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SCIENCE

Making Ceramics "Ductile"

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Distributed irreversible deformation in otherwise brittle ceramics (specifically, in silicon carbide and micaceous glass-ceramic) has been observed in Hertzian contacts. The deformation takes the form of an expanding microcrack damage zone below the contact circle, in place of the usual single propagating macrocrack (the Hertzian "cone fracture") outside. An important manifestation of this deformation is an effective "ductility" in the indentation stress-strain response. Control of the associated brittle-ductile transition is readily effected by appropriate design of weak interfaces, large and elongate grains, and high internal stresses in the ceramic microstructure.

Ceramics have been cited as the "materials of the future." However, ceramics are also notoriously brittle and are subject to catastrophic failure from the growth of a single dominant crack (*1*). They are limited in their use as load-bearing materials by a low intrinsic toughness and, correspondingly, a lack of ductility to absorb mechanical energy. Of the various mechanisms that have been advocated for imparting toughness to ceramics (*1*), the most widespread and practical is that of "bridging," in which frictional pullout of interlocking grains and second-phase particles retards crack-wall separation (*2, 3*). Toughness then becomes a rising function of crack size [so-called toughness-curve, or resistance-curve, behavior (*1*)]. A most important element in the enhancement of bridging is the controlled introduction of weak interfaces on the microstructural scale, to deflect the primary crack and thereby generate a more effective interlocking structure. One can also enhance bridging by coarsening and elongating the grain structure and by incorporating internal mismatch stresses (*1, 4, 5*). Thus to gain toughness in ceramics, one builds in microstructural heterogeneity. However, there is a price to pay. Toughness is improved, but only in the "long-crack" region. In the "short-crack" region, built-in weakness can enhance fracture at the microstructural level, reducing laboratory strength (6) and increasing the susceptibility to wear and erosion (*7, 8*).

In view of this tendency toward countervailing interrelations in toughness properties, surprisingly little effort has been made to understand the seemingly deleterious short-crack properties of heterogeneous ceramics. We argue that these same seemingly deleterious properties also have potential benefits. The impetus for this assertion comes from our recent contact study on a moderately tough, coarse-grain alumina (*9*). Our test uses the classical Hertzian configuration of an indenting sphere on a flat specimen surface (*10*). A rich literature exists for Hertzian tests on homogeneous brittle solids, notably glass, in which a cone-shaped crack (the Hertzian fracture) forms in a region of weak surface tensile stress outside the contact circle (*11–16*). No such literature exists for analogous tests on heterogeneous tough ceramics. In our study on coarse alumina, we found no well-defined cone crack but rather a damage zone of distributed intergranular microfractures in a region of high shear stress below the contact circle. Stress-induced intragrain twins appeared to act as essential crack precursors in the alumina by concentrating stresses at the weak grain boundaries. The subsurface microfracture damage zone expanded with repeat loading, indicating a pronounced "fatigue" characteristic.

We have performed Hertzian tests on ceramic systems whose microstructural heterogeneity was tailored by heat treatments. In their base homogeneous forms, these ceramics are classically brittle and exhibit the familiar cone fractures. In their heterogeneous forms, however, these same ceramics are subject instead to subsurface microfracture, leading to an effective "ductile" response in the contact behavior. The results have profound implications concerning the capacity of brittle solids to sustain mechanical damage and absorb energy. Most important, the results suggest ways in which such radical "brittle-ductile transitions" in the mechanical response of ceramics may be effected by controlled microstructural modifications.

In our Hertzian test, a hard sphere of radius r was loaded onto a flat specimen surface. From the load P and contact radius a, we plot indentation stress $p_0 = P/\pi a^2$ versus indentation strain a/r (*17*) to produce an indentation stress-strain curve. A special specimen configuration, consisting

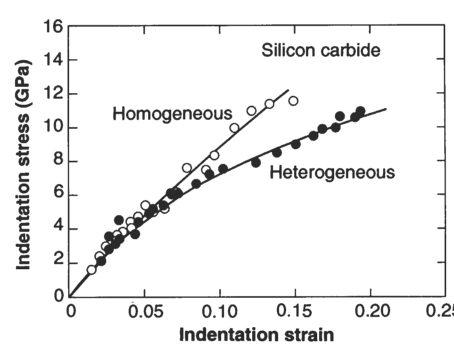

Fig. 1. Indentation stress-strain curve for silicon carbide in homogeneous fine-grain and heterogeneous coarse-grain forms. Data taken with tungsten carbide spheres in the radius range r from 1.58 to 12.7 mm.

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of two polished rectangular half-blocks bonded together with thin adhesive, allowed us to obtain section as well as surface views of the contact damage (*18*). Indentations were placed symmetrically across the surface trace of the bonded interface on the top surface. After indentation, the adhesive was dissolved and the half-blocks separated to reveal the subsurface damage. The top and side surfaces of a given half-block were then coated with gold and viewed in Nomarski illumination.

In our first example, we compared two forms of silicon carbide, a ceramic traditionally known for its innate hardness and brittleness. The first form, a commercially available monophase material (*19*), had a fine, equiaxed, well-bonded microstructure (grain size ≈ 4 μm). The second form, a material we developed by incorporating an yttrium-aluminum-garnet (YAG) sintering agent and subjecting the sintered material to a grain-growth heat treatment (*20*), had a coarsened, elongated microstructure (grains ~3 μm thick and 25 μm long) with weakened interfaces between matrix grains and boundary phases. The microstructural conditions in the second form were conducive to enhanced grain bridging and therefore to enhanced long-crack toughness.

The indentation stress-strain curve for the homogeneous form (Fig. 1, open circles) shows little nonlinearity, indicating essentially elastic-brittle behavior. By contrast, the curve for the heterogeneous form (Fig. 1, filled circles) tends toward a "yield" response, more characteristic of ductile solids. In micrographs of the contact damage, we observe a transition from well-defined cone fracture (Fig. 2A) to distributed subsurface microfracture (Fig. 2B) with the increase in microstructural heterogeneity. Higher magnification reveals a relatively uninterrupted (transgranular) crack path in Fig. 2A and locally deflected (intergranular) microfractures along the weak interphase boundaries in Fig. 2B.

An even more striking illustration of the effect of microstructural heterogeneity on the intrinsic mechanical response is afforded by a micaceous glass-ceramic (*21*). This ceramic has a respectable long-crack toughness (*22*). It conversely has a low short-crack toughness, a notable consequence of which is that the material is readily machinable (*23*). In its base glass state, the material is effectively homogeneous on the microscale. A controlled heat treatment crystallizes mica flakes (~1 μm thick and 10 μm long) in the glass matrix, with weak interphase boundaries, to produce the glass-ceramic. Whereas the indentation stress-strain curve for the base glass shows little nonlinearity, the curve for the crystallized glass-ceramic shows a dramatic yield turnover (Fig. 3). Half-surface and section micrographs of the contact damage in the base glass (Fig. 4A) reveal classical Hertzian cone fracture. In the glass-ceramic (Fig. 4B), the damage is contained wholly subsurface and has the macroscopic appearance of a plastic zone in metals (*18*). Higher magnification of the areas within the subsurface damage zones revealed local shear fractures along the mica-glass interfaces, with some linkage and coalescence in the glass matrix at heavier loads.

The distinctive change from classical cone fracture to distributed microfracture damage with increased microstructural heterogeneity (Figs. 2 and 4) denotes a new kind of brittle-ductile transition in ceramic materials. This transition is apparent as an increased nonlinearity and a correspondingly greater work of penetration in the indentation stress-strain functions (Figs. 1 and 3). It foreshadows a greater resistance to strength degradation and an enhanced capacity for energy absorption from spurious contacts and impacts.

Microscopically, the subsurface damage responsible for the ductility originates from some local shear-driven deformation process. The nature of this process is fundamentally different from the dislocation processes that operate in metals (*17*). Instead, the deformation originates at "shear faults" in the grain microstructure. The key is the presence of intrinsically weak interfaces to create and arrest the faults, and perhaps

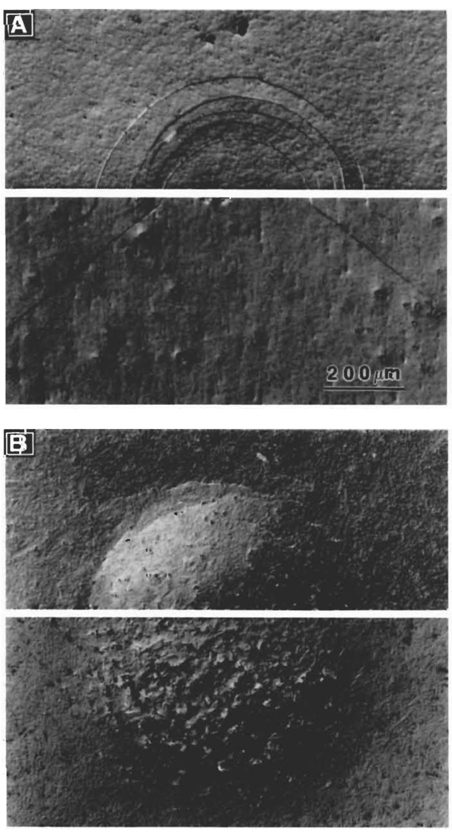

Fig. 2. Half-surface (top) and section (bottom) views of Hertzian contact damage in silicon carbide, from a tungsten carbide sphere of radius r = 3.18 mm at load P = 2000 N. (**A**) Homogeneous fine-grain form showing well-defined cone crack (stress p_0 = 7.44 GPa), (**B**) heterogeneous coarse-grain form showing distributed subsurface damage (stress p_0 = 7.20 GPa).

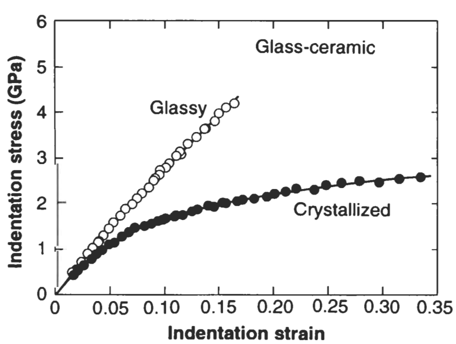

Fig. 3. Indentation stress-strain curve for glass-ceramic in base glass and crystallized forms. Data taken with tungsten carbide spheres in the radius range r from 0.79 to 12.7 mm.

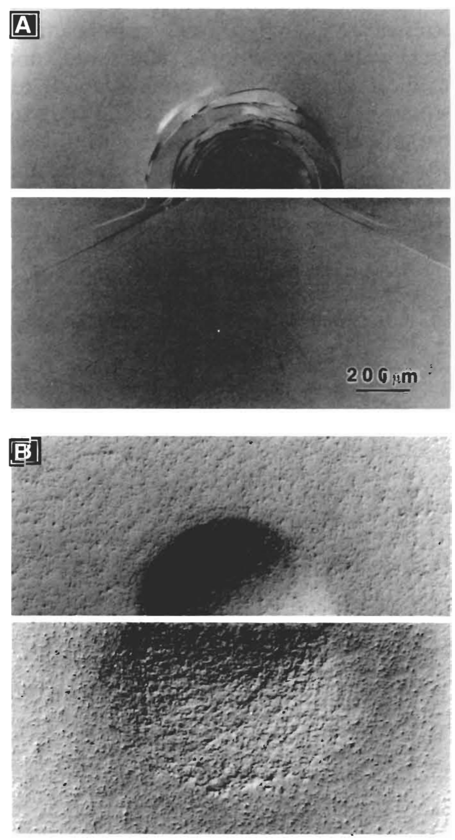

Fig. 4. Half-surface (top) and section (bottom) views of Hertzian contact damage in glass-ceramic, from a tungsten carbide sphere of radius r = 3.18 mm at load P = 1000 N. (**A**) Base glass form showing well-defined cone crack (stress p_0 = 2.88 GPa). (**B**) partially crystallized form showing distributed subsurface damage (stress p_0 = 1.87 GPa).

also to provide favored paths for ensuing extensile microcracks at the fault edges, within the subsurface Hertzian stress field. In the silicon carbide (Fig. 2), the shear faults form at the interfaces between the matrix grains and the grain boundary (YAG) phases. In the glass-ceramic (Fig. 4), they form at the mica-glass interface. This transition to apparent ductility in otherwise highly brittle ceramics is attributable to the large compressive component of contact stress fields (*13*, *16*). The deflection of any downward propagating surface ring cracks along grain or interface boundaries away from the tensile stress trajectories (*13*) suppresses the development of a single cone crack, and at the same time, the action of strong shear stresses on the weak planes in the confining subsurface zone promotes the development of a population of highly stabilized microcracks. The latter kind of distributed damage has been widely considered in the fracture of rocks (*24*), where hydrostatic compressive fields are the norm, but not in advanced ceramics, whose design has hitherto been based predominantly on single-crack mechanics.

Our results are of special relevance to the mechanical response of ceramics where highly localized mechanical or thermal stresses are likely (*10*), such as in bearings, local impact conditions, refractories, and medical implants (for example, tooth restoratives). The implication is that one may design ceramic microstructures to change the very nature of the damage behavior and so optimize the mechanical response to suit particular applications.

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