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Role of Microstructure in Dynamic Fatigue of Glass-Ceramics after Contact with Spheres

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Dynamic fatigue data are reported for fine- and coarse-grained micaceous glass-ceramics after contact damage with spheres. The strengths of indented specimens are measured at stressing rates from $\sim 10^{-2}$ to 10^{4} MPa·s^{-1} in water. The strength degradation is substantially faster in the coarse-grained structure, and is accelerated further by multicycle contact loading. Failures originate from contact sites in all cases but undergo a progressive transition from classical cone cracks to quasi-plastic microcrack zones with increases in the grain size and the number of contact cycles. The results highlight the particularly deleterious effect of quasi-plastic damage accumulation on lifetime.

I. Introduction

Ceramics are susceptible to slow crack growth in the presence of moist environments.[1,2] Slow growth of critical flaws leads to strength degradation, increasing the longer the stress is applied. Such effects are measured most conveniently in constant stressing rate experiments, in which flexure stresses are applied at given rates up to the point of failure. The slope of the resulting "dynamic fatigue" plot of strength versus stressing rate is an inverse measure of the crack velocity exponent.[3,4]

One way of generating dynamic fatigue data with relatively small scatter in the data and control in the flaw state is to test specimens with indentation cracks. Most experiments of this type have used Vickers indenters, with well-defined radial cracks.[5–7] In those cases, the failure conditions are amenable to fracture mechanics analysis, as well as to direct observation of crack growth to failure. Residual stresses around the indentation site can have a profound influence on the crack evolution, but can be accommodated within the fracture mechanics framework to allow accurate evaluation of crack velocity parameters.[5]

More recently, interest has turned to spherical indenters (Hertzian indentation), because of their special relevance to the lifetimes of ceramics for dental materials, bearings, cutting tools, etc.[8] Hertzian indentation uniquely enables one to follow the full evolution of lifetime-degrading contact cracks, from the inception of initial damage to final failure. As the ceramic microstructure is made more heterogeneous the damage undergoes a "brittle-to-quasi-plastic" transition:[9–11] in fine-grained ceramics, tensile-driven Hertzian cone cracks are initiated from the surface immediately outside the contact ("brittle mode"), whereas in coarse-grained ceramics, the damage takes the form of a "yield" zone containing closed shear microcracks below the contact ("quasi-plastic mode"). Although initially less deleterious than cone cracking, quasi plasticity can become highly deleterious in cyclic contact loading ("contact fatigue") as microcracks coalesce into radial cracks.[12]

Contact fatigue studies using spherical indenters in repeat loading have been well studied.[12–15] However, no systematic study has been made of the effect of quasi-plastic damage accumulation on the ensuing dynamic fatigue properties of ceramics. We examine this issue here by comparing dynamic fatigue data for two micaceous glass-ceramics tested in water: one material is relatively brittle with a fine grain structure and the other relatively quasi-plastic with a coarse grain structure. We show that the quasi-plastic damage mode is substantially more susceptible to fatigue than the conventional brittle mode, especially after cyclic contact.

II. Experimental Procedures

The materials used in this study were micaceous glass-ceramics (MGCs) (Corning, Inc., Corning, NY) used in previous studies of contact damage.[11,16,17] Two extreme microstructures, "fine" (F-MGC) and "coarse" (C-MGC), were produced using controlled aging heat treatments.[16,18] Table I shows basic details of the microstructure, along with fatigue parameters (to be determined below). Bar specimens 3 mm × 4 mm × 50 mm were cut from blocks of each MGC material and surface-polished to a 0.5 μm diamond finish for indentation-strength testing.

Hertzian indentations were conducted on the center top surfaces of the MGC bars using tungsten carbide (WC) spheres of radius $r = 3.18$ mm, in water. Single-cycle contact tests were made to peak loads 850 and 1200 N in the F-MGC and 500 and 1200 N in the C-MGC, at a fixed crosshead speed 0.2 mm/min (Model 1122, Instron Corp., Canton, MA). The lower of these loads in each case lies just above the thresholds for strength-degrading damage in the two materials—the higher load is a common value in the region of well-developed damage. Multicyclic contact tests were performed at the lower loads over 10^4 cycles at a frequency of 10 Hz (Model 8502, Instron Corp.).

Strength tests were conducted on the contact-damaged bars in four-point flexure (outer and inner spans 20 and 10 mm, respectively), with the contact damage site centered on the tensile side. The bars were broken in water at stressing rates from 10^{-2} to 10^{4} MPa·s^{-1}. All broken specimens were examined by optical microscopy (Nomarski illumination) to confirm failure from the contact damage site.

III. Results

Plots of strength σ_F versus stressing rate $\dot{\sigma}_a$ are shown in Fig. 1 for F-MGC and in Fig. 2 for C-MGC after single-cycle and

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Table I. Microstructures and Fatigue Parameters for Micaceous Glass-Ceramics

Heat-treatment conditions	Grain size‡ (μm)	Number of cycles, n	Dynamic fatigue parameters† N'	$\log \lambda'_P$§
		F-MGC		
1000°C, 4 h	0.3 × 1.0	1	36.6 ± 4.1	107 ± 12
1000°C, 4 h	0.3 × 1.0	10^4	20.9 ± 5.3	56.3 ± 14.4
		C-MGC		
1120°C, 4 h	1.2 × 8.0	1	21.3 ± 2.3	60.7 ± 6.8
1120°C, 4 h	1.2 × 8.0	10^4	13.4 ± 1.4	33.9 ± 3.7

†Standard errors. ‡Mica platelet thickness times plate diameter. §Evaluations for stresses in units of MPa, load in units of N, and time in s.

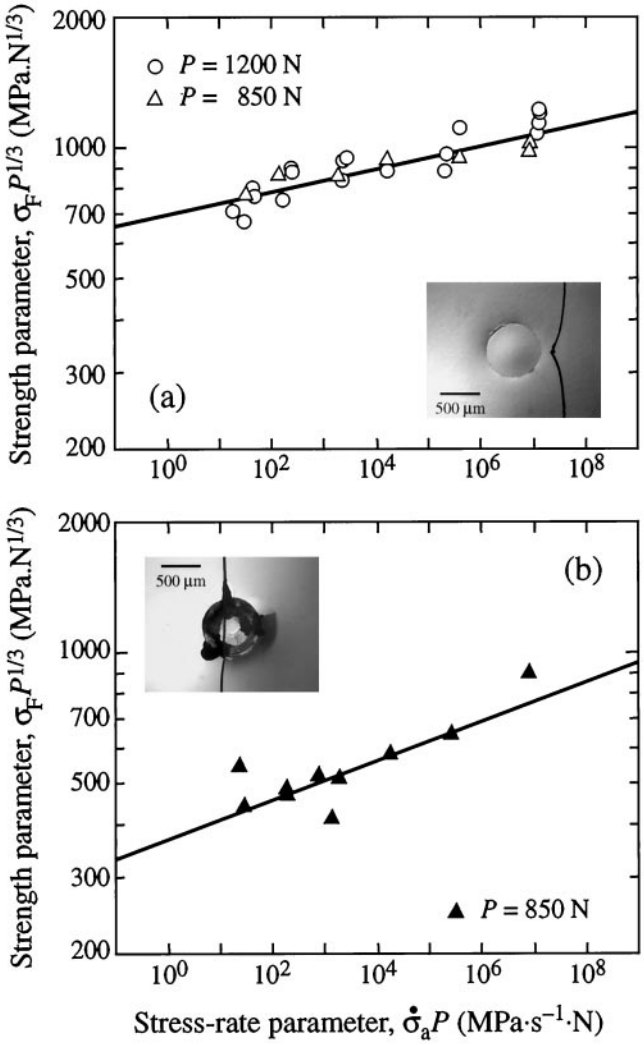

Fig. 1. Dynamic fatigue for F-MGC subjected to prior Hertzian indentation with WC spheres (r = 3.18 mm) in (a) single-cycle contact (n = 1) and (b) multicycle contact (n = 10^4), at loads P = 850 and 1200 N. All tests conducted in water. Open symbols represent failures from cone cracks, solid symbols represent failures from quasi-plastic zones. Micrographs shown in insets show failure sites at P = 850 N and stressing rate $\dot{\sigma}_a$ = 20 MPa/s.

multicycle Hertzian contact, for tests in water. Starting with a crack velocity relation $v \propto K^N$, with v the velocity, K the stress intensity factor, and N the crack velocity exponent, together with a stress-intensity relation $K \propto P/c^{3/2}$ for well-defined indentation cracks of characteristic dimension c at load P, one can derive a generalized dynamic fatigue relation:[7,19]

$$\sigma_F P^{1/3} = \lambda'_P(\dot{\sigma}_a P)^{1/(N'+1)} \quad (1)$$

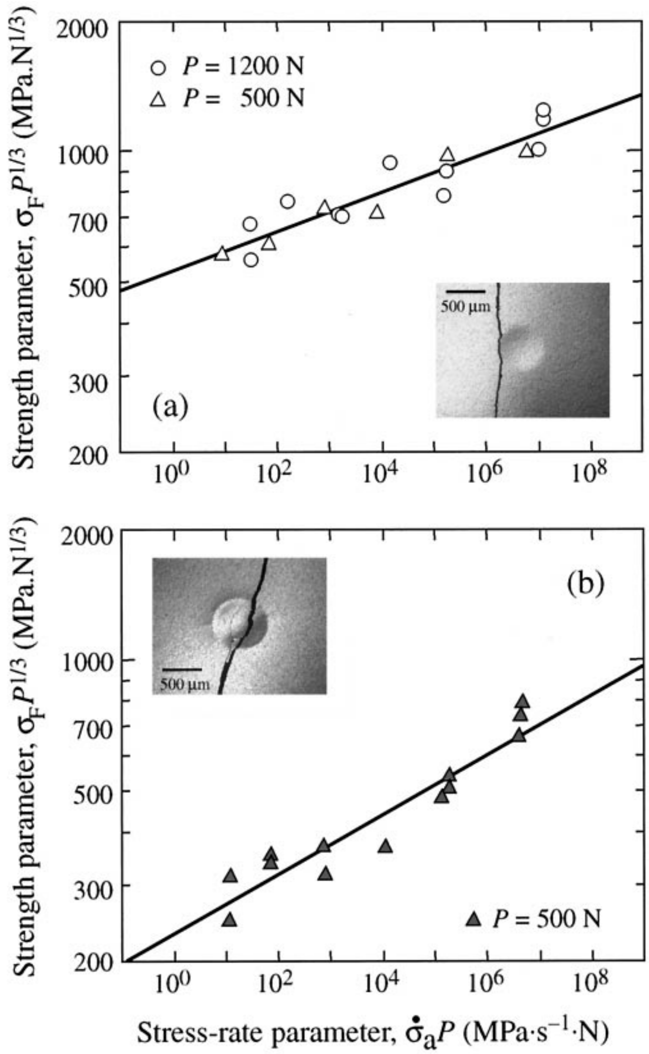

Fig. 2. Dynamic fatigue for C-MGC subjected to prior Hertzian indentation with WC spheres (r = 3.18 mm) in (a) single-cycle contact (n = 1) and (b) multicycle contact (n = 10^4), at loads P = 500 and 1200 N. All tests conducted in water. Open symbols represent failures from cone cracks, solid symbols represent failures from quasi-plastic zones. Micrographs shown in insets show failure sites at P = 500 N and stressing rate $\dot{\sigma}_a$ = 20 MPa/s.

N' and λ' are load-independent slope and intercept parameters: for flaws *without* residual contact stress, $N' \approx N$; for flaws *with* residual contact stress, $N' < N$.[5,20] Data at different P values may be reduced to universal plots by plotting $\sigma_F P^{1/3}$ vs $\dot{\sigma}_a P$. For both materials in Figs. 1 and 2, the data for each given loading condition (single-cycle or multicycle) fall on a universal curve, within the experimental scatter. The solid lines are best fits to the data sets, with appropriate N' and λ' values listed in Table I. Generally, the fits for the multicycle contact data fall below the single-cycle data, with steeper slopes.

Inset micrographs in Figs. 1 and 2 indicate the mode of failure in each case. In F-MGC (Fig. 1), failures originate from cone cracks in single-cycle loading but from quasi-plastic zones in multicycle loading; note the change from a cusplike fracture trace *outside* the contact site (Fig. 1(a)) to a trace *through* the contact site (Fig. 1(b)).[15] Radial cracks are evident in the latter case. In the C-MGC material (Fig. 2), failures occur exclusively from quasi-plastic zones; in this case the fracture trace passes through the contact site for both single-cycle loading (Fig. 2(a)) and multicycle loading (Fig. 2(b)), closer to the periphery in the former and more central in the latter. Radial cracks are not clearly apparent in Fig. 2, but were shown to be present by serial-sectioning the specimen from below and viewing in transmitted light.[12,14]

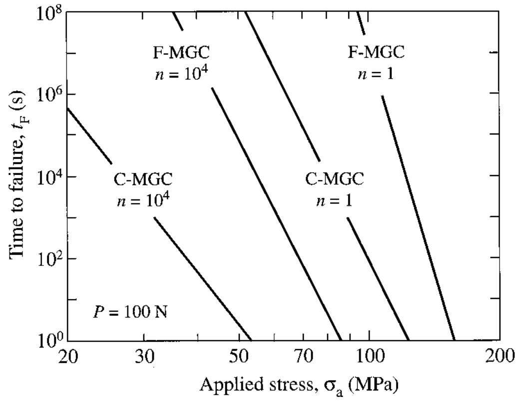

Fig. 3. Lifetime plots for F-MGC and C-MGC, for single-cycle and multicycle contacts at $P = 100$ N. Predictions from Eq. (2).

IV. Discussion

Dynamic fatigue tests on the F-MGC and C-MGC materials demonstrate the importance of microstructure on the lifetime properties for materials subjected to prior damage from contacts with spherical indenters. Specifically, we have shown that C-MGC is considerably more susceptible to dynamic fatigue—i.e., lower N' (Table I)—than F-MGC.

This increased susceptibility of C-MGC to fatigue is attributable to a progressive change in contact damage mode. A transition from simple cone fracture (brittle mode) to distributed microdamage (quasi-plastic mode) with increasing microstructural heterogeneity[16] and greater number of contact cycles is well documented in glass-ceramics,[11,16] as well as in other ceramics.[17] For single-cycle loading, such a transition is apparent in the micrographs: in F-MGC (Fig. 1(a)), flexural failure occurs exclusively from cone cracks, in C-MGC (Fig. 2(a)), from quasi-plastic zones. Cone cracks are characteristically free of residual contact stresses, so that the exponent N' at $n = 1$ for the F-MGC material in Table I may be considered to be a measure of the crack velocity exponent N.[5,21] In the C-MGC, the contact loads are not greatly in excess of the quasi-plastic damage threshold, and failure originates from individual microcracks at the periphery of the contact zone.[12,17] These microcracks are formed by shear stresses within the damage zone ("shear faults")[22–25] and are characterized by local residual driving stresses from unreversed sliding at the closed crack interfaces.[17] Residual stresses around critical flaws have been shown to enhance slow crack growth,[5,6,21,26] commensurate with the increased slope (reduced N') in C-MGC relative to F-MGC in Figs. 1(a) and 2(a).

For multicycle loading (Figs. 1(b) and 2(b)), the degree of quasi plasticity increases dramatically, even in the most-brittle ceramics,[15] indicative of a cyclically induced brittle–plastic transition.[13,27] At lower stressing rates, the quasi plasticity can generate radial cracks, from microcrack coalescence[12] (recall the inset micrographs in Figs. 1(b) and 2(b), with failures through the contact damage zones). Such radial cracks are much more dangerous than the individual microcracks from which they form and so degrade the strength more rapidly. This would explain the lower N' values at $n = 10^4$ in multicycle loading in Table I.

The results in Figs. 1 and 2 can be used to construct lifetime design diagrams for components subjected to a sustained applied stress σ_a. Starting with the same premises as those used in the derivation of Eq. (1), an equivalent relation for the time to failure t_F at fixed σ_a from indentation cracks formed at contact load P can be obtained:[20,21,26]

$$\frac{t_F}{P^{2/3}} = \frac{\lambda'_P}{(N' + 1)(\sigma_a P^{1/3})^{N'}} \tag{2}$$

An illustrative plot of predictions from Eq. (2), using the values of N' and λ'_P listed in Table I, is given in Fig. 3 for indentations made at $P = 100$ N (a level considered "typical" of contact forces in dental function[11,14]) for each MGC type and for $n = 1$ and 10^4. It is immediately evident that an increase in the grain size, as well as the number of contacts, can seriously degrade prospective lifetimes.

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