High-speed wear testing of selected ceramics in abrasive slurry

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Abstract. Due to increased production speeds in modern process environments, the loading conditions in sliding contact applications have become more challenging. Therefore it is important to have good understanding of wear mechanisms in high-speed sliding conditions.

A wear testing device was manufactured for the purpose of cost-efficient simulation of water-containing high-speed sliding conditions. Sliding speeds up to 40 meters per second can be achieved while abrasive-containing slurry is fed into the contact interface of material samples and a rubber coated drum. High-speed wear tests were run with three ceramic materials: silicon nitride, silicon carbide and partially stabilized zirconia. Wear behavior of these ceramics was analyzed as a function of sliding speed and slurry composition.

Results indicate that in mild wear region increase in sliding speed reduced overall wear, whereas in severe wear conditions increasing speed accelerated wear. A transition from mild to severe wear was observed for silicon carbide when tested in alumina-containing slurry. For the materials that had high enough hardness, wear rates were dictated by the respective order of fracture toughness. Visual inspection of the worn samples supported the interpretation of wear mode transition.

1. Introduction
Wear and friction exist in all applications that have mechanical contact interfaces and relative motion between surfaces. Wear in its different forms is the single most important reason for the need of constant maintenance and limited lifetime of mechanical components [1]. Increasing efficiency by decreasing lead time is the goal of every modern manufacturing process. For sliding contacts this translates into increasing sliding speeds, and thus increasing the amount of energy brought into the contact interfaces. This results in harsher conditions for materials to operate in. Some processes have already reached speeds exceeding 30 m/s and even higher speeds can be expected in the near future. On the other hand, sliding speeds above 10 m/s are rarely used in conventional wear tests (eg. pin-on-disc, ball-on-disc), therefore the testing data, even though conducted with comparable materials, cannot be used for reliably predicting wear behavior of the respective materials in industrial high-speed applications. Mechanisms of wear in harsh industrial sliding contacts are strongly dictated by the physical and chemical strain directed on the materials. Ultimately, material properties define the exact reaction of a surface towards certain loading parameters.

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Material development for specific industrial applications relies on extensive research and accumulated knowledge of specific conditions. Standard tests, like the ASTM G65 and different pin-on-disk setups offer comparable results for materials in specific conditions but due to the standardized nature of these methods, higher sliding speeds are often overlooked. A radical change in any of the environmental variables in an application produces a need for thorough material testing in those specific conditions. Only accurate simulation can give directions for reliably tailoring material properties to best suit the final application. Understanding wear mechanisms is the key connection between material properties and the environmental conditions.

2. Experimental procedure
A special piece of high-speed sliding equipment was constructed for the purpose of simulating the demanding conditions in modern, water-containing process environments. The basic operating principle of the machine resembles that of the common ASTM G 65 standard sand abrasion wear test, with a few prominent differences. Firstly, water-based kaolin slurry was used instead of dry silica sand. Another deviation from standard test was adjustable sliding speed, up to 40 m/s. The high-speed wear tests were conducted in a concealed environment with closed slurry circulation, due to messy nature of the abrasive medium and high sliding speeds.

Three very different, widely used structural ceramics were tested: silicon nitride (Si₃N₄), silicon carbide (SiC) and partially stabilized zirconia (Mg-PSZ, ZrO₂). It is important to note, that these generic names and chemical compositions apply to many different ceramics that have different tribological behavior. In this paper, however, we only concentrate on the general mechanical properties that are shown in table 1. Zirconia had the highest toughness but the lowest hardness. Silicon carbide was by far the hardest but also the most brittle material. Silicon nitride had sufficiently high hardness combined with better toughness properties than the carbide. Fracture toughness values were determined by Vickers indentation method, which is currently considered controversial method for that particular purpose [2,3]. In this study, however, materials have very large differences in properties and thus the simplified method was found useful.

| Material             | Hardness [HV₁] | Fracture toughness [MPa√m]ᵃ | Density [g/cm³] | E [GPa] |
|----------------------|----------------|-----------------------------|-----------------|---------|
| Si₃N₄ (tetragonal)   | 1670           | 4.60                        | 3.44            | 300     |
| SiC (tetragonal)     | 2900           | 3.40                        | 3.21            | 400     |
| PSZ (ZrO₂, tetragonal and monoclinic) | 1100 | 10.7                        | 5.68            | 190     |

ᵃVickers indentation fracture toughness test.

Understanding friction and wear in a process requires detailed knowledge of all the materials involved in the process. In this study the abrasive medium was delivered into the contact interface in form of abrasive slurry. The main abrasive used in these tests was ordinary, non-calcinated kaolin powder, which is one of the most common minerals used in industrial applications. The particle size was in the region of 0.1 to 2 microns. Kaolin is, in its non-calcinated state, very soft material with Mohs hardness in the region of 2…2.5. The characteristic lamellar shape of particles and the small particle size make kaolin powder a very mild abrasive. Another abrasive material used in high-speed sliding wear tests was coarse grained alumina (97Al₂O₃ + 3TiO₂) spraying powder. With boulder-like particle geometry and much higher hardness than that of kaolin, 9 on Mohs scale, this abrasive material was used for increasing the wear rates to a level where differences in wear behavior could be observed more reliably. The particle size of this powder was between 5 and 22 microns. The vast differences between the morphologies and particle size of the two abrasive powders can be seen in the figure 1.
Figure 1: SE-images of the two abrasive powders used for wear testing. Kaolin mineral (a) and alumina spraying powder (b). Note the identical scale bars.

Two different water-based slurry compositions were used: One with 10 wt% kaolin powder and another with 10 wt% kaolin and additional 5 wt% alumina spraying powder. Being a coarse powder, alumina did not mix very well with water. Using kaolin in both mixtures ensured roughly similar appearance and viscosity of the slurry. An adequate accuracy of composition was achieved with automatic water filling mechanism in the wear testing device. Some variation in slurry composition during wear testing is inevitable due to tendency of kaolin to deposit on the walls of the wear testing equipment.

The main measurable results acquired from the wear tests are the wear volumes calculated using mass losses of the samples and the known densities of the materials. Sample preparation preceding the weighing process was a very simple procedure where ceramic sample blocks were glued to AISI 316L stainless steel with water proof epoxy resin. After the resin had cured and all excess epoxy was removed, the samples were washed, cleaned ultrasonically and dried. The same cleaning procedure was conducted after the wear tests as well to ensure proper removal of kaolin residues and thus providing reliable weighing results. Characterization of the worn sample surfaces was conducted using scanning electron microscopy and digital photography.

3. Results and Discussion

Wear volumes divided by normal force and sliding distance translate to specific wear, which in some instances is also called severity of wear [4]. Figures 2 and 3 show a significant difference between the two slurry compositions used in the wear tests. In kaolin slurry, the specific wear of all three ceramics decreased when sliding velocity was increased. This is either an indication of running-in wear or possible evidence of the hydroplaning effect present in higher sliding velocities [5]. Adding alumina reversed the effect, so that the specific wear increased when sliding velocity was increased. Wear volume per unit of load and sliding distance increased with greater sliding velocity, which suggests the absence of running-in phenomenon. The respective order of materials by specific wear remained the same until the fourth test with hard abrasive slurry and high sliding velocity. In these demanding testing conditions silicon carbide experienced a noticeable increase in specific wear value.
Scanning electron microscope images show similarities in the fully worn surfaces (figure 4) of silicon nitride and silicon carbide. There are signs of pores acting as wear initiation points, so the high porosity of partially stabilized zirconia may have had a negative effect on its wear behavior.

Figure 5 shows a worn area of a SiC-sample where remnant of initial groove from sample preparation process can be seen. Grains of the bulk silicon carbide can be seen inside a groove not yet worn, surrounded by smoothly worn areas. Smoothed area is likely caused by trans-granular fracture mechanism associated with subsurface cracking [6–8] phenomenon combined with possible re-compacting of wear particles [9]. Severity of crack initiation and propagation is dependent on cyclic loading conditions caused by friction forces and hard particles passing the contact interface. Assuming that the primary mechanisms of wear are abrasion and subsurface intergranular cracking, we can conclude that surface smoothness is an indication of trans-granular crack propagation.

Figure 5. Close inspection of worn SiC surface still in the running-in stage of wear.
Visual inspection of the worn surfaces presented in figure 6 clearly shows that silicon nitride gains a smooth, shiny surface with all of the wear testing parameters. Increase in specific wear of silicon carbide seen in the wear test data is also evident from the digital images. By definition, mild wear results in surface roughness smaller than grain size which in turn leads to polished appearance of the sample surface. Mild wear also often includes different stages of oxidation and delamination, which inherently result in a lower wear rate due to the reforming cycle of the protective oxide layer [10]. Rough surface in turn is usually connected to severe wear mode, where trans-granular fracturing is the dominant mechanism of material removal. Other theories suggest, however, that smooth wear track does not always mean smaller (abrasive) wear rate than with rough surface [11]. We can eliminate this possibility by looking at the extremely low wear rates encountered in the conditions producing smooth surfaces.

| Sliding speed [m/min] | Slurry composition          |
|----------------------|-----------------------------|
| 500                  | Soft (10% kaolin)           |
| 2000                 | Soft (10% kaolin)           |
| 500                  | Hard (10% kaolin with 5% added alumina) |
| 2000                 | Hard (10% kaolin with 5% added alumina) |

**Figure 6.** Photographs of samples after the wear tests.

Different development in wear behavior when sliding speed is increased from 500 m/min to 2000 m/min was observed. Silicon nitride wore very little with all slurry compositions and sliding velocities, which can be mostly attributed to its high hardness and adequate fracture toughness. Silicon carbide experienced a sudden deterioration when tested with hard abrasive slurry in 2000 m/min speed. SE-image of the silicon carbide sample 3 (alumina-containing slurry, 500 m/min) show signs of mild wear mode: polishing caused by trans-granular fracture propagation and possible tribochemical reactions, since water is known to react with all of the tested materials [5,12]. However, evidence of any oxide layers inherent to tribochemistry was not found.

In this study, low wear rates in the soft abrasive slurry can be partially accounted to running-in wear phenomenon, since there is noticeable smoothing on all of the worn ceramic surfaces. Due to uninterruptible nature of the test configuration, periodic weighing was not possible.

Wear resistance of partially stabilized zirconia was the lowest in all wear tests, but the results were very consistent and predictable, as opposed to the silicon carbide. The apparent reason for the most wear of zirconia is the low hardness of the material. High fracture toughness is solely not enough in this kind of loading situations. Hardness above a certain threshold is necessary for resisting scratching and thus material removal in abrasive wear conditions.
4. Conclusions
Three main observations can be drawn out from the results and discussion shown above:

- The results acquired from the high-speed sliding wear tests conducted so far are encouragingly consistent. This new method can now be considered viable for extreme wear testing and even simulating conditions occurring in the industrial high-speed sliding applications.
- The well-known concept regarding severity of wear is feasible in analyzing wear behavior of different materials in our specific high-speed sliding testing environment. The characteristic smooth appearance of mild wear mode was lost with increased sliding speed for silicon carbide in hard abrasive slurry. This combined with significant increase in wear rate was interpreted as a transformation from mild to severe wear mode.
- The existence of critical threshold values for both hardness and fracture toughness is proposed for a material to remain in the mild wear region in extreme high-speed sliding applications. In this study, silicon nitride had the best combination of these two properties, which was apparent from its stable wear behavior and polished wear track that is characteristic to mild wear mode.

Further studies with more parameter variations need to be conducted to get more detailed information about the specific set of parameters needed to push silicon carbide from mild to severe wear mode. On the other hand, severe wear mode for silicon nitride is yet to be found with this specific wear testing method.

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