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Grain-Size Dependence of Sliding Wear in Tetragonal Zirconia Polycrystals

Yingjie He, Louis Winnubst,* Anthonie J. Burggraaf, and Henk Verweij

Faculty of Chemical Technology, Laboratory for Inorganic Materials Science, University of Twente, 7500 AE Enschede, The Netherlands

Paul G. Th. van der Varst and Bert de With'

Faculty of Chemical Technology, Laboratory of Solid State Chemistry and Materials Science, Eindhoven University of Technology, 5600 MB Eindhoven, The Netherlands

Using a pin-on-plate tribometer with the reciprocating motion of SiC against yttria-doped tetragonal zirconia polycrystal (Y-TZP) plates, the friction and wear of Y-TZP ceramics were investigated as a function of grain size in dry N_2 at room temperature. The results showed that the overall wear resistance increased as the grain size of Y-TZP ceramics decreased. For grain sizes ≤0.7 µm, the wear results revealed a Hall-Petch type of relationship $(d^{-1/2})$ between wear resistance and grain size. In this case, the main wear mechanisms were plastic deformation and microcracking. For grain sizes ≥0.9 µm, the wear resistance was proportional to the reciprocal of the grain diameter. In this regime, delamination and accompanying grain pullout were the main mechanisms. In this case, the phase transformation to monoclinic zirconia had a negative effect on the wear resistance of TZP ceramics. The coefficient of friction tended to be higher for fine-grained TZP-SiC couples than for coarse-grained TZP-SiC couples, whereas, for a specific regime of grain size, the coefficient of friction was almost independent of the grain size.

I. Introduction

THE wear resistance of ceramics is affected by microstructural parameters, such as grain size, porosity, and grain-boundary behavior. The results obtained by Marshall et al. show that the high-purity and coarse-grained alumina ceramics reveal a low grinding resistance. As a first-order approximation, a relation between wear resistance and the reciprocal square root of grain size has been found in abrasive-wear tests with oxide ceramics. A Recently, Cho et al. used fracture mechanics to demonstrate the existence of a Hall-Petch-type relation between wear-damage stress and grain size of polycrystalline alumina ceramics. Furthermore, Liu and Fine extended the fracture-mechanics analysis in the Cho work to quantify the dependence of sliding wear on grain size for polycrystalline alumina, in the case of grain-boundary microfracture.

At present, considerable attention is given to properties and possible applications of nanocrystalline ceramics. 7.8 Ultrafine-grained yttria-doped tetragonal zirconia polycrystal (Y-TZP) ceramics show improved high-temperature mechanical properties and low-temperature aging resistance in water, especially

R. O. Scattergood—contributing editor

at 150°-250°C.10 Recent studies10,11 indicate that a high toughness (8-9 MPa·m1/2) can be obtained in these ceramics without the occurrence of an irreversible martensitic transformation. High wear resistance of nanocrystalline materials may be expected, especially if little or no porosity is present. 4,12 To optimize TZP ceramics for high-wear-resistant application, identification of the friction and wear behavior of TZP ceramics, as a function of grain size, is necessary. For zirconia ceramics, the amount of transformed TZP, as influenced by the grain size, can affect mechanical properties, wear resistance, and wear mechanisms. Therefore, the present investigation also is concerned with the influence of the phase transformation of TZP on the wear rate. The wear tests on Y-TZP ceramics have been conducted on materials made with two types of powders: a nanocrystalline powder prepared by a chloride precipitation method and a commercially available powder (TZ-3Y, Tosoh, Tokyo, Japan).

II. Experimental Procedures

(1) Wear Tests

Wear experiments were performed with a pin-on-plate tribometer, as shown in Fig. 1. A polished silicon carbide (SiC) sphere (pin) with a diameter of 4 mm mounted in a copper tube slides reciprocally against a stationary Y-TZP plate. Tests were conducted at room temperature in a dry N_2 atmosphere (humidity <1%) under a normal load of 8 N, corresponding to the initial Hertzian contact pressure of 858 MPa. The track length was 10 mm with a frequency of 4 Hz (for the pin), corresponding to a maximum sliding velocity of 12.5×10^{-2} m/s or an average velocity of 8×10^{-2} m/s. The testing time was $\sim\!20$ h, corresponding to a sliding distance of 5.8 km. The friction force

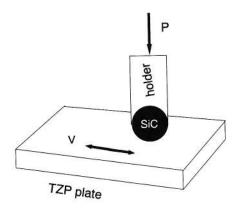


Fig. 1. Schematic diagram of pin-on-plate wear rig. Pin moves reciprocally on a static plate.

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*Author to whom all correspondence should be addressed.

Table I. Friction and Wear Properties for Sliding Wear of a SiC Ball on a TZP Plate

Sample	Grain size (μm)	Friction coefficient, f_e	Roughness, R_a (TZP)* (nm)	Wear rate, K (TZP) (× 10^{-6} mm ³ ·(N·m) ⁻¹)	Fraction monoclinic	
					Polished	Worn surface
	A 100 C TABLE 1	CONT. INTO C. T. S. SAMPRONS	ZY5.7 material	100 - 100 March 100 - 100 - 100 March 100 Marc		300
G1 ZY5.7 [†]	0.18	0.42 ± 0.08	1482	1.08 ± 0.22	0	0
G2 ZY5.7	0.36	0.38 ± 0.07	1787	1.82 ± 0.16	0	0
G3 ZY5.7	0.50	0.36 ± 0.05	2214	2.38 ± 0.40	0	0
G4 ZY5.7	0.70	0.38 ± 0.06	2743	2.63 ± 0.28	<3	5–8
G5 ZY5.7	0.90	0.30 ± 0.06	4602	4.74 ± 0.45	<3	50-60
G6 ZY5.7	1.20	0.33 ± 0.04	5866	6.67 ± 0.08	20	50-60
G7 ZY5.7	1.50	0.31 ± 0.05	7428	8.00 ± 0.70	50	50-60
			TZ-3Y material			
A1-TZ-3Y	0.34	0.50 ± 0.08	2068	1.60 ± 0.30	0	0
A2-TZ-3Y	0.90	0.38 ± 0.08	6493	5.95 ± 0.60	<3	40-45

*Center line average roughness33 measured along the direction perpendicular to sliding. 'This specimen was prepared by sinter forging at 40 MPa.

was determined by a force transducer. Friction forces were simultaneously sampled under external triggering control with a computerized data-acquisition system and were continuously measured during the tests.¹³

After wear testing, the specimens were cleaned with ethanol in an ultrasonic bath and then dried at 120°C for 4 h in a drying stove and subsequently cooled while blowing with dry N_2 . The specific wear rate, K_w (= K_{pin} for the SiC pin and K_{plate} for the Y-TZP plate), was obtained as

$$K_{w} = \frac{W_{m}}{\rho LP} \tag{1}$$

where W_m is the mass loss, ρ the density, L the total wear path length, and P the normal load.

(2) Sample Preparation and Characterization

Commercially available SiC spheres with mirror-polished surfaces (GIMEX Technische Keramiek B.V., Geldermalsen, The Netherlands) were used in all cases as pin materials. The plates were ZrO₂ with 5.7 mol% YO_{1.5} (ZY5.7), prepared by gel-precipitation synthesis, and TZ-3Y (with the same composition as ZY5.7) (Tosoh Europe B.V., Amsterdam, The Netherlands). The gel-precipitation (or "chloride") method is based on precipitation of a 0.2M HCl solution of ZrCl₄ and YCl₃ with ammonia. The average crystallite size was 8 nm for the chloride powder and 34 nm for the TZ-3Y powder. More powder properties are given in Ref. 14. All powders were uniaxially pressed at 50 MPa in a rectangular die and then isostatically pressed at 400 MPa and sintered for 2–5 h at temperatures of 1150°–1570°C in a tube furnace. The average grain size was

measured using the linear-intercept technique¹⁵ on scanning electron microscopy (SEM) pictures. The bulk density of >98% was determined for all sintered samples using the Archimedes technique in mercury. For the specimens of 0.18 μ m grain size with 98% density, sinter forging was used as described by He *et al.*¹⁶ and Boutz *et al.*¹⁷ The specimens for wear tests were finally polished, using 0.05 μ m Al₂O₃ powder, to a final roughness of <80 nm. Finally, all specimens were annealed at 950°C for 10 min (heating and cooling rate of 2°C/min). The Vickers hardness (H_{ν}) was determined with a Vickers indentor (Isser Stedt Garant 250 RD) at a load of 98 N. The hardness values were almost the same (12.8 \pm 0.4 GPa) for the specimens with different grain sizes used in this study.

The surface roughness of the plates and profiles of some wear tracks on the plate were measured with a profilometer (Model Dek-Tak 3030, Sloan, Santa Barabara, CA). Worn tracks were analyzed by SEM (Model JSM-35CF, JEOL, Tokyo, Japan). The phase composition of TZP ceramics before and after sliding wear were measured by X-ray diffractometry (XRD) with $CuK\alpha$ radiation (Model PW 1710, Philips Scientific Instruments, Almelo, The Netherlands). The volume fraction of monoclinic zirconia was determined by XRD using the equation of Toraya *et al.*¹⁸ Adhesive particles on the worn surface and wear debris were analyzed using energy-dispersive X-ray analysis (EDAX).

III. Results

(1) Tribological Properties of TZP Plates

To investigate the influence of grain size on wear behavior of TZP ceramics systematically, all samples have almost the same

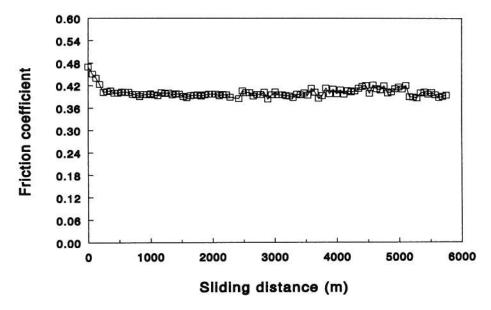


Fig. 2. Coefficient of friction of specimen G1–SiC couple as a function of sliding distance, under a normal load of 8 N and at an average velocity of 0.08 m/s.

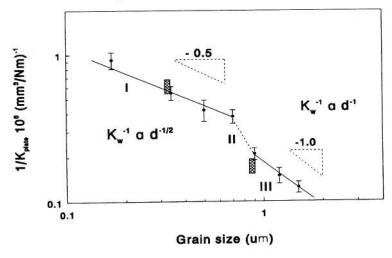


Fig. 3. Wear resistance as a function of average grain size for TZP ceramics with an SiC pin sliding at an average velocity of 0.08 m/s and a normal load of 8 N. Results with an error bar on the plotted curve are for ZY5.7 materials. Crosshatched area gives results for TZ-3Y materials.

density, to eliminate the influence of porosity on wear resistance. In the present study, the relative densities of all samples are 98%–98.5%. The friction and wear properties are summarized in Table I. The friction coefficient, f_c , is an average of at least three measurements obtained after the running-in stage. The friction coefficients as a function of time (distance) indicate that, for all tests, a running-in time of <3.3 h (about 950 m) exists, after which a stationary value is reached. Figure 2 gives an example of the experimental results. For ZY5.7 ceramics, stationary f_c values of 0.36–0.42 are obtained and are independent of grain size in the grain-size region \leq 0.7 μ m. A relatively

low f_c value (\sim 0.3) is observed for the coarse-grained ceramics (>0.9 μ m). The same trend is observed for TZ-3Y ceramics, in which f_c values of 0.50 and 0.38 are found for the materials with grain sizes of 0.34 and 0.90 μ m, respectively.

As shown in Fig. 3, the wear resistance $(K_{\text{plate}}^{-1}, \text{ which is the recipocal of the wear rate, } K_{\text{plate}})$ as a function of grain size can be divided in two parts. One part (region I in Fig. 3) is the grain-size region <0.7 μ m, whereas the other part (region III in Fig. 3) is in the grain-size region >0.9 μ m; region II in Fig. 3 must be regarded as a transition range. Using the power relation $K_{\text{plate}}^{-1} \propto d^{-n}$ (d is the average grain size), as indicated by Zum

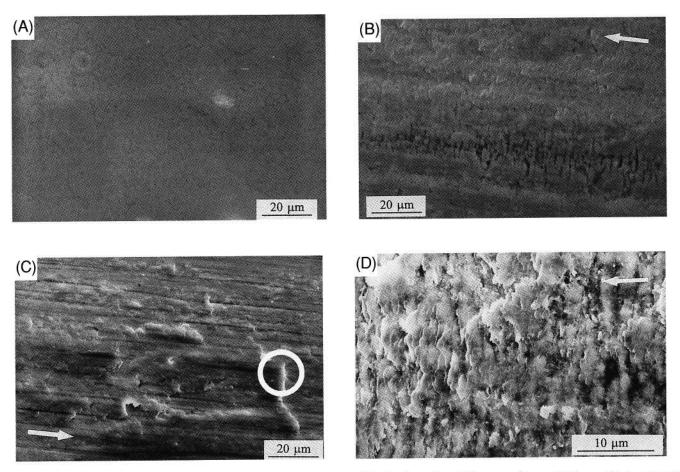


Fig. 4. Morphologies of specimen G1 with a grain size of $0.18 \mu m$. ((A) polished surface, (B and C) worn surface, and (D) magnified version of Fig. 4(B)). Arrow indicates one direction of the reciprocal sliding.

Gahr *et al.*,¹⁹ values of n = 0.5 for region I and n = 1.0 for region III are obtained. These results reveal a Hall–Petch-type dependence for region I. Interestingly, the change of n values from 0.5 to 1.0 correlates to the onset of the phase transformation to monoclinic zirconia. From Table I, the martensitic phase transformation of TZP ceramics can be induced by fracture or sliding-wear processes in region III but not in region I. For specimen G5, 50%–60% of TZP is transformed to the monoclinic phase, which is a larger percentage than that induced by single-edge notched beam (SENB) fracture. Evidence has been given that cycling stresses enhance the phase transformation.²⁰

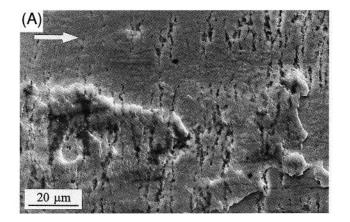
(2) Morphology of Worn Surfaces of TZP Plates

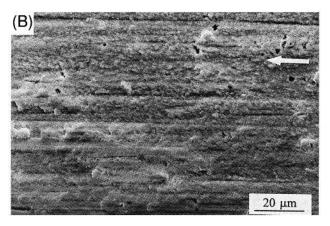
Typical SEM micrographs of the worn surface of ZY5.7 ceramics after sliding 5.8 km are presented in Figs. 4-7. The micrographs of the polished surfaces before sliding are similar for all specimens. As an illustration, a micrograph of a polished sample is shown in Fig. 4(A). Figures 4(B)-(D) show the wear tracks of ZY5.7 ceramics with a grain size of 0.18 µm. The morphologies observed indicate that plastic deformation, microcracks, microcutting or microploughing, and adhesion of wear debris occur during dry sliding. In this case, plastic deformation and microcracking are the major wear mechanisms. The plastic deformation of the materials is attributed to the large shear stresses acting on the loaded zone. Some of the plastic deformation may result from adhesion and smearing of small wear debris particles.21 Fatigue processes due to repeated abrading on the top surface and the corresponding plastic deformation of the surface may result in microcracking. Microcracking patterns are found perpendicular to the sliding direction, as shown in Fig. 4(B). These microcracks are more often present at the turning point of the wear track than in the middle. In Fig. 4(C), typical plastic deformation patterns, one of which is marked with the white circle, are shown. Figure 4(D) indicates

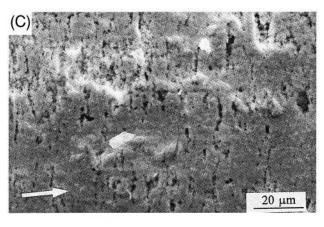
some plastic deformation, accompanied by cracklike patterns. Wear debris adheres easily to the microcracked parts. Similar wear mechanisms occur in specimens G3 and G4 but with a larger contribution of microcracking and less plastic deformation (Fig. 5). Overall observations of the morphologies of specimens G1–G4 indicate that a change in the grain size of the TZP plate in this regime does not result in a drastic change in wear morphology or in wear mechanism.

However, the specimens with grain sizes of 0.9–1.5 μm, specimens G5 and G7, give a difference in wear morphology (Figs. 6 and 7), if compared to the finer-grained ceramics. The wear tracks show a pronounced surface roughness. Figures 6(A) and (B) show a detachment of the deformed surface layer for specimen G5, and Fig. 7(A) shows a similar detachment for specimen G7. A large and deep layer has been removed from the plastically deformed surfaces by a delamination process, as shown in Figs. 6(C) and 7(B). Figure 7(C) is a high-magnification view of a portion of the wear pattern that is generated on the subsurface after delamination. Fracture at the subsurface is observed to be mainly of the intergranular type, with very limited transgranular fracture. Pits due to grain or particle pullout are found.

A more detailed examination of the wear tracks reveals an absence of microcracks at the contact surface in Figs. 6 and 7. This reflects the influence of the phase transformation from tetragonal to monoclinic resulting in a volume increase. For specimens G1–G4, where no transformation occurs, significant evidence of microcracking at the contact surface can easily be found. The delamination wear for region III is caused by nucleation and propagation of microcracks at a subsurface level. Specimens G5 and G7 show the same fracture behavior; only the degree of wear increases as the grain size increases. The morphology of specimens G5 and G7 also shows more wear particles adhering to the contact surface, forming elevated, smooth regions (see Fig. 6(D)). For TZ-3Y ceramics, inspection







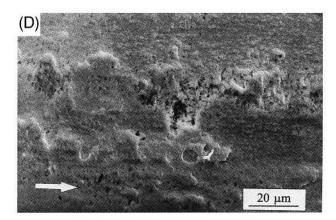


Fig. 5. Morphologies of specimens (A and B) G3 and (C and D) G4 with grain sizes of 0.50 and 0.70 μm, respectively; (A) and (C) are taken from the turning point area; (B) and (D) are from the middle of the wear track. Arrow indicates one direction of the reciprocal sliding.

of wear-track morphologies indicates that specimen Al behaves similar to the fine-grained ZY5.7 materials, whereas specimen A2 reveals wear tracks similar to those of coarse-grained specimens G5 and G7.

The roughness (R_a) values, perpendicular to the sliding direction, of worn TZP surfaces after 5.8 km of sliding tests are given in Table I. The average R_a value strongly depends on and increases with grain size. The R_a data are related to particle removal at the contact surfaces. According to Van den Berg and de With, ²² the R_a values give an indication of the geometry of both contacting surfaces. In turn, a larger R_a value results in a decrease in the number of contact points and, hence, at certain loads, causes an increase in local contact pressure. The increased contact pressure leads to an increased shear stress, which may, in turn, affect the wear process.

IV. Discussion

(1) Wear Resistance and Hall-Petch-Type Relation at Grain Sizes ≤0.7 μm

The experimental results shown an unambiguous relation between grain size of Y-TZP ceramic and wear resistance under unlubricated sliding conditions. In region I with grain size $\leq 0.7~\mu m$, the dependence of the wear resistance $(K_{\rm plate}^{-1})$ of TZP ceramics on grain size can be described as $K_{\rm plate}^{-1} \propto d^{-1/2}$ (Fig. 3). This result is similar to that of Rice and co-workers, 3.4 who found a Hall–Petch-type relation between the wear resistance and the average grain size, d, in MgO and Al_2O_3 ceramics during abrasive wear. This also was reported by Zum Gahr et al., 19 for TZP ceramics in unidirectional sliding, who suggested that this should be expected if plastic deformation or brittle fracture is considered as the dominant process. The Hall–Petch relation was first introduced by Hall²³ and Petch²⁴ in

polycrystalline metals undergoing plastic deformation controlled by dislocation pileup. The yield stress, σ_y , was proportional to the inverse square root of d. Fracture is predicted to occur as a result of the nucleation of microcracks by the interaction of slip planes or dislocation pileups at grain boundaries. Evidence for plastic deformation in ceramic materials also has been given by other investigators. 21,25,26

In the present study, plastic deformation and accompanying microcracking of TZP are clearly observed from detailed SEM micrographs of the worn surfaces (see Figs. 4 and 5). Here, the surface-removal process is controlled by microfracture, which is caused, in turn, by plastic deformation and/or flaws. Fracture mechanics cited by Cho et al.5 are adopted to provide a theoretical understanding of this process. They supposed that extended microcracking can be generated in ceramics from grain-boundary flaws. These flaws are either present or generated by stress concentration at deforming elements. Using the models of Cho et al.5 and Liu et al.,6 local microfracture may occur when the damage-induced stress, σ_D , exceeds the strength of the material. This stress, σ_D , is the sum of three stresses: the accumulated stress, σ_A , during the sliding process; the tensile stress, σ_T , due to the applied stress field; and the internal stresses, 6 σ₁, resulting from the thermal expansion anisotropy of the grains.

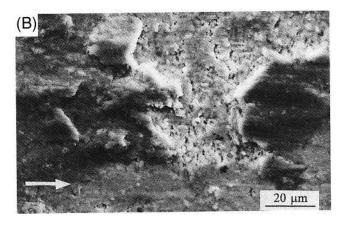
$$\sigma_D = \sigma_A + \sigma_T + \sigma_I \tag{2}$$

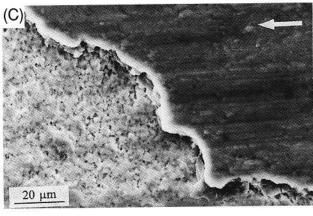
The critical condition for local microfracture is that the sum of the stress-intensity factors must be larger than or equal to the intrinsic grain-boundary toughness. Assuming that the size of the initial flaws is proportional to d and that grain-boundary microcracks are initiated from these flaws, the critical σ_D can be expressed as

$$\sigma_D = \sigma_l \left(\frac{d_c^S}{d} \right)^{1/2} \tag{3}$$

where d_e^s is the critical grain size corresponding to microfracture, under the action of the internal stress alone, caused by







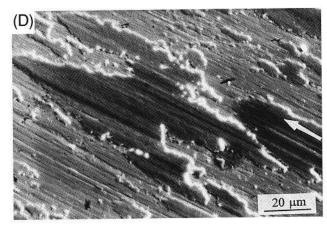
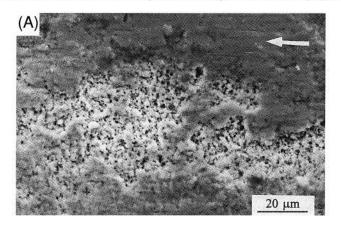
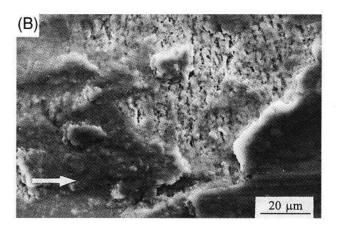


Fig. 6. Morphologies of specimen G5 with a grain size of 0.90 µm. Arrow indicates one direction of the reciprocal sliding.





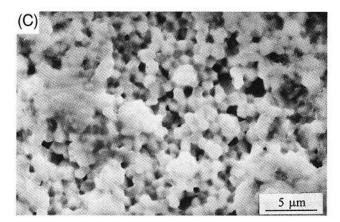


Fig. 7. Morphologies of specimen G7 with a grain size of 1.50 μm ((A) general worn surface, (B) wear scar after delamination, and (C) magnified version of Fig. 7(A) at the subsurface area after delamination). Arrow indicates one direction of the reciprocal sliding.

thermal anisotropy ($\sigma_T=0$ and $\sigma_A=0$). In this case, the critical σ_D acting in the ceramics depends on the grain size as a type of Hall–Petch relation ($d^{-1/2}$). A similar relation also was suggested by Winnubst $et~al.^{27}$ in brittle ceramics. They found that the fracture energy is correlated with grain size (proportional to $d^{-1/2}$). Using energy-conservation arguments and the Cho et~al. model, 5 Wang $et~al.^{28}$ developed an empirical model to express the volumetric wear loss in terms of extrinsic and intrinsic properties of ceramics. Wear particles are thought to be produced when the accumulation of energy during the sliding process exceeds the energy of new surface formation. The critical energy density, with dimensions of stress (see Wang $et~al.^{28}$) has been proposed as being proportional to σ_D . Thus, the wear volume, V, can be expressed as

$$V = C \left(\frac{\sigma_{\text{max}}}{\sigma_D} \right) \frac{LP}{H_{\text{v}}} \tag{4}$$

where C is a constant, L the sliding distance, P the normal load, and σ_{\max} the maximum external tangential stress (related to the friction coefficient f_c and the normal load P). For comparison with the results presented in this study, Eq. (4) is rewritten in terms of K_{plate}^{-1} :

$$K_{\text{plate}}^{-1} = \left(\frac{V}{LP}\right)^{-1} = C' \frac{\sigma_D H_{\text{v}}}{\sigma_{\text{max}}}$$
 (5)

where C' is a constant. Inserting Eq. (2) into Eq. (5), the wear resistance K_{plate}^{-1} is obtained as

$$K_{\text{plate}}^{-1} = C' \frac{H_{\text{v}}}{\sigma_{\text{max}}} \sigma_l \left(\frac{d}{d_c^S}\right)^{-1/2}$$
 (6)

This expression clearly reveals a Hall-Petch-type relation between wear resistance and grain size. For the present study on Y-TZP with average grain sizes $\leq 0.7 \, \mu m$, the critical grain size, d_c^S , and the internal stress, σ_I , are assumed to be constant. The variables f_c and H_v are independent of the grain size in the present range and, hence, also are constant. Therefore, in this region, the experimental results agree with the theoretical model qualitatively described in this paper. The actual wear mechanisms observed in grain-size region (I)—plastic deformation and microcracking are the dominant mechanisms—indicate that Eq. (6) can indeed be used for Y-TZP ceramics.

(2) Wear Resistance and Phase Transformation at Grain Sizes ≥0.9 µm

For Y-TZP with grain sizes $\geq 0.9 \mu m$ (region III of Fig. 3), the results show a wear resistance inversely proportional to the grain size (power of -1). A change is observed, as a function of grain size, from mild wear (region I) by plastic deformation and microcracks to severe wear (region III) by delamination. Also, a phase transformation is observed from the tetragonal to the monoclinic phase in grain sizes ≥0.9 µm. The transformation of TZP particles is induced by the stress during reciprocating sliding. As a result of this transformation, a compressive layer is formed at the worn surface. Hence, the formation and propagation of microcracks on the contact surface are prevented, and earlier formed microcracks can be healed. In this way, the transformation process results in a positive influence on wear resistance. On the other hand, a tensile stress can develop beneath the compressive layer, forming internal stresses in local areas. These internal stresses promote microcrack nucleation at, for instance, pores and grain boundaries. Fatigue lateral microcracks also can be produced at weak interfaces. The removal of particles starts when these subsurface cracks intersect at the contact surface of the body. Altogether, the removal of the material is identical to that described in the delamination theory of wear.²⁹ As grain sizes increase, the

internal stresses increase, because of more zirconia phase transformation, subsequently leading to a large degree of delamination. The high wear rate in partially stabilized zirconia (PSZ) or TZP, due to the delamination, also has been reported elsewhere in reciprocating-sliding processes30 or grinding by diamonds.31

For such delamination-wear processes, the damage-induced stress, σ_D , expressed in Eqs. (2) and (3) can be used for the present case as well. The only difference with the situation in fine-grained materials is that an internal stress introduced by the phase transformation also must be included. In combination with a microcracking theory of Fu and Evans,32 another empirical expression for the wear rate can be developed. This wear model is based on material removal by grain pullout, which is similar to the wear mechanisms in the present study. Using the wear model of Liu et al.,6 the wear rate, as a function of grain size, d, is expressed as

$$K_{\text{plate}} = \frac{1}{\nu P} \frac{\mathrm{d}V(t)}{\mathrm{d}t} = \frac{c\dot{\sigma}_D}{d_c^S \sigma_I \nu P} d \tag{7}$$

where V(t) is the wear volume, t the sliding time, v the average sliding velocity, c a coefficient with the dimensions of volume loss, and $\dot{\sigma}_D$ the damage-stress accumulation rate $(d\sigma_D/dt,$ which is independent of grain size⁵); d_c^s and σ_l are the same as in Eq. (3). Equation (7) shows that the wear resistance is inversely proportional to the grain diameter.

V. Conclusions

The wear resistance in TZP ceramics is increased by a factor of 8 when the grain size decreases from 1.5 to 0.18 µm. A Hall-Petch-type relation between wear resistance and grain size is observed for grain sizes ≤0.7 µm. In this case, the main wear mechanisms are plastic deformation and microcracking.

For relatively coarse-grained materials, with grain sizes ≥0.9 µm, the wear resistance of the material is proportional to the reciprocal of the grain diameter, where delamination and accompanying grain pullout are predominant during sliding wear. In this case, partial zirconia phase transformation can be induced by shear stresses during sliding.

The occurrence of a zirconia phase transformation prevents the formation of microcracks on the top surface. On the other hand, this transformation weakens the grain boundaries at the subsurface layer by using internal stresses that promote delamination and grain pullout, resulting in more wear particles in the coarse-grained TZP materials.

For grain sizes ≤0.7 µm, the value of the friction coefficient is independent of the grain size. For the coarse-grained material (≥0.9 µm), the friction coefficient is slightly less than that of the fine-grained material. Large amounts of SiC wear debris generated during sliding are responsible for this lower friction coefficient.

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