attempted to show the limitation of the Evans and Charles approach for transformation-toughened ceramics, especially when studying residually stressed surfaces. Indeed, the use of their approach can substantially alter the form of the indentation data, and hence confuse its interpretation.

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Reply to 'Comments on "Crack-size dependence of fracture toughness in transformation-toughened ceramics"'

In recent years, several investigators have used an indentation technique for determining fracture toughness, K_c, of brittle materials. Two approaches, both based upon the indenter crack approximated as a penny-shaped crack under point loading at the centre, have been suggested. Evans and Charles [1] used data from various materials of known fracture toughness (K_c) values and generated a master curve by plotting $(K_c\Phi/Ha^{1/2})(H/\Phi E)^{0.4}$ against c/a, where H is the hardness, E is the Young's modulus of elasticity, c is the crack radius and a is half the indent diagonal. The other approach, proposed by Anstis *et al.* [2] sets $K_c = \chi P/c^{3/2}$ with χ being a material dependent parameter. Green [3], in the preceding discussion of a recent paper by Ikuma and Virkar [4], suggests that the technique of Evans and Charles [1] leads to erroneous results and that one must use the method given by Anstis *et al.* [2]. The present authors have also noted that the two indentation techniques often give differing results and a discussion of this point is warranted. As will be shown in the following, however, the contention by Green [3] that the problem lies in the validity of the Evans and Charles [1] approach is without basis. The objective of our response is twofold. Firstly, we will examine Green's [3] data and assess the validity of the concept of the crack growing out of the zone of compression. Secondly, we will examine our data using both of the techniques and attempt to sort out the source of the discrepancy.

Green [3] suggests that the increase in K_c (as determined using the method of Evans and Charles [1]) with increasing $c^{1/2}$ observed in our and his work is the result of nonlinearity in the $(K_c\Phi/Ha^{1/2})(H/\Phi E)^{0.4}$ against c/a curve at low values of c/a. By contrast, the method of Anstis *et al.* [2] yielded K_c independent of c [3]. In Fig. 1, the data of Ikuma and Virkar [4] on $ZrO_2 + 4.5\,mol\,\%\,Y_2O_3$ is replotted. K_c calculated using the method of Anstis *et al.* [2] is also plotted in the same figure. Note that K_c against $c^{1/2}$ using this technique actually decreases with increasing $c^{1/2}$. For similarly prepared samples, Green [3] finds K_c independent of $c^{1/2}$. However, when K_c decreases with increasing $c^{1/2}$, Green [3] suggests that this

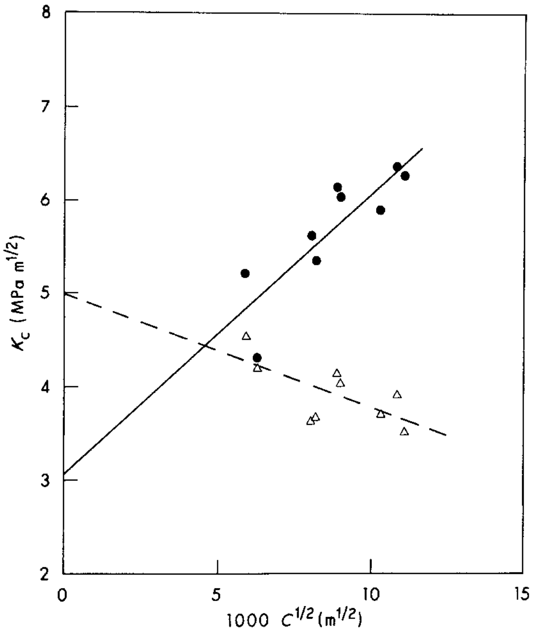

Figure 1 K_c of ZrO_2 + 4.5% Y_2O_3 plotted as a function of $c^{1/2}$; K_c determined using (●) the Evans and Charles [1] technique and (△) the method of Anstis *et al.* [2].

implies that the crack is growing out of the zone of compression as the crack becomes larger.

Fig. 2 of Green [3] shows $P/c^{3/2}$ determined as a function of $c^{1/2}$ for samples that were annealed and polished, and those that were subjected to a thermochemical treatment involving a diffusional step which removes the stabilizing oxide (e.g. Y_2O_3 or CeO_2) from the near surface region [5]. For annealed samples $P/c^{3/2}$ was found to be independent of $c^{1/2}$ but for samples subjected to the thermochemical treatment, $P/c^{3/2}$ was found to decrease with increasing $c^{1/2}$. Assuming $\chi \approx 0.07$ (using reasonable values for E and H), the difference in $P/c^{3/2}$ for these two treatments corresponding to $c = 100\,\mu$m gives a compressive surface stress on the order of 405 MPa assumed uniform to a depth of 100 μm. Clearly, as the stress is not uniform, the surface stress must be greater than 405 MPa while that at a depth of 100 μm should be lower. A similar calculation for $c = 25\,\mu$m gives a compressive stress on the order of 900 MPa; i.e. the surface stress must be greater than 900 MPa. (This calculation assumes that the plot in Fig. 2 of Green can be extrapolated. If, however, the crack is contained within the zone of compresion below 100 μm, then $P/c^{3/2}$ against $c^{1/2}$ below 100 μm will be expected to increase with $c^{1/2}$. In that case, however, the magnitude of surface compressive stress of 405 MPa will be nearly uniform to a depth of about 100 μm.) However, X-ray analysis [5] only indicated a surface stress of the order of 550 MPa. These calculations using Green's [3] data indicate that substantial compressive stresses must be present to a significant depth provided we assume that $K_c \propto P/c^{3/2}$.If one assumes a bend strength of 500 MPa and $K_c \approx 5$ MPa m$^{1/2}$ for the annealed and polished samples, the critical flaw size will be of the order of 125 μm. The implication then is that the samples subjected to the thermochemical treatment should exhibit strength enhancement of at least 200 to 300 MPa. However, no such increase in strength was observed [6]. The lack of increase in strength then implies that the zone of compression must be quite shallow. Then, the decrease in $P/c^{3/2}$ with increasing $c^{1/2}$ cannot be due to the crack growing out of the zone of compression as suggested by Green [3], but some other explanation must be sought. In fact, K_c cannot be linearly proportional to $P/c^{3/2}$ as assumed by Green [3]. A further confirmation of the incorrectness of Green's [3] hypothesis is obtained via the following example.

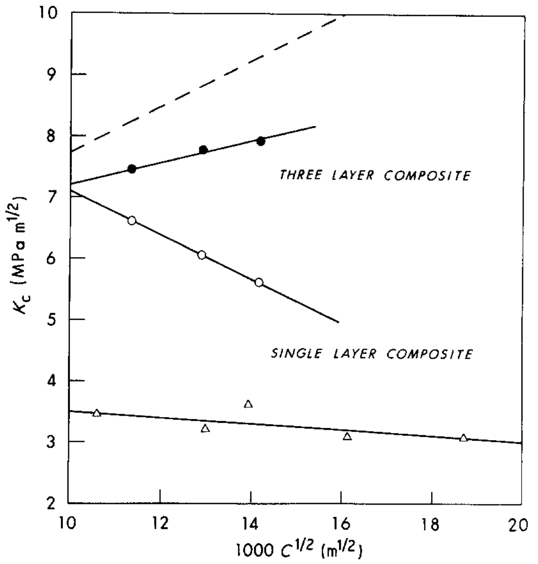

Figure 2 K_c of three layer composites determined using the techniques of (●) Evans and Charles [1] and (○) Anstis *et al.* [2]. Note that K_c determined using the technique of Anstis *et al.* [2] decreases with increasing $c^{1/2}$ despite the fact that the crack is entirely contained within the zone of compression. In these samples, c/a was below 2.5.--- predicted. Single layer composite (△) using the technique of Anstis *et al.* [2].

Recently, Cutler *et al.* [7] fabricated Al_2O_3 + 15 wt % ZrO_2 composites in such a way that the surface region contained unstabilized ZrO_2 while the bulk region contained Y_2O_3 doped ZrO_2(3% Y_2O_3). Upon cooling to room temperature (from sintering temperature), some of the zirconia in the surface layers transformed to the monoclinic form while zirconia in the bulk was all tetragonal. The surface layer of a 6 mm thick bar sample was at least 300 μm in thickness (typically in excess of 500 μm). A strain gauging technique showed that the magnitude of the compressive stress in the surface layer was ~ 330 MPa. Bend strength data confirmed the existence of substantial compressive stresses in that the strength was similarly increased over single layer samples made of Al_2O_3 + 15 wt % unstabilized ZrO_2. K_c was determined as a function of $c^{1/2}$ using the method of Anstis *et al.* [2] for both the single layer sample and the three layer composite. Fig. 2 shows the plots of K_c against $c^{1/2}$. For the single layer sample, K_c was found to decrease with increasing $c^{1/2}$ although the decrease was slight. The three layer composite, by contrast, showed a substantial drop in K_c with increasing $c^{1/2}$. The largest c was 200 μm in this measurement. A crack of this dimension, however, is entirely contained within the zone of nearly uniform compression in the three layer composite. Thus, one should expect an *increase* in K_c with $c^{1/2}$ as indicated by the broken line. This result clearly demonstrates that a decrease in K_c with increase in $c^{1/2}$ cannot be interpreted as though the crack is growing out of the zone of compression. (Ironically, if one were not aware of the presence of compressive stresses in these or Green's samples, the decrease in K_c with increase in $c^{1/2}$ may in fact be interpreted as though a tensile stress exists in the surface region.) K_c estimated using the method of Evans and Charles [1] is also shown in the same figure. Note that K_c so determined actually increases with $c^{1/2}$ as expected.

In these experiments on the three layer composites, the typical value of c/a was between 1.80 to 2.65 which corresponds to the nonlinear part of the master curve given by Evans and Charles [1]. An estimate (assuming H ≈ 16 GPa) of c/a ratio for the Al_2O_3 + 30% ZrO_2 sample of Green [3] (with compressive stresses) for $c \approx 100\,\mu$m gives $c/a \approx 1.58$. At such low values of c/a, it is doubtful that the technique of Anstis *et al.* [2] is applicable. For very short cracks (low c/a ratio), the assumption of a penny-shaped crack with point loading at the centre may be an over simplification. The residual force caused by the plastic deformation under an indent is not a point force but a distributed force. For large c/a ratios, the assumption of a point loaded crack is reasonable but not for small c/a ratios. We know that for a point loaded penny-shaped crack

$$K = \frac{P}{(\pi c)^{3/2}} \qquad (1)$$

where P is the point force. Also, for a penny-shaped crack under a uniform stress σ,

$$K = \frac{2\sigma c^{1/2}}{\pi^{1/2}} \qquad (2)$$

If we distribute the point force on the entire crack surface as an extreme case of distributed force, $\sigma = P/\pi c^2$. Then,

$$K = \frac{2P}{(\pi c)^{3/2}} \qquad (3)$$

i.e. the K is twice that for a point loaded crack when the force is distributed over the entire surface. This illustrates one of the reasons why $K_c \neq \chi P/c^{3/2}$ at too low a value of c/a. It is interesting to note that estimate of c/a for the Al_2O_3 + 30% ZrO_2 samples of Green [3] indicates that for the samples with compressive stresses, c/a ranges from 1.58 to 2.47 while for samples that were annealed and polished, c/a ranges from 2.52 to 3.02. Thus, the trends observed by Green [3] can not be due to the crack growing out of the zone of compression – rather it is the result of the inapplicability of the technique of Anstis *et al.* [2] at too low a value of c/a. Estimates of c/a ratios for the annealed and tempered glass plates used by Marshall and Lawn [8] (assuming $H \approx 5.6 \times 10^9$ Pa) indicates that for the tempered plate, c/a ranges from about 1.41 to about 2.20 while that for the annealed plate ranges from 2.66 to 5.06. The linear range in the tempered glass occurs for c/a ratios greater than 2.0; the ratio Marshall and Lawn [8] assume to be acceptable (i.e. the ratio beyond which $K_c = \chi P/c^{3/2}$ is valid). It is conceivable that c/a will have to be even greater than 2.0 for $K_c = \chi P/c^{3/2}$ to be valid. It is clear that in the work of Green [3], this condition is not satisfied (c/a is typically below 2.0 in Green's

[3] samples subjected to thermochemical treatments; for Al_2O_3 + 30% ZrO_2, c/a varies between 1.58 to 2.47 while that for β''-Al_2O_3 + 15% ZrO_2, c/a varies between 1.47 to 2.19, assuming $H \approx 12\,\mathrm{GPa}$).

It is clear that for small values of c/a, the technique of Anstis *et al.* [2] does not yield reliable values of K_c (as discussed by Anstis *et al.* [2]). For obtaining reliable results, $c/a > 2.0$ as pointed out by Marshall and Lawn [8]. In the case of three layer composites, it is possible to get K_c increasing with $c^{1/2}$ using the technique of Anstis *et al.* [2], provided c/a is nearly constant for the range of c values and/or is greater than 2.5. Often, however, for well developed cracks, c/a is found to be below 2.5. In such cases, K_c as determined by the technique of Anstis *et al.* [2] gives an apparent decrease in K_c with increase in $c^{1/2}$. Decrease in $P/c^{3/2}$ observed by Green [3] is, as stated before, simply the result of using the technique beyond the range of validity and the interpretation that the crack is growing out of the zone of compression is incorrect.

Whether the technique of Evans and Charles yields reliable values of K_c at small values of c/a remains to be established. It is important to note, however, that the technique of Evans and Charles [1] is somewhat empirical and through curve fitting probably makes corrections for low c/a ratio. As such, it is possible that at low c/a values, the Evans and Charles [1] technique may yield reliable values. In the work of Ikuma and Virkar [4], c/a was often lower than 2.0. It is for this reason that the method of Evans and Charles [2] was preferred. (It is worth noting that K_c determined using the Evans and Charles [1] approach by extrapolating to $c^{1/2} = 0$ (Fig. 1) gives a value of $3.1\,\mathrm{MPa\,m^{1/2}}$ which agrees well with $K_c = 3.5\,\mathrm{MPa\,m^{1/2}}$ obtained using DCB technique [4].) Large scatter in the data often results when attempting to use the Evans and Charles [1] technique at low values of c/a. The region of low c/a values needs to be thoroughly examined experimentally before a reliable master curve (if at all possible) can be made. Clearly, more work is needed to resolve these critical issues.

Based on this work, the following conclusions can be drawn;

1. The observation by Green [3] that $P/c^{3/2}$ decreases with increasing $c^{1/2}$ is not the result of the crack growing out of the zone of compression. It relates to the inapplicability of the technique of Anstis *et al.* [2] for low values of c/a.
2. For low values of c/a, our results indicate that the Evans and Charles [1] approach gives more realistic values of K_c. However, no attempt is made here to generalize this conclusion. Further analysis and calibration at low values of c/a is necessary. Based on this, the contention by Green [3] that the technique by Evans and Charles yields incorrect values of K_c is without basis.

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