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Sliding Wear of ZrO$_2$–Al$_2$O$_3$ Composite Ceramics

Y. J. He,$^a$ A. J. A. Winnubst,$^a$ A. J. Burggraaf,$^a$ H. Verweij,$^a$
P. G. T. van der Varst$^b$ and G. de With$^b$

$^a$University of Twente, Faculty of Chemical Technology, Laboratory for Inorganic Materials Science, PO Box 217, 7500AE, Enschede, The Netherlands

$^b$Eindhoven University of Technology, Faculty of Chemical Technology, Lab. of Solid State Chemistry and Materials Science, PO Box 513, 5600 MB Eindhoven, The Netherlands

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Abstract

The friction and wear behaviour of two ZrO$_2$–Al$_2$O$_3$ composite materials, ADZ (20 wt% Al$_2$O$_3$ dispersed in yttria-doped ZrO$_2$ matrix) and ZTA (Al$_2$O$_3$–15 wt% ZrO$_2$), were investigated. Sliding wear tests were carried out on these materials under dry conditions using a ball-on-plate configuration. The effects of grain size and zirconia phase transformation on friction and wear behaviour were examined. Sinter forging was applied to determine the role of grain boundaries on the wear process. It was found that for ADZ composites the wear resistance increased with decreasing grain size. Only limited grain pull-out occurs in these systems, which also explains the fact that in this case sinter forging has no positive effect on wear resistance. For ZTA composites a compressive surface caused by zirconia phase transformation has a beneficial effect on wear resistance. Here grain pull-out is the main wear mechanism which explains the positive effect of sinter forging ZTA on wear resistance. © 1997 Elsevier Science Limited.

1 Introduction

High performance ceramics can be obtained through microstructural engineering. The microstructure can be optimized by control of, for example, grain size, porosity and grain boundary morphology and composition. The optimization of the microstructure is an important factor for improvement of properties such as strength, toughness, hardness and wear resistance. In Ref. 1 a Hall–Petch type relation was found between wear rate and grain size for yttria-doped tetragonal zirconia (Y-TZP) ceramics against SiC with plastic deformation and microcracking as the main wear mechanisms. The same relationship was also found by Wu et al. for alumina materials. Using fracture mechanics, Cho and co-workers and Wang et al. demonstrated that a Hall–Petch type relation exists between the critical stress for wear transition (from mild to fracture-controlled severe wear) and grain size for ceramics. On the other hand, Liu et al. combined the fracture mechanisms used by Cho et al. with a microcracking controlled theory to demonstrate that the wear rate is directly proportional to the grain size for alumina ceramics which was also observed for Y-TZP where the wear mechanism consists of delamination and grain pull-out. The grain size has been recognized as an important factor in characterizing the wear behaviour of ceramic materials.

In Y-TZP ceramics the toughness is directly related to the amount of the tetragonal phase and the transformability from tetragonal to monoclinic zirconia induced by an external stress. Compared to Y-TZP ceramics, polycrystalline Al$_2$O$_3$ shows a lower bulk density, higher Young's modulus and hardness, lower linear thermal expansion coefficient and higher thermal conductivity but, on the other hand, inferior mechanical properties. ZrO$_2$–Al$_2$O$_3$ composite ceramics may be very attractive materials due to the combination of both ZrO$_2$ and Al$_2$O$_3$ properties. Introduction of zirconia in an alumina matrix (zirconia-toughened alumina = ZTA), leads to a composite material with increased toughness. Stress-induced phase transformation toughening and/or stress-induced microcrack toughening are here the toughening mechanisms. Under certain conditions microcrack toughening occurs in ZTA by the presence of the monoclinic zirconia formed during cooling after ceramic processing resulting in stresses around the zirconia particles. This latter toughening mechanism is used effectively for increasing fracture toughness, but the wear resistance is reduced as compared to pure alumina.
It has been found that composites with 20 wt% alumina in a zirconia matrix (alumina dispersed in a zirconia matrix = ADZ), exhibit improved toughness and enhanced thermal shock resistance and hardness when compared to zirconia ceramics. In addition, the elastic modulus of these composites is higher, resulting in an increase in the critical grain size for thermally induced phase transformation. These improvements could be advantageous for tribological applications. It has been reported that composite ceramic cutting tools exhibit much better cutting performance than single-phase Y-TZP.

In Refs 14 and 15 it has been shown that the mechanical properties, e.g. bending strength and fracture toughness, are increased by a factor of about 1.5-2 when sinter forging techniques are used to sinter TZP and ZTA materials. Such an increase in mechanical properties confirms that the grain boundaries are improved by strengthening the bonding between the grains due to creep deformation in the sinter forging process.

In this paper the wear characteristics of ZTA and ADZ composites with a certain range of average grain sizes will be described. The influence of grain boundary strengthening on wear is studied. Through a better knowledge of the relation between wear and grain size/grain-boundary morphology it is possible to find the optimal microstructure for special tribological applications.

2 Experimental Procedure

A homogeneous \( \text{ZrO}_2-\text{Al}_2\text{O}_3 \) composite with a composition of 5 atom% Y-doped zirconia and 20 wt% (about 28 vol%) \( \text{Al}_2\text{O}_3 \) was obtained by the ‘\( \alpha \)-alumina’ suspension method. In this method, commercially available \( \alpha \)-alumina (AKP-50, Sumitomo Chemical Co. Ltd) powder was used with a particle size of 0.1–0.3 \( \mu \)m. A stable suspension with a concentration of 300 g/l was prepared at a pH of 3 by ultrasonic treatment (Branson Sonifier 450) and ball milling. The pH value was adjusted with HCl. A prolonged milling time of more than 24 h was very effective in reducing the alumina particle size in the suspension and obtaining a stable suspension with a high concentration. A mean particle size of 0.23 \( \mu \)m with a narrow size distribution was measured in the stable suspension. This stable \( \alpha \)-alumina suspension was added to a dilute hydrochloric acid solution (0.2 M) containing \( \text{YCl}_3 \) (Cerac 99.9%) and \( \text{ZrCl}_4 \) (Merck, p.a.). The mixed solution was subsequently precipitated in an excess of \( \text{NH}_4\text{OH} \) (Merck 25%). A gel was obtained, and washed thoroughly with an ammonia/water mixture to remove all chloride and then washed three times with ethanol to remove the residual water. The dried gel was calcined at 550°C for 2 h.

The ZTA powder with a composition of 85 wt% \( \text{Al}_2\text{O}_3–15 \) wt% \( \text{ZrO}_2 \) (10 vol%) was prepared by a gel-precipitation method, where a diluted metal chloride solution was hydrolysed in an excess of \( \text{NH}_4\text{OH} \). This process is described in detail in Ref. 16. The powder used here was calcined at 1100°C for 2 h.

The powders were isostatically compacted at 400 MPa and sintered isothermally at 1400–1550°C for 4 h (heating rate 2°C/min) to obtain a microstructure with different grain sizes. The samples used for sinter forging were pre-sintered at 1100–1200°C (heating and cooling rates: 2°C/min) to a density of about 75% to improve compact strength. The samples were subsequently machined plan-parallel. Sinter forging experiments were performed for 15 min under a constant load corresponding to an initial stress of 40 MPa which is defined with respect to the dimensions just before loading. The sinter forging heating schedule was: heating from ambient temperature to 1150°C at a heating rate of 600°C/h and then at 120°C/h to 1400°C and 1450°C for ADZ and ZTA, respectively. More details of the sinter forging set-up are given in Ref. 16. Densities of green and sintered compacts were measured in Hg by the Archimedes’ technique. All specimens used for wear tests have densities of at least 98% of the theoretical value. The effects of residual porosity on wear characteristics can therefore be eliminated in the present study. The linear intercept technique was used to determine the average grain sizes in two-phase polycrystalline ceramics from SEM photographs as described by Wurst and Nelson.

Friction and wear tests were carried out on a ball (pin)-on-plate wear tester. A polished SiC sphere with a diameter of 4 mm mounted in a copper tube slides reciprocally against an ADZ or ZTA plate in a dry \( \text{N}_2 \) atmosphere (humidity <1%). A normal load of 8 N was used, corresponding to an initial Hertzian contact pressure of 858 MPa. Track length and frequency are resp. 10 mm and 4 Hz corresponding to a maximum sliding velocity of 12.5 \( \times \) \( 10^{-2} \) m/s or an average velocity of 8 \( \times \) \( 10^{-2} \) m/s. The configuration and experimental conditions are described in detail in Ref. 1. Prior to wear experiments all specimens were annealed at 1300°C for 10 min and ultrasonically cleaned in ethanol. After wear testing, the specimens were cleaned again in the same way. The wear volume was calculated from the weight loss, which was measured using an electronic balance (accuracy: 1.0 \( \times \) \( 10^{-5} \) g). The worn surfaces were analysed by a JSM-35CT (JEOL) scanning electron
microscope (SEM). The surface roughness before and after wear testing was examined using a profilometer (Dek-Tak 3030, Slom, Santa Barbara, USA). The phase composition was measured on initial and worn surfaces using an X-ray diffractometer with Cu Kα radiation (Philips PW1710) by continuous scanning of 2θ values of 26–36° with a step size of 0.015°. At each step the intensity was counted for 10 s. The volume fraction (Vm) of monoclinic ZrO2 was calculated by means of Toraya et al.'s method.18 The integrated peak intensities of all (111) reflections were scanned. Intensities were adjusted for the background.

3 Results and Discussion

3.1 ADZ ceramics

The friction coefficient, f, and the wear rate (Kplate and Kpin) after sliding wear are listed in Table 1. Each result is an average of at least three pairs of specimens. The sliding distance for each sample is about 5.8 km. The values of the friction coefficient are obtained when the system is in a steady state after a running-in time of less than 130 min (about 624 metres sliding). The f values do not show significant variations with grain size.

Using a ring-on-block tribometer Zum Gahr et al.20 also indicated that the friction coefficient was independent of grain size for TZP ceramics with the grain size ranging from 0.55 μm to 1.65 μm. The data in Table 1 show that the grain size of the composites obviously has an influence on wear resistance. The wear resistance of the materials increases with decreasing grain size in ADZ composites. The relationship between grain size and wear resistance, however, is a complex issue, since both ZrO2 and Al2O3 grain sizes determine the final results. The grain sizes of both ZrO2 and Al2O3 increase by almost the same factor, so a similar trend in the relationship between wear rate and grain size holds for the composite. It is important to note that the results of ADZ composites presented here are obtained for grain size as the only variable. A Vickers microhardness value of 15 GPa and a three-point bending SENB fracture toughness value of 8 MPa m0.5 are measured for all samples.

In order to investigate the influence of the sinter forging technique applied to ADZ composites, wear tests were carried out on sinter forged specimens (ADZ-S) and compared with results of pressureless sintered specimens (ADZ-I). In both cases, specimens have similar grain sizes of 0.3 and 0.4 μm for ZrO2 and Al2O3, respectively. For the ADZ-S both f and Kplate do not differ from those for pressureless sintered ADZ, within the experimental error. This means that in ADZ composites, the sinter forging process does not improve the wear resistance.

Compared to single-phase Y-TZP the addition of 20 wt% alumina to Y-TZP results in an obvious increase of Vickers' hardness from 13 to 15 GPa. The ADZ with high hardness can hence also have a different wear behaviour too. The wear rate of single-phase Y-TZP with a grain size of 0.3 μm as analysed under identical tribological conditions with the same tribometer is 1.7 × 10⁻⁶ mm³/N m,1 which is about four times higher than that of ADZ (0.40 × 10⁻⁶ mm³/N m; see Table 1).

Figures 1 and 2 show scanning electron micrographs of unworn and worn tracks of ADZ composites with small and large grain sizes. Under the present testing conditions, a mild wear occurs in all ADZ materials. The worn surface of fine-grained ADZ is very smooth with only small amounts of grain pull-out. The wear track of the coarse-grained ADZ became rougher (see Fig. 2(b)), but still no evidence of brittle fracture was present. All wear tracks are characterized by the absence of microfracture. Some wear debris is adhered to the worn surface, especially at the edge between the worn and unworn parts (Figs 1(c) and 2(c)). A pronounced difference in the tangential forces during sliding is observed between ADZ composites and Y-TZP.1 Tested specimens of ADZ scarcely exhibit any microcrack-linked patterns on the worn surfaces or at the turning points. On the other hand, obvious geometric microcrack-linked patterns are easily and often

### Table 1. Friction and wear properties of sliding wear of a SiC ball on ADZ ceramic plates

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Grain size (μm)</th>
<th>ZrO2</th>
<th>Al2O3</th>
<th>Kplate (×10⁻⁴)</th>
<th>Kpin (×10⁻⁴)</th>
<th>Friction coefficient (μ)</th>
<th>m-ZrO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>ADZ-S</td>
<td>0.3</td>
<td>0.4</td>
<td>0.4</td>
<td>0.43 ± 0.04</td>
<td>1.4 ± 0.3</td>
<td>0.37 ± 0.03</td>
<td>0</td>
</tr>
<tr>
<td>ADZ-I</td>
<td>0.3</td>
<td>0.4</td>
<td>0.4</td>
<td>0.40 ± 0.02</td>
<td>1.3 ± 0.1</td>
<td>0.34 ± 0.04</td>
<td>1</td>
</tr>
<tr>
<td>ADZ-II</td>
<td>0.7</td>
<td>0.9</td>
<td>0.9</td>
<td>0.84 ± 0.04</td>
<td>1.5 ± 0.2</td>
<td>0.37 ± 0.04</td>
<td>0</td>
</tr>
<tr>
<td>ADZ-III</td>
<td>0.8</td>
<td>1.1</td>
<td>1.1</td>
<td>1.04 ± 0.06</td>
<td>1.8 ± 0.2</td>
<td>0.35 ± 0.03</td>
<td>&lt;1</td>
</tr>
</tbody>
</table>

ADZ-S: sinter forged specimens at 40 MPa with the same grain sizes as ADZ-I.
found on the worn surface of Y-TZP, which are a response to the tensile stresses which develop behind the rubbing contact resulting in a larger wear rate.

The worn track of sinter forged ADZ-S specimen reveals the same wear mechanism as in ADZ-I. In both cases scanning electron micrographs (Fig. 3) do not show any obvious difference. This observation is in agreement with wear rate results.

3.1.1 General discussion of ADZ
Observation of the wear tracks indicates that the contact surface is smooth for fine-grained ADZ. Hence, the contact forces are distributed over a wide area. A relatively rough surface is observed for coarser-grained ADZ, which is caused by fragmenting and by the pull-out of some larger grains. This type of surface gives higher contact pressures at local asperities resulting in larger tangential stresses behind the trailing edge of rubbing contacts, which consequently causes more wear loss. About 10 vol% of monoclinic zirconia is formed during the wear test of the coarse-grained ADZ (ADZ-III in Table 1). This phase transformation is accompanied by a volume increase for zirconia of 3–5%. Forces accompanied by this volume increase can weaken the grain boundaries resulting in a grain pull-out during wear tests.
As mentioned before, the main wear mechanism for fine-grained (non-transformed) ADZ is mild wear without any brittle fracture or formation of microcracks. For such a wear mechanism, the grain boundaries do not play an important role in the wear process. Therefore sinter forging for improving the grain-boundary strength hardly reveals an improvement in wear resistance in comparison with pressureless sintered specimens.

The difference in wear mechanism between ADZ and Y-TZP\textsuperscript{1} can be explained in terms of the difference in hardness. Using a quasi-static model, Evans \textit{et al.}\textsuperscript{22} established a theoretical treatment of ceramic wear as a function of hardness. They found that the wear rate is inversely proportional to a certain power of the hardness. Fischer \textit{et al.}\textsuperscript{23} however, indicated that the contact area between two ceramic materials is determined either by plastic deformation or, more probably in ceramics, by elastic deformation. The contact area between harder ceramic surfaces will be less than that between relatively softer ceramic surfaces. The contact stresses are therefore larger and, consequently, the tendency for fracture wear is larger. A higher hardness may therefore cause inferior rather than superior wear resistance. As suggested by Fischer \textit{et al.}\textsuperscript{23} this effect is only present for brittle materials in which the wear process
is controlled by brittle fracture. Because of its high fracture toughness (8 MPa√m) ADZ exhibits a ductile behaviour and has a high resistance to microcrack formation. As a result of these properties, the wear of ADZ composites occurs only by polishing and to a lesser extent by grain pull-out. Thus, the effects of hardness of the materials can be different from the case studied by Fischer et al. Wang et al. developed a semi-empirical wear model based on an energy balance and predicted that the wear volume is inversely proportional to the indentation hardness of polycrystalline materials. He et al. also used this model for ZrO$_2$-Al$_2$O$_3$ (5-20 vol% ZrO$_2$) composite materials and found that the higher hardness of the composite materials had beneficial rather than detrimental effects on the wear resistance. These literature results strongly support the results obtained in this study.

### 3.2 ZTA ceramics

Wear tests were performed on ZTA ceramics with various grain sizes which were prepared by sinter forging and pressureless sintering. The friction coefficient ($f_s$) and wear rate ($K$) data are listed in Table 2. The results are given as average values of three or four similar tests. It was found that sinter forged specimens have a wear rate which is a factor of 2-3 lower if compared to pressureless sintered specimens. The friction coefficient values are the same within experimental error. As for the effects of grain size, it can be seen from Table 2 that the coarse-grained ZTA-II reveals a lower wear rate than the fine-grained ZTA-I. After wear testing, a larger amount of tetragonal zirconia in ZTA-II has transformed to the monoclinic phase compared to ZTA-I.

Figure 4 shows SEM pictures of a ZTA-I surface before and after a wear test. The polished surface prior to the wear tests, with a roughness <50 nm, is shown in Fig. 4(a). The zirconia grains (white particles) are distributed homogeneously in the alumina matrix (black particles). After sliding wear, some microcracking perpendicular to the sliding direction and scaling patterns are found at the turning points of the wear tracks as shown in Fig. 4(b). No microcracks exist in the middle of the wear track. A high magnification SEM micrograph of the wear track (Fig. 4(c)) reveals that the wear loss of ZTA-I is caused by a polishing process and accompanying grain pull-out at places where some pits are left. Wear debris fills some local deep pits especially at the edges of the track and is smeared out during the sliding process. No plastic deformation was observed after wear tests.

For ZTA-S specimens, prepared by sinter forging, no microcracking or scaling patterns were observed. In Fig. 5, a high magnification picture of the wear tracks indicates almost the same wear mechanism as that in pressureless sintered ZTA-I. Grain pull-out in ZTA-S is, however, less than in ZTA-I.

In comparison with ZTA-I, the coarse-grained ZTA-II reveals the same wear mechanisms (Fig. 6), but no microcracks are found in the wear tracks. The worn surface of ZTA-II has a higher roughness than of ZTA-I.
Table 2. Friction and wear properties of sliding wear of the SiC ball on ZTA ceramic plates

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Grain size (μm)</th>
<th>ZTA plate K (× 10^5) (mm²/N m)</th>
<th>SiC ball K (× 10^5) (mm²/N m)</th>
<th>Friction coefficient (f)</th>
<th>m-ZrO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZTA-I</td>
<td>0.3 0.8</td>
<td>1.44 ± 0.31</td>
<td>1.1 ± 0.2</td>
<td>0.33 ± 0.05</td>
<td>5 15</td>
</tr>
<tr>
<td>ZTA-I</td>
<td>0.3 0.8</td>
<td>3.40 ± 0.90</td>
<td>1.0 ± 0.2</td>
<td>0.37 ± 0.03</td>
<td>6 14</td>
</tr>
<tr>
<td>ZTA-II</td>
<td>0.5 1.6</td>
<td>1.28 ± 0.34</td>
<td>1.2 ± 0.4</td>
<td>0.36 ± 0.05</td>
<td>22 39</td>
</tr>
</tbody>
</table>

"ZTA sinter forged at 40 MPa with the same grain size as ZTA-I.

3.2.1 General discussion of ZTA

The observation of worn surfaces indicates that polishing and grain pull-out are the main wear mechanisms in ZTA ceramics. Microcracks exist at turning points in pressureless sintered ZTA-I, while such microcracks have not been found in sinter forged ZTA-S. The grain pull-out during the wear process in the ZTA-I is more pronounced than in ZTA-S. The strengthening of grain boundaries by sinter forging prevents

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Fig. 4. Scanning electron micrographs of ZTA-I materials: (a) the polished surface; (b) the worn surface with some cracking and scaling patterns; (c) a high magnification of the worn surface. The arrows indicate one direction of the reciprocal sliding.
may result from compressive stresses at the surface caused by zirconia transformation from tetragonal to monoclinic accompanied by a grain volume increase of 3-5%, while no microcracks are formed because of the alumina matrix with higher Young's modulus than the zirconia second phase. The experimental results show that before wear testing the percentage of monoclinic zirconia which is spontaneously transformed during cooling after annealing is larger in coarse-grained ZTA than in fine-grained ZTA. The constraining force caused by the alumina matrix is in general higher in the bulk than in the surface layer. So the fraction of monoclinic zirconia in the polished surface is higher than that in the bulk. During the sliding process, a certain amount of tetragonal zirconia transforms resulting in an increase in the compressive stress in the surface layer with increasing zirconia grain size yielding an increase in compressive surface stresses. According to Green et al., the compressive stress in the surface layer can be calculated as follows:

\[ \sigma_c = \frac{1}{3} \frac{\Delta V}{V} \frac{EV_v}{(1 - \nu)} \]  

where \( \sigma_c \) is the biaxial compressive stress in the surface layer, \( E \) and \( \nu \) are the Young's modulus (375 GPa) and the Poisson's ratio (0.3), respectively for the alumina matrix, \( \Delta V/V \) is the fractional molar volume increase associated with the phase transformation (3-5%). \( V_v \) is the volume fraction of transformed zirconia, which is the difference between the amount of monoclinic zirconia at the worn surface, \( V_{worn} \), and in the bulk, \( V_b \). The minimum value of \( V_v \) is the volume fraction of zirconia transformed from the tetragonal to monoclinic phase during the sliding wear process (also, during cooling after sintering, some higher amount of monoclinic ZrO₂ might be present at the surface). Because the total amount of ZrO₂ in ZTA is about 10 vol%, the volume fraction \( V_v \) of transformed zirconia in the surface layer corresponds to:

\[ V_v = (V_{worn} - V_b) \times 10 \text{ vol}\% \]  

From eqns (1) and (2), the compressive stresses due to the transformation during sliding wear are calculated to be 57 MPa and 121 MPa for fine-grained ZTA-I and coarse-grained ZTA-II, respectively. The larger the compressive stress, the greater the resistance to crack propagation resulting from tensile stresses, hence leading to a decrease in wear loss. Therefore, it is suggested that the higher wear resistance in the coarse-grained ZTA-II is due to the larger compressive stress layer formed at the surface.
From the discussion given above it can be seen that the tetragonal zirconia transformation in ZTA has a positive effect on the wear resistance. This is different from the results of Y-TZP,\(^1\) where the tetragonal zirconia transformation results in a tensile shear stress underneath the sliding surface. This stress promotes subsurface cracks to propagate along grain boundaries, thereby increasing the wear rate. This different function of the tetragonal zirconia transformation may be related to the amount of zirconia and its situation in different ceramics. For ZTA materials, the zirconia particles are homogeneously dispersed in the alumina matrix, so the transformed zirconia particles are separated by the alumina matrix. The tensile stresses are limited to a local area. For this situation, the tensile stress in the subsurface may not have an obvious effect. Therefore, in case of ZTA materials, the tetragonal zirconia transformation (without induced microcracks) produces a superior rather than inferior wear resistance. The increase in wear resistance of ZTA ceramics due to transformation-toughening mechanisms is also reported by other investigators.\(^6\)

**4 Conclusions**

The effects of grain size and grain-boundary morphology of two zirconia–alumina composites on sliding wear are investigated using a pin-on-plate tribometer under a Hertzian contact pressure of 858 MPa.

During the wear tests of alumina dispersed in Y-TZP (ADZ) the amount of tetragonal zirconia transforming to the monoclinic crystal structure increases with increasing grain size resulting in a decrease in wear resistance. The volume increment caused by this phase transformation weakens the grain boundaries which results in grain pull-out.

In fine-grained ADZ, where no phase transformation occurs during wear tests, a decrease in wear rate of a factor 4 is observed if compared to single-phase Y-TZP with the same zirconia grain size. This improvement can be attributed to the higher hardness of the composite.

In these ADZ composites no wear track shows any microfracture while only a small amount of grain pull-out was observed. Sinter forging, which can improve the grain-boundary strength, therefore has no influence on the tribological properties of these systems.

For composites of \(\text{ZrO}_2\) in an alumina matrix (ZTA) also a larger amount of monoclinic zirconia was observed with increasing grain size but in this case the wear rate decreases. The higher wear resistance for coarser-grained ZTA is due to the larger compressive stress layer formed at the surface, without the formation of any subsurface cracks.

The wear mechanism in these ZTA composites is mainly polishing and grain pull-out, while microcracks arise at the turning points. Here strengthening of the grain boundaries by sinter forging has a positive effect on wear resistance. A decrease in wear rate by a factor of 2–3 is obtained if sinter forging is applied on ZTA.

**References**


