Tribological Properties of Nano-Sized ZrO$_2$ Ceramic Particles in Automotive Lubricants

The demand for decreasing CO$_2$-emission and harmful material content of the exhaust gas of passenger cars requires the improvement of the entire powertrain including the applied lubricants. One of the possible future engine lubricants can be the nano-sized ceramic particles, which can provide positive tribological properties also in the presence of non-metallic surface materials. This paper presents the experimental investigation of ZrO$_2$ nanoceramic powder as a lubricant additive. The tribological performance of the lubricant samples was experimentally investigated on a ball-on-disc translation tribometer. An optimum concentration was found at 0.4 wt%, where the wear scar diameter on the ball specimen was reduced by more than 40% compared to the reference sample. The SEM-analysis confirmed the mending mechanism theory: nanoparticles were revealed to aggregate between the asperities resulting in a significantly smoother contact surface.

Keywords: tribology, zirconium-dioxide, nano-ceramic, lubricant, additive, engine.

1. INTRODUCTION

The emission regulations in Europe are becoming continuously strict, which requires the development of the engine and vehicle component even further. One of the most important development - fields is the reduction of different losses in the internal combustion engines, e.g. thermal and mechanical losses including frictional losses. During the last years, engineers and researchers have elaborated several solutions to increase the efficiency of the engines like using low-viscosity lubricants, low-friction coatings, or optimization of the mechanical loads of components. To take advantage of the maximum potential of these solutions, they have to be suited together.

Lubricants are one of the most important parts of the piston engines. Their roles are to separate the rigid surfaces, prevent them from wear and corrosion and decrease the frictional losses. To fulfil these challenges, modern engine lubricants have to be applied together with different additives. Lots of lubricant additive types can provide their improving effects due to polarity difference and they can form a protective nano-scale layer on the rubbing surfaces. In the last years, the compatibility of the lubricant additives and the exhaust gas after-treatment systems became vital to the harmful emission products. The additives containing e.g. phosphor or zinc can form ash during their burning process sealing the channels in the particle filters, or they can cause damages in the catalyst systems of the vehicles.

These tribological losses will lead to different energy losses and failures in the mechanical systems due to friction and wear, and these losses can strongly influence the fuel consumption and component lifetimes of the engines and vehicles. Different statistics are presenting that one third of the world’s primary energy consumption is directly connected with the friction, and the wear is responsible for 60% of the failures in mechanical engineering. Besides the lubrication failure can cause more than 50% of accidents involving machinery equipment [1].

One of the possible future lubricant additives can be nanoparticles. These particles can be made of a huge variety of materials. According to their material, they can provide quite different properties. However, this huge amount of variety requires research activities to characterise their properties and their optimal mixing concentration in different types of lubricants. These types of particles can have lots of influence factors regarding tribology, e.g. particle size, material, the property of building agglomerations, concentration, or attendance of surface-active solving agents. To determine these influence factors, wide research activities have to be carried out.

In this paper, the usability of selected zirconium-dioxide nano-scaled ceramic particles will be summarised and discussed as lubricant additives.

2. NANO-SCALED LUBRICANT ADDITIVES

ISO/TS 80004 standard defines nanoparticle as a small object of a bulk material that behaves as a whole unit with three external dimensions in the nanoscale, whose longest and shortest axes do not differ more than a factor of 3. Nanoparticles are solid particles ranged between 1 and 100 nm in size [2]. Around the particle, there is a surrounding interfacial layer, which fundamentally affects most of the particle’s physical and chemical properties. This interfacial layer generally consists of ionic groups or other molecules. Because of this
layer nanoparticles can exhibit size-related properties significantly different from either fine particles or bulk materials [3].

To apply this kind of nanoparticles as lubricant additives, some key preparation steps are crucial: homogenisation and mixing the particles into the fluid and the selection of the proper material, size and concentration. These key factors have to be experimentally investigated before wide application in passenger cars.

The average particle diameter of these additive types can influence their tribological properties drastically: L. Pena-Parás et al. [4] have investigated the influence factors of different particle sizes of TiO$_2$ nanoparticles. They have discovered that only particles with initial diameters smaller than the average surface roughness can decrease the friction and wear. However, if the particles are initially too small, the particles can agglomerate to form much larger micron-scale clusters, such that the friction and wear cannot continue to decrease.

Different working mechanisms were reported in the literature to describe the investigated results. Zhang et al. [5] summarized the different mechanisms for nanoparticles, which can be seen in Figure 1:

a) Rolling (or ball bearing) mechanism: the nanoparticles will work as nano-scale balls to roll between the rubbing surfaces and change the sliding friction to sliding-rolling one.
b) Mending mechanism: the nanoparticles can be collected in the grooves of the rubbing surfaces resulting in a smoother contact surface.
c) Polishing mechanism: the nanoparticles will polish the roughness peaks of the surface leading to a smoother surface, which reduce the running-in phase of the rubbing surfaces.
d) Protective film mechanism: the nanoparticles can attach to the rubbing surfaces (e.g. with secondary bindings or via polarity difference) and they can form a protective tribofilm between the sliding surfaces.

Zirconium dioxide (ZrO$_2$, also named as zirconia) is the white crystalline oxide of zirconium transition metal. Generally, zirconia is produced by the calcination (heating to high temperature in oxygen-containing medium to earn thermal decomposition) of zirconium containing compounds and ores. Zirconia occurs naturally in its mineral, known as baddeleyite. Normally zirconia occurs with a monoclinic crystalline structure, but this structure varies with temperature. Three different phases are known depending on its temperature. Zirconia has a monoclinic structure under 1170 °C, tetragonal between 1170 °C and 2370 °C, and cubic above 2370 °C in atmospheric pressure as shown in Figure 2. As the temperature rises, the degree of symmetry in the crystalline structure increases.

Zirconia has high chemical resistance, even the strong concentrated acids can barely attack zirconia. Zirconia has strong corrosion-resistant properties without the typical brittleness of technical ceramics. Compared to other common and advanced ceramics zirconia has exceptional physical strength and mechanical properties at room temperature. The density of zirconia is between 5.68 and 6.08 kg/m$^3$, depending on its crystal structure. Zirconia has a higher hardness (~1200 HV), bending (900-1200 MPa) and compressive strength (2000 MPa) compared to steels, with lower fracture toughness (7-10 MPa.m$^{1/2}$) and similar Young’s modulus (~210 GPa) and thermal expansion coefficient (11*10$^{-6}$/K). Stabilizing zirconia increases its low fracture toughness. Because of these prominent properties, zirconia is one of the most studied engineering ceramic materials.

Zirconia is in the focus of research in lubricant nanoadditives. The effect of different zirconia nanoparticles on thermophysical characteristics and rheological properties of newly developed synthetic oils were investigated with various methods [8],[9].

Tribological properties of zirconia nanoparticles were investigated in many aspects. Frictional losses are the main sources of waste energy in mechanical systems. To reduce friction, it is crucial to investigate the tribological properties of zirconia doped lubricants. In recent years, numerous articles reported the friction decreasing effect of zirconia nanoparticles from 5 to 28% [10]. With the development of zirconia nanoparticles in recent years, attempts were made to improve the properties of nanoparticles as lubricant additives. Friction decreasing properties can be reduced more (by 27-45%) if the zirconia is surface activated [10],[11]. Zirconia-composite nanoparticles can reduce friction by around 16% [12]. A previous study clarifies the self-
lubricating mechanism of the zirconia-composite materials with different oxide ceramics, therefore the decreasing in the coefficient of dry friction. ZrO₂/CuO composite resulted in the best in friction decreasing by 40% [13]. Some papers reported about 20-30 nm diameter zirconia nanoparticles coated with 15 wt% alumina by sol-gel method. Microtribological experiments on very small loads (0.03-0.1 N) resulted in 20-63% decrease in friction, compared to the reference nanoparticles (Al₂O₃/Al₂O₃) [14].

Lifetime and wear rate are coherent parameters. Reducing the wear results longer lifetime for the parts. Wear modifying effects of zirconia nanoparticles were investigated in many articles. Wear properties are still ambiguous, papers report wear modifying effects of zirconia from 37% decrease to 66% increase in wear rate [10],[11]. Multiaxially loaded cyclopentanes can support zirconia in tribological processes. A hard, protective ceramic-like tribofilm was formed on the surfaces prevented severe wear, seizure and damped the tribological impacts [15]. Zirconia doped lubricant showed lower wear rates and enabled to increase the maximum contact pressure (extreme pressure) compared to the reference. Zirconia tolerates the high pressure (over 400 MPa) and pressure (extreme pressure) compared to the reference. Wear properties are still ambiguous, papers report wear modifying effects of zirconia from 37% decrease to 66% increase in wear rate [10],[11]. Multiaxially loaded cyclopentanes can support zirconia in tribological processes. A hard, protective ceramic-like tribofilm was formed on the surfaces prevented severe wear, seizure and damped the tribological impacts [15]. Zirconia doped lubricant showed lower wear rates and enabled to increase the maximum contact pressure (extreme pressure) compared to the reference. Zirconia tolerates the high pressure (over 400 MPa) and pressure (extreme pressure) compared to the reference. Wear properties are still ambiguous, papers report wear modifying effects of zirconia from 37% decrease to 66% increase in wear rate [10],[11]. Multiaxially loaded cyclopentanes can support zirconia in tribological processes. A hard, protective ceramic-like tribofilm was formed on the surfaces prevented severe wear, seizure and damped the tribological impacts [15].

3. INVESTIGATION METHODS

Before a proper engine lubricant including nano-additives will be able to be used in an operating engine, wide investigations of the tribological properties are necessary. To carry out these research efficient, essential measurements with simple testing specimens have to be used.

Most scientific papers describe the following investigation method to characterise different lubricant samples and during the experiments these were also used:

- Preparation of the lubricant-additive mixture
- Execution of the tribological properties with selected testing specimens and investigation method
- Analysis of the wear on the contacting surfaces with different microscopes
- Evaluation and explanation of the results (frictional losses, wear parameters, working mechanisms)

The used homogenisation and mixing procedures can be divided into two main groups: one-step and two-step methods. The two-step procedure separates the production of the additive and the distribution into the liquid lubricant. With this method, several possibilities are existing for the preparation of lubricant-additive samples, even to produce a sample with multiple types of additives.

The used homogenisation procedure contained 3 minutes of magnetic stirrer mixing step with the revolution of 1000 rpm, and 30 minutes of ultrasonic homogenisation on the temperature of 50 °C. The lubricant sample was stirred magnetically again until the lubrication pipes were filled up with the sample.

For the investigations, neat Group 3 base oil was used without any added additive. This kind of base oil is widely used to produce low-viscosity lubricants for passenger cars. The results made with this oil was used as the basis of the nanoparticle-added samples. To characterise the tribological properties of the selected ZrO₂ nanoparticle, 6 lubricant samples with different nanoparticle concentration were prepared with the presented homogenisation method.

The experimental investigation was made with an Optimol SRV5 tribometer. This machine enables to continuously measure the friction coefficient of the analysed tribo-system. The tribometer provides an oscillating movement with sine velocity pattern. The rubbing specimens were lubricated via the peristaltic oil pump of the tribometer. The setup of the tribometer and the used lubrication system can be seen in Figure 3.

For the experiments standardised testing specimens were used: a ball specimen with diameter of 10 mm and a disc specimen with diameter of 24 mm and height of 7.9 mm. The specimens are standardised in the norm ISO 19291:2016 [21].

For the measurement, a self-developed testing program was used. The specimens were moved with a 1 mm stroke and 50 Hz frequency translation movement pattern. Continuous oil flow was realised during the experiments with the oil flow rate of 225 ml/h. Both the specimens and the lubricant sample were heated up to 100 °C. The 30 seconds low-load phase (50 N) was followed by a high-load step (100 N) with the time duration of 2 hours.

The tribometer can record two different friction coefficient values during the whole measurements with high recording frequency.

- COF (coefficient of friction) value: the maximum friction coefficient value during one stroke, which arises usually from the dead centres of the oscillation movement, representing the property of the system under boundary lubrication condition.
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- FAI (friction absolute integral) value: an average value calculated from the area under the friction coefficient values recorded with high frequency, representing the property of the system under hydrodynamic lubrication regimes.

The surfaces of the worn specimens were analysed via digital (Keyence VHX-1000), confocal (Leica DCM 3D) and scanning electron microscopes (HIROX SEM-4000M).

- Analysis with a digital microscope: The dimensions of the worn surface picture can be defined with this microscope. The measured dimensions (WSD: wear scar diameter on the ball specimen) will be used to compare different lubricant sample variations. Besides, the signs of the wear mechanisms can also be analysed.

- Confocal microscope analysis: This type of microscope enables to scan the whole worn surface and its surrounding areas. This scanned 3-dimensional surface can be analysed to define other wear parameters, e.g. wear depth, wear volume, surface roughness.

- Scanning electron microscope with EDX sensor: The scanning electron microscope can produce pictures about the surfaces with higher magnitude, compared with the light microscopes. With the help of these high-magnitude pictures, the proper wear mechanisms can be defined. To add an EDX sensor to the measurements the proper material composition of the surface can be determined.

### 4. EXPERIMENTAL RESULTS

The measurement equipment is able to record the selected measurement values (like friction coefficient values) in every second during the experiment. As it can see in Figure 4, the friction coefficient values can be shifted around and the results have to be evaluated: the friction coefficient values from the end of the measurements are taken as comparison values.

![Figure 4. The time dependence of the recorded friction coefficient values during the tribometer measurements](image)

With every concentration, at least 3 independent measurements were carried out for the statistical evaluation and the results of each experiment were compared to determine the optimum concentration. As a comparison basis, the results with neat Group 3 base oil was used. After each measurement, the wear scar on the ball specimen was recorded via a digital microscope. This type of microscopes also enables to measure the wear scar diameter on the specimen.

To analyse the tribological effects of the nano-ceramic particles, the results of the chosen reference are crucial. Neat Group 3 base oil (provided by MOL-LUB Kft.) was chosen as a reference oil sample and its tribological properties were widely analysed with the described measurement method. The recorded digital microscope image of the applied specimen can be observed in Figure 5.

![Figure 5. Digital microscope image of the disc specimen](image)

The microscopic evaluation of the reference specimen illustrates a heavily damaged tribological surface. The worn surface shows a high amount of fatigue wear which can be identified by the small craters on the surface. On the surface, no sign of the burned lubricant can be established which can be explained with the high amount of worn material: due to the high wear, the burned oil molecules were also removed from the surface resulting in almost oil-free worn surface.

The surface of the disc with reference oil was analysed via a scanning electron microscope to be able to compare the results with the analysed ZrO₂ nanoparticle. Figure 6 clearly illustrates the main wear mechanism of this reference oil: huge fatigue wear crates (pitting) can be identified on the surface which refers to a high overload of the investigated system. The neat Group 3 base oil was not able to protect the rubbing surfaces against the wear, which should be one of the main aims of the lubricants. This result proves the necessity of the additives in every lubricant of today because the lack of these additives would produce fatal damages in the components of the engines and every lubricated machine.

![Figure 6. SE-Scanning Electron Microscope image about the surface of disc specimen with neat Group 3 reference oil, A) middle-stroke, B) dead-centre](image)

The bar chart on Figure 7 clearly shows the difference between each concentration: the addition of 0.1 wt% ZrO₂-additive into the base oil has increased the frictional losses slightly, however, the wear scar diameter on the ball surface has been reduced. The additional concentration increase has led to a slight decrease in friction and wear, until the 0.4 wt% value, which can be considered as the optimum concentration of the investigated nanoparticle. Over 0.4 wt% both the friction
coefficients and the wear scar diameter have started to rise. One exception can be observed: the 0.3 wt% have shown higher wear on the investigated specimens.

The optimum concentration of the ZrO$_2$-based oil mixture can be determined by the value of 0.4 wt%. In this concentration, the WSD (wear scar diameter) value was reduced by 40% with negligible average friction coefficient increase (1%).

Figure 7. Comparison of the experimental results of analysed lubricant samples with various nanoparticle concentration

Figure 8 represents the digital microscopic images about the applied testing specimens with 0.4 wt% nanoparticle concentration. This worn area illustrates the damage of the specimens: the surface is slightly worn with the mechanisms of abrasion (wear grooves along the surface) and signs of the burned oil can also be determined on the surface.

Further wear values were measured via Leica DCM 3D confocal microscope. This microscope enables to scan and evaluate the surface of the specimens. The wear scars including a certain unworn area of the disc specimen were scanned by this microscope, and they were evaluated with evaluation software of the machine to earn the volume value of the worn surface: the worn surfaced was enclosed by lots of lines, the average height-value of the unworn areas was calculated and the volume under this height-value in the enclosed area is considered as the wear volume. Figure 9 is an example of the scanned surface which enables the evaluation of the wear volume inside the enclosed area.

Figure 11 compares the investigated lubricant samples according to the caused wear volume. The reference oil (neat Group 3 base oil without additives) provides not enough wear resistance property because the wear volume is quite high. This tendency correlates with reality because these kinds of lubricants require high additive content to reach their maximum tribological potential. The application of ZrO$_2$ material in different concentrations decreases the wear volume drastically: the highest wear reduction property is provided by the lubricant with the ZrO$_2$ concentration of 0.4 wt% and the value of the reduction is over 86%. The tendency of the two different wear values (wear scar diameter on the ball specimen and the wear volume on the disc specimen) is identical.

Figure 10. Comparison of the investigated concentrations according to their occurred wear volume

The evaluation of the cross-sectional profiles by the worn disc specimens (Figure 11) illustrates the depth contribution of the worn areas and these profiles represent the dead-centre areas at every concentration. The evaluation of the reference proves the tendency of the former experiments that the neat Group 3 base oil without additives is not able to protect the rubbing surfaces from the tribological damages. However, the addition of these ZrO$_2$ nanoparticles into the lubricant reduces the wear depth drastically: the wear volume and the wear depth have been reduced until the optimal 0.4 wt% concentration.
The overdose of nanoparticles may interrupt the established protective tribofilm between the surfaces by removing ZrO$_2$ particles from the surface. The lack of protective film leads to higher wear and a higher amount of self-adhered metal material on the surface. The signs of the self-adhered material can be seen in Figure 11, at the concentration of 0.5 wt%: small peaks can be observed, marked with green colour. The adhesion of the materials between the ball and disc specimen increases both the frictional losses and wear volume. Higher ZrO$_2$-content in the analysed lubricant cannot provide further positive tribological properties: more ZrO$_2$ nanoparticle causes larger agglomerates which increases the 3-body abrasion wear mechanism on the rubbing surfaces, reducing the lifetime of the lubricated components.

![Figure 12. SE-Scanning Electron Microscope image and EDX mapping picture about the surface of disc specimen with 0.4 wt% ZrO$_2$ nano-additive, A) middle-stroke, B) dead-centre](image)

The worn surfaces of the disc specimens were investigated via scanning electron microscope (HIROX SH-4000M), with the magnitude of 1000x. This magnitude allows analysing the structure of the surface, the marks referring to the wear mechanisms and the distribution of the additive materials on the surface. The results of the SEM-analysis about the disc specimen with 0.4 wt% ZrO$_2$ content can be seen in Figure 12. Two main wear mechanisms can be established on the worn surface: abrasion (horizontal wear grooves) and pitting (fatigue wear crates), however, the damage level of the surface is significantly lower, compared with the reference result. Besides, the crates on the surface are also smaller which clearly indicates the positive wear reduction properties of the nano-sized ZrO$_2$ particles with the concentration of 0.4 wt%. The distribution of the zirconium (Zr) and oxygen (O) elements on the worn surface reveals the information that the ZrO$_2$ particle used as lubricant additives could withstand the cleaning effects of the ultrasonic cleaner and chemical fluids. The presence of remained ZrO$_2$ particles can be explained with the van der Waals forces between the particles and the surface of the ball and disc specimens: the strength of these forces strongly depend on the surface and by this kind of tiny particles, the van der Waals forces, especially in the wear grooves are stronger than the separation ability of the cleaning process. The quantitative analysis of the Zr-distribution on the worn surface resulted in a significant amount of Zr element, which is illustrated in Table 1.

![Table 1. Quantitative element analysis on the worn surface of the disc specimen with 0.4 wt% ZrO$_2$ lubricant sample](image)

The presence of the ZrO$_2$ on the worn surface enables to deduct the function mechanism of them: they may fill up the grooves on the surfaces, they may form a protective film on the surface via van der Waals forces or they can act as nano-bearings between the surfaces to protect them. To prove the proper function mechanism, further SEM analysis with higher magnitudes were carried out. The investigation was accomplished in the 3D Lab of the Institute of Physical Metallurgy, Metalforming and Nanotechnology in the University of Miskolc with a Helios G4 PFIB CXe scanning electron microscope.

Figure 13 illustrates the found ZrO$_2$-agglomerate on the surface with the magnitude of 35,000x: a huge number of nanoparticles have formed an agglomeration, connecting to each other with van der Waals forces. Besides, the nanoparticles can also formulate van der Waals forces between them and the metal surfaces, these forces are stronger in the surface grooves. This phenomenon can also be seen in Figure 13: nanoparticles can be identified in the grooves of the worn surface. The in the grooves founded nanoparticles can refer to the mending mechanism (see in Figure 1).
This paper has presented the possibility of using ZrO\textsubscript{2} nanoparticles as lubricant additives. The tribological properties of the analysed nanoparticle-base oil mixture were investigated by a translator tribometer and with a ball-on-disc tribosystem. The wear on the ball and disc specimens were examined with digital-confocal, and scanning electron microscopes to understand its working mechanism. The results can be summarised as follows:

- An optimum concentration of ZrO\textsubscript{2} nanoparticles in the base oil was identified, its value is 0.4 wt\%. The lubricant sample with this NP concentration was able to reduce the wear scar diameter by 40\% with less than 1\% average friction coefficient increase. The analysis of the wear volume showed a higher wear decrease with this concentration: the wear volume was decreased by more than 86\%.
- The most dominant wear mechanisms were also identified: abrasion and fatigue pitting. The amount of these wear mechanisms is significantly lower, compared with the reference measurements without ZrO\textsubscript{2} nanoparticles.
- The scanning electron microscope analyses have revealed a significant amount of ZrO\textsubscript{2} material on the worn surface, even after multiple thorough ultrasonic cleaning. The particle can form agglomerates via secondary van der Waals forces and they can be collected in the surface and wear grooves on the rubbing surfaces, resulting in a smoother contact surface.

The ZrO\textsubscript{2} nanoparticles as lubricant additives have revealed an interesting application in engine lubricants or in any lubricated machines. Its wear reduction properties are more than promising. Further, reality-nearby investigations (e.g. fired engine test bench measurement including exhaust gas analysis) must be carried out to use this kind of additive in the running engines of the passenger cars in the future. This additive may replace some of the additives which can block the channels of the particle filters or can cause serious damages in the catalysts of the vehicles.

5. SUMMARY

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ТРИБОЛОШКА СВОЈСТВА НАНО-СТРУКТУРНИХ КЕРАМИЧКИХ ЧЕСТИЦА ZrO₂ У АУТОМОБИЛСКИМ МАЗИВИМА

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Потреба да се код возила смањи емисија CO₂ и садржај штетних материја у издувним гасовима захтева pobољшање целокупног погонског склопа укључујући и мазив. Једно од мазива за мотор могле би да буду керамичке честице нано димензија које показују добра триболовска својства на површинама од неметала. Рад приказује експериментална истраживања нанокерамичког праха ZrO₂ као адитива у мазиву. Испитиване триболовских перформанси обављено је експерименталним путем коришћењем ball-on-disc трибометра. Утврђено је да је оптимална концентрација при 0,4 теж%, при чему је пречник бразготине од хабања на узорку кугле смањен за више од 40% у поређењу са референтним узорком. СЕМ анализе је потврдила теорију механизма поправљања: показано је да се нано честице акумулирају у храпавостима и да се тако добија додирна површина много веће глаткоће.