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From steel to plastics: friction reducing diamond-like carbon films

Zusammenfassung

Kunststoffe werden in vielen Anwendungen eingesetzt. Ihre tribologischen Eigenschaften sind von großem Interesse, um Reibung und Verschließ zu reduzieren. Dadurch kann die Lebensdauer der Komponenten verlängert werden. Dies sind beispielsweise Anwendungen mit Zahnrädern. Die Reibung von Kunststoffen kann durch eine Beschichtung mit diamantartigen Kohlenstoffschichten (DLC, diamond-like carbon) reduziert werden. Die Schichten werden mittels plasmaunterstützter chemischer Gasphasenabscheidung (PECVD) auf die Bauteile abgeschieden. Die tribologischen Eigenschaften der unbeschichteten und beschichteten Substrate wurden mit Hilfe von Universal Material Tester (UMT1 und UMT3) Systemen in oszillierender Pin-on-Plate Kontaktgeometrie untersucht. Die tribologischen Untersuchungen wurden trocken bei Umgebungsbedingungen mit Geschwindigkeiten von 10 mm/s oder 200 mm/s und einem Hub von 11 mm für fünf Minuten durchgeführt. Als Gegenkörper wurde eine unbeschichtete 100Cr6 Stahlkugel mit einem Durchmesser von 10 mm verwendet. Für einige Untersuchungen wurde auch der Gegenkörper mit einer diamantartigen Kohlenstoffschicht beschichtet. Zunächst wurde eine Grundcharakterisierung der Beschichtung auf Stahlsubstraten durchgeführt. Dazu wurden Schichten bei einer Biasspannung zwischen -400 V und -1200 V in Schritten von 200 V abgeschieden. Alle beschichteten Substrate zeigten eine reduzierte Reibung im Vergleich zum unbeschichteten Substrat, wobei die besten Ergebnisse mit der größten Biasspannung erzielt wurden. Bei einer Normalkraft von 2 N und einer Geschwindigkeit von 200 mm/s wurde bei Verwendung eines unbeschichteten Gegenkörpers der Reibungskoeffizient um 57 % von 0,53 auf 0,23 reduziert. Eine weitere Reduzierung des Reibungskoeffizienten um bis zu 61 % von 0,16 auf 0,06 wurde bei Verwendung eines beschichteten Gegenkörpers erzielt. Zusätzlich zur reduzierten Reibung konnte bei Anwendung von beschichteten Substraten und Gegenkörpern keine Verschleißspur beobachtet werden. Bei der Abscheidung der a-C:H Schichten auf Kunststoffsubstrate (Ultramid A4H PA66) konnte der Effekt der Reibungsreduzierung ebenfalls gezeigt werden. Die tribologischen Messungen mit einer Normalkraft von 2 N und einer Geschwindigkeit von 10 mm/s zeigten eine Reibungsreduzierung um 25 % von 0,24 auf 0,18. Tests zur verbesserten Tragfähigkeit mit Kräften zwischen 2 N und 25 N, entsprechend initialen Hertz'schen Flächenpressungen zwischen 69 MPa und 160 MPa wurden mit beschichteten Ultramidsubstraten und beschichteten Stahlkugeln als Gegenkörper durchgeführt. Mit steigender Normalkraft stieg der Reibungskoeffizient nur leicht von 0,04 auf 0,07 an.

Abstract

Plastics are used in a wide field of applications. Their tribological properties are of crucial interest to reduce friction and wear. Thereby the lifetime of the components can be enhanced, for example at applications using gears. The friction of plastics can be reduced by applying diamond-like carbon (DLC) coatings using a plasma-enhanced chemical vapor deposition (PECVD) process. The tribological properties of the coated and uncoated substrates were investigated using Universal Material Tester (UMT1 and UMT3) systems with oscillating pin-on-plate contact geometry. The tribological tests were run dry under ambient conditions with velocities of 10 mm/s or 200 mm/s and a stroke length of 11 mm for five minutes. As counterpart an uncoated 100Cr6 steel ball with a diameter of 10 mm was used. For specific tests the counterpart was coated with a diamond-like carbon film as well. In the first instance a basic characterization of the coating was done on steel substrates. Therefore the bias voltage was varied between -400 V and -1200 V in steps of 200 V. All coated substrates showed a reduced friction compared to the uncoated one, achieving the best results with the highest bias voltage. At a normal force of 2 N and a velocity of 200 mm/s the coefficient of friction was reduced by 57 % from 0.53 to 0.23 using an uncoated counterpart. Further reduction of the coefficient of friction up to 61 % from 0.16 to 0.06 was achieved by using a coated counterpart. Additionally to the reduced friction no wear track could be observed using coatings on substrate and counterpart. Applying the a-C:H coatings on plastics substrates (Ultramid A4H PA66), the effect of the friction reduction could be observed as well. The tribological tests using a normal force of 2 N and a velocity of 10 mm/s showed a reduced friction by 25 % from 0.24 to 0.18. Improved loading capacity tests with varied forces between 2 N and 25 N, representing initial Hertzian pressures between 69 MPa and 160 MPa, were performed using a coated Ultramid substrate and a coated steel ball as counterpart. With increasing force the coefficient of friction was only slightly increasing from 0.04 to 0.07.

1. Introduction

The tribological properties of plastics are of crucial interest for numerous industries because plastics are used in many applications. Reduced friction and wear are important to enhance the lifetime of components. Applying diamond-like carbon coatings to reduce friction and wear on metal substrates is used since several years for applications [1]. Nowadays the deposition on plastics is in the focus of research, too. Using friction and wear reducing coatings on plastics components, for example gears, would make it possible to use plastics gears with improved wear resistance instead of

steel components. In this article the authors present the transfer of DLC coatings from metal to plastics substrates. The low coefficient of friction was maintained.
Regarding deposition of diamond-like carbon (DLC) coatings on plastics it is important to consider the glass transition temperature of plastics. The glass transition temperature of the investigated polyamide (Ultramid A4H PA66) is approximately 65 °C. Due to the low glass transition temperature of the polyamide, a process which is carried out at temperatures below 65 °C is necessary. Therefore the coatings were applied using a plasma-enhanced chemical vapor deposition (PECVD) process. With this method coatings with hardness up to 30 GPa [2] could be applied at temperatures below 60 °C.

2. Experimental details

2.1 Substrate preparation

The tribological investigations were done on stainless steel 1.4301 and on Ultramid A4H (PA66) substrates. The Ultramid substrates were produced using an injection molding machine and the samples had a size of 75 mm x 25 mm x 3 mm. The dielectric constant depends on the water content and is in the range of 3.2 to 5.0.
For analytical investigations regarding the hardness of the coatings silicon substrates with a size of approximately 10 x 10 mm were used.

2.2 Film preparation

The steel and plastics substrates were coated with a diamond-like carbon (DLC) coating using PECVD processes. The deposited diamond-like carbon films are amorphous hydrocarbon (a-C:H) coatings. The PECVD processes were carried out in a vacuum chamber at a pressure of about $2*10^{-2}$ mbar. Due to the high deposition rate toluene [3] (C_7H_8) with a purity of 99.9 % was used as precursor. The coatings were deposited using different bias voltages. For a good adhesion on the metal substrates sputtered chromium and plasma polymerized SiC_xH_y from a tetramethylsilane precursor were used as adhesion layer. For the application on Ultramid substrates only SiC was used.

2.3 Experimental setup

The coefficient of friction of the uncoated and coated substrates was measured using Universal Material Tester (UMT1 and UMT3) systems [4] with oscillating pin-on-plate contact geometry (Figure 1).
The tribological tests were run dry under ambient conditions for five minutes with velocities of 10 mm/s or 200 mm/s and a stroke length of 11 mm. As counterpart an uncoated 100Cr6 steel ball with a diameter of 10 mm was used. For specific tests the counterpart was coated with a diamond-like carbon film as well. The steel and polyamide substrates were glued on a steel plate to be fixed in the UMT system. The directions of tool traces on the samples regarding manufacturing process were in line with oscillating movement. Different normal forces were used and are named when the results were discussed.

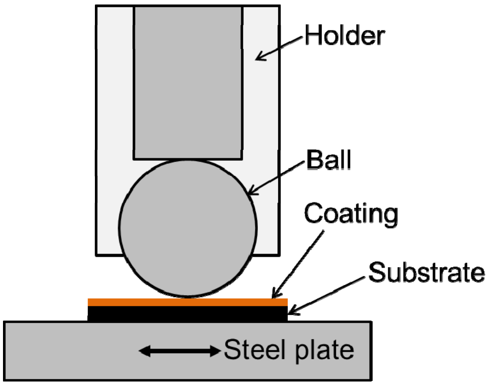


Figure 1: Schematic of the pin-on-plate contact geometry.

The hardness of the coatings was measured using nanoindentation experiments. Therefore the UMT1 system was used in a vibration as well as thermal and acoustic isolation enclosure with a Nanohead2 module. The obtained force versus indentation depth curves were analyzed using the model of Oliver and Pharr [5]. The indentations were done on coated silicon substrates.

3. Theory

The applied diamond-like carbon films are a-C:H coatings and consists of carbon and hydrogen. From a theoretical point of view a voltage of -100 V per C-Atom in the precursor molecule has to be applied on the substrate to prepare hard coatings [6].
Generally carbon has three different hybridizations, namely sp^1, sp^2, and sp^3 [7]. In a DLC coating the ratio between the sp^2 and sp^3 part dominates the properties of the coating. The strong sp^3 bonding part is responsible for the name diamond-like. A simple structural model for a DLC coating is a stochastic mixture of sp^2 and sp^3 bonds. The more sp^3 bonding the more hardness is resulting. Depositing the DLC coating out of a gas phase using hydrocarbon (toluene) as a precursor, the coating will have parts of hydrogen. Hydrogen is only able to build one bond, so there will be no cross-linking incorporating hydrogen in the coating. A high hydrogen part reduces the hardness of the coating but can lead to a reduced friction. The challenge for the deposition on plastics is to deposit the film-forming particles with high kinetic energy without heating up the coating and thereby the substrate itself.

4. Results and discussion

4.1 Diamond-like carbon films on steel

For a basic characterization of the coatings steel substrates were used. In a first step the bias voltage for the deposition process was varied between -400 V and -1200 V in steps of -200 V. The resulting five coatings were analyzed regarding the hardness and coefficient of friction. With increasing bias voltage the hardness of the films on silicon wafers was increasing in the investigated bias regime (Figure 2). It is expected that the hardness would be decreasing above a critical bias voltage due to film growth mechanisms [6] of ion bombardment and therewith again decreasing sp^3 content.

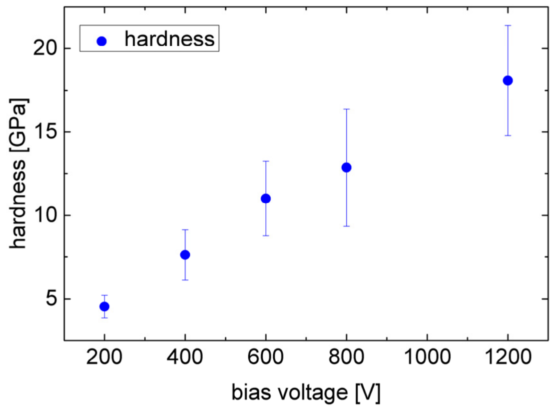
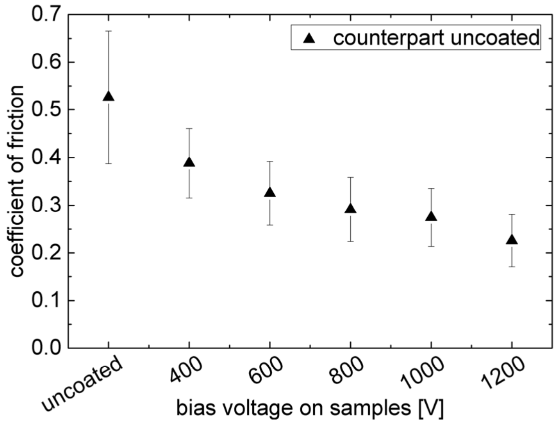


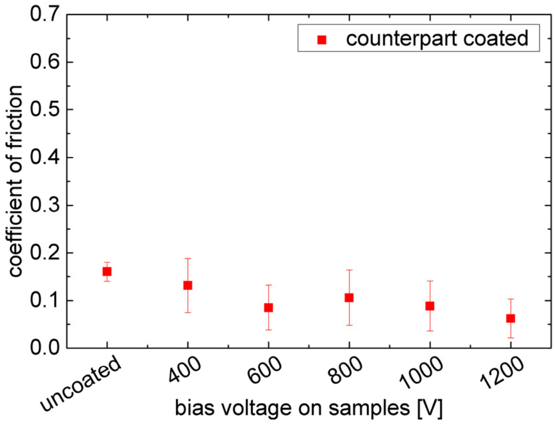
Figure 2: Hardness of the DLC coatings in dependence of the applied self bias voltage.

Regarding friction of the coated steel substrates the coefficient of friction was decreasing with increasing bias voltage resp. hardness in comparison with the uncoated reference (Figure 3a). The coefficient of friction decreased linear with increasing bias voltage. For the uncoated reference the coefficient of friction after five minutes tribological measuring time was 0.53. Applying the DLC coating on the steel substrates leaded to a reduced coefficient of friction of 0.23 at the highest bias voltage of -1200 V.

DLC coatings are well known to reach low friction. Therefore further tribological tests were performed using a coated steel ball as counterpart to achieve lower coefficients of friction. The results using a coated steel ball as counterpart revealed further reduced friction for all samples (Figure 3b). As expected the highest friction occurred for the uncoated reference. In that case the coefficient of friction was 0.16. In accord with the measurements done with the uncoated counterpart, the measurements showed a decreasing coefficient of friction down to 0.06 with increasing bias voltage. Significant lower values are not expected with DLC coatings [8].



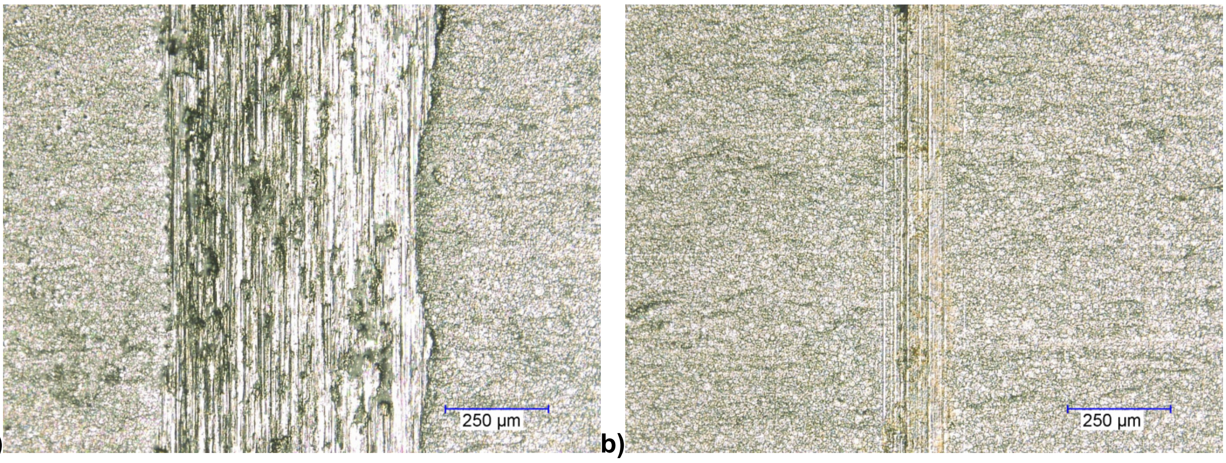
a)



b)

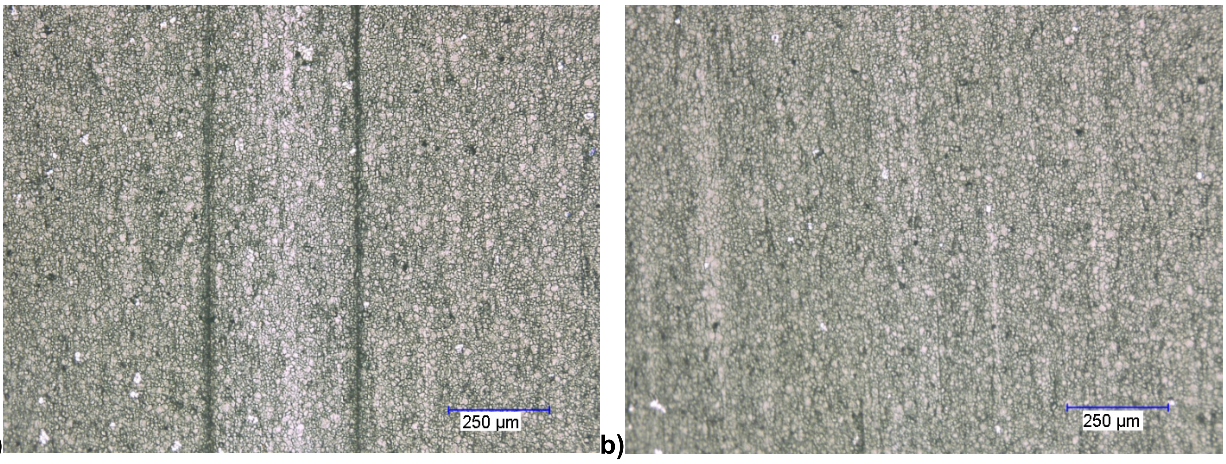
Figure 3: Coefficient of friction of uncoated and with different bias voltages coated steel substrates after five minutes of tribological measuring time with a) an uncoated and b) a coated 100Cr6 ball. Normal force 2 N (initial Hertzian pressure 652 MPa), 200 mm/s.

In addition to the friction wear is an important factor for industrial applications and the lifetime enhancement of components. Therefore the samples of the tribological tests were examined under an optical microscope. For the uncoated steel references wear tracks could be seen using uncoated as well as coated counterpart (Figure 4). Using the uncoated counterpart the trace is much broader and deeper comparing with the result obtained against a coated counterpart. The wear of the uncoated steel substrate was reduced using a DLC coated counterpart.



a) b)
Figure 4: Microscope pictures of the wear tracks of uncoated steel substrates after five minutes tribological measuring time against an a) uncoated and b) DLC coated 100Cr6 ball (compare Figure 3).

A further improvement appeared on the coated steel substrates (Figure 5). A wear track appeared only at the tribological test against the uncoated counterpart. The wear of the DLC coated steel substrate was reduced using a DLC coated counterpart. With regard to Kovalev et al. [9] it is possible to speak about ‘zero wear’. Kovalev et al. described a model for ‘zero wear’ on DLC coated steel substrates in which asperities underwent plastic deformation on a nanometer scale, but were not destroyed and no particles were formed.
The wear results seen in the microscope pictures (Figure 4 and Figure 5) correspond to the measured coefficients of friction. The highest value of 0.53 occurred for the uncoated steel substrate with the uncoated counterpart. The lowest value of 0.06 was determined for the coated substrate with the coated counterpart.



a) b)
Figure 5: Microscope pictures of the wear tracks of DLC coated steel substrates (-1000 V, compare Figure 3) after five minutes tribological measuring time against an a) uncoated and b) DLC coated 100Cr6 ball.

The result on dry running steel substrates is: Introducing DLC coatings a significant reduction of friction and wear occurs. The more components were coated the more is the effect. An analogue result was found by Scholz et al. [10] for the lubricated case with improvement using coatings on both parts and a ‘zero wear’ reaction in the best combination.

4.2 Diamond-like carbon films on polyamide

As next step the coatings were tested on plastics substrates. In this article Ultramid was used as polyamide substrate. At first a coating thickness variation in the range of 1 µm to 4 µm in steps of approximately 1 µm was done. As can be seen in the photograph (Figure 6), there were no flaking or cracks in the coatings at all thicknesses. Regarding the ongoing tribological tests that mean that the coating adhesion on the Ultramid was very well.

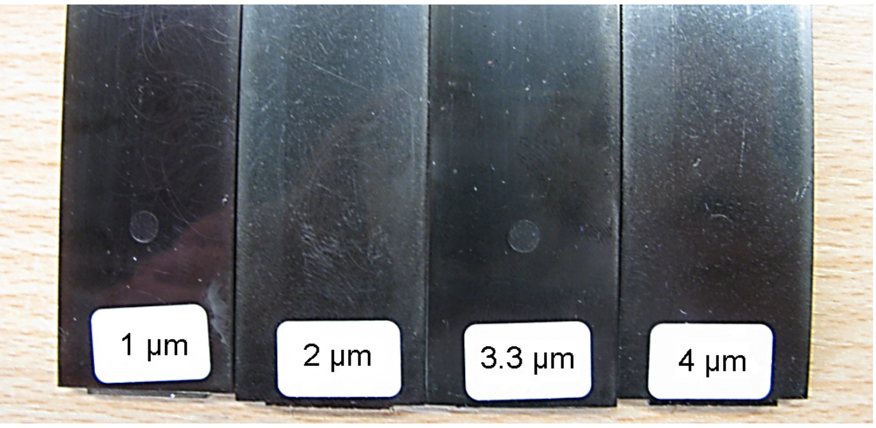


Figure 6: DLC coated Ultramid substrates with different film thicknesses.

The coated substrates were rubbed with nonwoven web to figure out the necessary thickness for a sufficient load bearing capacity. At film thicknesses of 1 µm and 2 µm the load bearing capacity of the coating was not satisfactory, the substrate under the coating was pressed in due to the mechanical load. Above a film thickness of 3 µm the films withstood this test. The necessary load bearing capacity was achieved.
Resulting from the tests on steel substrates a coating with a hardness of 18.6 GPa measured on a silicon substrate was applied on the plastics substrates. Moreover it is possible to run this process economical. According to the tribological measurements done on the steel substrates the first tests on plastics were performed with an uncoated counterpart (Figure 7a). Comparing the results of the uncoated and coated substrates a reduced friction by 25 % from 0.24 down to 0.18 was achieved.
Further tribological tests were performed using a coated Ultramid substrate and a coated counterpart as a conclusion from the tests done with the steel substrates. This time the tribological tests were done with different normal forces between 2 N and 25 N, representing initial Hertzian pressures between 69 MPa and 160 MPa. With increasing normal force the coefficient of friction was only slightly increasing from 0.04 to 0.07 (Figure 7b). This means, that the coefficient of friction of this coating was independent of the substrate material, because the coefficient of friction was in the same region as for the contact between coated steel substrate and coated counterpart (compare Figure 3b). Furthermore these tests demonstrate that the load bearing capacity of the DLC coating was sufficient enough for the Ultramid substrate. The investigated initial Hertzian pressures exceed the occurring pressures in gear applications. That means the DLC coating is suitable for gear applications using plastics gear wheels for example.

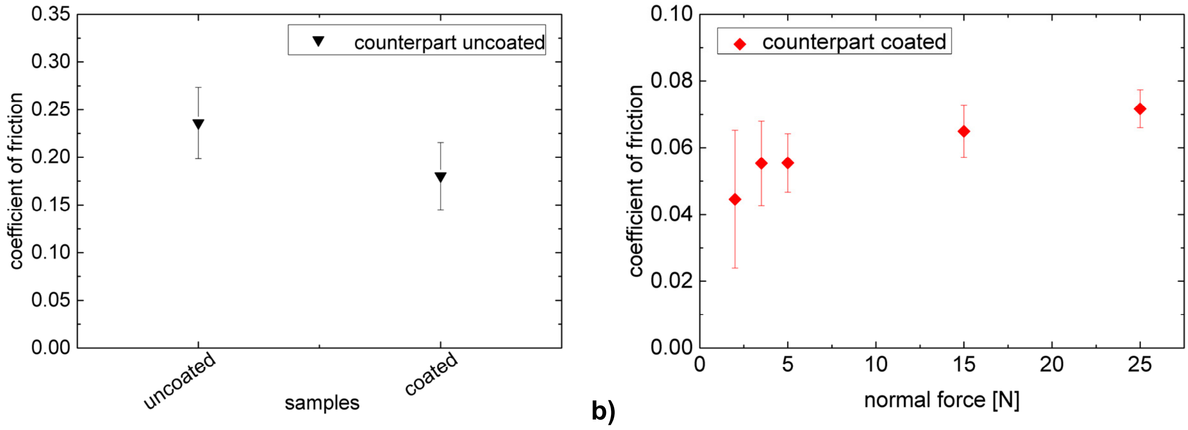


Figure 7: a) Coefficient of friction of uncoated and DLC coated Ultramid substrates against an uncoated counterpart. 2 N, 10 mm/s. b) Coefficient of friction at different normal forces of a DLC coated sample against a DLC coated 100Cr6 ball. 10 mm/s.

5. Conclusion

The tribological measurements on steel substrates showed a reduced friction for all DLC coated substrates using different bias voltages. With increasing bias voltage the coefficient of friction was decreasing. The best value of 0.06 was achieved using DLC coatings on substrate and counterpart. Moreover no wear track could be observed using a coated counterpart against a coated substrate.
The friction reducing diamond-like carbon coatings could be transferred successfully to Ultramid substrates maintaining the same low coefficient of friction. Moreover the load bearing capacity of the DLC coating is sufficient enough for that kind of PA66 substrate.
In summary one can conclude that it is possible to use diamond-like carbon coatings on plastics to reduce friction and wear of the components and thereby enhance the lifetime.

6. Acknowledgement

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