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Influence of Porosity on Friction and Wear of Tetragonal Zirconia Polycrystal

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

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

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

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

Sliding wear properties of ultra-fine-grained (180 nm) yttria-doped tetragonal zirconia (Y-TZP) ceramics were examined with porosities from 1.5% to 7%. On a pin-on-plate tribometer under dry-N₂ conditions, e.g., wear rates of the material increase by a factor of 5 by increasing porosity with a factor of 5 (from 1.5 to 7.0 vol%). In all cases no (irreversible) phase transformation to monoclinic zirconia took place during wear tests. The results for the relatively dense nanostructured materials showed significant evidence of plastic deformation and less microcrack formation. The morphology of the wear tracks in these ceramics indicate that the degree and amount of microcracks on the contact surface increased with porosity. A change in wear mechanism is observed.

I. Introduction

POLYCRYSTALLINE tetragonal zirconia (TZP) ceramics are promising in tribological applications due to their high flexural strength, hardness, and fracture toughness at room temperature or elevated temperature.^{1,2} The presence of porosity generally has a negative influence on mechanical properties, since pores can cause stress concentration, resulting in low strength.³ The presence of pores, distributed at grain boundaries, weakens the grain boundaries and leads to a reduction of fracture energy. It is found that for TZP ceramics the variation of fracture energy (γ), Young's modulus (E) and fracture toughness (k_{IC}) with residual porosity follows an exponential relation.⁴⁻⁶ It is expected that the wear characteristics of ceramics are also affected largely by residual porosity.

Wear loss of ceramic materials is governed by both plastic deformation and fracture.⁷ Porosity in ceramic materials can promote surface and subsurface cracking, favoring the occurrence of granular wear particles in the contact area.⁸

The purpose of the present investigation is to study the dependence of friction and sliding wear on porosity of ultra-fine-grained TZP ceramics with an average grain size of 180 nm. Samples were selected with different porosity in the range of 1.5–7.0 vol%. Ultra-fine-grained TZP ceramics were found to be specially suitable since (1) The grain size could be kept constant at a variable porosity, because ZY5.7 shows a

very slow grain growth at 1150°C in a large range of dwell times.⁹ (2) The stress-induced phase transformation from tetragonal to monoclinic is almost completely suppressed by the ultra-fine grain size.^{6,10} A tetragonal-to-monoclinic phase transformation has a significant effect on the tribological properties of TZP ceramics during sliding wear,^{8,11} resulting in a more complex wear process.

II. Experimental Procedure

Nanocrystalline and weakly agglomerated zirconia powders (Y-TZP) are prepared by a gel-precipitation method using metal chlorides as precursor chemicals. More details about the preparation process have been reported elsewhere.¹² The specimens are uniaxially pressed in a die at 80 MPa to a rectangular form and subsequently isostatically pressed at 400 MPa. The ultra-fine-grained Y-TZP specimens with grain size 180 nm are densified at 1150°C for varying times by pressureless sintering or by sinter forging¹³ to obtain 1.5%–7.0% porosities. Porosity was measured using the Archimedes technique in Hg and grain size by SEM pictures using the linear intercept technique.¹⁴ The surface of TZP specimens was subsequently polished with 0.05 μm alumina powder. Subsequently the specimens were ultrasonically cleaned for 45 min in ethanol and then annealed at 950°C for 10 min (heating and cooling rates 2°C/min) to remove the residual stresses produced by machining and polishing. The pin materials used in all tests are commercially available sintered SiC spheres (diameter 4 mm) with a mirror-polished surface (Gimex Technische Keramiek B.V., Geldermalsen, The Netherlands).

A wear testing system is used with a reciprocally moving pin-on-plate rig with a sinusoidal velocity and a load of 8 N (858 MPa) against a stationary TZP plate. All tests were carried out unlubricated in dry nitrogen gas with a relative humidity of less than 1% and at room temperature. The track length was 10 mm, the average sliding velocity 0.08 m/s, and the testing time about 20 h, corresponding to a sliding distance of 5.8 km. After the wear experiments, the samples were ultrasonically cleaned with ethanol for 30 min and dried at 120°C for 4 h. The samples were then cooled in dry N₂ gas and weighed. The weight losses of the worn pin and plate were converted to a volume loss using the apparent densities of the materials involved. The wear rate, K_w ($\text{mm}^3/(\text{N}\cdot\text{m})$), was calculated from these data. More experimental details are given in Ref. 11.

The phase composition of the contacting surfaces of TZP before and after sliding tests was analyzed with an X-ray diffractometer with $\text{CuK}\alpha$ radiation (PW 1710 diffractometer, Philips Scientific Instruments, Almelo, The Netherlands). The surface roughness of the samples was measured with a profilometer (Dek-Tak 3030, Sloan, Santa Barbara, CA). The wear tracks were observed using a scanning electron microscope (SEM, JSM-35CF, JEOL). The energy-dispersive X-ray (EDX)

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*Member, American Ceramic Society.

[†]Author to whom all correspondence is to be addressed: University of Twente, Faculty of Chemical Technology, Laboratory for Inorganic Materials Science, P.O. Box 217, 7500AE, Enschede, The Netherlands.

technique was used to analyze the worn surface and wear debris.

III. Results and Discussion

All TZP specimens were fully tetragonal zirconia in the bulk before the wear experiments. After sliding wear tests, no monoclinic (*m*) zirconia was detected on the worn surfaces. This could be expected, since the grain size of 180 nm is much less than the critical size where stress-induced transformation of tetragonal zirconia to the monoclinic phase may occur.

The average roughness of the Y-TZP plate surfaces before and after sliding wear is shown in Table I. The center line average roughness, R_a , is defined as the arithmetic mean deviation from the mean line through the complete profile. The mean line is defined as the line that has equal total areas of profile above and below.¹⁵ The raw data were measured by a profilometer after a 5.8 km sliding test at conditions specified before. The roughness values of the initial polished surface, which are less than 80 nm, are nearly independent of bulk porosity. After the sliding tests, much larger roughness values were found, increasing with porosity.

The wear rate of the TZP plate increases by a factor of 5 by increasing porosity a factor of 5 (from 1.5 to 7.0 vol%) as given in Table II. Each result was obtained by testing at least three samples. In Fig. 1, $\ln(1/K_w)$ versus porosity is plotted, which reveals a linear relationship. The slope of the curve in Fig. 1 equals -23 . In this way the data can be fit (over this limited porosity range) by an exponential relation:

$$1/K_w = 1/K_w^0 \exp(-23P) \quad (1)$$

where $1/K_w^0$ is the wear resistance of a pore-free TZP material ($(N \cdot m)/mm^3$). P is the (fractional) porosity. An exponential relation between wear resistance and porosity, with an identical slope of -23 , is also given by Hines *et al.*¹⁹ for polycrystalline alumina.

For the TZP ceramic plates, the morphologies of the worn surfaces are shown in Figs. 2 to 5. Materials S7P and S5P with porosity of 7.0 and 5.0 vol%, respectively (Figs. 2 and 3), reveal large cracks obviously developed after rubbing due to tensile stresses between the contact surfaces. The cracks exhibit geometric and periodic patterns and are perpendicular to the sliding direction. Large cracks are formed through weak links by fatigue wear processes, forming fish-scale patterns at the surface. The numbers of cracks at the turning point (Figs. 2(A) and 3(A)) are much higher than those in the middle of the wear tracks (Figs. 2(B) and 3(B)). Adhesion of a small amount of

wear debris on the TZP ceramics is also found. Microfracture is found occasionally and decreases with decreasing porosity.

Figures 4 and 5 show the wear tracks of materials with porosities of 3.0 and 1.5 vol%, respectively. Both reveal similar wear characteristics, and plastic deformation is clearly present on these worn surfaces. A part of the plastic deformation results from adhering and smearing of a thin wear debris layer. The features of the contacted surface do reveal, however, plastic deformation on the real ceramic top surface. Some microcracks occur on the wear tracks, but the degree of fracture and the amount of microcracks are much less than in samples S7P and S5P. Altogether, the analyses of the morphologies indicate that, upon decreasing the porosity from 7.0 to 1.5 vol%, an enhanced amount of plastic deformation and a reduction of cracking and grain pull-out by intergranular fracture is present.

As established by other investigators,^{16,17} crystalline ceramics can be deformed plastically by slip and/or twinning and even by grain boundary sliding like the mechanism of ceramic superplasticity.¹⁸ The presence of pores reduces the possibility of grain boundary sliding. The nucleation of microcracks is more likely to occur predominantly from pores, because pores cause high stress concentration, resulting in enhanced cracking along grain boundaries. An interconnected grain boundary microcracking pattern is generated during a reciprocal fatigue process, which is more pronounced and more serious with larger pores and/or higher porosity. These cracks are a major source of wear in ceramic materials. For nearly dense samples, a relatively strong grain boundary prevents the formation of cracks. In this case a stress concentration at grain boundaries can significantly enhance the plastic deformation before crack nucleation and growth, as can be seen from the SEM graphs of samples S3P and S1P. It appears that in more dense materials, fewer particles are removed around pores, because the pores are embedded firmly and well-separated from each other in the solid matrix.¹⁹ For sample S1P prepared by sinter forging, the improved grain boundary may further enhance the wear resistance of TZP (see Fig. 1).

According to the results in Table II, the values of f under stationary conditions are reduced with decreasing porosity except the specimen with the highest density (S1P), where f is slightly increased again to 0.42. It was mentioned earlier that microcracking is an important wear mechanism for porous ceramics. Removal of material by pull-out of grains or fracture in microcracking regions leads to surface roughness. As a result, friction increases substantially, favoring the occurrence of wear particles in the contact area. Sample S1P reveals plastic deformation at the contact area as a result of a high applied load. According to Rice,¹⁷ this plastic deformation may result in an increase in f . In addition to this, the relatively smooth sliding surface obtained in the dense sample S1P leads to an increase of the cohesive force between two contact surfaces due to a reduction of adsorbed air films, similar to vacuum conditions.²⁰ It is found that a high f under vacuum can be explained by strong adhesive forces acting between exposed fresh surfaces, while an introduction of gases into the vacuum causes a decrease in friction by adsorption layer lubrication.

Generally, static friction is much larger than dynamic friction. During sliding, the static friction often occurs at the turning point of wear tracks because the sliding direction is changed at the turning point. The relatively larger tangential friction force at the turning point of the wear track causes higher surface fatigue than in the middle. So microcracks are more concentrated at the turning point because of higher friction coefficient at the turning point.

Table I. Characteristics of TZP Plates with an Average Grain Size of 180 nm and Varying Densities before and after Sliding Tests*

Sample code	P (vol%)	Center line average roughness, R_a (nm)		
		Polished surface	to wear track	⊥ to wear track
S7P	7.0	<80	2295	5012
S5P	5.0	<80	1831	2948
S3P	3.0	<80	1700	2166
S1P*	1.5	<80	1150	1482

*Specimens were sinter forged at 40 MPa for 25 min.

Table II. Friction and Wear Properties for Sliding of SiC Balls against TZP Plates

Sample code	Wear rate of TZP plate K_{TZP} , $\times 10^{-6}$ ($mm^3/(N \cdot m)$)	Wear rate of SiC pin K_{SiC} , $\times 10^{-6}$ ($mm^3/(N \cdot m)$)	Steady-state friction coefficient (f)
S7P	5.18 ± 0.30	4.90	0.46 ± 0.07
S5P	3.10 ± 0.30	4.30	0.39 ± 0.08
S3P	2.10 ± 0.24	4.00	0.36 ± 0.06
S1P	1.08 ± 0.22	3.42	0.42 ± 0.08

IV. Conclusions

Plastic deformation and microcracking are the two basic wear mechanisms for ultra-fine-grained Y-TZP ceramics. The presence of pores promotes the formation of surface or subsurface cracks. The wear resistance decreases by a factor of 5 if the porosity increases from 1.5 to 7.0 vol%.

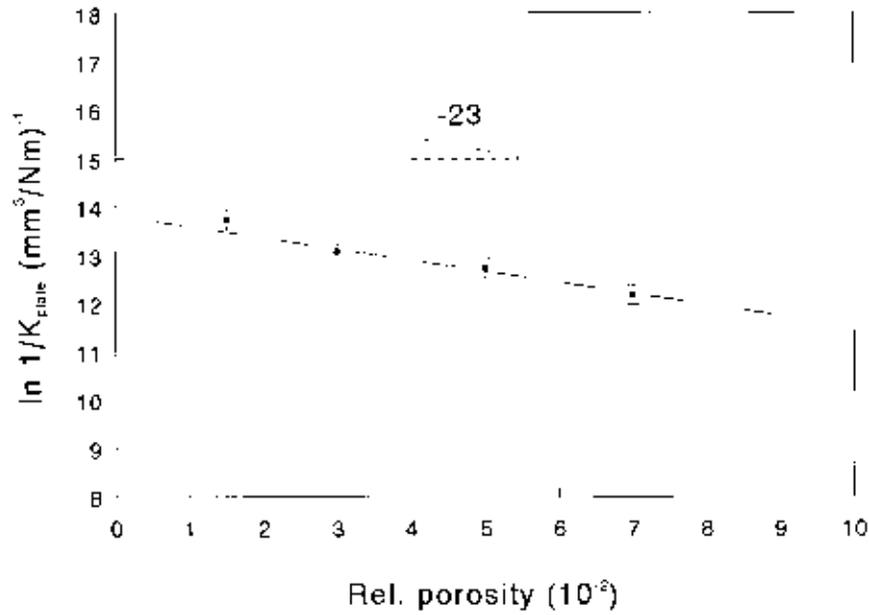


Fig. 1. Logarithmic wear resistance versus porosity of TZP specimens at a normal load of 8 N and an average velocity of 0.08 m/s during sliding against a SiC pin.

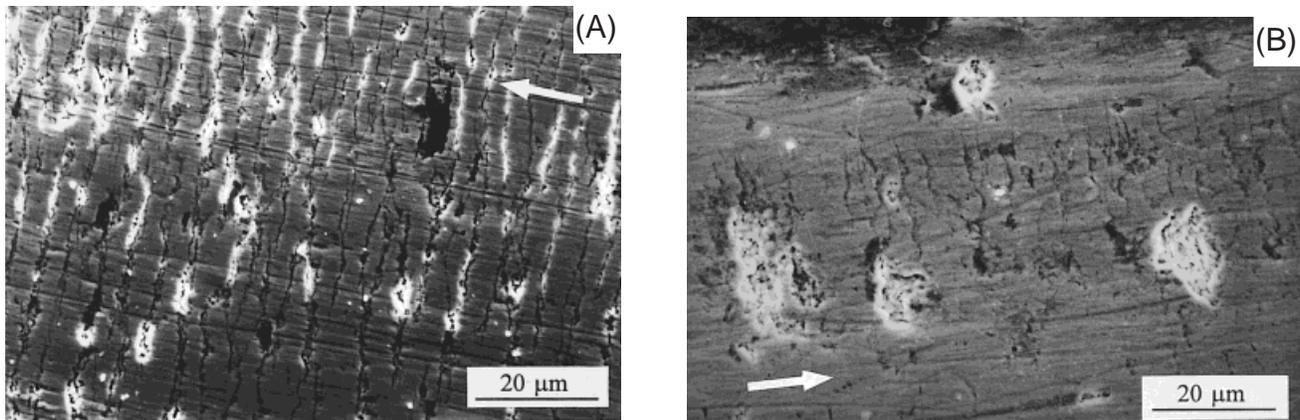


Fig. 2. Scanning electron microscopy of the wear track of S7P: (A) turning point area; (B) the middle of the wear track. The arrow indicates one direction of the reciprocal sliding.

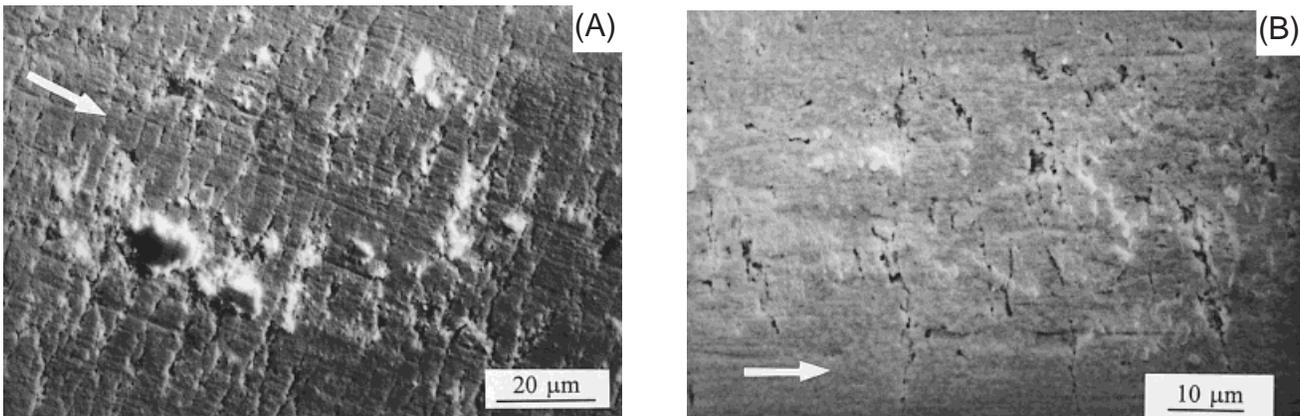


Fig. 3. Scanning electron microscopy of the wear track of S5P: (A) turning point area; (B) the middle of the wear track. The arrow indicates one direction of the reciprocal sliding.

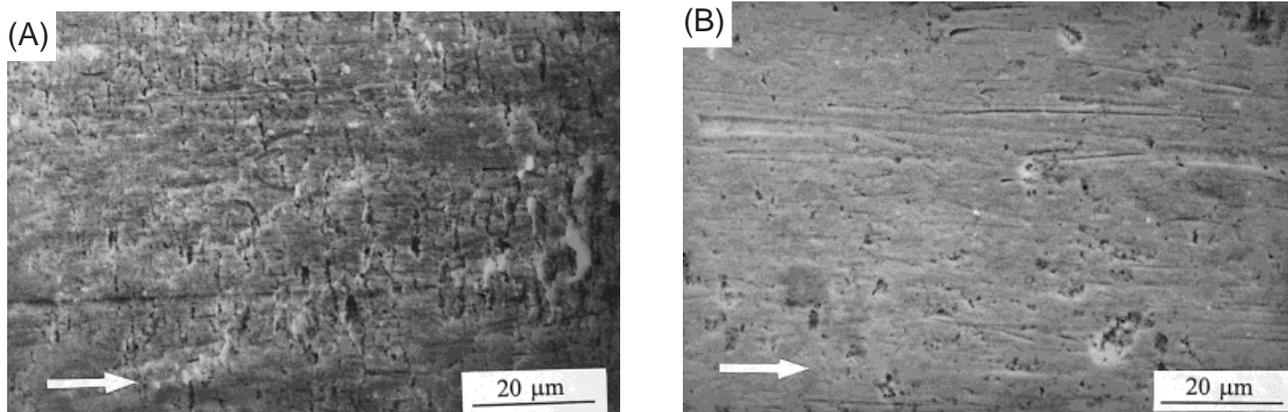


Fig. 4. Scanning electron microscopy of the wear track of S3P: (A) turning point area; (B) the middle of the wear track. The arrow indicates one direction of the reciprocal sliding.

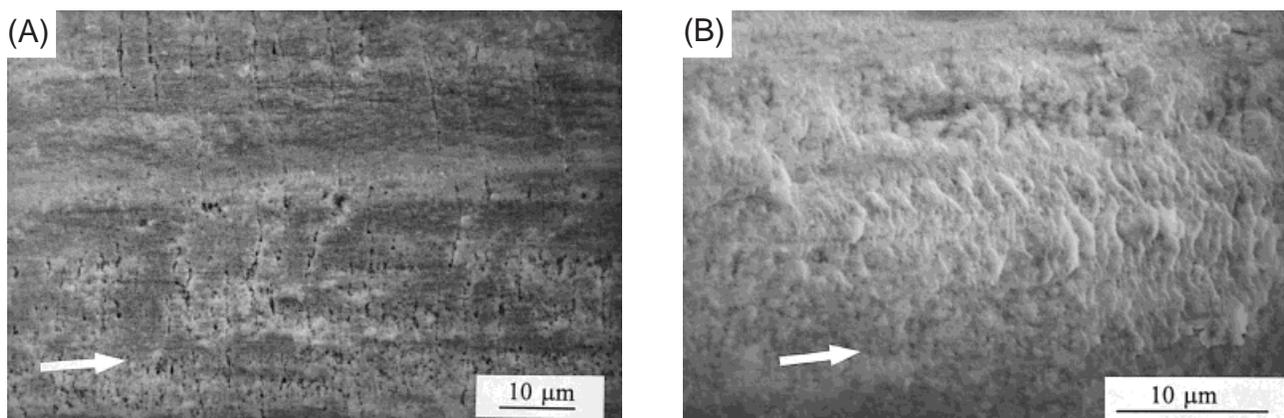


Fig. 5. Scanning electron microscopy of the wear track of S1P: (A) turning point area; (B) the middle of the wear track. The arrow indicates one direction of the reciprocal sliding.

For relatively dense materials, more plastic deformation occurs, resulting in a slight increase in friction coefficient f . Surface roughness decreases with decreasing porosity. During sliding wear of ultra-fine-grained TZP ceramics (180 nm), no irreversible zirconia phase transformation takes place. The wear rates of SiC material are influenced by those of the TZP counter material.

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