.3278	0.3042	0.2850	0.2691	0.2555
0.3	1.1037	0.7504	0.5749	0.4762	0.4139	0.3709	0.3390	0.3141	0.2941	0.2775	0.2634
0.4	1.0918	0.7847	0.6119	0.5073	0.4393	0.3920	0.3571	0.3301	0.3085	0.2907	0.2757
0.5	1.0783	0.8207	0.6579	0.5508	0.4773	0.4247	0.3855	0.3553	0.3311	0.3113	0.2948
0.6	1.0636	0.8574	0.7124	0.6081	0.5316	0.4741	0.4299	0.3951	0.3671	0.3442	0.3250
0.7	1.0480	0.8940	0.7744	0.6806	0.6062	0.5468	0.4986	0.4592	0.4266	0.3993	0.3762
0.8	1.0322	0.9301	0.8434	0.7694	0.7059	0.6514	0.6042	0.5634	0.5278	0.4967	0.4694
0.9	1.0165	0.9659	0.9192	0.8759	0.8357	0.7985	0.7640	0.7319	0.7021	0.6744	0.6486
1.0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000

which is in agreement with the work of Stallybrass.[13] Moreover, it is seen from Eq. (6) that $K_I \to 0$ as $c_1 \to 0$, i.e., for complete crack closure. It is clear therefore, that the partially-closed crack solution is a smooth continuation of the open crack solution and as the crack approaches complete closure, $K_I \to 0$.

For cases where the surface crack is completely open at failure, Eq. (1) can be simplified, as $c_1=0$ and the integrals for a wide variety of residual stress distributions are relatively simple to perform.

B. Determination of Closure Distance

In order to derive the stress intensity factor, for a given loading it is necessary to be able to determine the closure distance (c_1). Numerical approaches have been introduced to determine c_1 for surface cracks.[8-11] For illustrative purposes, however, an approximate analytical approach will be used here, based on the work of *Thresher and Smith*,[5] who consider the partial closure of an internal crack. In their study they showed that for a loading of the type given by Eq. (3), the open crack length (2a) can be determined by solving the following equation,

$$\sum_{m=0}^{G} \gamma_{2m} \frac{\Gamma(m+\frac{1}{2})}{\Gamma(m+1)} \left(\frac{a}{a_o}\right)^{2m} + \sum_{m=1}^{H} \gamma_{2m-1} \frac{\Gamma(m+\frac{1}{2})}{\Gamma(m+1)} \left(\frac{a}{a_o}\right)^{2m-1} = 0 \quad , \tag{8}$$

where

$$\gamma_m = \sum_{n=m}^{I} \alpha_n \binom{n}{m} (1-(a/a_o))^{n-m} (-1)^m \tag{9}$$

where $\binom{n}{m}$ are the binomial coefficients. This approach ignores the influence of the free surface for the case of an edge crack, but its simplicity should identify the important variables in controlling crack closure.

C. Strengthening Due to Residual Surface Stresses

Approaches have been outlined in the previous sections to determine K_I and closure distance for a surface crack under an arbitrary loading. For cases where the crack is under residual compression, one expects to obtain strengthening of the body. Let us consider, therefore, a surface crack under the combined influence of a residual stress distribution given by Eq. (3) and an applied tensile stress σ_A. The stress intensity factor (from Eq. (6)) is given by

$$K_I = \sqrt{(\pi a_o(1-c_1^2))} \quad \left(\sum_{n=0}^{I} \alpha_n H_n(c_1) + \sigma_A H_o(c_1) \right) \quad . \tag{10}$$

The failure condition is given by $K_I = K_C$ and $\sigma_A = \sigma_F$. In the absence of surface stresses, $K_C = 1.1215\ \sigma_F^\circ \sqrt{(\pi a_o)}$, where σ_F° is the base strength of the material. Using this failure criterion, one obtains

$$\left(\frac{\sigma_F}{\sigma_F^o}\right)^{-1} = \frac{\alpha_o(1-c_1^2)^{1/2}}{1.1215\ \sigma_F} \left(\sum_{n=0}^{I} \left(\frac{\alpha_n}{\alpha_o}\right) H_n(c_1) + \frac{\sigma_F}{\alpha_o} H_o(c_1) \right) \quad , \tag{11}$$

and thus one can estimate the strengthening due to surface compression. For the special case of a completely open crack

$$\left(\frac{\sigma_F - \sigma_F^0}{\sigma_F^0}\right) = -\ (\alpha_o + 0.6089\alpha_1 + 0.4686\alpha_2 + 0.3932\alpha_3 + 0.3449\alpha_4$$
$$+ 0.3107\alpha_5 + 0.2850\alpha_6 + 0.2647\alpha_7 + 0.2482\alpha_8$$
$$+ 0.2343\alpha_9 + 0.2226\alpha_{10} + \ldots\ldots\ldots\ldots) \tag{12}$$

D. Example of Strengthening due to Tempering

As an example of the theoretical approach, let us consider a body containing the residual stress distribution

$$P(x_1) = \sigma_0(1-3(2a_0x_1/h-1)^2) \qquad (12)$$

where h is the width of the body. This distribution has been suggested[9] to be representative of stresses introduced by tempering. In applying the analysis to a finite body it is expected to run into errors for large values of (a_0/h). The first step in the analysis is to determine c_1 in terms of σ_A/σ_0 using Eq. (8). The situation when $\sigma_A/\sigma_0 = 0$ is shown in Fig. 2. It is found that the crack is completely closed for $(a_0/h) < 0.211$, which is the point where $P(x_1) = 0$, i.e., when the crack is within the compression zone. For larger values of (a_0/h), the crack is partially closed. The approximate approach for calculating closure distance is in reasonable agreement with the numerical analysis of Bakioglu et al[9] for $(a_0/h) < 0.4$. The stress intensity factor solution for this situation is calculated from Eq. (10) and Table 1. The results are shown in Fig. 3 and are compared with the numerical data of Bakioglu et al[9] and the solution that assumes the crack to be completely open. It is found the agreement with the numerical results is excellent. The values of K_I for the partially-closed crack are somewhat higher than the open crack solution. Indeed for values of a_0/h between 0.21 and 0.39, the open crack solution gives negative values of K_I. It follows therefore that not only will the strengthening be less than that for an 'open' crack but there is also a range of conditions for which $K_I > 0$ that is not predicted for an 'open' crack. This latter effect is important as subcritical crack growth processes can occur once $K_I > 0$.

The strengthening due to tempering calculated from Eq. (1) is shown in Fig. 4. It is found that when the surface crack is initially within the compression zone ($a_0/h < 0.21$). The strengthening increases as (a_0/h) or $(\sigma_F^\circ/\sigma_0)$ decrease. For situations where $0.21 < \alpha_0/h < 0.4$ some strengthening is possible but if the base strength of the body (σ_F°) is low or the surface compressive stress is high, weakening or spontaneous failure can occur. Finally, for $(a_0/h) > 0.4$, no strengthening is possible and if σ_F°/σ_0 is small then spontaneous failure can occur. The arrows in Fig. 4 indicate the minimum

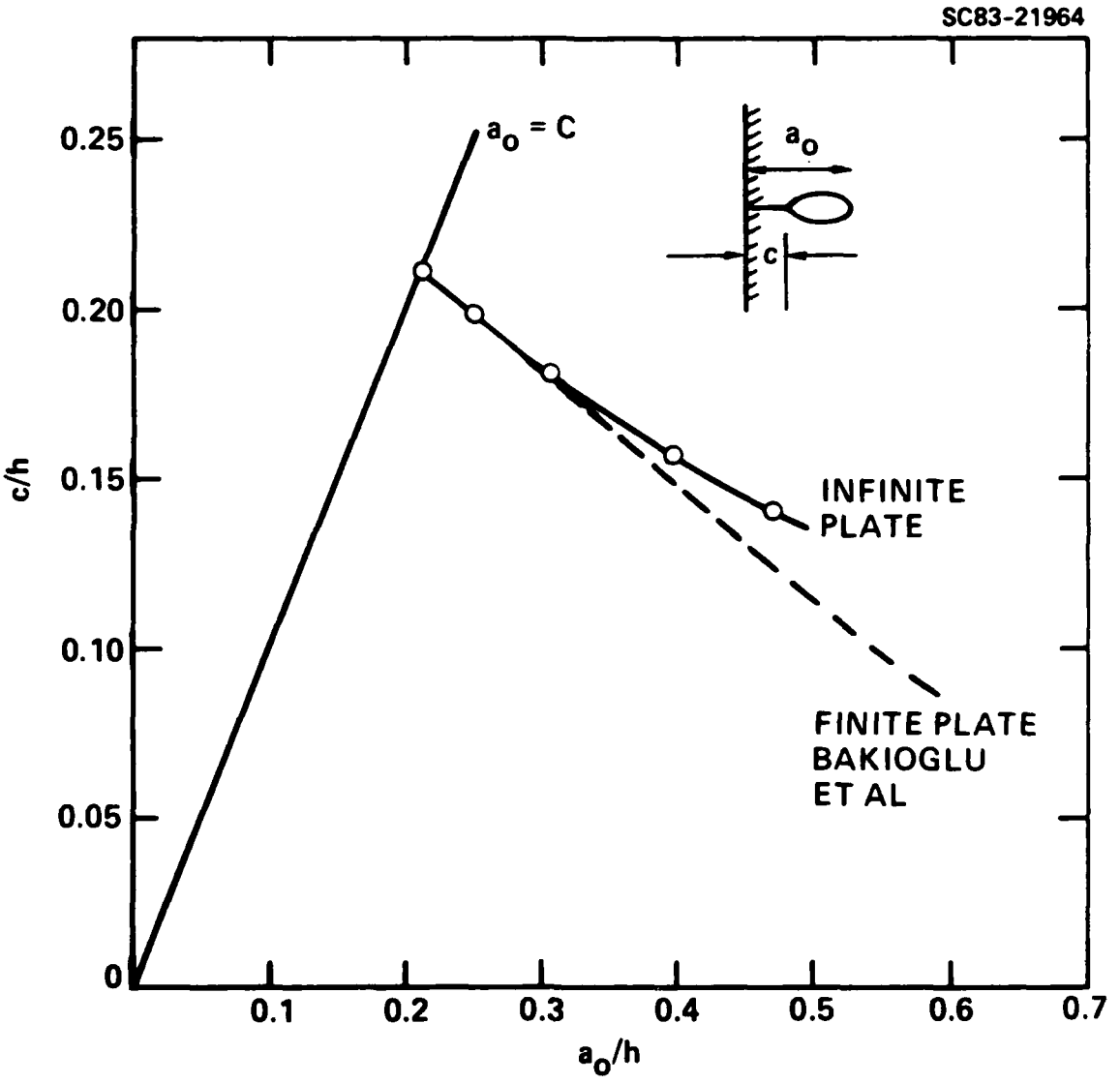

Fig. 2 Comparison of calculated closure distance for tempering with previous work (Ref. 9) in absence of applied stress.

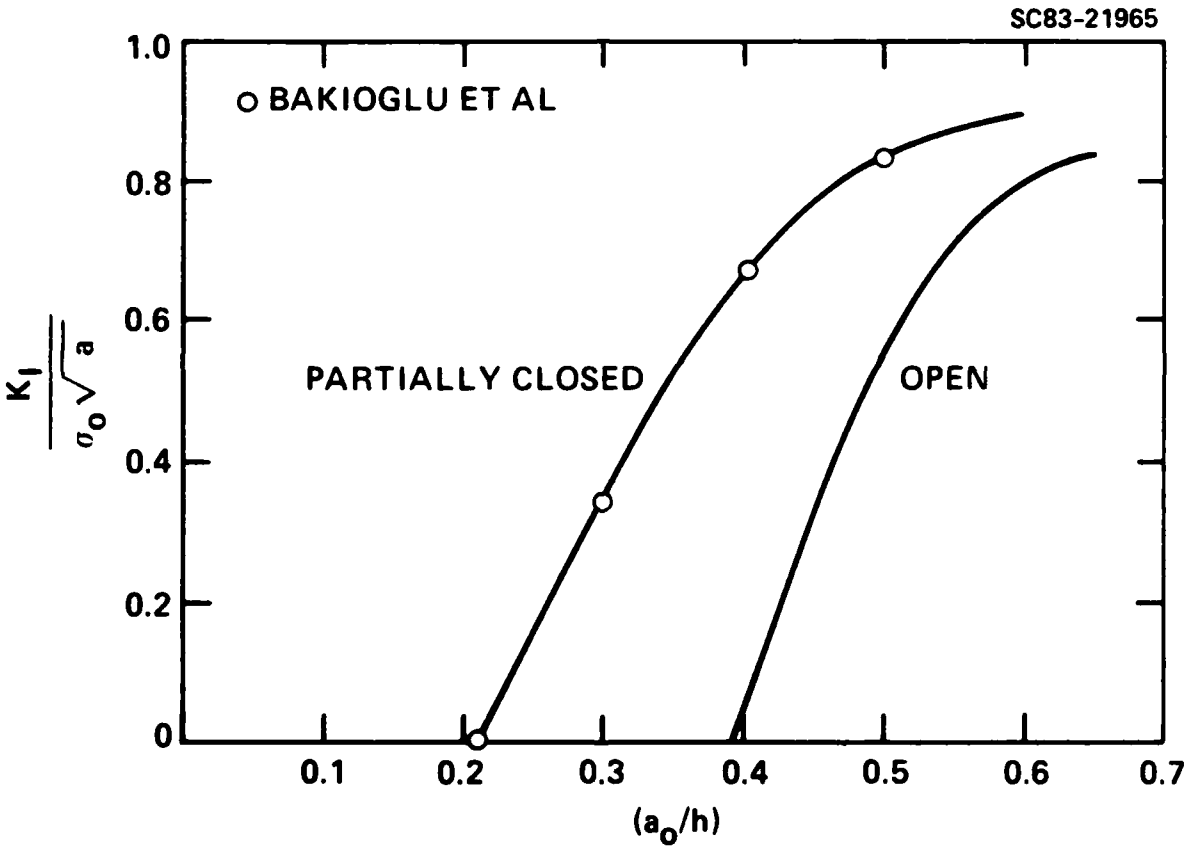

Fig. 3 Comparison of stress intensity factor for a surface crack in a tempered plate with previous work (Ref. 9) and to open crack solution.

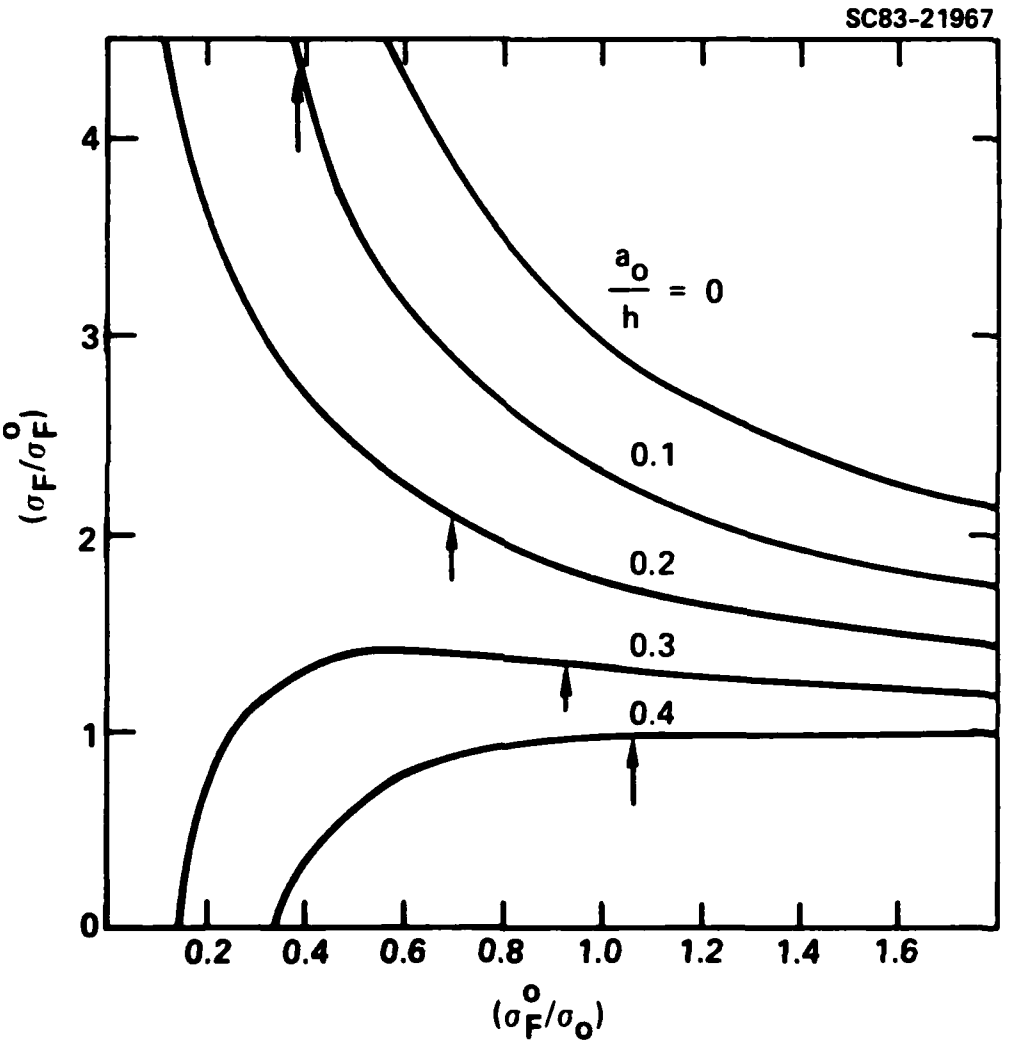

Fig. 4 Strengthening due to tempering in terms of base strength of material, surface residual stress and crack size.

value of σ_F°/σ_0 for which the crack is completely open. For lower values, the crack is partially closed at failure and the strengthening is less than that predicted for an open crack.

CONCLUSIONS

An approach has been outlined for calculating the stress intensity factor of a surface crack that is subjected to a combination of applied and residual stresses, and incorporates the effect of crack closure. Using an approximate expression for determining the closure distance in terms of the applied and residual stresses, it is then possible to estimate the strenthening obtained for a particular residual stress distribution. This assumes that failure still occurs by extension of the surface crack and not from some alternate flaw population.

The outlined approach was applied to the procedure of tempering. It was found that the approach agreed well with previous work. The influence of partial crack closure was found to give higher values of K_I than that predicted assuming the crack to be open and in particular there is a range of conditions for which $K_I > 0$ that is not predicted at all for an 'open' crack. This effect gives smaller amounts of strengthening than predicted for open cracks and a wider range of conditions in which subcritical crack growth processes could occur. For the particular example, it was found that if the surface crack is initially within the compression zone the amount of strengthening increases as the crack size to body size or the base strength to compressive stress ratios decrease. If the crack tip is within the tensile region, however, both weakening or spontaneous failure of the body are possible.

ACKNOWLEDGEMENTS

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A TECHNIQUE FOR INTRODUCING SURFACE COMPRESSION INTO ZIRCONIA CERAMICS

No. 18

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ABSTRACT

A technique is presented whereby compressive surface stresses are introduced into transformation-toughened ZrO_2 ceramics, by removing the stabilizing oxides such as Y_2O_3 from the surface. The advantages of this approach over other techniques are discussed, and experimental data confirming the presence of compressive surface stresses and subsequent improved resistance to indentation cracking are described.

It has been noted in transformation-toughened ZrO_2 ceramics that surface grinding causes the tetragonal ZrO_2 to transform to monoclinic.[1-5] The transformation involves a volume increase and thus places the surface in compression. Stresses as high as 1GPa have been measured experimentally.[6] Such compressive stresses are expected to lead to improvements in contact resistance and can lead to strengthening.[2,4,7] Other techniques such as impact or quenching have been suggested for introducing these compressive stresses.[8,9] The aim of this communication is to report a new technique for introducing these stresses and to report preliminary results on the improved contact resistance (to indentation) of these materials.

In certain of the transformation-toughened materials, it is found necessary to alloy the ZrO_2 with an oxide such as Y_2O_3 or CeO_2,[10] in order to retain the tetragonal phase. Therefore, if a material once fabricated with such an addition is heat-treated in ZrO_2 powder containing no alloy addition, the alloying oxide is expected to be removed from the surface region due to the concentration difference. Once this occurs, the chemical free energy associated with the ZrO_2 transformation will be increased in the surface region and the transformation is expected to proceed. This effect is confirmed in Table 1, where the apparent fraction of ZrO_2 in the monoclinic phase (not corrected for x-ray penetration) is determined from the areas under the {111} tetragonal and monoclinic x-ray diffraction peaks (CuKα radiation). It was found that after the heat treatment the amount of monoclinic ZrO_2 is substantially increased when compared to either the as-fired or annealed (after grinding) surface. Moreover, it was found that a greater fraction of ZrO_2 was transformed by the heat treatment than by surface grinding or impact. It thus appears that the Y_2O_3 removal technique is an efficient way of introducing compressive stresses into these materials. It is expected that the depth of the compressive zone can be controlled by the heat treatment temperature and time and that the damage associated with the other techniques such as grinding or impact, will not be a factor in the Y_2O_3 removal approach. Included in Table 1 are the measured values of the surface residual stresses using a previously-described x-ray diffraction technique,[6] confirming that compressive stresses are present.

Table 1

Fraction of Monoclinic ZrO_2 for an Al_2O_3/30 vol.% ZrO_2* Composite for Various Surface Treatments

Surface Treatment	Monoclinic Fraction	Surface Stress (MPa)
As fired	<0.02	-
Impact (220 SiC Grit)	0.10	-
Ground (320 Grit Diamond)	0.11	-550
Annealed (1400°C-16 h)†	<0.02	-180
Y_2O_3 Removal		
-1400°C (1 h)	0.31	-140
-1400°C (4 h)	0.60	-330
-1400°C (16 h)	0.67	-550

* ZrO_2 alloyed with 2.2 mole % Y_2O_3, fabricated by slip casting.
† After surface grinding.

The surface of a heat-treated specimen is compared in Fig. 1 with an annealed surface. It was found that a thin layer of the ZrO_2 powder adheres to the polished surface of the composite. This layer is ~5 μm in depth and was removed by polishing before obtaining the monoclinic ZrO_2 content data in Table 1.

In order to substantiate that the heat-treated surfaces were more resistant to contact damage, Vickers hardness indentations were made on polished surfaces of the as-fabricated and heat-treated composites. The radius of the radial cracks (c) around the indentations were measured for various indentation loads (P), and the results are given in Table 2. It was found that the crack size for a given load was substantially less for the heat-treated surfaces. At higher loads the difference between the two surfaces becomes less, presumably because the crack size is substantially

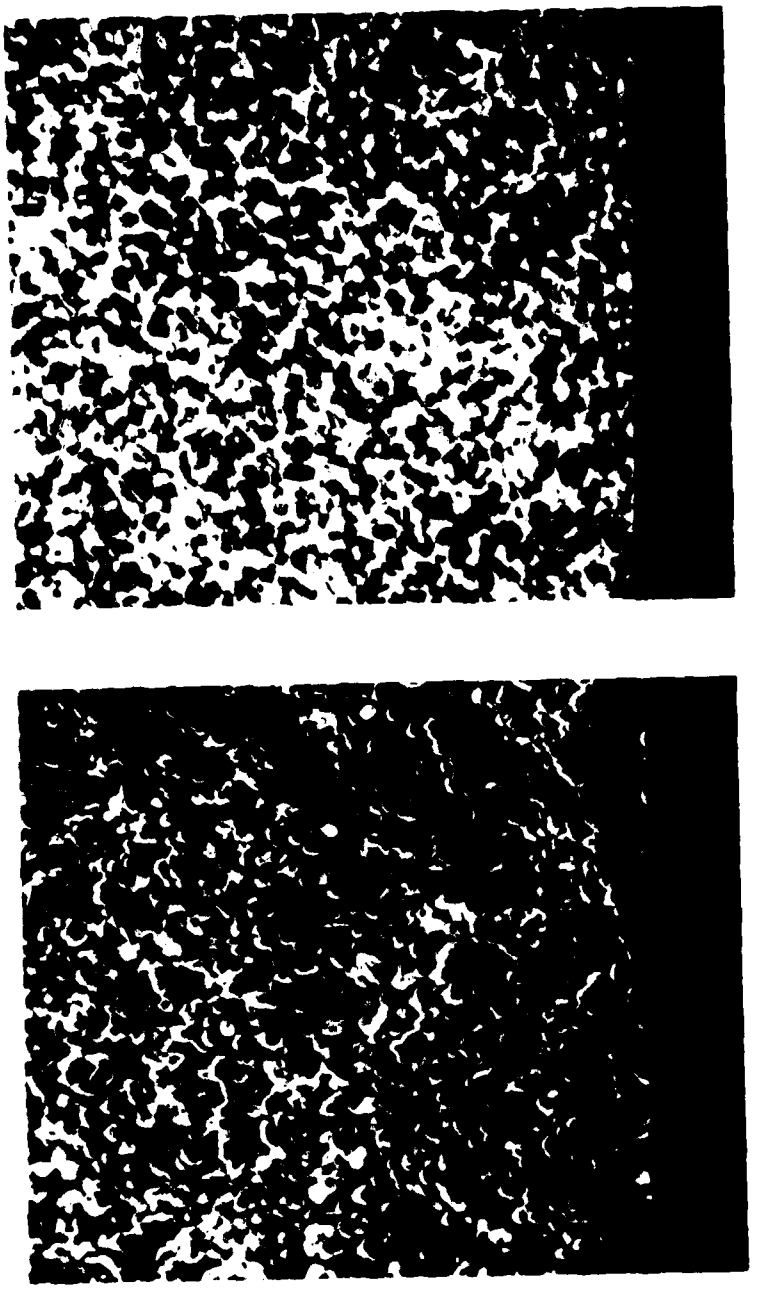

Fig. 1 Comparison of surfaces of Al_2O_3/30 vol.% ZrO_2 (2.2 mol.% Y_2O_3) after heat treatment in unstabilized ZrO_2 powder (left) and annealing in air (right). Conditions 1400°C/4 hours.

Table 2

Comparison of Indentation Crack Sizes for As-Fabricated and Heat-Treated Surfaces

Surface Treatment	P(N)	c(μm)	$P/c^{3/2}$ ($MPa \cdot m^{1/2}$)
A. As-fabricated	98	134	63.2
	147	191	55.7
	196	229	56.6
	294	290	59.5
B. Y_2O_3 Removal			
-1400°C (1 hr)	98	91.6	112
	147	129	100
	196	176	83.9
	294	223	88.3
-1400°C (4 h)	98	82.3	131
-1400°C (16 h)	98	80.0	137

larger than the depth of the compressive zone. Included in Table 2 is the value of $P/c^{3/2}$, which is usually a constant for materials where the crack growth is controlled by the residual stress associated with the indentation.[11,12] It was found that $P/c^{3/2}$ was approximately constant for the as-fabricated material, but decreases with increasing load for the heat-treated materials. The higher values of $P/c^{3/2}$ for the heat-treated surfaces are a result of the surface compressive stresses acting to reduce the stress intensity factor of the radial cracks.[13] As the load is increased, however, the crack size will become much larger than the compressive zone depth, and thus the effect of the surface stresses will be reduced.

Finally, it appears that use of the indentation technique could be a useful and rapid way of determining the optimum heat treatment conditions.

ACKNOWLEDGEMENTS

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RESIDUAL SURFACE STRESSES IN Al_2O_3-ZrO_2 COMPOSITES

No. 19

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ABSTRACT

Compressive surface stresses can be introduced into transformation-toughened ZrO_2 ceramics by a variety of techniques. These techniques are discussed and compared for Al_2O_3/ZrO_2 composites, particularly with respect to the transformation and residual stress profiles. Previous work on the direct measurement of the surface stresses and their influence on mechanical properties, such as strength, is reviewed.

1.0 INTRODUCTION

It is recognized that the transformation of tetragonal ZrO_2 to its monoclinic structure can be used to toughen certain ceramics.[1-8] The toughening mechanism has been analyzed theoretically by several authors.[9-13] This transformation toughening, hence, has the potential to strengthen these materials. In addition, however, it has been shown that the ZrO_2 phase transformation can be induced at the surface of these materials. Such surfaces would be expected to be placed in compression as the unconstrained transformation occurs martensitically and involves a volume increase.[14,15] In such situations if the material fails from flaws within the compression zone, an additional strengthening mechanism exists, i.e., compressive surface strengthening. The surface phase change was first noted by Garvie and his coworkers,[1,16] when they noted that surface grinding increased the amount of monoclinic ZrO_2 at the surface. Moreover, they noted that removal of the transformed layer by polishing led to a slight strength decrease (~20%). The influence of surface grinding on the strength of transformation-toughened materials has been studied by other groups.[17-19] For example, in Al_2O_3/ZrO_2 composites, it has been shown that annealing of ground surfaces can lead to a dramatic strength decrease.[18] This was interpreted as the removal of the surface compressive stresses. In order to optimize the influence of surface grinding, Gupta[19] studied the effect of changing the grit size of the grinding wheel on the strength of transformation-toughened ZrO_2. In this work it was found that a maximum occurred in the strengthening as a function of the grit diameter. The increase in strength was interpreted in terms of an increasing depth of the compressive zone, while for very coarse grit, strength-degrading microcracks were observed in the surface.

In addition to strengthening, compressive surface stresses are expected to lead to improvements in other properties of transformation-toughened materials, such as improved resistance to contact damage.

The aim of this paper is to review the techniques used by the authors to 1) introduce surface compression, 2) measure the depth and magnitude of these stresses, and 3) predict the influence of surface compression on strength.

2.0 TECHNIQUES FOR INTRODUCING SURFACE COMPRESSION

As indicated in the introduction, surface grinding can be used to introduce surface stresses in transformation-toughened materials. In addition, other techniques have been suggested, such as impact,[20] surface chemical reactions to form monoclinic ZrO_2,[21] and low temperature quenching.[22] These techniques are best understood in terms of the thermodynamics of the constrained ZrO_2 phase transformation. It has been shown that the minimum work (W) required to cause the transformation of a constrained ZrO_2 inclusion is given by[23]

$$W = -|\Delta G_c| + \Delta U_{SE} f + \Delta U_s / D \qquad (1)$$

where ΔG_c is the chemical free energy change of the unconstrained ZrO_2 phase transformation, ΔU_{SE} is the strain energy change, (1-f) is the reduction of strain energy caused by accompanying surface phenomena, ΔU_S is the change in surface energy associated with the surface phenomena and D is the inclusion diameter.

In order to induce the surface phase transformation, therefore, W could be supplied by external sources such as grinding or impact stresses. Alternatively, techniques for increasing $|\Delta G_c|$ (e.g., low temperature quenching) or grain size (D) could provide means of inducing the phase change. Indeed Eq. (1) suggests a myriad of ways that the surface transformation could be introduced. One technique that appears to have great potential has been recently investigated by the authors.[24] In some transformation-toughened materials, it is found necessary to add an alloying oxide (stabilizer) such as Y_2O_3 or CeO_2 to the ZrO_2 in order to retain the tetragonal phase. If such materials once fabricated are heat-treated in ZrO_2 powder containing no alloying oxide, the stabilizer will be removed from the surface of the fabricated body (increasing $|\Delta G_c|$) and will allow the transformation to proceed. This technique eliminates sources of damage that can accompany techniques such as surface grinding or impact and as shown in Table 1 leads to larger fraction

Table 1

Fraction of ZrO_2 Transformed to Monoclinic by Various Techniques for a Sintered Al_2O_3/30 vol. ZrO_2 Composite*

Technique	f
Sintered Surface	<0.02
Ground Surface (320 grit diamond)	0.11
Impacted Surface (220 grit SiC)	0.10
Y_2O_3 Removal 1400°C/1 hour	0.31
1400°C/4 hours	0.60
1400°C/16 hours	0.67

*ZrO_2 contained 2.2 mole per cent Y_2O_3

3.0 DETERMINATION OF TRANSFORMATION PROFILE

It is important in work on inducing phase transformations at surfaces to determine the transformation profile, which for transformations that involve volume changes, will be related to the residual stress profile. In the early work of Pascoe and Garvie,[16] an iterative process of x-ray diffraction and polishing was used. In this type of process, the monoclinic content is the mean of the zone penetrated by the x-rays, weighted exponentially by the proximity to the surface. Figure 1, for example, shows a comparison of the apparent monoclinic profiles for a ground surface and a surface in which the Y_2O_3 has been removed. These data were acquired using CuKα radiation, for which the penetration depth is ~5 μm (Al_2O_3/30 vol% ZrO_2), so the form of the true profiles is probably substantially different from that given in Fig. 1. The use of CrKα radiation would give a more exact profile as the penetration distance is much smaller (~2 μm). These types of polishing experiments do give information on the transformation depth, i.e., the depth at which the apparent surface monoclinic content is the same as the bulk monoclinic content, but the difficulties in obtaining the true profile, their accuracy and their laborious nature makes the approach rather unattractive. More rapid techniques for estimating the transformation depth[25,26] and the true surface monoclinic content[26] have been suggested but these depend on assumptions about the transformation profile.

An alternative approach for obtaining the transformation profile is to cross section the specimen and use a technique such as Raman Microprobe Analysis to measure the monoclinic content. This approach has been used previously to determine the ZrO_2 phase transformation in the vicinity of fracture surfaces.[27] Table 2 summarizes the transformation depth for the Y_2O_3 removal specimen as measured by the various techniques discussed in this section. It is found that the two calculation techniques underestimate the transformation depth, presumably because they both assume a uniform transformation zone.

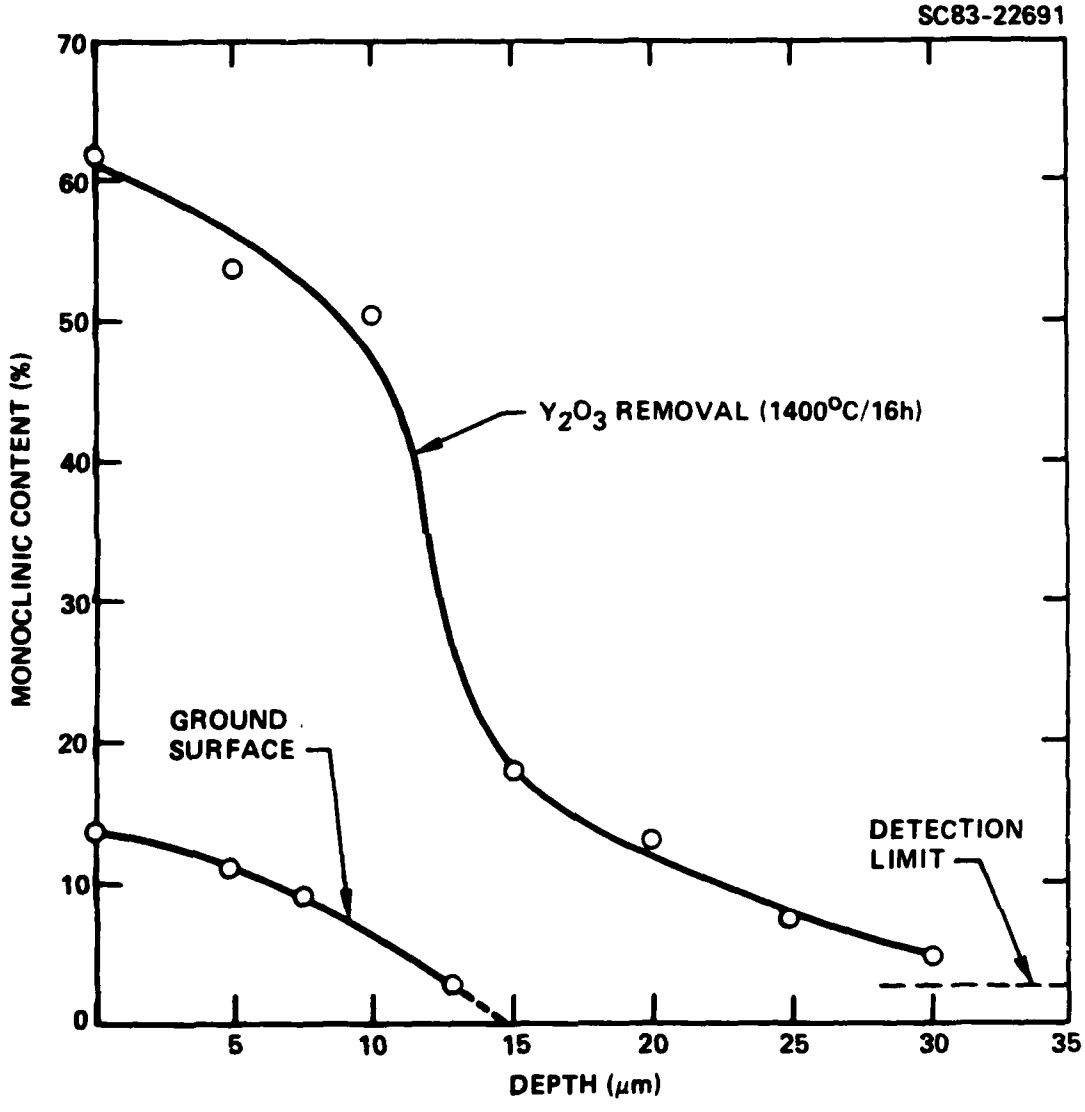

Fig. 1 Comparison of apparent monoclinic content as a function of distance from surface for a ground surface and a surface from which Y_2O_3 was removed (Al_2O_3/30 vol.% ZrO_2).

Table 2

Transformation Depths for Al_2O_3/30 vol% ZrO_2 (2.2 mole% Y_2O_3)*

Technique	Transformation Depth (μm)
Polishing	~32
Kosmac et al[25]	6
Garvie et al[26]	8

*Heat treated in ZrO_2 powder to remove Y_2O_3 (1400°C/16 hours)

4.0 MEASUREMENT OF MAGNITUDE AND DEPTH OF SURFACE STRESSES

The mechanical response of materials containing residual surface stresses is expected to depend on the profile of these stresses. This profile could be calculated theoretically. For example, the transformation profiles discussed in the last section could be used to calculate the stress profile using the volume change associated with the transformation. The stresses can be calculated from thermal stress theory because of the complete correspondence between stresses developed by concentration differences and stresses developed by temperature differences. For example, for an infinite slab whose surfaces have been transformed, the residual stresses parallel to the surface are biaxial and are given by[28]

$$\sigma_R(x) = \sigma_R(y) = \frac{V_v E \varepsilon}{(1-\nu)} [f_a - f] \qquad (2)$$

where E is Young's modulus of the slab, ν is its Poisson's ratio, ε is the strain associated with the volume change, V_v is the volume fraction of ZrO_2 in the composite, f is the fraction of ZrO_2 transformed at a point and f_a is the average fraction of ZrO_2 transformed. For the ZrO_2 transformation, one therefore expects the surface to be in compression whereas the interior of the body will be under a compensating tension. It is clear that this approach could be modified by theoretically predicting the transformation profile and then using Eq. (2) to predict the stress profile. Clearly, either of the approaches still require experimental confirmation and, hence there is a need for direct measurement of the residual stresses. One technique, based on x-ray diffraction, has been used by the authors to measure the residual stresses due to grinding on both Al_2O_3 and transformation-toughened Al_2O_3/ZrO_2 composites.[23,29] In this technique a given set of planes in the surface of a specimen are examined as a function of their angular rotation with respect to the surface. After a suitable calibration of the x-ray elastic constants of these planes, the strains are used to calculate the residual stress. For this work, it was found necessary to use CrKα radiation so that the penetration depth of the

x-rays was small.[23,29] In order to determine the stress profile, the compressive layer can be removed by polishing the specimen and re-determining the residual stress in successive steps. An example of this is shown in Fig. 2 for the ground surface of Al_2O_3/30 v/o ZrO_2,[23] along with a correction that was applied to the raw data to compensate for stress relaxation when a portion of the surface is removed.[30] It should be noted that the depth of the stresses is similar to the transformation depth (Fig. 1). Although Eq. (2) has not been verified from the complete transformation profile it was found that the magnitude of the residual stresses at the surface measured by the x-ray diffraction technique did correlate reasonably well with Eq. (2). For this calculation the value of f (at the surface) was calculated using the technique of Garvie et al[26] described earlier, which was then substituted in Eq. (2), assuming f_a = 0 (maximum value of residual stress). The calculated and measured residual stress for a variety of Al_2O_3/ZrO_2 composites are compared in Table 3.[23,31] As can be seen, compressive surface stresses as high as 1 GPa have been measured in these materials.

Table 3

Comparison of Measured and Calculated Residual Surface Stress in Al_2O_3/ZrO_2 Composites

Vol.% ZrO_2 (m/o* Y_2O_3)	f	Type of Surface	Surface Stress (MPa) Measured	Calculated
7.5 (1.4)	0.53	Ground	-440	-300
15 (1.4)	0.59	Ground	-570	-440
30 (2.4)	0.16-0.25	Ground	-450-680	-310-480
50 (2.4)	0.17	Ground	-780	-480
60 (3.6)	0.41	Ground	-1010	-1300
30 (2.2)	0.85	Y_2O_3 Removal	-550	-1600

*m/o mole%

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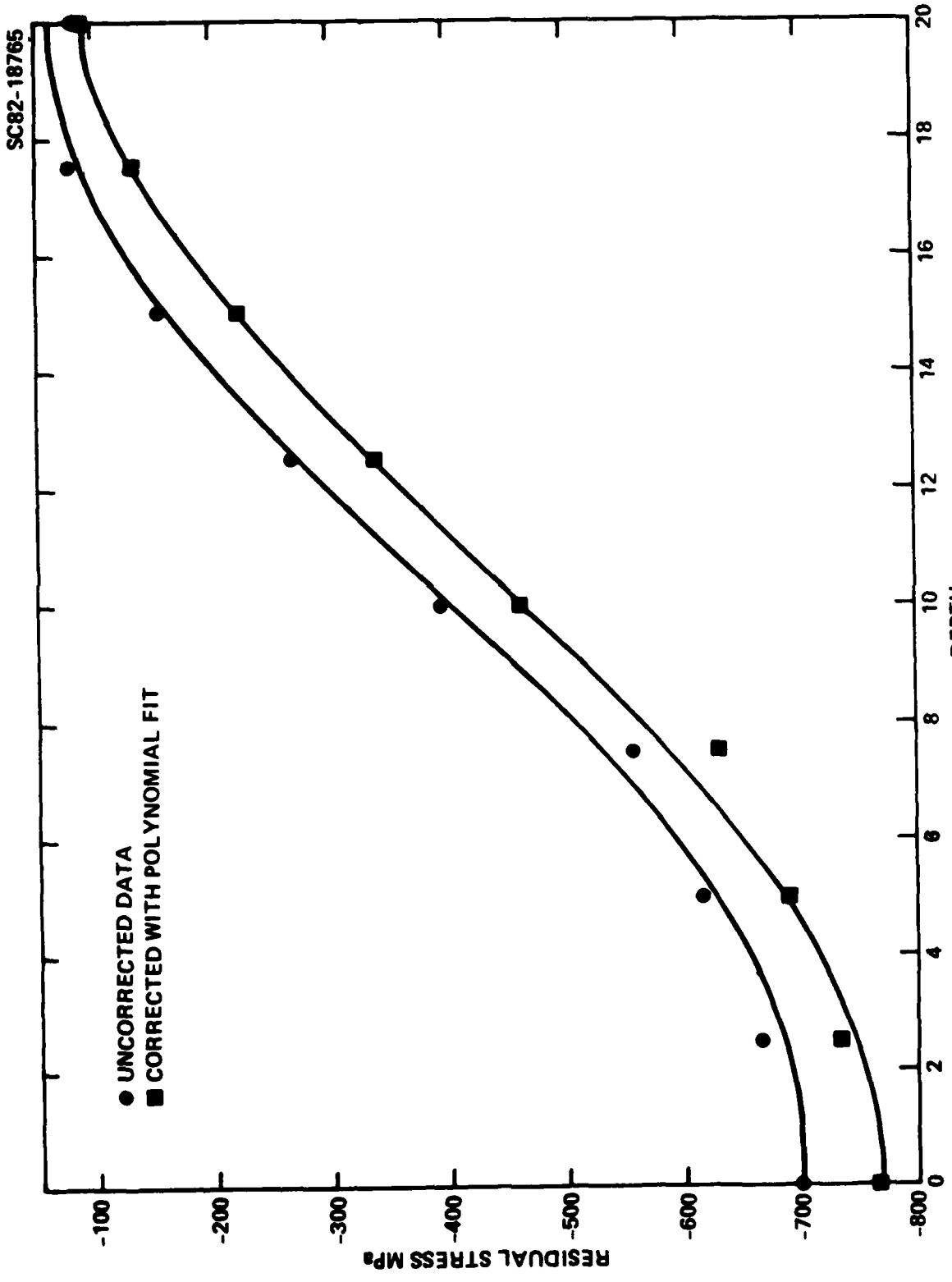

Fig. 2 Measured residual surface stress for a ground Al_2O_3/30 vol% ZrO_2 composite as a function of depth removed by polishing.

5.0 INFLUENCE OF SURFACE COMPRESSION ON STRENGTH

For materials that fail from surface flaws, it is expected that compressive surface stresses would give rise to strengthening. Using the simple residual stress distribution in Fig. 3, Lawn and Marshall[31] have shown that the residual stress component of the stress intensity factor (K_I^R) is given by

$$K_I^R = m\sigma_o(\pi a_o)^{1/2} M \tag{3}$$

where

$$M = (\frac{2}{\pi} \sin^{-1} \delta_1)(1 + \frac{\delta_1}{2d_1}) - \frac{2}{\pi\delta_1} (1-(1-\delta_1^2)^{1/2}) - \frac{\delta_1}{2d_1} \text{ when } \delta_1 < 1 \ ,$$

$$M = (1 - \frac{2}{\pi\delta_1}) \qquad \text{when } \delta_1 > 1 \ , \tag{4}$$

m is a free surface correction, $\delta_1 = \delta/a_o$ and $d_1 = d/a_o$. During failure there will also be an applied component of the stress intensity factor K_I^A which is given by

$$K_I^A = mY\sigma_A(\pi a_o)^{1/2} \tag{5}$$

where σ_A is the applied stress and Y is a constant that depends on the crack loading and geometry. For conditions where the crack is completely open at failure, i.e., there is no contact between the opposing crack faces, the total stress intensity factor (K_I^T) is given by simply adding Eqs. (3) and (5). For example, in a tensile test Y = 1 and ignoring the free surface correction (m = 1), one obtains

$$K_I^T = K_I^A + K_I^R = \sigma_o(\pi a_o)^{1/2} M + \sigma_A(\pi a_o)^{1/2} \tag{6}$$

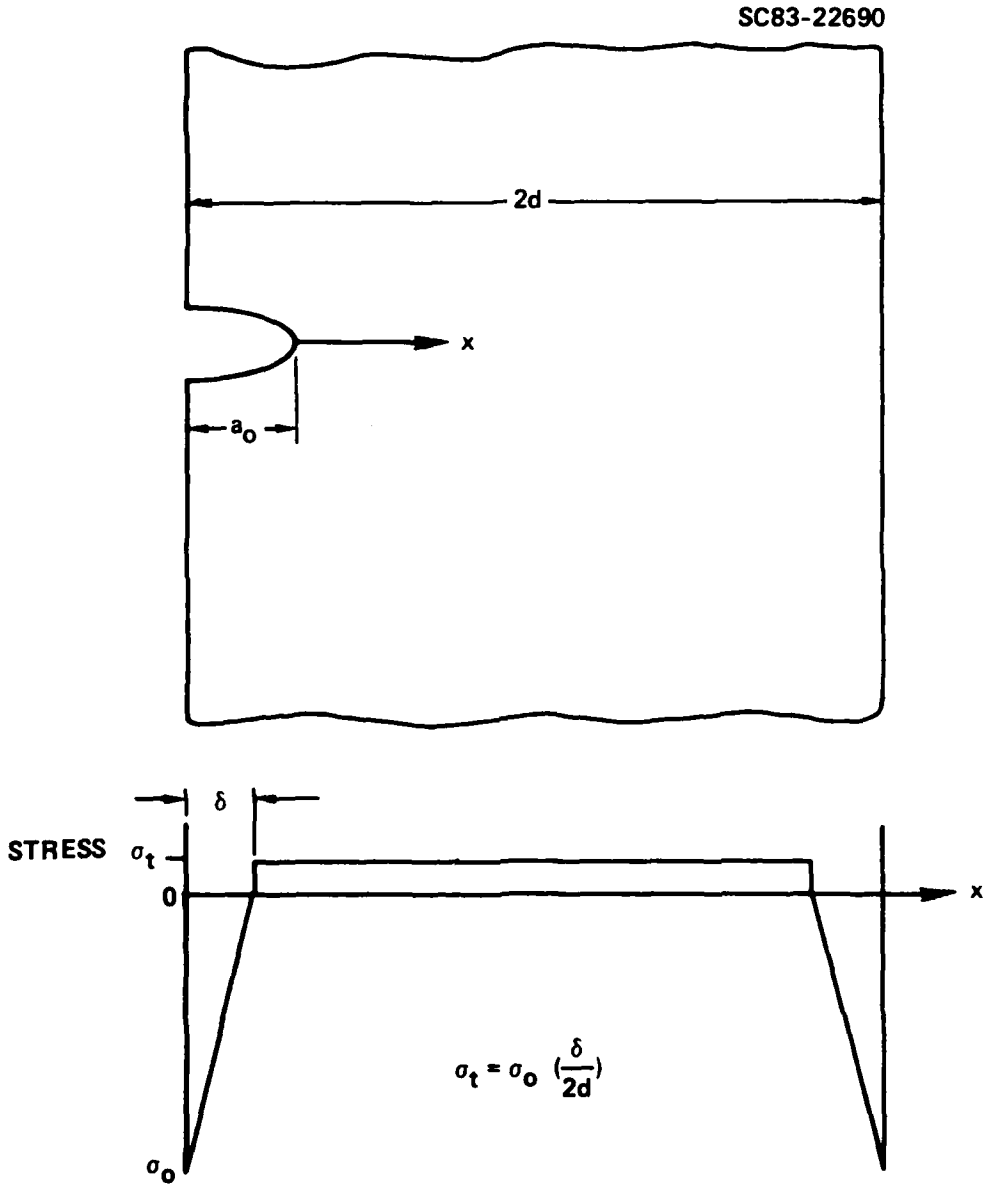

Fig. 3 Simple residual stress distribution for a plate under surface compression.

At failure $K_I^A + K_I^R = K_c$, $\sigma_A = \sigma_f$ and substituting $K_c = \sigma_f^o(\pi a_o)^{1/2}$ where σ_f^o is the strength in the absence of surface compression, one obtains

$$\frac{\sigma_f}{\sigma_f^o} = [1 - M(\frac{\sigma_o}{\sigma_f^o})] \tag{7}$$

This equation is illustrated in Fig. 4 as a function of (σ_o/σ_f^o) and δ_1, for the case where $d_1 = 50$. This example is similar to that analyzed by Swain[32] and as can be seen the strengthening is linearly dependent on (σ_o/σ_f^o). It is worth noting, however, that when $d_1 < 4$, the residual tensile stresses in the body can give rise to weakening.

As pointed out earlier, this type of approach does not account for partial crack closure when simple superposition of K_I^R and K_I^A cannot be used. This effect is expected to be important when σ_o is large, i.e., when the compressive surface stress keep the crack closed at the surface. Such effects have been analyzed by one of the present authors and it is found when the crack is partially closed, $K_I^T > 0$ for conditions not predicted by Eq. (6).[33,34] It was found in general that K_I^T is greater than that predicted by Eq. (6) when the crack is partially closed, though both solutions will merge when the surface crack becomes completely open. In terms of strengthening, this implies that the solution for a partially closed crack predicts less strengthening than Eq. (7). Using a previous analysis,[34] the strengthening incorporating the influence of crack closure is included in Fig. 4 for the cases where $\delta_1 > 1$. For the case where $\delta_1 = 2.0$, the crack was found to be completely open at the failure condition for $\sigma_o/\sigma_f^o < 10$. The small differences between Eq. (7) and the closure analysis are presumably a result of ignoring the free surface correction. For the case where $\delta_1 = 1.0$, however, the surface crack is partially closed at failure for $\sigma_o/\sigma_f^o > 5$ and the strengthening is less than predicted by Eq. (7). In order to approximately determine the influence of crack closure when $\delta_1 < 1$, the following approach was taken. When σ_o is very large compared to σ_f^o the crack will be closed to a depth δ. In the limit, therefore, the surface crack will be so strongly

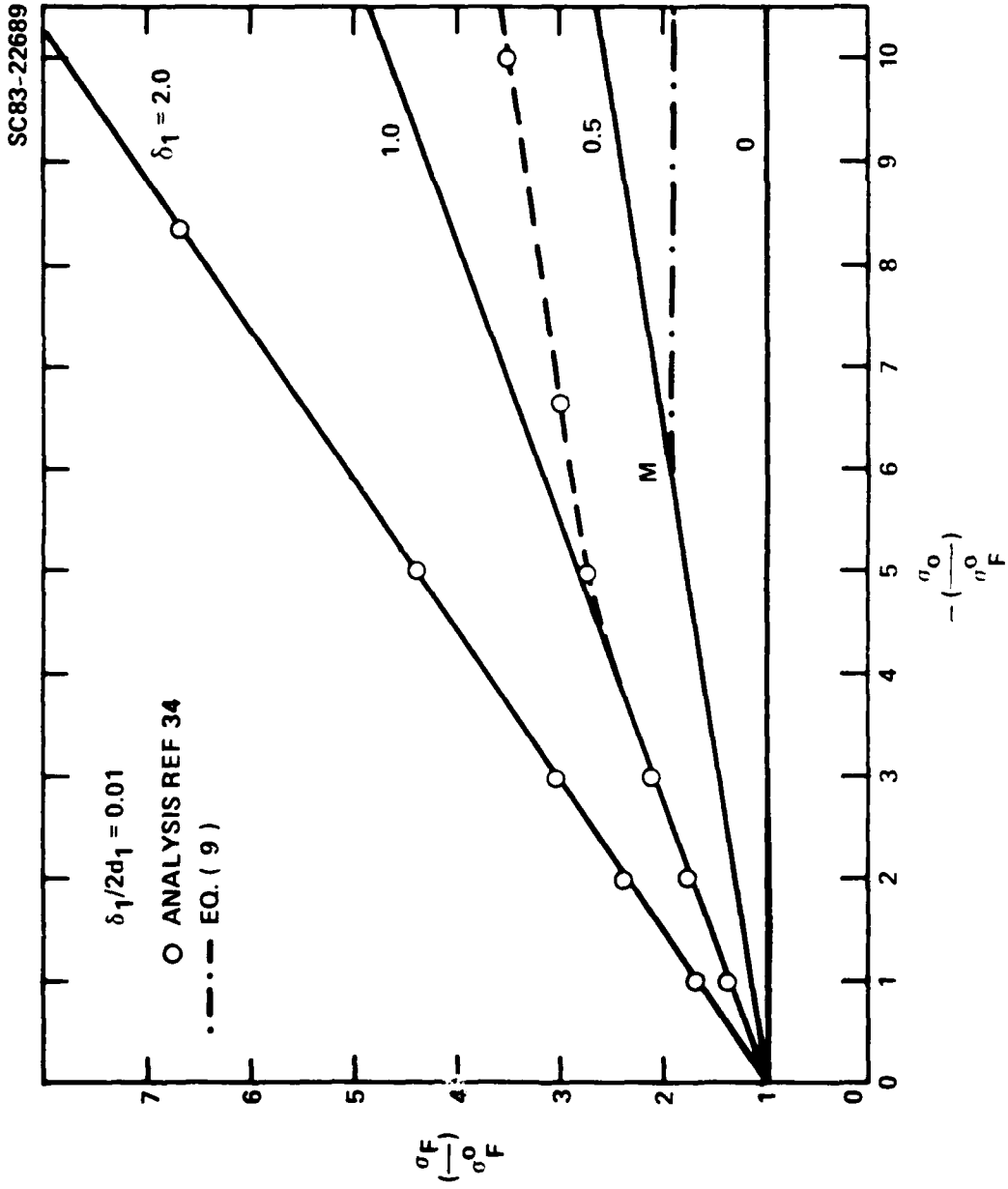

Fig. 4 Potential strengthening for a material subjected to a simple residual stress distribution (Fig. 3).

pinned at the surface that it will act more like an internal crack of length $(a_o - \delta)$. For this case, the total stress intensity factor will be

$$K_I^T = \left(\frac{\sigma_o \delta_1}{2d_1} + \sigma_A\right)\left(\pi\left(\frac{a_o - \delta}{2}\right)\right)^{1/2} \tag{8}$$

and the strengthening will be

$$\frac{\sigma_f}{\sigma_f^o} = \left(\frac{2}{1 - \delta_1}\right)^{1/2} - \frac{\sigma_o \delta_1}{2\sigma_f^o d_1} \tag{9}$$

This equation is plotted in Fig. 4 for the case where $\delta_1 = 0.5$, and is found to intersect that predicted by Eq. (7) at M. The strengthening taking account of crack closure is therefore expected to be given by Eq. (7) at low values of σ_o/σ_f^o and by Eq. (9) at high values. From this approximate approach it appears that σ_f/σ_f^o must go through a maximum when for cases when $\delta_1 < 1$.

As discussed previously, the depth of the compressive zone in transformation-toughened ZrO_2 ceramics is relatively shallow (~20 μm). Therefore, in order to obtain substantial strengthening, failure must occur from rather small surface cracks, perhaps ~50 μm or less. For sintered Al_2O_3/ZrO_2 composites, this has been found not to be too common as several alternative flaw populations exist, e.g., agglomerates or voids.[35] The effects can, however, be more striking on hot-pressed materials. Figure 5 shows the difference in strength obtained for hot-pressed Al_2O_3/ZrO_2 composites that are surface ground and those that are surface ground and subsequently annealed. For a hot-pressed Al_2O_3/30 vol.% ZrO_2 (2 m/o Y_2O_3) composite, the use of SiC particle impact to induce surface compression was examined and the results are shown in Fig. 6. For small SiC particles, some

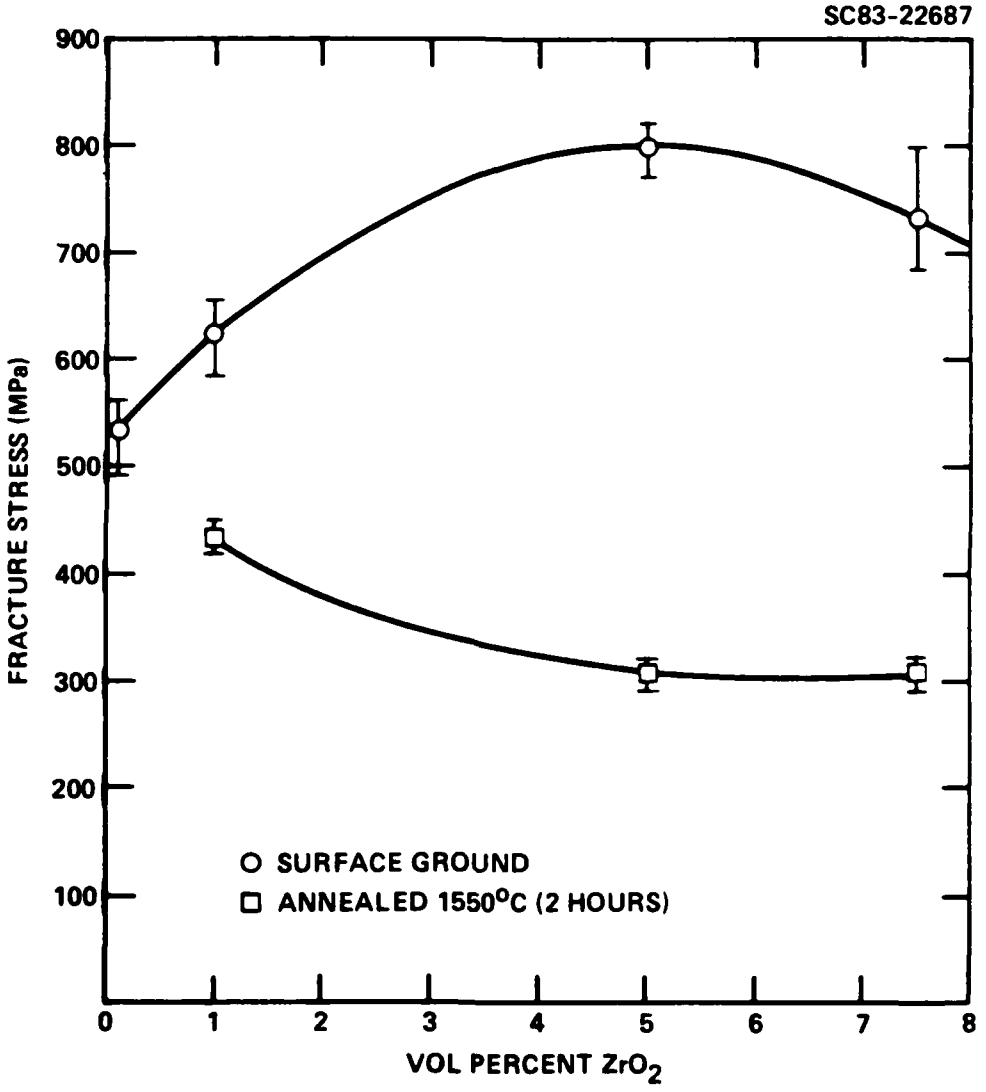

Fig. 5 Comparison of strengths of hot-pressed Al_2O_3/ZrO_2 composites after surface grinding and subsequent annealing.

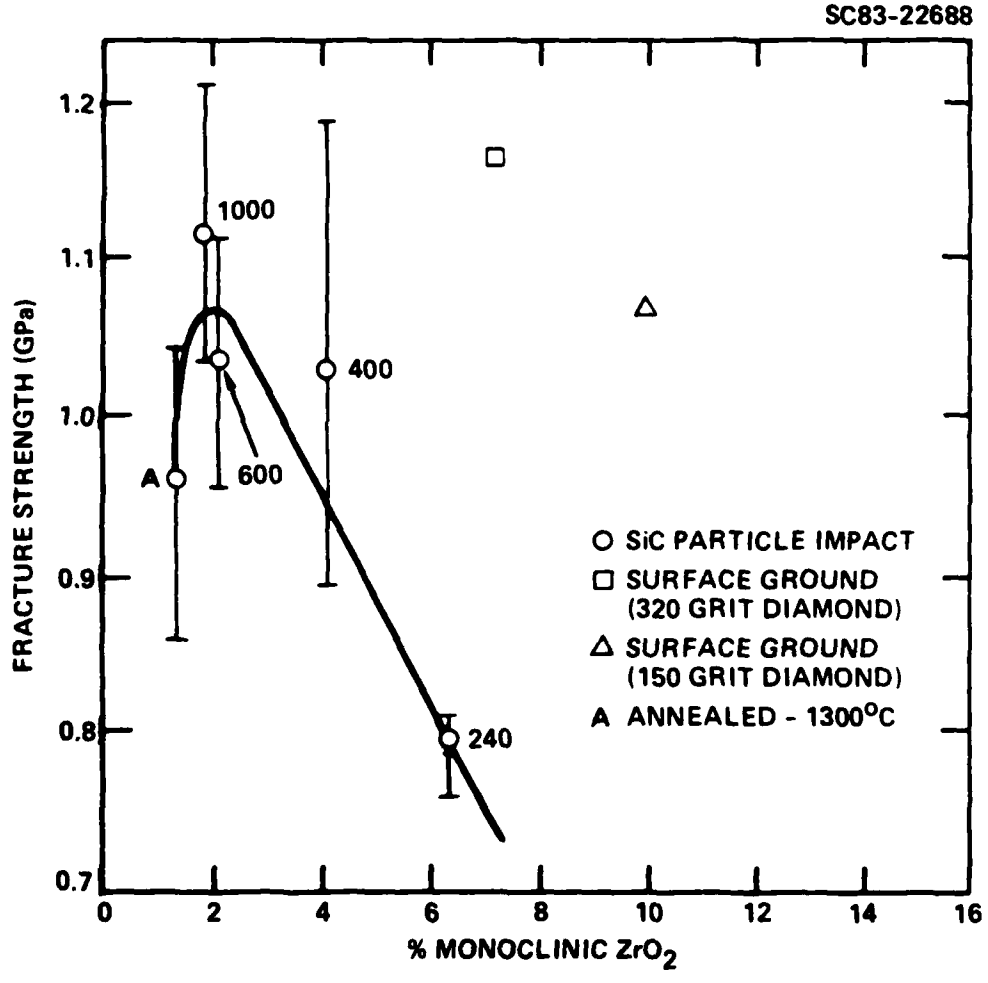

Fig. 6 Influence of impact of SiC particles on the strength of hot-pressed Al_2O_3/30 vol.% ZrO_2 (2 mol.% Y_2O_3). The numbers indicate grit size of SiC used.

strengthening was observed but for larger sizes, the strength was found to decrease. In these latter materials, more surface ZrO_2 was transformed to monoclinic but the cracks introduced by the impact must be larger than those previously present and hence led to strength degradation. The influence of surface grinding for the same composite is included in Fig. 6.

Even in the absence of strengthening, the presence of surface compressive stresses are expected to be beneficial for improved resistance to contact damage, e.g., wear resistance. Table 4 compares the radius of the radial cracks produced at Vickers hardness indentations for annealed specimens and specimens in which the Y_2O_3 was removed from the surface. It is clear that the surface compression has led to a substantial decrease in crack size particularly at low loads. At the higher loads, the crack sizes are substantially larger than the compressive zone size and the difference between the two surfaces becomes less pronounced.

Table 4

Comparison of Indentation Crack Sizes for As-Fabricated and Heat-Treated Surfaces

Surface Treatment	P(N)	c(μm)*
A. As-fabricated	98	134
	147	191
	196	229
	294	290
B. Y_2O_3 Removal		
-1400°C/1 hour	98	91.6
	147	129
	196	176
	294	223
-1400°C/4 hour	98	82.3
-1400°C/16 hour	98	80.0

* Radial crack radius

Finally, it is worth remembering that the surface compression will be relieved if the material is subjected to a high temperature. Figure 7 shows the apparent fraction of monoclinic ZrO_2 (determined by x-ray diffraction) after annealing for 2 hours at various temperatures. It is generally expected that once the material is raised above the transformation temperature (~1100°C), the monoclinic ZrO_2 would transform to tetragonal and provided no grain growth occurs during annealing, the tetragonal ZrO_2 would remain down to room temperature when the specimen is cooled. Although this is true for a substantial amount of the monoclinic ZrO_2, annealing temperatures as high as 1600°C were required before the monoclinic ZrO_2 content returned to its bulk value. This process was also found to be time-dependent, so it appears other processes must be involved. Previous work[36] indicated that this was probably a result of some microcrack healing that must occur before all the monoclinic ZrO_2 produced by grinding is re-transformed back to tetragonal ZrO_2 by annealing.

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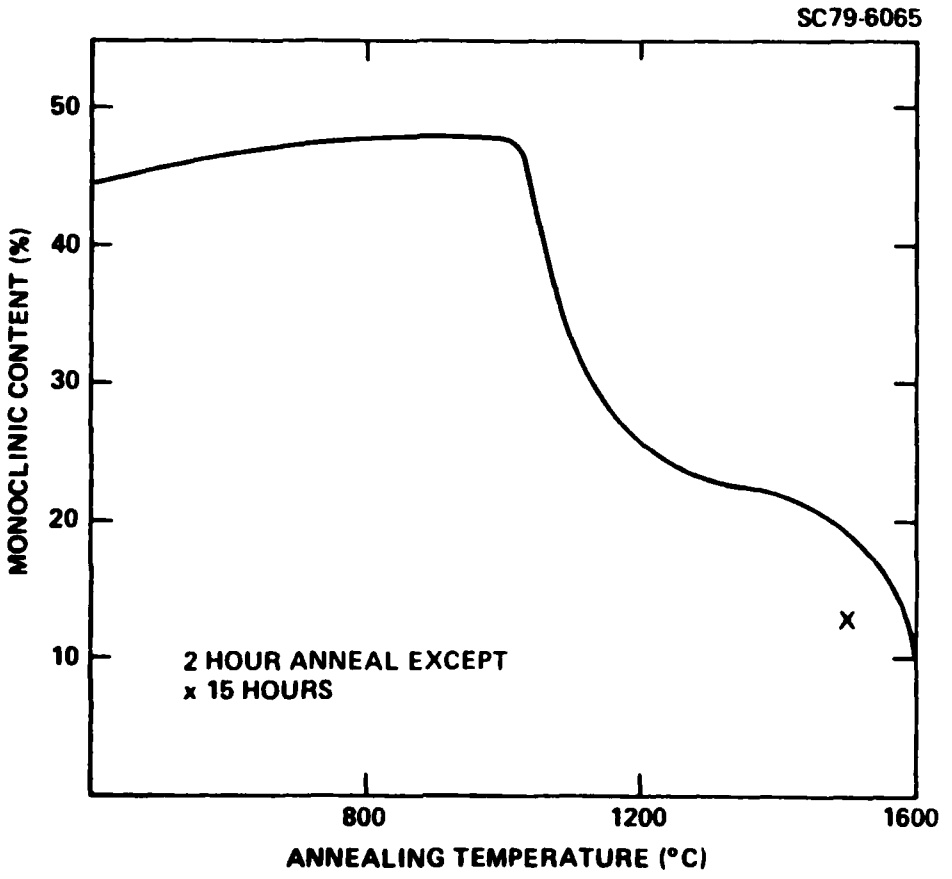

Fig. 7 Influence of annealing on apparent surface monoclinic content for a hot-pressed Al_2O_3/7.5 vol.% ZrO_2.

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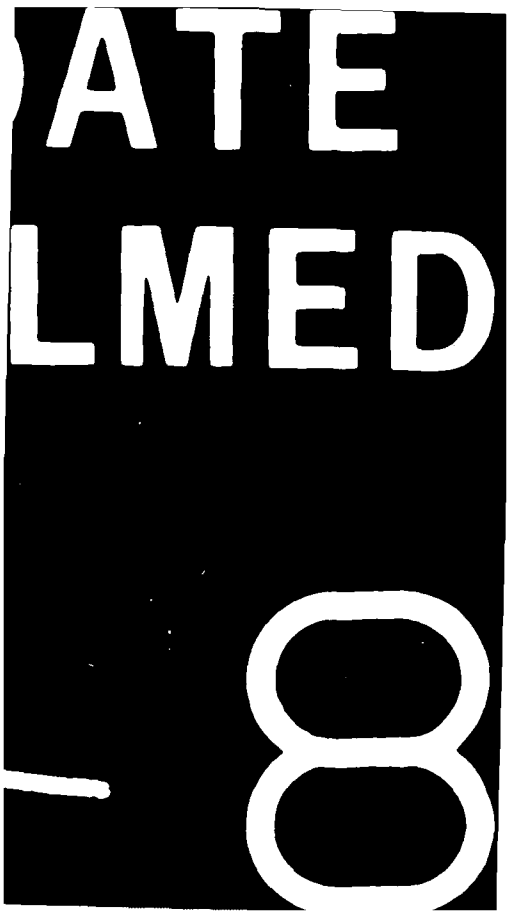
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