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Wear Behaviour of Partially Stabilized Zirconia at High Sliding Speed

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Abstract

The wear behaviour of three partially stabilized zirconias (PSZs) against steel was investigated on a pin-on-disk wear machine under dry conditions (from 5 m/s to 50 m/s, at 5 N load). In order to maintain plane-on-plane contact, the ceramic pins (dia. = 5 mm) were ground on the wear machine before the wear tests. Particular behaviour of PSZ ceramics was found with extremely heavy wear only in a special range of sliding speed between 10 m/s and 20 m/s and much lower wear was found outside this range. The heavy wear was accompanied by a phase transformation (tetragonal–cubic), which was identified by analysing the wear debris by means of XRD. Delamination mechanism was observed by analysing the worn surfaces of the pin and the wear debris with SEM attached to an EDAX system. The wear resistance of Mg–PSZ was generally better than that of Y–PSZ under the present conditions, especially at high sliding speeds (> 20 m/s).

Das Abriebverhalten dreier teilstabilisierter Zirkonoxidwerkstoffe (TSZ) gegen Stahl wurde mit einer 'pin-on-disc' Apparatur unter trockenen Bedingungen bei 5 N im Geschwindigkeitsbereich von 5 m/s bis 50 m/s untersucht. Um einen guten Kontakt der Berührungsflächen zu erreichen, wurden die keramischen Stite (5 mm Durchmesser) vor den Verschleiß tests auf der Versuchsapparatur eingefahren. Das TSZ zeigte speziell im Geschwindigkeitsbereich von 10 m/s bis 20 m/s einen extrem hohen Verschleiß, der jedoch außerhalb dieses Bereichs weitaus geringer war. Der hohe Abrieb ging mit einer durch eine röntgenographische Untersuchung des abgetragenen Materials nachgewiesenen Phasenumwandlung tetragonal–kubisch einher. Die REM/EDAX Analyse der geschädigten Oberfläche des Stifts und des Abriebs lassen auf einen schichtenweisen Abtragungsprozeß schließen. Das Abriebverhalten des Mg–TSZ war unter den gegebenen Bedingungen im allgemeinen, speziell jedoch bei hohen Geschwindigkeiten (> 20 m/s), besser als das des Y–TSZ.

On a étudié la résistance à l'usure par l'acier de trois zircones partiellement stabilisées (PSZ) à l'aide d'une machine d'usure aiguille-sur-disque en milieu sec (de 5 à 50 m/s, 5 N). Afin de maintenir le contact plan-sur-plan, les aiguilles céramiques (dia. = 5 mm) avaient été préalablement passées à la machine d'usure avant les essais. La PSZ présente un comportement particulier avec une usure extrêmement forte à l'intérieur de la gamme de vitesses comprises entre 10 et 20 m/s et une usure bien moindre à l'extérieur de ce domaine. La forte usure était accompagnée d'une transformation de phase (tétragonale–cubique), identifiée sur les débris d'usure par diffraction X. L'analyse de la surface usée des aiguilles et des débris par EDAX couplé au MEB a permis la mise en évidence d'un mécanisme de délamination. Dans ces conditions, la résistance à l'usure de la Mg–PSZ était en général supérieure à celle de la Y–PSZ, particulièrement pour les vitesses élevées (> 20 m/s).

1 Introduction

Recently, considerable attention has been drawn to partially stabilized zirconia (PSZ) ceramics, because of their special mechanical, chemical and thermal properties. In addition to low thermal conductivity and good corrosion resistance, improved fracture toughness makes PSZ ceramics very attractive as thermal barriers and wear-resistant materials, especially in complex environments. For several

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years, many encouraging results have been obtained in mining industry and metallurgical applications.[1,2] The good wear resistance of PSZ ceramics has also been verified in some engine elements.[3] However, their potential applications are still limited by the lack of reliable engineering data concerning the friction and wear behaviour.

In order to investigate the possibility of larger tribological applications of PSZ ceramics, a number of studies have been carried out to investigate their sliding characteristics in a variety of conditions. Scott[4] studied the wear behaviour of a self-mated Mg–PSZ couple on a pin-on-flat tribometer. At very low sliding speed, rapid wear of Mg–PSZ was found only in a narrow range of environmental temperature (about 200°C). Scott suggested that the particular wear behaviour of Mg–PSZ depended on the amount of transformable phase in the sample.[4] Aronov[5] also observed a narrow range of temperature when examining the wear behaviour of Mg–PSZ against itself on a pin-on-plate reciprocating wear machine, but in this temperature range the wear of Mg–PSZ decreased by three orders of magnitude. A phenomenological model was thus proposed to explain the strengthening mechanism of PSZ during the friction, concerning the phase transformations by frictional heating.[5] A similar phase transformation was also observed by Ishigaki & Nagata[6] when analysing by laser Raman the particles generated during a wear test of a self-mated Y–PSZ on a pin-on-disk wear machine.[6] Woydt & Habig[7] studied the effects of sliding speed and environmental temperature on wear behaviour of a self-mated Mg–PSZ couple on a pin-on-disk wear machine. Some similar effects of the two test parameters on the wear rate of Mg–PSZ were observed with a transition speed at ambient temperature, and a transition temperature with a relatively low speed. According to XRD (X-ray diffraction) and TEM (transmission electronic microscope) analysis, the low wear–high wear transition is related to a phase transformation (tetragonal to cubic) in the sample.[7] High wear rate of PSZ was found by Stachowiak[8] when testing PSZ/PSZ with a pin-on-flat high frequency wear machine. The delamination and plastic deformation were considered as the main wear mechanisms of PSZ in this case.[8] Fisher *et al.*[9] examined the wear behaviour of tough and brittle zirconias in different environments. It was shown that the effects of environment were influenced by the fracture toughness of the zirconias tested.[9] The wear behaviour of zirconia (TZP) under lubricated conditions was studied by Birkby *et al.*[10] It was found that the wear mechanism was related to the powder composition and the microstructure of the surface layer.[10]

Considering the results above, the wear behaviour of zirconia ceramics seems to be very sensitive to the structure of the material, and to the test parameters, such as temperature, environment and sliding speed. The wear mechanisms are often complicated. In this paper, experimental results of PSZ/steel are presented. The wear mechanisms involved were studied by means of SEM (scanning electronic microscope) with EDAX (energy dispersion analysis by X-ray) attachment and XRD.

2 Wear Apparatus

The wear tests were carried out on a high-speed pin-on-disk wear machine, which is shown schematically in Fig. 1. The linear speed at the contact point may be set from 5 m/s to 60 m/s. Load is applied by means of a dead-weight system which is linked to the pin holder by a wire in the direction of the axis of the disk. Linear wear is measured by two laser distance sensors fixed on the carriage. One is in front of the wear track of the disk to take into account possible thermal expansion and wear of the disk, another is opposite the pin holder. The variation in the difference between the two sensors measurements gives the linear wear. The accuracy of the sensors is to ±1 μm and the percentage error of the measurement is less than 5% for a linear wear from 50 μm to 500 μm. The loading and frictional forces were measured with a piezoelectric sensor. The wear machine was located in a sealed room in which the air humidity was controlled by a humidity regulating system.

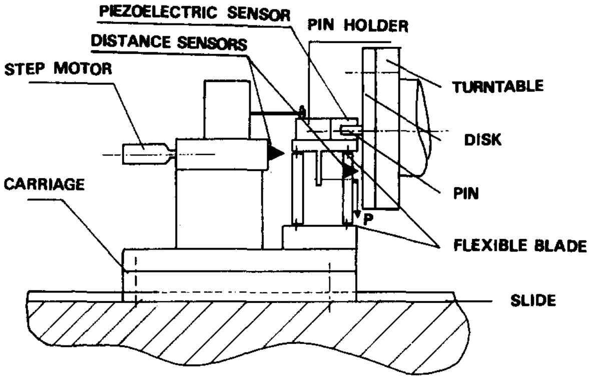


Fig. 1. Schematic representation of wear machine of high sliding speed.

3 Materials

The zirconias used for the investigations were provided by three French companies: Demarquest,

Table 1. Properties of materials

Materials	*Chemical composition (wt%)*	*Density ($g\ cm^{-3}$)*	*Thermal conductivity ($Wm^{-1}\ °C^{-1}$)*	*Flexural strength (MPa)*	*Young's modulus (GPa)*	*Hardness ($kg\ mm^{-2}$)*
Mg–PSZ	MgO: 2–3; ZrO_2: 96 HfO_2: bal.	5·6	0·92	580	141	1 409 HV 843 HK
Y–PSZ1	Y_2O_3: 3; ZrO_2: bal.	6·08	2·9	1 000	205	1 987 HV 1 347 HK
Y–PSZ2	Y_2O_3: 5·4; ZrO_2: bal.	5·98	3·0	800	200	1 482 HV 1 259 HK
Steel	C: 0·95; Mn: ·3; Si: ·3; Cr: 1·5; Fe: bal.	7·8	46	2 200	210	72 HRC

Tensile strength. HV: Vickers Hardness. HK: Knoop Nardness. HRC: Rockwell Hardness.

SCT and Ceramique et Composite. The magnesia-doped (9 mol%) partially stabilized zirconia (Mg–PSZ) consists of three phases: cubic, tetragonal and monoclinic. The average grain size is about 30–50 μm. The Y–PSZ1 was sintered with 2 mol% of Y_2O_3 at 1500°C. It consists mainly of tetragonal phase with a small amount of monoclinic precipitate. Another Y–PSZ ceramic, called Y–PSZ2, was sintered with 4 mol% of Y_2O_3. The grain size of the two Y–PSZ ceramics is about 0·5 μm.

The disks were made of french grade 100C6 steel (AISI52100). The surface roughness of the disks was between 0·6 and 0·8 μm after grinding. The wear track diameter of the disks was 160 mm. Other properties of the materials are listed in Table 1.

4 Experimental Procedure

In order to have the two surfaces parallel at the start of the wear tests, the pin was ground on an abrasive paper fixed to the disk before each new test. The load was 5 N and the speed was 5 m/s for the grinding. The grinding continued until the wear scar depth of pin attained 50 μm. The pin surface was then washed in freon and the disk surface in acetone.

The wear of pin was evaluated by linear wear and wear rate. The linear wear value is obtained by using following equation:

$$Ui = (Pi - Di) - (Po - Do) \qquad (1)$$

where Pi is the distance between the first distance sensor and pin holder at the moment i, and Di is the distance between the second distance sensor and the steel disk at the same time. Po and Do are the corresponding values at the start of the wear test.

The wear rate is expressed in mm^3/m and is calculated by following equation:

$$Vi = \pi R^2 Ui/d \qquad (2)$$

where R is radius of pin, Ui is the linear wear

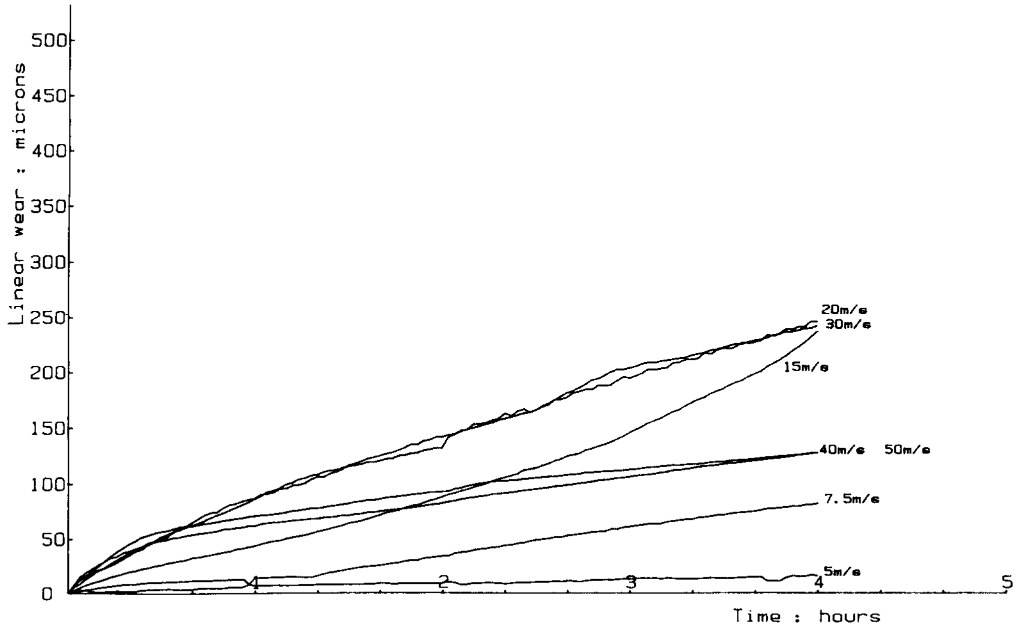


Fig. 2. Linear wear evolutions of Mg–PSZ pins (5 N, 50% RH).

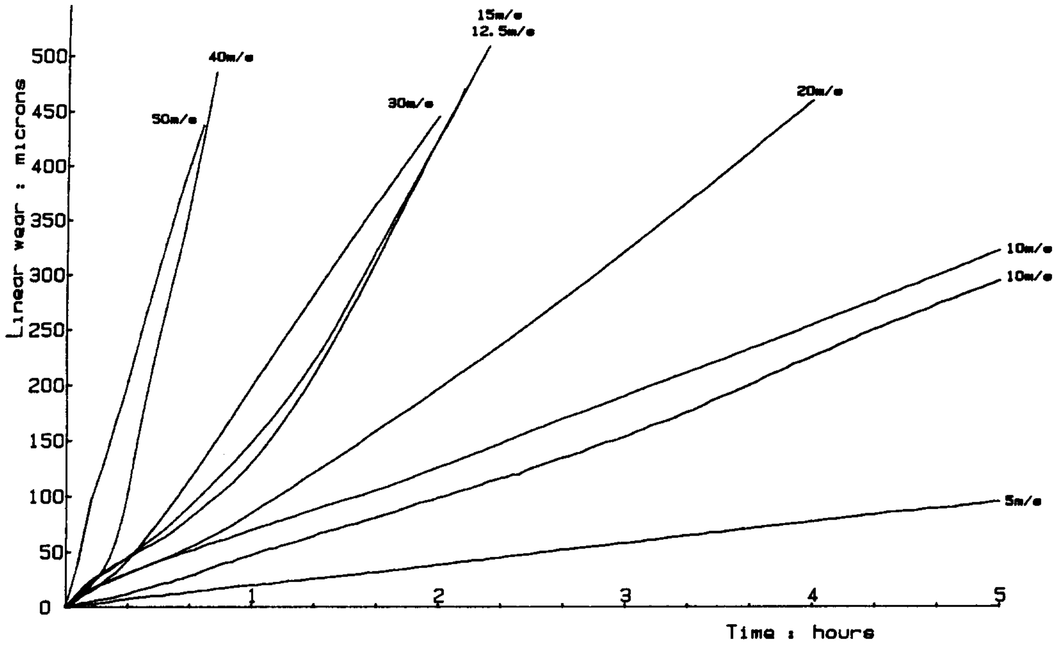


Fig. 3. Linear wear evolutions of Y–PSZ1 pins (5 N, 50% RH).

obtained from eqn (1), and *d* is the distance slid during a certain time. *Ui* was measured every 2 min. The final value of wear rate *V* was obtained by a linear regression of *Vi* versus sliding distance.

The humidity in sealed room was maintained between 45% and 55% and the temperature was about 22°C during the wear tests.

5 Wear Results

Linear wear of Mg–PSZ pin versus time is presented in Fig. 2. Moderate wear was found when the sliding speed was lower than 10 m/s or higher than 30 m/s. It is interesting to note that rapid and unstable wear was observed at 15 m/s. Similar phenomena were found in the case of Y–PSZ1 at 12·5 m/s and 15 m/s (Fig. 3), and in the case of Y–PSZ2 at 10 m/s and 15 m/s (Fig. 4).

Figure 5 showed the effect of sliding speed on the wear rate of PSZ pins. The wear rate here was calculated by linear regression from the linear wear presented previously. In the case where the linear wear was not stable at the beginning, only the stable part was considered. The most rapid wear was found between 10 m/s and 20 m/s for the three PSZ samples

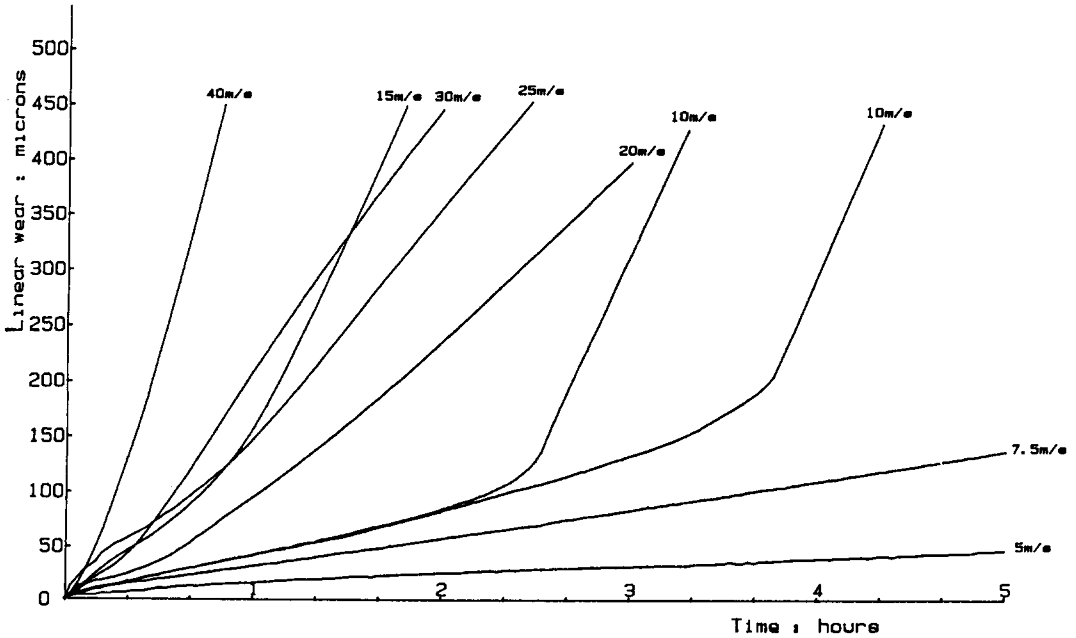


Fig. 4. Linear wear evolutions of Y–PSZ2 pins (5 N, 50% RH).

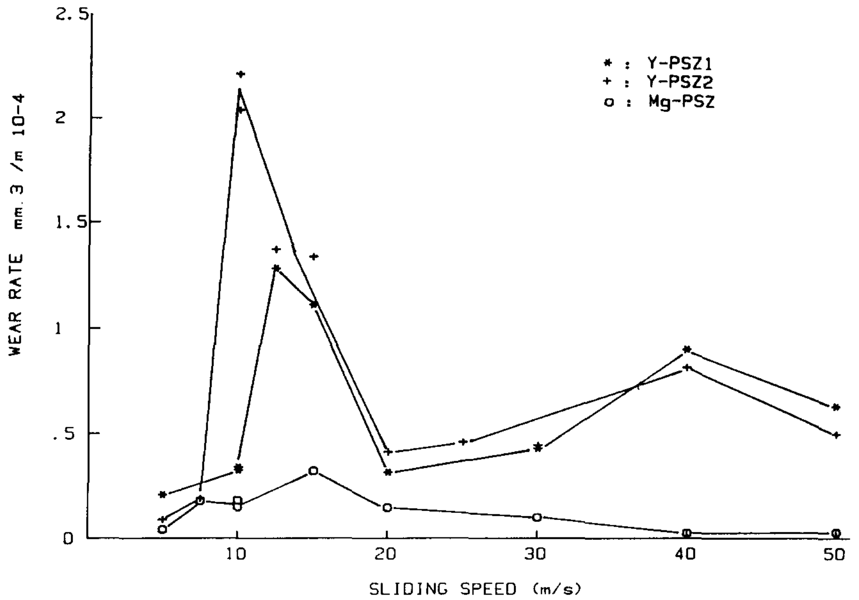


Fig. 5. Effects of sliding speed on the wear rate of PSZ pins (5 N, 50% RH).

tested. The wear rate of Y–PSZ increased when the speed increased from 20 m/s to 40 m/s and then decreased. In contrast, the wear rate of Mg–PSZ decreased when the speed increased from 15 m/s to 50 m/s. At 40 m/s, for example, the wear rate of Mg–PSZ was about 40 times lower than that of Y–PSZ.

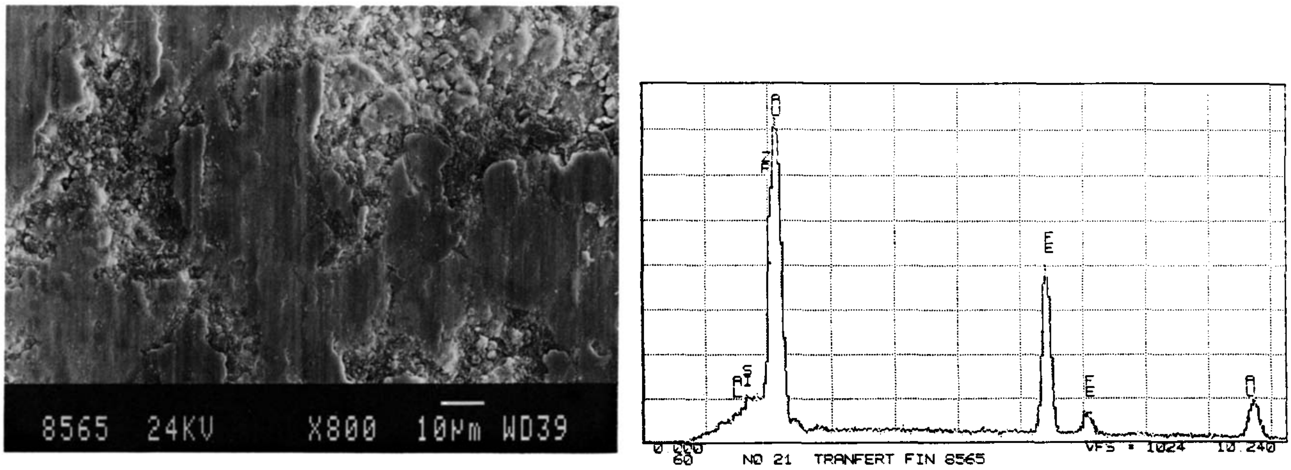
6 Worn Surface Analysis

In order to study the wear mechanisms of PSZ pins, the worn surface were analysed by SEM with EDAX attachment and by XRDA.

It was found that metallic transfer from steel disk to ceramic pin should be a main wear mechanism when the speed is lower than 10 m/s. Figure 6 shows an example of worn surfaces of PSZ pins after the wear tests. A metallic element-containing layer was identified by EDAX. This explained the small wear rate of the PSZ pin under these conditions.

Because of the high wear rate of PSZ in the speed range between 10 and 20 m/s, more attention was paid to analysing the worn surface tested at these speeds. Figure 7 shows some examples of worn surfaces of PSZ pins tested at 15 m/s. Cracks were found on the worn surfaces. A surface layer was observed on a Y–PSZ1 pin. The layer seemed to be separated from the pin by a crack.

On the other hand, a large amount of wear debris was produced during the wear tests when the speed was between 10 and 20 m/s. The wear debris was generally in plate form. Figure 7 also shows an example of the observation of wear debris produced during the wear test Y–PSZ1/steel at 15 m/s. When the debris is compared with the surface layer on the



(a) (b)

Fig. 6. (a) Scanning electron micrograph of worn surface of Mg–PSZ pin after test at 10 m/s and (b) X-ray dispersion spectra.

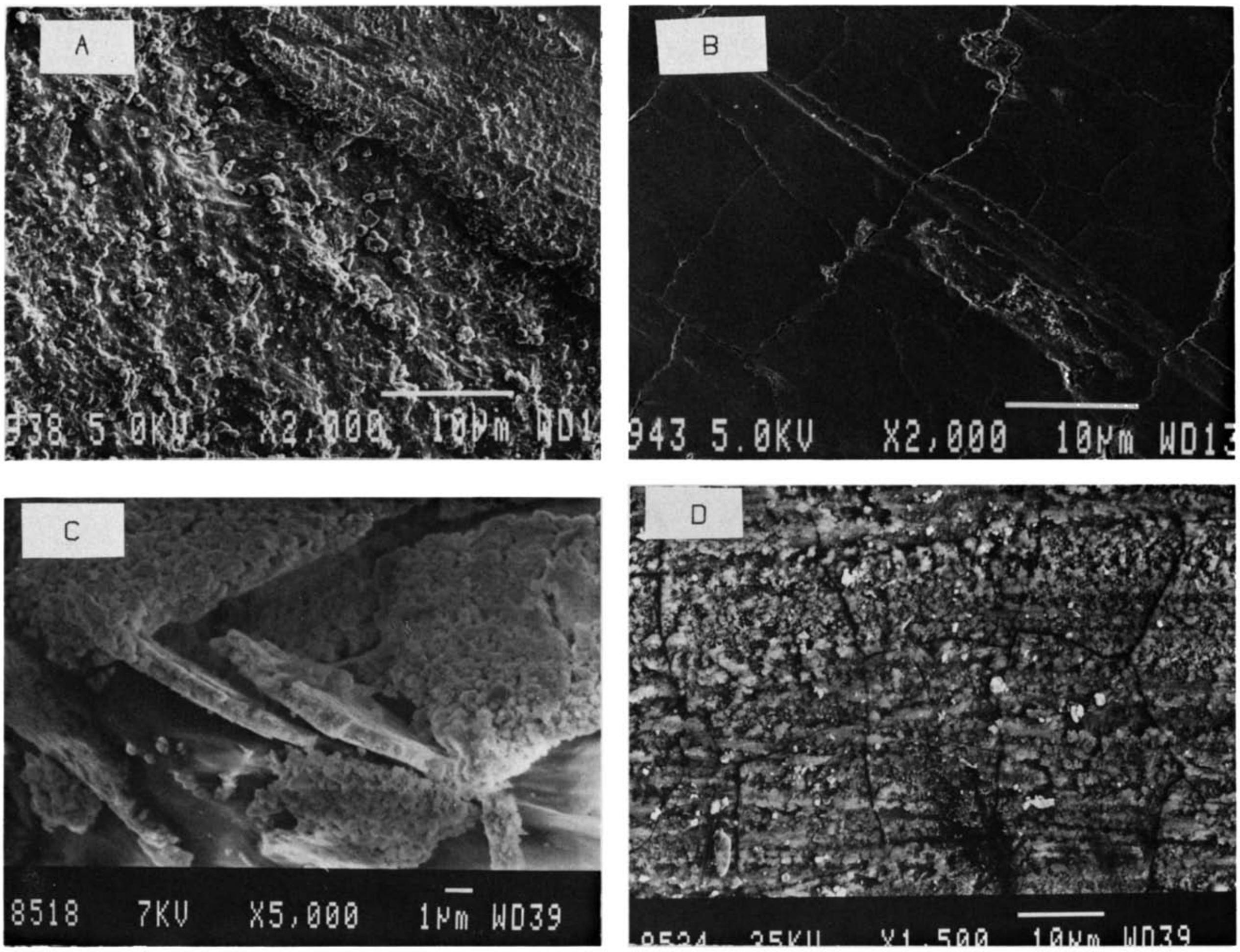


Fig. 7. Scanning electron micrographs of worn surface and wear debris after test (15 m/s). A, Y–PSZ1 pin; B, Y–PSZ2 pin; C, wear debris of Y–PSZ1/steel; D, Mg–PSZ pin.

Y–PSZ1 pin, it is reasonable to imagine delamination of the PSZ pin during the wear test. It is suggested that the delamination of the pin surface may be the main cause of the heavy wear of the PSZ pin in this case.

The structure of the debris was also analysed by XRD. Figure 8 shows the comparison between the results for a Y–PSZ1 pin after test and that for the wear debris produced during the test of Y–PSZ1/steel. It was found that the wear debris was only composed of cubic phase of ZrO_2. The transformation tetragonal–cubic generally takes place at high temperature, accompanied by a change of volume. Thus, the delamination of the PSZ pin in this case was probably related to the phase transformation involved.

The wear surface of PSZ pins tested at different speeds were also analysed by XRD. In the case of Mg–PSZ pins, no evident relative variation between the cubic phase and tetragonal phase was found when the speed increased from 5 m/s to 50 m/s. However, a considerable increase of the monoclinic

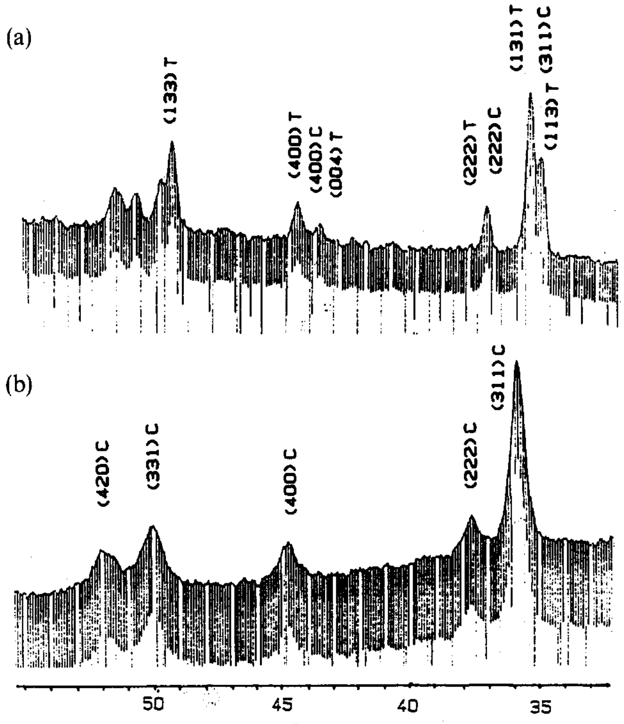


Fig. 8. X-Ray diffraction spectra of (a) worn surface of Y–PSZ1 pin and (b) wear debris after test (15 m/s).

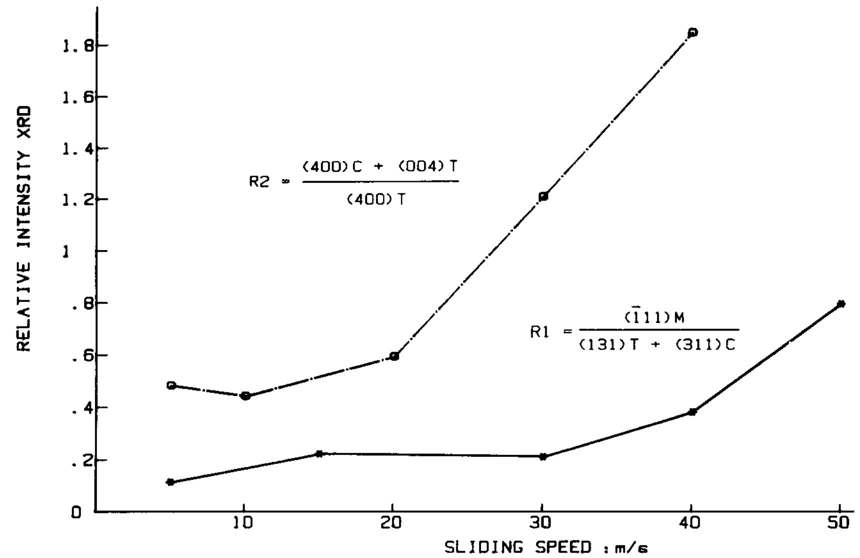


Fig. 9. Evolution of the phases in PSZ worn surfaces versus the sliding speed. R1, Mg–PSZ; R2, Y–PSZ.

phase was found when the speed was higher than 30 m/s (R1 in Fig. 9). For Y–PSZ1 pins, no pic of the monoclinic phase was found for the worn surfaces, but a relative increase in the cubic phase was observed when the speed increased from 10 m/s to 40 m/s (R2 in Fig. 9).

7 Discussion

The decrease in wear resistance of the PSZ pins between 10 m/s and 20 m/s shows the important effects of sliding speed on their wear behaviour. However, the effect of sliding speed is not independent of other parameters. The variation of the speed affects not only the relative velocity of two surfaces in contact, but also the temperature at the interface and probably the vibrational behaviour of the tribometer. According to frequency analysis of friction and load forces, no evident change of frequency was observed between 5 m/s and 50 m/s for the PSZ/steel couples. Although the bulk temperature measured by a thermocouple (1 mm from the contact surface) was only 50°C when the speed was 10 m/s, sparks were observed at the interface after the low wear–high wear transition point. Considering the low conductivity of zirconias, the surface temperature should be much higher than that of the bulk. In addition, significant variations of bulk temperature were observed while the wear rate changed to a high value for several tests. It was supposed that the phase transformation (tetragonal–cubic) should be due to the increase of surface temperature.

The same phase transformation was also observed by Woydt & Habig when examining the wear behaviour of a self-mated ZrO_2 couple on a pin-on-disk machine,[7] but the transition speed was 1 m/s at ambient temperature. If 10 m/s were considered as the transition speed in the present study, the main reason for this difference could be attributed to the higher conductivity and larger wear track diameter of the steel disk used in the present work.

The good wear resistance of Mg–PSZ at higher speeds (>30 m/s) gave some encouraging data for the tribological applications of Mg–PSZ, but the wear mechanism involved is not entirely understood. The increase of monoclinic phase seemed to be a positive factor for the wear resistance of Mg–PSZ (Fig. 9). However, the reason for this increase is still uncertain. The high wear rate of Y–PSZ at high speeds (>30 m/s) might be related to the increase of cubic phase in comparison with that at lower speeds.

8 Conclusions

(1) Rapid wear of both Mg–PSZ and Y–PSZ was found in a special range of sliding speed (between 10 and 20 m/s with 5 N load and 50% relative humidity).
(2) The main wear mechanism of PSZ pins in this range of speed is delamination, due to the phase transformation (tetragonal–cubic).
(3) The wear resistance of Mg–PSZ is better than that of Y–PSZ, especially at higher sliding speeds (>30 m/s).

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