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Strength Degradation from Contact Fatigue in Self-toughened Glass-ceramics

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We investigated strength degradations from cyclic contact fatigue in self-toughened glass-ceramics. Hertzian indentation was used to induce cyclic contact load. Dynamic fatigue was also performed with changing stress rates from 0.01 to 10000 MPa/sec. After that, strength data and fracture origins were analysed. As the number of contact cycles increased or stressing rate decreased, severe strength degradation occurred by as much as 50% because of radial cracks developed from microcrack coalescence.

Key words: Cyclic contact fatigue, Dynamic fatigue, Hertzian indentation, Strength degradation, Microcrack coalescence, Radial crack

I. Introduction

Conventional researches on the mechanical properties of ceramic materials are generally based on the improvement of fracture toughness because most brittle ceramics show critically lower fracture toughness under tensile stresses.1) For several decades many researchers have succeeded in increasing fracture toughness by *in situ* microstructure control.1-7) Microstructures that include abnormally elongated grains of high aspect ratio are controlled by starting powders or sintering conditions. Crack resistant (R-curve) behavior in these *in situ* toughened ceramics arises from frictional tractions associated with the pullout of elongated grains by crack bridging.8) Therefore, it has been expected that the tailoring of microstructure contributes to increase reliability and lifetime of ceramics.

However, *in situ* growth of elongated grains may deteriorate other mechanical properties such as fracture strength or fatigue characteristics.9) Even though it is possible to enhance both strength and toughness properties at the same time in silicon nitride (Si_3N_4) or silicon carbide (SiC) material by α/β phase transformation, improvement in all mechanical properties including hardness, wear resistance, and fatigue resistance from *in situ* tailoring of microstructures is not universal.10) Moreover, improvement of fracture toughness by grain growth, after the α/β transformation is completed, causes deterioration of strength or wear resistance. Therefore, it is still controversial that "high toughness" guarantees improved performance of the material. In engineering applications where contact stresses are important, evaluation of strength after contact fatigue is a crucial parameter in the evaluation of material performance. For example, in the case of dental restorations, repetitive cyclic contact stresses result in concentrated damage accumulation, leading to deleterious reductions in material strength.11,12) Therefore, in this study, we report how the degradation of strength from mechanical fatigue critically affects the lifetime of *in situ* toughened materials by the evaluation of strength degradation from contact fatigue in self-toughened glass-ceramics.

Most ceramic products experience fatigue stresses, where cyclic or non-cyclic stresses are susceptible. Understanding the material behavior under fatigue conditions is indispensible for the prevention of fracture by fatigue. It is usual to classify the fatigue into static fatigue, dynamic fatigue and cyclic fatigue based on the loading type. Over several decades fatigue studies have not been studied because of the catastrophic failure of brittle ceramics. However, since *R*-curve behavior became an issue, the importance of the research of fatigue has increased because the crack of *in situ* toughened ceramics propagates stably.13-17) More recently, contact fatigue studies using spherical indenters in repeat loading have been extensively studied.18-23)

In this paper, we describe how the strength degrades in self-toughened glass-ceramics from cyclic contact fatigue or dynamic contact fatigue after inducing cyclic contact damage deliberately with spheres.24,25)

II. Experimental Procedure

1. Test material

The material used in this study was a commercial mica-containing glass-ceramics (Macor, Corning Inc., Corning,

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NY), which consists of 55% mica and 45% borosilicate glass. Macor composition in weight % is 46% SiO_2, 14% MgO, 16% Al_2O_3, 10% K_2O, 8% B_2O_3, and 6% F.

The sintered microstructure consists of interconnected network structure of randomly oriented interlocking mica flakes in a borosilicate glass matrix. The individual mica flakes are approximately 10 μm of basal plane and 1~2 μm of thickness.

2. Cyclic contact fatigue test

Bar specimens 3 mm×4 mm×50 mm were cut and machined from Macor block, 100 mm diameter×70 mm thickness. Then the surfaces were polished to 1 μm diamond finish for fatigue and strength testing. Cyclic fatigue was performed to induce contact damage on the center of the surface by indentation with tungsten carbide (WC) spheres of radius r=3.18 mm, for number of cycles n up to 10^6 at frequency f=10 Hz and loads up to P=200 N. The cyclic wave was applied periodically on the surface in a havesinusoidal wave form. Most of the experiments were conducted in a water environment and some experiments were conducted in air (relative humidity ~50%). All specimens were examined in an optical microscope (Nomarski illumination) after gold (Au) coating. The sectional view of the damage zone was obtained using the bonded specimen technique.[24]

Strength tests were conducted on the contact-damaged bars in four-point flexure (outer and inner spans are 20 and 10 mm, respectively), with the contact damage site centered on the tensile side. The bars were broken in air and water environments in rapid loading, 500 mm/min (t<20 msec). Unindented specimens were also broken under the same conditions for comparison.

3. Dynamic fatigue test

Dynamic fatigue tests were conducted by carrying out strength tests on the contact damaged bars in the same four-point flexure, at different stressing rates. 10^{-2} to 10^4 $MPas^{-1}$ by control of cross head speed, from 0.0017 mm/min to 500 mm/min. All broken specimens were also examined by optical microscope (Nomarski illumination) to confirm the failure origin.

III. Results and Discussion

1. Cyclic contact fatigue and strength degradation

The microstructure of Macor glass-ceramics used in this study is shown in Fig. 1.[26] The microstructure has a high density >99% without any pores and a distributed grain size of 10 μm in the basal plane and 1~2 μm thickness. Individual microcracks develop at interfaces, and ultimately coalesce. The microcracking occurs along the mica-glass weak interface rather than along the cleavage plane of mica, suggesting that the mica-glass interfaces are weaker than the mica cleavage plane. The hardness and fracture toughness measured by Vickers indentation are H=2.5 GPa and T_0=1.9 $MPam^{1/2}$, respectively. The inherent strength measured at rapid speed (< 20 msec) in air is about 170 MPa.

However, the initial strength of the Macor glass-ceramics is degradaded as the number of cycles increase in the cyclic loading from 0 to maximum load P=200 N, as shown in Fig. 2. The strength losses begin to accelerate after $n=10^4$, especially in a water environment. It is thought that the presence of water is to enhance the strength degradation during the contact fatigue because of pronounced crack propagation.[1] The strength falloff after the number of cycles, $n=10^5$, at the contact load, P=200 N, is critical to the lifetime of the material even if the glass-ceramics is self-toughened. The results are noteworthy because recent studies[26,27] have focussed on the damage tolerance of the quasi-plastic ceramics such as self-toughened glass-ceramics. The present study shows that the rapid strength falloff at multiple numbers of cycles is critically detrimental to the lifetime of tough and heterogeneous ceramics even though the damage tolerance of the quasi-plastic ceramics is better than that of fine-

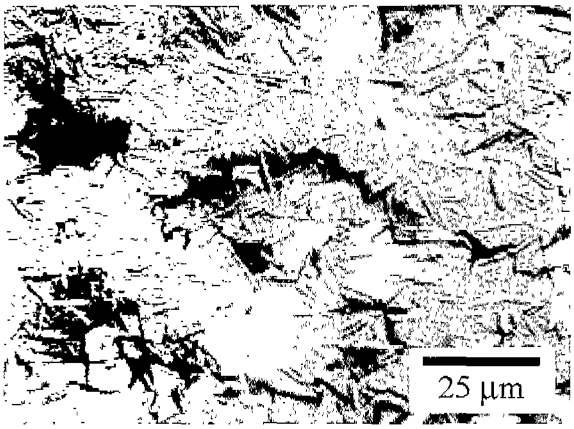

Fig. 1. Optical micrograph showing microstructure and microcracks in Macor glass-ceramics. Viewed in Nomarski illumination.

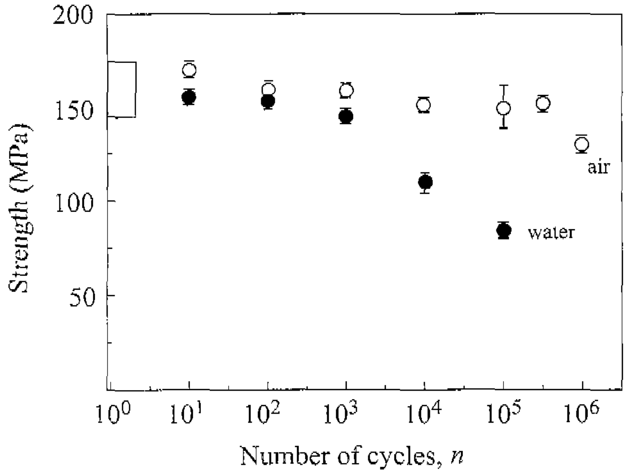

Fig. 2. Inert strength as a function of number of contact cycles n, indentation with WC sphere of radius r=3.18 mm, at maximum load P=200 N. Unfilled symbols indicate test results in air, filled symbols in water. Box at left axis are "laboratory" strength(unindented specimen).

grained brittle and homogeneous ceramics at smaller number of cycles.

The strength degradation is closely related to the contact damage formed under the cyclic loading. Fig. 3 shows the surface damage morphology from the cyclic contact fatigue in sequences of increasing n. Fig. 3 compares the contact damage for (a) number of cycles, $n=10^4$ and (b) number of cycles, $n=10^5$ at the maximum load, P=200 N in water. The surface impression from the cyclic contact fatigue consists of an array of closed shear faluts with microcracks at their ends as shown in Fig. 1. The severity of damage is clearly enhanced by the increase in number of cycles. Note the appearance of the radial cracking (arrow is indicated in the figure). The comparison of (b) with (a) shows further propagation of radial cracking from the contact periphery as the number of cycles increases.

The radial cracks propagate in two ways as the number of cycles increases. First, the radial cracks propagate stably in a radial direction as shown in Fig. 4(a). This crack propagation mode is more easily observed during cyclic contact damage in an air rather than a water environment. The second mode is the development of surface fretting along with the propagation of the radial cracks. The material is pushed-out as the radial crack propagates. This propagation mode is easily observed in a water environment. Fig. 4(b) indicates that the radial cracks propagate from the subsurface into

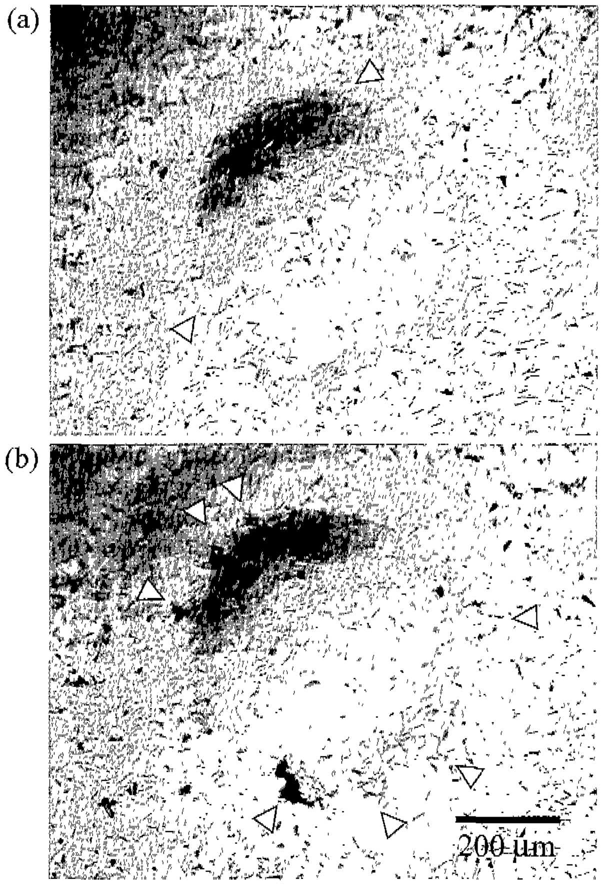

Fig. 3. Optical micrographs of Hertzian indentation sites, comparing damage after contact fatigue test at load P=200 N and number of contact cycles, n, (a) 10^4 cycles and (b) 10^5 cycles. All tests in water environment. Note the appearance of radial cracks at contact periphery after cycling.

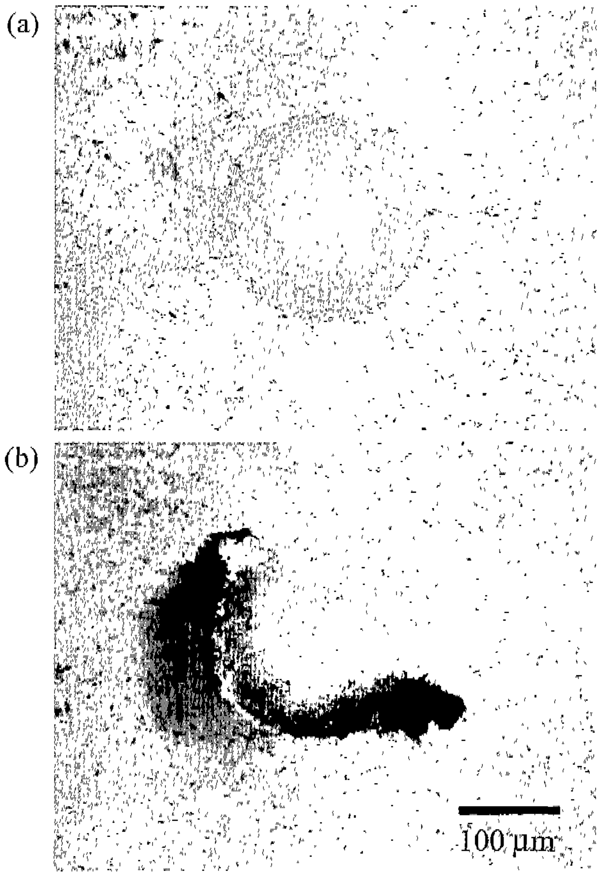

Fig. 4. Optical micrographs of Hertzian indentation sites comparing damage after contact fatigue in condition of (a) P =750 N, $n=10^4$, in air and (b) P=250 N, n=10^3, in water.

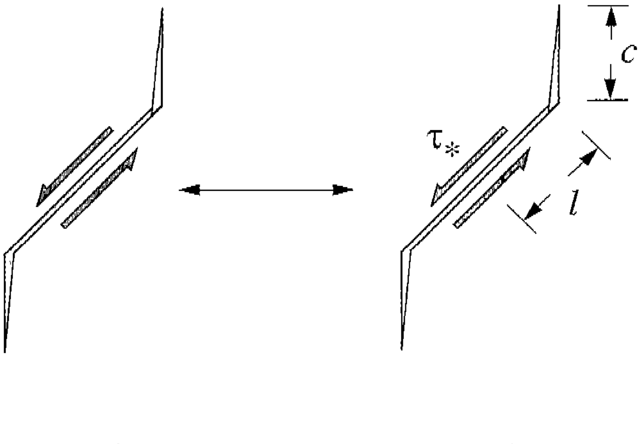

Fig. 5. Schematic diagram showing microcrack coalescence Critical shear stress, τ^*, on shear fault with half-length, l, produces wing cracks with length of c. Coalescence more occurs as the distance d decreases or c increases.

the surface, which causes the surface fretting.

In brittle ceramics such as amorphous glass the damage takes the form of classical macroscopic cone cracks outside the contact region. In tougher ceramics such as glass-ceramics, however, the damage takes the form of a quasi-plastic damage zone, which consists of microcracks formed by shear stress.[24] Although the strength is not critically degradable before the microcracks coalescence, we believe that the rapid strength degradation occurs when the radial cracks propagate from the microcrack coalescence.[21,23] Recently, a model of contact damage accumulation from cyclic loading with spheres in tough ceramics was esbablished.[21] This study proposed that the strength degradation is strongly related to microcrack coalescence. In heterogeneous ceramics, elongated grains tend to show enhanced long-crack toughness, mostly by grain bridging. However, the contact deformation along the weak grain boundary is associated with the activation of discrete "shear faults," from which microcracks initiate in the subsurface region at stresses above the critical shear stress, τ^*. Macroscopically, the damage occurs in a region of hydrostatic compression and shear stress, which contains multiple microcracks. A schematic diagram of microcrack coalesence between two shear faults with spacing d is shown in Fig. 5. In cyclic contact fatigue, the wing microcracks with length c from the shear fault with a characteristic dimension l coalesce progressively by shear stress at the sliding fault. The microcrack coalescence is developed into a radial crack and ultimate material removal, which causes severe strength degradation. Fig. 6 shows intensified damage of the glass-ceramics from a higher contact load, P=700 N. In this case we show the half-surface (Fig. 6(a)) as well as the side view (Fig. 6(b)). Comprehensive damage has occurred from microcrack coalescence, radial crack development, and material removal in both the half-surface and the side surface.

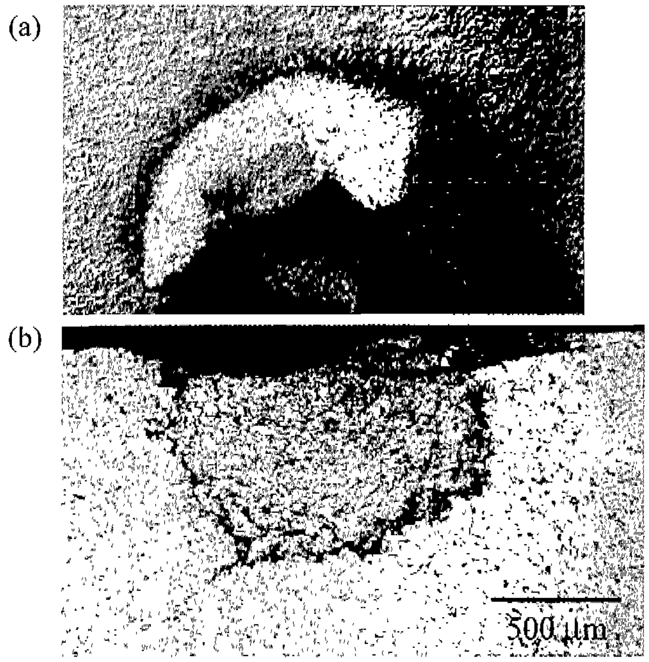

Fig. 6. Contact fracture in Macor glass-ceramics, after contact fatigue at P=700 N and n=10^4, in air. Each photograph indicates damages of (a) half-surface and (b) side view. Note microcrack coalescence and developments into radial cracks lead to material loss and failure.

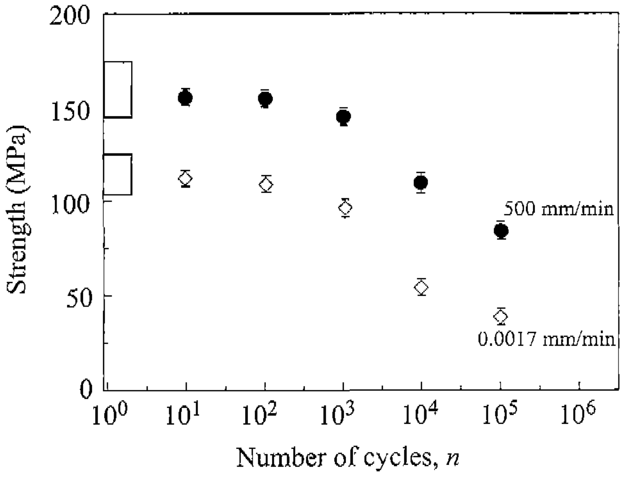

Fig. 7. Inert strength as a function of number of contact cycles, n, indentation with WC sphere of radius r=3.18 mm, at maximum load P=200 N. Circle symbols indicate test results in fast fracture (500 mm/min) and diamond symbols in slow fracture (0.0017 mm/min).

2. Dynamic fatigue and strength degradation

Fig. 7 plots strength data as a function of number of cycles after cyclic contact fatigue on Macor ceramics, each at a fixed peak load P=200 N, for different crosshead speeds (500 mm/min and 0.0017 mm/min) in water. The tendency towards lower strengths at slower crosshead speeds is in good agreement with prior results, which showed that slow growth of critical flaws leads to strength degradation.[28,29]

Plots of flexural strength versus stressing rate are shown in Fig. 8 for Macor glass-ceramics after cyclic contact fatigue at fixed n=10^4 and variable load, P=100, 200, 300, or 500 N. Firstly, glass-ceramics after contact fatigue at higher loads showed lower strengths because the flaw size produced by contact stress depends on the load. Secondly, lower stressing rates (slower crosshead speed) during strength testing resulted in lower strengths due to slower crack growth. The results of dynamic fatigue testing indicate that the ceramic is more susceptible to distributed micro-damage at longer application time due to tension stresses subjected to prior-indented damage. The best fit[22] of the graph results in load-independent slope parameter, N'=20.25. Generally, this fitting result shows steeper slopes than fine-grained materials, which indicates deleterious effect of self-toughened glass-ceramics.[22] The graph includes failure origins in bend specimens of Macor, for dynamic fatigue. Open symbols indicate that most of the specimens break away from indentation sites or from contact periphery, black symbols failure from centralized fracture paths through the indented damages. It is noteworthy that the data at the conditions of 200

N, 10 MPa/sec and 300 N, 100 MPa/sec deviate from the linear line. The lower strengths at these two data points are closely related to the change of failure origin from away locations to centralized paths. Therefore, we believe that the lower strengths are due to the development of radial cracks into the surface from the microcrack coalescence. It is certain that the radial crack, developed from growth and coalescence of microcracks in the microcrack damage zone formed from cyclic contact fatigue, ultimately exhibits lower strength during dynamic fatigue.

Fig. 9 shows failure origins on broken strength-test specimens containing indentations from cyclic contact fatigue at indentation load, (a) 100 N and (b) 200 N with number of cycles n=10^4, for decreasing crosshead speed in dynamic fatigue. Whereas the failure origin is changed from inside to outside of contact damage formed at prior-indented load, P =100 N, the failure origin moved from outside to a centralized path at load, P=200 N. In case of P=100 N and 500 mm/min, it is conjectured that the failure is from one of the critical mirocracks rather than from a radial crack because of the smaller size of the damage zone. We have confirmed that fracture does not always occurr from the central zone and that failure occurs away from the indentation sites at slower stressing rates. On the other hand, the fracture through the central zone under a condition of P=200 N and 0.0017 mm/min speed is caused by a radial crack from microcrack coalescence because of the change of failure origins. Fig. 10 confirms that centralized fracture paths through the indentation sites are related to radial cracks. We stopped the loading just before fracture occurred on the same specimen after we had checked the maximum load to failure. Surface micrographs are shown in Fig. 10. Fig. 10(a) shows the optical micrographs for contact damage area before and after the applied load, 182 N with a crosshead speed of 2 mm/min during dynamic fatigue. Fig. 10(b) corresponds to the case of slower crosshead speed, 0.0017 mm/min at the same applied load. We have confirmed that the radial crack is initiated from the indentation sites for a 0.0017 mm/min speed while nothing is changed for a 2 mm/min speed. Therefore, we verify that the failure fracture that broke through the indentation sites was from radial crack on the surface developed from microcracks coalescence in the subsurface damage zones.

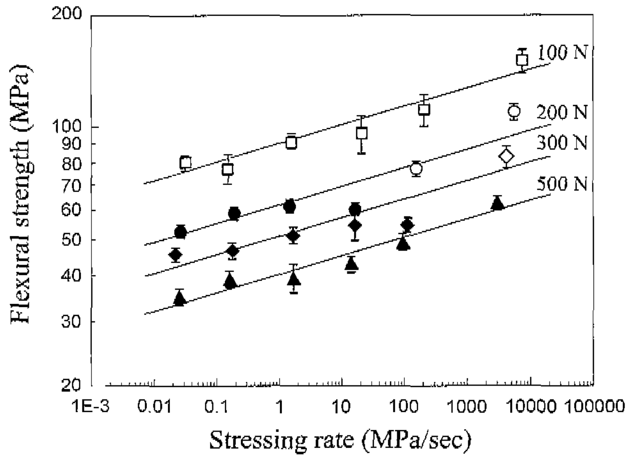

Fig. 8. Dynamic fatigue for Macor glass-ceramics subjected to prior Hertzian indentation with WC spheres at different indentation loads, P=100, 200, 300 and 500 N with n=10^4 contact cycles. All tests conducted in water. Open symbols represent failures from outside of contact sites, solid symbols represent failures from center in contact sites.

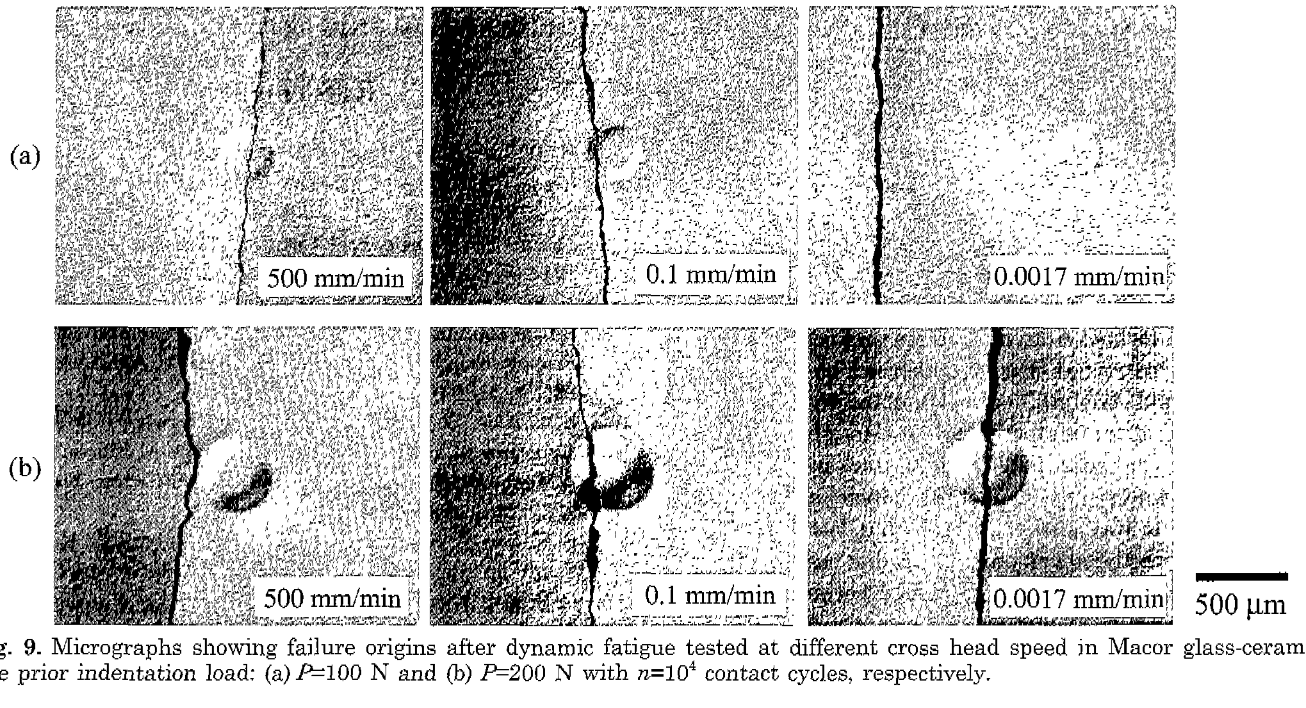

Fig. 9. Micrographs showing failure origins after dynamic fatigue tested at different cross head speed in Macor glass-ceramics The prior indentation load: (a) P=100 N and (b) P=200 N with n=10^4 contact cycles, respectively.

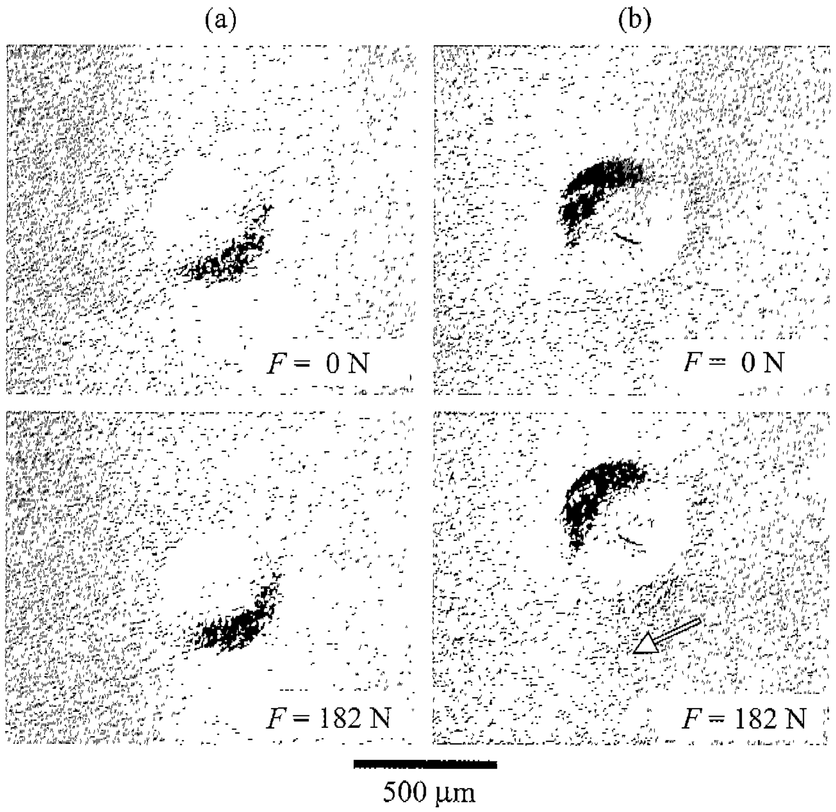

Fig. 10. Micrographs of contact sites after stopped at each load, F, during dynamic fatigue test. Tests conducted at different cross head speed: (a) 2 mm/min and (b) 0.0017 mm/min. All tests in water. Note developed radial crack during slow fracture. The prior indentation conditions are P=200 N and n=10^4.

The implication is that quasi-plastic damage accumulation is very deleterious on the lifetime of self-toughened ceramics if microcrack coalescence occurs by multiple cyclic fatigue and slow crack growth during dynamic fatigue. The appearance of radial cracking decreases the strength of self-toughened materials.

IV. Conclusion

We investigated strength degradations from cyclic contact fatigue and dynamic fatigue in self-toughened glass-ceramics. The strength degradation was substantially rapid at critical cycles according to the increase in number of cycles, and was accelerated further by the development of radial crack formed from interaction and coalescence of microcracks. The radial cracks, which act as failure origins, led to rapid strength degradation and the end of the useful lifetime of the material. The self-toughened glass-ceramics were very susceptible to slow crack growth in the presence of water environments, which also caused strength loss. It was also confirmed that lower strength was related with the development of radial cracks. Therefore, we concluded that self-toughened ceramics with a coarse grain and weak interface structure are substantially more susceptible to fatigue, especially after cyclic fatigue and dynamic fatigue.

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