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Original Research Article

Measurement of chemical and geometrical surface changes in a wear track by a confocal height sensor and confocal Raman spectroscopy

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ABSTRACT

Geometrical and chemical changes in the wear track can cause a drift in friction level. In this paper, chemical and geometrical surface changes in wear tracks are analyzed. For this, a setup with a confocal height sensor was developed to measure the local height changes on the wear track, combined with confocal Raman spectroscopy to determine the chemical changes at the surfaces. Pin-on-disc experiments were performed at room temperature and at elevated temperature (600 °C) to understand the material behavior between mild and severe wear regimes. The wear tracks developing between the two ceramics, alumina (Al_2O_3) and zirconia (Y-TZP), were analyzed using these techniques. The results of confocal height sensor showed significantly more geometrical changes in surface roughness at 600 °C compared to the test conducted at room temperature. The developed roughness in the wear track was approximately 250 times larger at 600 °C due to the higher degradation of the mechanical properties of ceramic. Further, material transfer was observed for the test conducted at 600 °C using Raman Spectroscopy. Material transfer at room temperature is difficult to observe because surface changes are less evident in mild wear regimes. The results show that the changes in the micro-geometry of the surface and the chemical compositions of the surface influence the friction level and wear processes. The confocal height sensor and Raman Spectroscopy were used to measure and understand the geometrical and chemical changes occurring on the surface of a wear track during sliding in a single setup.

1. Introduction

Friction and wear play an important role in the effective functioning of mechatronic systems where a precise positioning accuracy over a long working life is desired. The structure of two sliding surfaces, the operating variables and mechanical or chemical interaction of the components in the system influence friction and wear [1,2].

Ceramics are used in many applications, due to their low thermal conductivity and superior mechanical properties, chemical and electrical resistance. Alumina (Al_2O_3) and zirconia (ZrO_2) are widely used in industry as insulators in power transmission systems, optics, journal bearings, MEMS systems and so on [3].

Various measuring techniques are available to investigate the wear between ceramics. Optical methods based on light

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scattering are widely used to study the change in the surface topography [4,5]. Confocal microscopy can be used to analyze the micro-geometry of surfaces [6,7]. Miyake et al. [8] used confocal laser microscopy to determine wear by observing profiles of microgrooves in Al–Al_2O_3 composite surface. The advantages of using confocal microscopy are high spatial resolution together with a high accuracy and the possibility to be applied for different kinds of materials. In a confocal chromatic optical setup, the chromatic light is used to get complete parallelization of the depth scan, so the rough surfaces within the measuring range are measured [4,9].

Atomic force microscopy (AFM), energy dispersive microscopy (EDS) and X-ray photoelectron spectrometry (XPS) are common techniques to measure the presence of elements on the surface [10,11]. Sample pre-preparation is required for some of the mentioned techniques, which is a disadvantage to study the wear and surface changes. During the measurement there is a possibility of damaging the surface due to the test conditions. Raman spectroscopy is a non-intrusive technique, where sample preparation is not necessary. Confocal Raman spectroscopy is more suitable for ceramics, because of high spatial resolution and reduced fluorescence effect [11–13]. Many studies have been done on zirconia [14–16] and alumina [17,18] using Raman spectroscopy. For example, the structural phase transition of an alumina (Al_2O_3) was observed by Cava et al. [19]. The ability of the Raman spectroscopy to detect phase changes in the contact occurring due to mechanical stresses was demonstrated by authors [15–19].

The present research work in this paper focuses on the analysis of the wear track made by alumina (Al_2O_3) on zirconia (ZrO_2) at temperatures of 25 °C and 600 °C. A comparison between mild and severe wear regimes [12,15] indicates that at 600 °C material transfer is more evident between Al_2O_3 ball and ZrO_2 plate. In order to do this, a measurement setup composed of a fiber optics based confocal height sensor and a confocal Raman spectroscopy sensor has been developed. The latter was used to investigate the molecular composition of the wear track. The advantage of the setup is to measure the surface properties in a wear track. Further, it can be used in combination with tribological testers for an in-situ measurement. Wear tracks on ceramic contacts that have been operating respectively in the mild and severe wear regimes [20–22] will be analyzed and the surface changes will be compared.

2. Pin on disc tests

Friction measurements were carried out using a high temperature pin-on-disc tribometer at room temperature and at 600 °C. An alumina (Al_2O_3) ball with a diameter of 10 mm was sliding against a zirconia (Y-TZP) plate. A normal load of 5 N was applied with a sliding velocity of 0.1 m/s and a sliding distance of 1 km. Information of the sample preparation is described in our previous publication [10,23]. The properties of materials used in this study are summarized in Table 1.

The coefficient of friction measured as a function of sliding distance is shown in Fig. 1 for two temperature conditions. In both the cases, the transition from the initial to the steady state coefficient of friction was observed at the beginning of experiment. The average coefficient of friction obtained at room temperature was found to be 0.5 and 0.8 at 600 °C.

The difference in coefficient of friction is due to mechanical properties of ceramics and wear behavior described by Adachi et al. [20] and Wang et al. [24]. At high temperature conditions severe wear takes place and friction level is higher as compared to room temperature.

3. Surface measurement setup

A photograph of the experimental setup is shown in Fig. 2(a), and in Fig. 2(b) the schematic is shown.

The confocal height sensor (STIL CL1-MG210) is connected through an optical fiber to a controller (CCS Prima). The chromatic light from the controller is measuring the local surface height with a working distance of 3.3 mm and a nominal measuring range of 100 μm. The spot diameter of the sensor is 2 μm and the height resolution is 5 nm. Data acquisition is done by a computer. The Raman setup is composed of a laser (Ventus Laser) with wavelength 532 nm and power 50 mW. The green light from the laser is transmitted through optical fibers to a sensor (Horiba Super Head-532) with an objective lens of 50× (N.A. 0.5). Scattered light from the sample coming back through the sensor is sent to a

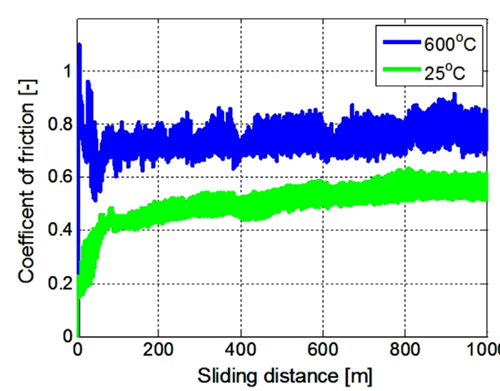

Fig. 1 – Friction level of 3Y-TZP sliding against Al_2O_3 at room temperature and at 600 °C.

Table 1 – Properties of material used in testing [22].

Material	Hardness (GPa)	Elastic modulus (GPa)	Bending strength (MPa)	Fracture toughness (MPa $m^{1/2}$)	Grain size (μm)	CLA surface roughness (nm)
Alumina	20±0.8	320	340	3.4±0.4	5	15
3Y-TZP	13±0.4	210	450±10	4.2±0.2	0.45	25

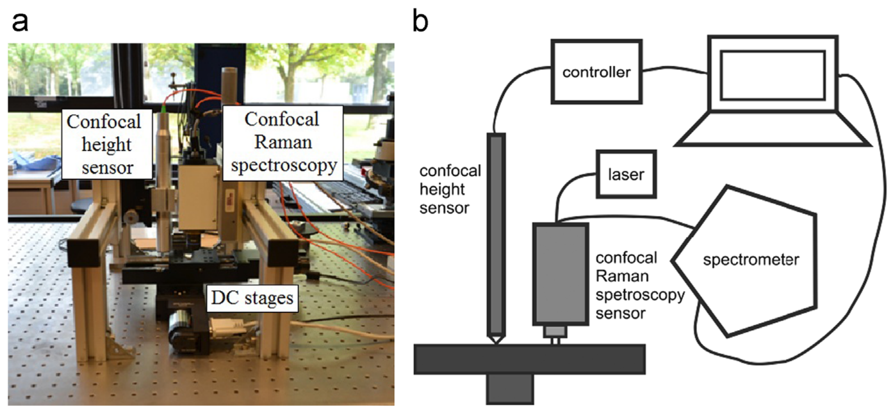

Fig. 2 – Confocal height sensor and confocal Raman spectroscopy setup (a) picture and (b) schematic layout.

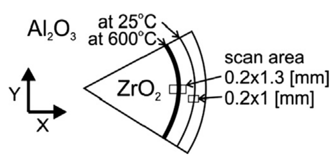

Fig. 3 – Area analyzed on the disc sample.

spectrometer (iHR320) by fiber optics. The measured spectrum is continuously monitored and recorded by the computer. The linear translation stages with the DC motor are moving the sample in two directions (*X* and *Y*) for measurement. The accuracy of the stages is 1 μm. A Labview program was written and used to control the system and record the data.

4. Results

The schematic representation of the areas studied from the worn sample is shown in Fig. 3.

The area measured by the confocal height sensor across the wear track at room temperature is 0.2 mm in X direction and 1 mm in Y direction. For high temperature an area of 0.2 mm in X and 1.3 mm in Y direction is measured due to severe wear.

The result of the surface height measurements obtained with the confocal height sensor is presented in Fig. 4, where the overall view of the scanned area is visible as a 3D image. The wear track made at room temperature and at high temperature is shown in Fig. 4(a) and (b).

The profile across the wear track for images in Fig. 4 is represented in Fig. 5(a) and (b) respectively for room temperature and at 600 °C. The horizontal axis corresponds to measured points across wear track and the vertical axis represents the height changes.

The peaks with various heights were observed in the wear track due to wear process, which is visible in Figs. 4 and 5. These peaks suggest also that material from counter surface was transferred to the disc.

At room temperature the wear track is significantly smaller in size as compared to that at 600 °C where the contact is operating under severe wear conditions [22].

Material transfer observed in the profile of the wear track was confirmed by the confocal Raman spectroscopy. Measurements were conducted at three locations on the disc, one outside the wear tracks and one for each wear track. The wear track across the disc at room temperature is very narrow of approximately 200 μm. In this wear track no material transfer was observed. The results obtained for the disc at 600 °C are presented in Fig. 6.

Fig. 6 shows the measured light intensity against the Raman shift outside and inside the wear track. Outside the wear track only Raman shifts of zirconia are visible at 141, 253, 462, 638, 2439 cm^{-1} [16]. Inside the wear track besides zirconia, an alumina bond can be observed at 4436 cm^{-1} for high temperature conditions [18]. The intensity and amount of alumina bonds are lower compared to zirconia, which indicates that only small amounts of Al_2O_3 are found in the wear track. The wear track tested under room temperature conditions does not show significant changes in the spectrum as compared to the spectrum measured outside the wear track. At room temperature the hardness of the ceramics is higher. Further, the changes in the surface roughness at room temperature are lower than at 600 °C.

The ball used in the high temperature test was also measured at two spots, one in the wear scar and one outside the wear scar. The Raman shifts obtained for alumina ball at two locations are presented in Fig. 7.

The Raman shifts for alumina bonds outside and on the wear scar of the ball are visible at 4079, 4393, 4689 and 4822 cm^{-1}. In the wear scar, a zirconia transfer layer after the wear test was measured. The formation of a transfer layer during sliding of ceramics has been reported in literature [10,25]. The shifts at 155, 278, 477, 642 and 2428 cm^{-1} represent zirconia bonds. When compared to the zirconia disc, those shifts have different values and correspond to the monoclinic phase of zirconia. It can be concluded that zirconia during the wear process has changed phase from a tetragonal to a monoclinic phase. The tetragonal to monoclinic transformation is ascribed to the presence of thermal stresses due to the sliding contact and poor thermal conductivity of zirconia [26]. Further, on the ball organic substances were found at 1469 and 1772 cm^{-1}.

5. Summary and conclusion

A confocal height sensor and a confocal Raman spectroscopy measurement setup were used to investigate the wear track

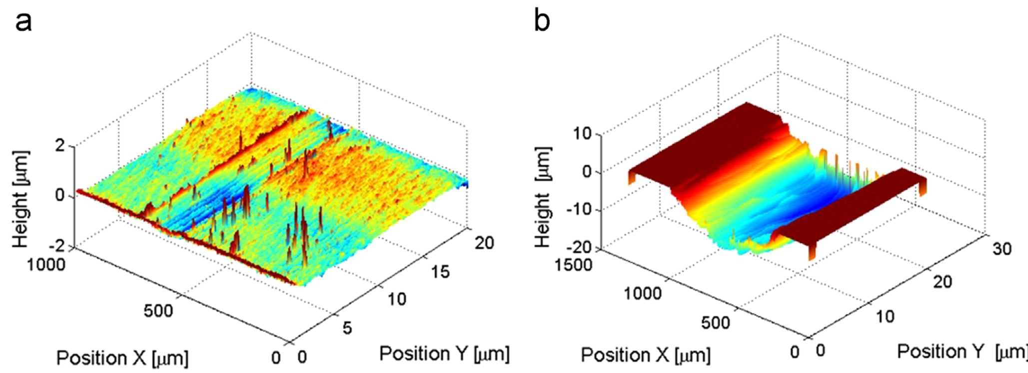

Fig. 4 – Confocal height sensor image in 3D view of wear tracks. (a) 3D view of disc at room temperature and (b) 3D view wear track at high temperature.

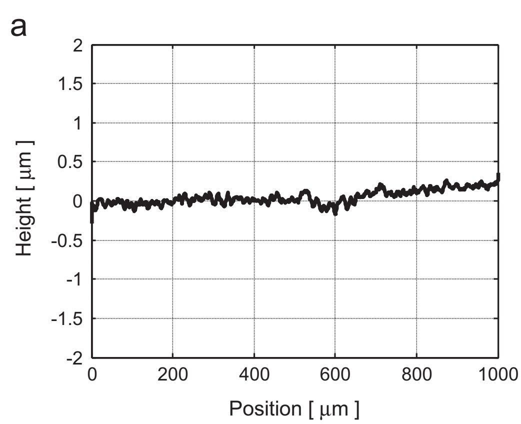

Fig. 5 – The surface height profile across the wear tracks. a) profile across the wear track at room temperature and b) profile across the wear track at high temperature.

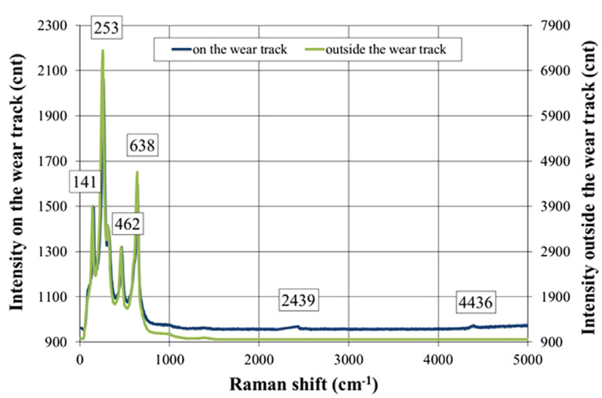

Fig. 6 – Raman spectrum outside and inside the wear track of the zirconia disc.

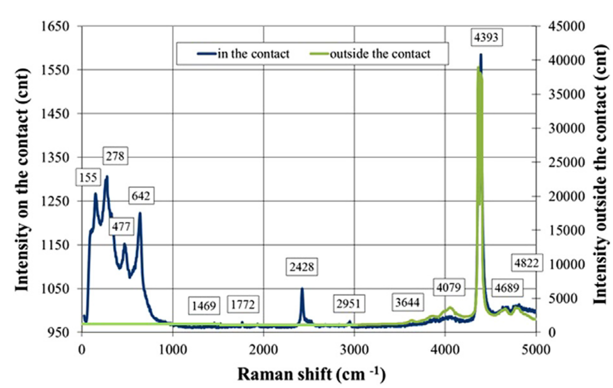

Fig. 7 – Raman spectrum of alumina ball inside the wear scar and outside the wear scar.

generated on a ceramic sample at room temperature and at 600 °C. The geometrical changes were measured by the confocal height sensor. The results clearly show the surface topography changes in and outside the wear track. The difference of mild and severe wear regimes due to temperature conditions was observed by comparing the surface roughness. At room temperature, where mild wear takes place the resulting wear track is narrow compared to the wear track obtained at 600 °C. The coefficient of friction was significantly lower at room temperature than in high temperature conditions. The confocal Raman spectroscopy shows chemical changes in the wear tracks of both the disc and the ball. The measured material compositions inside and outside the wear track are different, which indicates that material transfer occurred during the wear process. The transfer was observed from the pin to the disc as well as from the disc to the pin. Material transfer has already been reported to occur in many ceramics [27]. With the increase in temperature, the hardness of ceramics decreases. Generally, materials with larger expansion coefficient tend to soften with increasing temperature [24]. Based on our observation at 600 °C the decrease in the mechanical properties resulted in more material transfer when compared to room temperature conditions under sliding test. During material transfer between alumina and zirconia, a phase transformation from tetragonal to monoclinic was observed in case of zirconia after the wear tests. The wear debris structure changes indicate that during wear process in high temperature the particles of Y-TZP can transform to structure of monoclinic ZrO_2. Confocal Raman spectroscopy was successfully used to investigate the wear tracks and formation of a transfer layer in the wear track. The measurement system can be used in future to analyze composite layers of ceramics like for example in self-lubricating composites.

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