

Friction and Deformation Behavior of Elastomers

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Abstract

This research paper is about investigating the mechanisms of elastomeric friction at low velocities. To do so, different experimental setups were performed to analyze friction, adhesion and surface energy among others. The tested materials were EPDM samples with variations in the carbon black content. It was found, that at least for low velocities, the real contact area has the main impact on the friction of elastomers. This contact area seems to be highly influenced by the hardness or other bulk properties of the elastomers, which are modified by the varying carbon black content.

Keywords

Elastomers, Friction, Adhesion, Tribology, Carbon Black Variation, Contact Area

1. Introduction

Reducing friction and wear of elastomeric seals is still a current topic for industry and science. Usually coatings or lubricants are used for this reduction. In order to develop more efficient solutions, this work is about to identify the key mechanisms for reduced friction. In essence, the friction behavior of elastomers is characterized by two phenomena—by adhesion and hysteresis [1]. Typically, at low velocities the friction of elastomers is dominated by adhesion and at high velocities by hysteresis [1] [2]. The authors already investigated these influences for an uncoated and also a plasmapolymeric coated FKM, in an oscillating-pin-on-plate and also a rotating ball-on-disc test setup [3]. To avoid the influence of wear and a high temperature, this actual article focuses on tribological experiments at low velocities. Additionally, to the tribological investigations, this work also considers the adhesion, the surface energy, the roughness and the dif-

ferent contact areas of the elastomeric materials. The aim is to gain a better understanding of how friction reduction is influenced.

2. Materials and Methods

For the investigations EPDM (Ethylene-Propylene-Diene-Rubber) plates with varying Shore-A hardness values were used as the substrate material. The hardness values were 40, 50, 60 and 70 Shore-A. Hereinafter the elastomers are called EPDM40, EPDM50, EPDM60 and EPDM70. The hardness was only varied by the carbon black addition. More carbon black content leads to a harder elastomer. The thickness of these plates was 2 mm. Different geometries were stamped or cut out of the plates, depending on the experimental requirements. Another part of the sample preparation was also cleaning. Two different cleaning methods were investigated, which are explained in the following.

In the first cleaning procedure, the samples were placed in an ultrasonic bath for one hour at 60°C. The bath is filled with a solution of 98% of demineralized water and 2% of a surfactant cleaner. Subsequently, the samples are rinsed with demineralized water, blown with compressed air and stored for at least 24 hours to ensure, that potential residual moisture can evaporate. The second cleaning procedure was about wiping the elastomers with acetone. For the wiping, a lint-free cloth was used. The degree of cleaning of these two methods is compared in Section 3.1 with XPS measurements.

2.1. X-Ray Photoelectron Spectroscopy (XPS)

X-ray photoelectron spectroscopy was used to differentiate the two cleaning methods, which were investigated in this article. XPS is based on the external photoelectric effect, in which photoelectrons are released from a solid surface, through X-ray. The determination of the kinetic energy of these electrons, allows conclusions about the chemical composition and the electronic nature of the sample surface. The information depth is about 10 nm and all elements, except hydrogen and helium, are detectable. The spot diameter was 650 µm.

For the measurements, a VG 220i-XL-System with the following parameters was used: magnetic lens mode, 0° declining angle of the photoelectrons, monochromatized $Al_{K\alpha}$ -excitation, constant analyzer Energy-Mode (CAE) with 70 eV matching energy in superimposed spectra and 20 eV in energetically high-resolution line spectra.

2.2. Atomic Force Microscopy (AFM)

The roughness and also the adhesion measurements on a molecular scale were carried out by using an atomic force microscope “EasyScan 2” from the company “Nanosurf”. Depending on the measurement, two different modes were used, which are explained subsequently.

To measure the roughness of a material, the AFM scans the surface with an oscillating cantilever, which gets disturbed by surface forces, e.g. Van der Waals

forces. The scanning process is performed in a tapping mode. This means, that the cantilever oscillates near to its resonance frequency of 190 kHz and only softly taps the surface in the returning points. The surface forces affect the oscillation frequency of the cantilever, from which the surface topography can be deduced. For the investigations in this article, an aluminum reflective coated cantilever, with a tip diameter smaller than 20 nm, was used. Furthermore, a free vibration amplitude of 399.9 mV was applied and the measurements were performed under ambient conditions. Five images with a size of $90 \times 90 \mu\text{m}$ were taken from each sample. For each of those images, three roughness profiles were extracted to calculate R_a according to DIN EN ISO 4287. These 15 R_a values were used to calculate the average roughness for the sample and error bars represent the standard deviation from the according mean value.

The AFM can also measure the adhesion of a surface, by using force spectroscopy in the static mode. For this static mode another kind of cantilever was used. In this mode the tip of the relevant cantilever starts $10 \mu\text{m}$ above the surface and then moves $11 \mu\text{m}$ downwards. Afterwards the tip withdraws to the starting position. Simultaneously to this procedure, the deflection of the cantilever is measured to get force-distance curves, which are allowing conclusions about the adhesion of the surface. A detailed description of this adhesion measurement method can be found in [4]. The measurements were performed at room temperature and a relative humidity of 30% to 50%. To avoid an influence of the different electric charges between the cantilever and the substrate, the specimen holder and the scan head were electrically connected. Also, a holding time was integrated before each measurement. Here the cantilever was hold in contact with the substrate for five minutes, whereby the electric imbalance was neutralized.

Five different spots were examined on each sample. For each spot, the maximum deflection of the cantilever was taken and the average of these five values was used for the evaluation. The investigations were performed with two different cantilevers, with varying tip diameter:

- 1) A silicon cantilever, with a tip diameter of less than 20 nm, a force constant of 0.2 N/m and a resonance frequency of 13 kHz.
- 2) A silicon cantilever, with a force constant of 0.2 N/m, a resonance frequency of 13 kHz and a polymethylmethacrylate (PMMA) tip, whose diameter is $1.5 \mu\text{m}$.

2.3. Surface Energy Determination

The surface energy of a material, allows conclusions about the adhesion of the surface. In order to determine this surface energy, contact angle measurements were performed. In this article, a contact angle measuring system “G2” from the company “Kruess” was used for the investigations. Respectively three samples of each material were tested, to gain three surface energy values. The average of these three values for each material was taken for the discussion and error bars represent the standard deviation from the according mean value. Six different

liquids were used to calculate the surface energy. The used liquids were: Benzyl alcohol, n-Decane, Glycerol, Ethylene glycol, diiodo-Methane and water. The contact angle values have been recorded continuously during the drop application. This approach, also known as dynamic contact angle measurement, excludes randomly caused deviations over course of the drop application. The measuring time was 30 s, starting 5 s after the deposition of the 6 μl drops. The dosing speed of the drops was 12 $\mu\text{l}/\text{min}$ and the method according to Wu [5] was chosen for the surface energy calculation, because it delivers better results for low-energy systems.

2.4. Tribological Setups

The friction behavior of the materials was investigated with two different setups. The setups differ in terms of surface pressure, contact surface, motion etc.

A pin-on-disc setup was implemented, to measure the friction force in case of a rotary motion (see **Figure 1**). To perform this setup, a “Universal Material Tester (UMT3)” system from the company “CETR” was utilized. This device presses the face of a rolling bearing, with a defined normal force onto an elastomeric disc. The disc is moving rotary and simultaneously the friction forces as well as the adjusted normal force are recorded. The used rolling bearing has a chamfer of 0.4 mm. Because of this chamfer, the real contact area and thus the real surface pressure can only be exactly calculated, when the true penetration depth is known. However, due to the yielding of the sensors and other machine elements, the penetration depth cannot be determined in an appropriate way. Therefore, the chamfer is disregarded for the calculation of the contact surface and also the surface pressure. **Table 1** shows the test parameters for this setup. The tests were performed without lubrication. For this setup one sample was tested for each material.

Additionally, a tensile setup was applied to measure the friction force in case of a linear motion (See **Figure 2**). This setup was carried out on a tensile testing machine “Z020” from the company “Zwick/Roell”. A 5 N force sensor, from the type “Kap-TC” was used. In contrast to the pin-on-disc setup mentioned above, here a square elastomer plate was pulled horizontally over a stainless steel surface. Simultaneously the frictional force was recorded. **Table 2** shows the testing parameters, which were used. The tests were carried out without lubricants. In this setup, three samples were tested for each material.

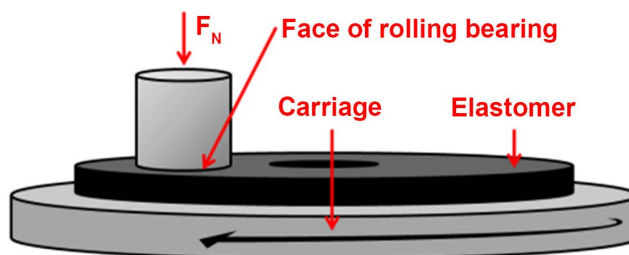


Figure 1. Scheme of the pin-on-disc setup.

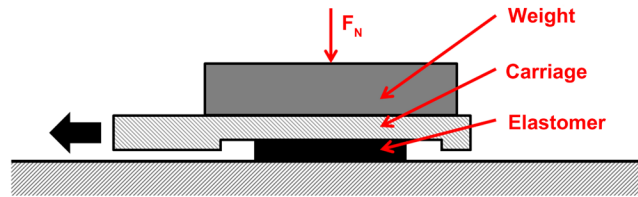


Figure 2. Scheme of the tensile setup.

Table 1. Testing parameters for the pin-on-disc setup.

Normal force	4.7 N
Surface pressure	0.06 MPa (calculated without chamfer)
Nominal contact area	78.54 mm ² (calculated without chamfer)
Mean circumferential velocities	0.2 mm/s
Test duration	10 s
Temperature	30 °C
Test specimen	Steel rolling bearing (Ø 10 mm, chamfer 0.4 mm)
Mean test radius on disc	19.25 mm
Acceleration time of the rotary drive in the beginning of the test after contact	0.05 s

Table 2. Testing parameters for the tensile setup.

Normal force	1.43 N
Surface pressure	0.004 MPa
Nominal contact area	400 mm ²
Linear velocity	0.2 mm/s
Test duration	60 s
Temperature	Room temperature
Test specimen	elastomer plate (20 × 20 mm)
Test distance	12 mm
Acceleration time of the traverse in the beginning of the test	<0.8 s

2.5. Macroscopic Pull-Test

In order to determine the adhesion of the substrates on a macroscopic level, also the “Universal Material Tester (UMT3)” system from the company “CETR” was utilized. With this setup, the face of a rolling bearing is pressed onto the substrate surface and then withdrawn vertically (See **Figure 3**). The separation forces were recorded and used for the evaluation of the adhesion. Before the separation a holding time of 10 s was scheduled, to ensure that the elastomer has completely adapted to the surface of the rolling bearing.

Chateaubinois *et al.* investigated in [6], that this adaption never exceeds 1000 s. However, Voll investigated on a similar setup, that already 1 s seems to be sufficient for this adaption [7]. Three samples were tested per each material to get an average. **Table 3** shows the test conditions.

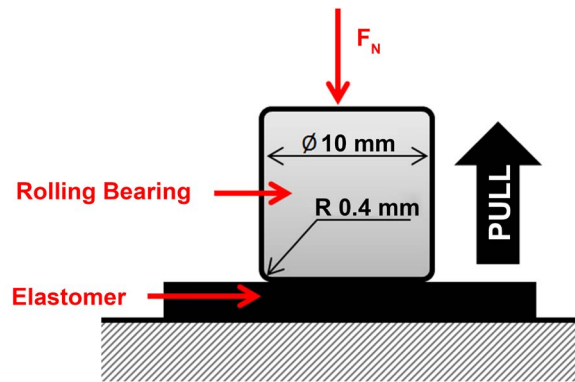


Figure 3. Schematic setup for the pull-test.

Table 3. Testing parameters for the pull-test.

Normal force	40 N
Surface pressure	0.51 MPa (calculated without chamfer)
Nominal contact area	78.54 mm ² (calculated without chamfer)
Holding time	10 s
Vertical separation velocity	10 mm/s
Temperature	Room temperature
Test specimen	Steel rolling bearing (Ø 10 mm, chamfer 0.4 mm)
Sample geometry	Discs with the diameter of 23 mm

2.6. Investigation of the Contact area

The following section explains how the contact area was investigated. The aim was to observe, how the contact areas of the different EPDM materials are influenced by varying surface pressures. To do so, 20 × 20 mm EPDM plates were cut out and placed under a light microscope (Keyence VHX-600). Then a thin glass plate (15 × 15 mm) was placed on the surface. On top of this glass plate, a metal disc with a small hole for the observation is positioned and burden with various weights (See **Figure 4**). Images were taken with a 500× enlargement and afterwards converted into black and white images by setting a threshold value. An example for this procedure is shown in **Table 4**. With this procedure, the contact area of the substrates can be determined and observed with different surface pressures. The surface pressure was varied from 0 to 0.51 MPa.

3. Results and Discussions

In the following section the results of the various tests are described and discussed, to gain a better understanding of the primary friction mechanisms.

3.1. Evaluation of the Cleaning Methods

The results of the XPS measurements for both cleaning methods, and also for an uncleaned reference, are shown in **Table 5**. It can be seen, that wiping the

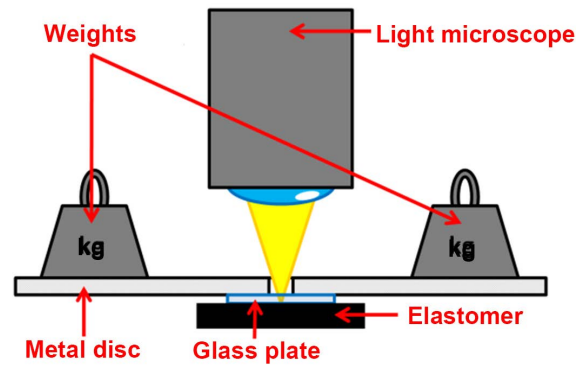


Figure 4. Setup for the contact area investigation (based on [8]).

Table 4. Exemplary procedure for the contact area determination on EPDM50.

Nominal surface pressure/normal force	0.006 MPa/1.45 N	0.01 MPa/4.05 N	0.05 MPa/11.59 N
Image with 500x enlargement			
Black & white conversion			
Real contact area	3 %	22.4 %	61.8 %

Table 5. XPS results for different cleaning methods.

Sample	C (at%)	O (at%)	Zn (at%)	Si (at%)	Ca (at%)	S (at%)	N (at%)	Na (at%)	Cl (at%)
EPDM50, uncleaned reference	84.9	9.8	0.6	1.5	0.9	1.1	0.9	0.3	0.2
EPDM50, 1 h US-bath cleaning in a surfactant cleaner solution	96.9	2.1	0.1	0.4	-	0.2	0.2	0.1	-
EPDM50, wiped with acetone	85.4	10.4	0.4	1.5	1.6	0.4	0.4	-	-

samples surface with acetone seems not to clean efficiently, because the values for the elements stay nearly the same, when comparing them to the uncleaned reference.

It can be assumed, that the contaminations are blurred on the surface, rather than to be removed. Cleaning the samples in an US-bath, using a surfactant solution, seems to be more efficient, when considering the carbon content as an indicator for the cleanliness. The elastomers consist mainly of carbon and after

this cleaning procedure more of this pure material can be identified with the XPS measurement. Building on these results, the standard cleaning method for the following measurements is to clean the samples for one hour and at 60°C in an US-bath, filled with a surfactant solution.

3.2. Surface Characterization

In this section the results of the roughness and contact angle measurements are presented and discussed. Before measuring the contact angles and determining the roughness of the EPDM substrates, the samples were cleaned for one hour in an ultrasonic bath, filled with a surfactant solution.

In **Figure 5** the roughness values (R_a) for the different substrates are illustrated. It can be seen, that the four EPDM substrates have nearly the same roughness. The measurements were performed with an AFM, which was described in section 2.2. Since all material surfaces have almost the same roughness, the influence of roughness is neglected in following discussions.

In **Figure 6** the different polar and also the disperse parts of the materials are presented. The results were measured with the dynamic drop procedure, which is explained in section 2.3. The surface energy slightly increases with increasing hardness, except for the EPDM50. When looking at the polar parts, there is no clear trend identifiable. EPDM40 and EPDM70 have a higher amount of polar parts than EPDM50 and EPDM60. The connection between adhesion and surface energy has already been investigated in other articles, but without clear correlations [9]. Based on the assumption, that a higher surface energy results in higher adhesion, the adhesive forces should increase with increasing hardness. Therefore, it can also be assumed that the friction force also will, because the main friction mechanism in this low velocity area is adhesion. In the following sections, these theses are evaluated.

3.3. Tribological Investigation

In the following section, the results of the tribological investigations are shown and discussed. The results were divided into the static friction force and the

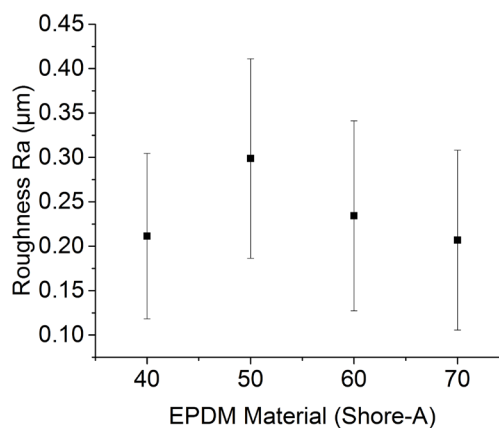


Figure 5. Roughness values (R_a) for EPDM materials.

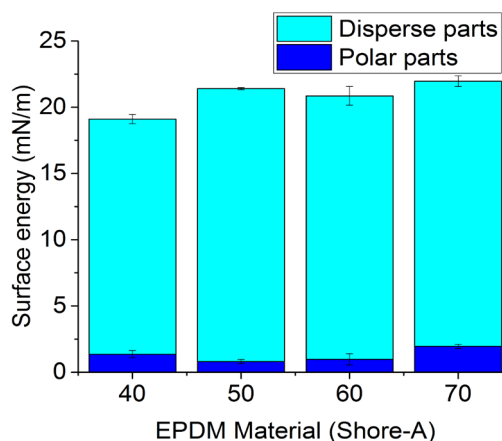


Figure 6. Surface energy values for EPDM materials.

dynamic friction force. The static friction force is the highest force, which is necessary to move the sample out of a stationary position. The dynamic friction force is the force, which is required to move the sample when it is already in motion. The tests were performed with the tensile and the pin-on-disc setup, which are described in Section 2.4.

Considering possible influences due to acceleration effects in the setups, the development of the static friction force is evaluated in the following. For the tensile setup, the acceleration phase is less than 0.8 s. The corresponding static friction force was found to be approx. 8 s after the beginning of the test. For the pin-on-disc setup, the acceleration phase was 0.05 s. Thereby the static friction force was found after approx. 1 s of testing time. That means the acceleration phases can be neglected for both tests. The static friction force was observed, when the target velocity of 0.2 mm/s is already reached. So it can be assumed, that the static friction force applies for the target velocity.

Figure 7 shows the static friction force for the different substrate materials. All the samples were moved with 0.2 mm/s out of a stationary position. It can be seen, that with increasing Shore-A hardness, the static friction force decreases. Furthermore, the graph shows higher friction for the pin-on-disc setup, although it has a smaller nominal contact area. The main reason for this is most probably, that due to the higher normal force and surface pressure, the real contact area should be greater for the pin-on-disc setup. The real contact areas for the different setups are investigated in Section 3.5.

Figure 8 shows the dynamic friction force for both tribological setups. The results are similar to the results of the static friction force measurement. The dynamic friction decreases with increasing hardness for both setups. EPDM40 shows stick-slip motion, when performing the test on the tensile setup. This behavior was observed for all three tested EPMD40 samples, so an experimental artifact can be excluded. Further investigations have to clarify, why it was not observed for the other materials. Furthermore, again the friction force on the pin-on-disc setup is higher than on the tensile setup, equally to the static friction force measurements.

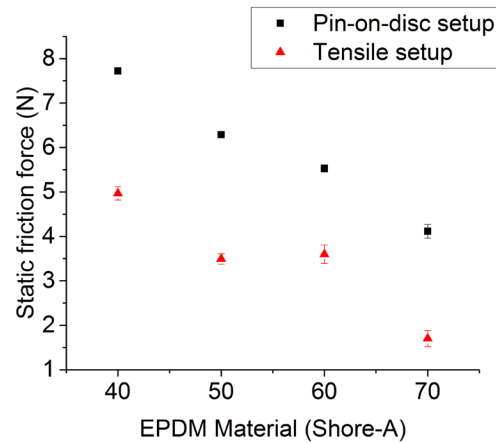


Figure 7. Static friction force for EPDM materials.

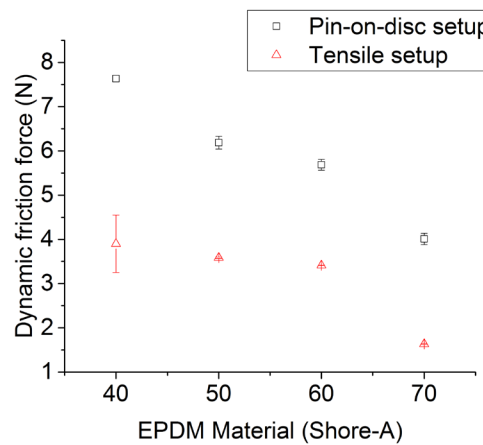


Figure 8. Dynamic friction force for EPDM materials.

These results do not meet the expectations, when comparing them to the surface energy determination from the previous section. It was observed, that the surface energy slightly increases with increasing hardness and thereby the friction should also increase. However, the opposite was observed. The friction force decreases significantly with increasing hardness for these two setups. This means, that there has to be another factor, which has much more weight on the friction, at least for those two setups. Voll & Popov investigated in [10], that the adhesion force increases with increasing viscosity. Based on the assumption, that the viscosity increases with increasing carbon black content [11] [12], the friction force should also increase for these experiments. Furthermore, the “simplest adhesion model” in [4] suggests, that the adhesion should increase with an increased shear strength. When considering, that the shear strength increases with increasing Shore hardness, the friction force should also increase for the applied experiments. So both theories seem not to fit for these results.

Adhesion is the main mechanism for friction in the low velocity area, in which these tribological tests were performed. So in the following section the adhesion of the materials is investigated.

3.4. Investigation of the Adhesion Force

Complementary to the tribological experiments from the previous chapter, the adhesion of the materials is investigated in this section more directly. For this purpose, a macroscopic pull-test and measurements with an AFM were carried out. The pull-test was performed with the procedure mentioned in Section 2.5. and the AFM measurements as described in Section 2.2.

In contrast to the tribological experiments in the previous section, the macroscopic pull-test was performed with a much higher surface pressure. The intention was to gain high adhesion forces, to better differentiate between the substrate materials as investigated in [13]. **Figure 9** shows the results of the pull-tests for the EPDM materials. Additionally, the influence of the two cleaning methods mentioned in Section 3.1. was investigated, by using differently cleaned sample batches of each material. It can be seen that the adhesion rises with increasing hardness. An exception here is EPDM60, which has also relatively high standard deviations. Furthermore, the adhesion seems to be higher when wiping the samples with acetone instead of cleaning them in an US-bath, although the XPS measurements from Section 3.1. showed, that cleaning the samples in an ultrasonic bath should make them cleaner. A reason for this could be that wiping the sample surface with acetone, leaves residues that cause higher adhesion. That means in other words, that a properly cleaned elastomeric surface shows less adhesion. Furthermore, a correlation can be found when comparing these results with the surface energy determination from Section 3.2. The surface energy was slightly increasing with increasing Shore hardness and thereby the adhesion should also increase. These macroscopic adhesion measurements revealed the same tendency. Even the slight decrease in the surface energy of EPDM60, is expressed in a decreasing adhesion force, when comparing it with EPDM50 and EPDM70. Nonetheless, the interpretation must be done carefully, due to the high standard deviations for EPDM60 in this macroscopic pull-test.

Based on the assumptions that the viscosity and the shear strength are increasing with increasing Shore hardness/carbon black content, these results do also fit to both theories mentioned in Section 3.3. The adhesion force increases with increasing viscosity and shear strength [4] [10].

In contrast to that, the results of the macroscopic pull-test do not fit to the tribological tests mentioned in section 3.3. An increasing hardness led to decreasing friction, but also to increasing adhesion. In the low velocity range, in which the tribological tests were performed, the friction force should be proportional to the adhesion.

The adhesion of the substrates was also investigated with an AFM. In contrast to the previously mentioned macroscopic adhesion measurement, this microscopic measurement is less affected by the surface roughness of the materials, due to the use of the extremely fine tips. **Figure 10** shows two exemplary roughness profiles and two AFM cantilever tips, which were true to scale. One profile line for uncoated EPDM40 and additionally a second profile line for plasma-polymeric coated EPDM40.

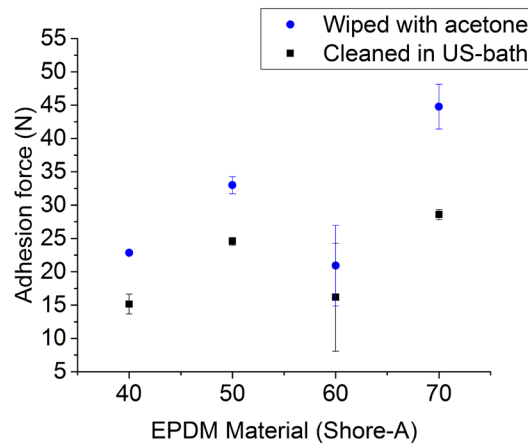


Figure 9. Adhesion of EPDM materials, measured with a macroscopic pull-test.

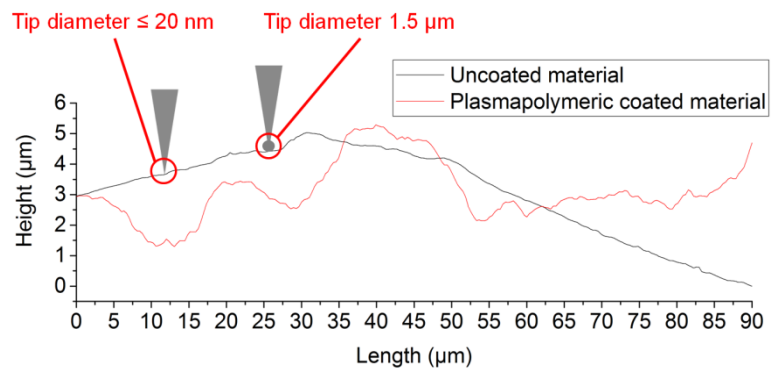


Figure 10. Profile-lines of uncoated and coated EPDM40, measured with AFM in tapping mode.

Figure 11 shows the results, measured using a cantilever with a tip diameter of approx. 20 nm. A plasmapolymeric coated EPDM40 sample (R_a 0.55 μm) was also tested to verify this measurement method. The deposited coating is an adhesion lowering coating and it should show a reduced deflection. A description of the deposition process and the coating can be found in [3]. There is a slight tendency that the deflection at pull-off decreases with increasing Shore-A hardness, which means that the adhesion decreases with increasing hardness. As expected the plasmapolymeric coated sample shows reduced adhesion, so this measurement method seems to be validated. To verify these results and to have a greater surface acting, another test was carried out using a cantilever with a tip diameter of 1.5 μm . The following **Figure 12** shows the results of this measurement. A plasmapolymeric coated EPDM40 sample was tested as well. Again there is a slight trend visible, in which the deflection/adhesion decreases with increasing hardness. The plasmapolymeric coated sample shows lowered adhesion. As expected, the deflection increases when using the cantilever with a tip diameter of 1.5 μm , because of the greater contact surface. The influence of the different cantilever tip masses on the deflection magnitude has to be investigated in further researches.

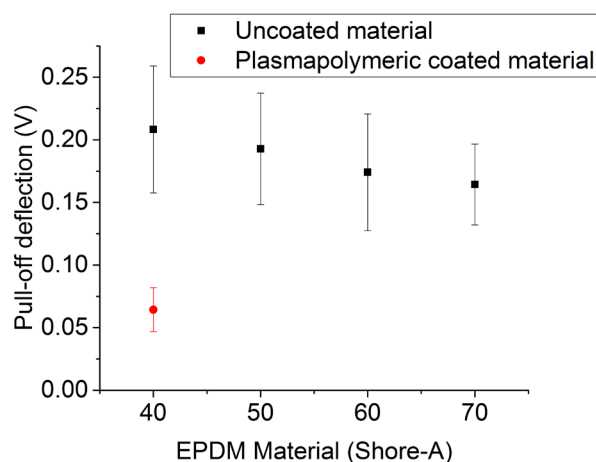


Figure 11. Deflection of the AFM cantilever at pull-off; Tip diameter ≤ 20 nm.

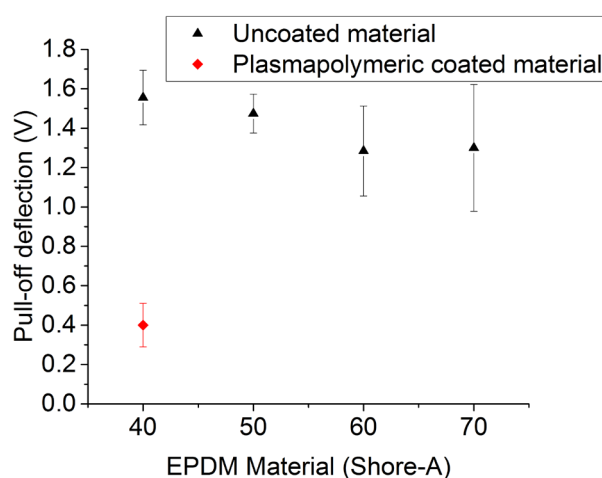


Figure 12. Deflection of the AFM cantilever at pull-off; Tip diameter $1.5 \mu\text{m}$.

It is hard to say whether the observed results show clear trends or just statistic deviations and actually there is no significant difference between the EPDM materials observable. When taking the observed trends into account, the results of both experiments do not fit to the results of the macroscopic pull-tests and also the surface energy determination. Both, the surface energy and the macroscopic adhesion, increased with increasing material hardness.

3.5. Investigation of the Contact area

This section is about investigating the contact area of the different EPDM materials with varying surface pressures. The applied procedure is described in section 2.6. In **Figure 13**, the results of the contact area investigations are shown. As expected, the real contact area increases with increasing surface pressures, as already investigated in literature [6] [14] [15]. Further **Figure 13** shows the real contact area is decreasing with increasing hardness. More exactly the softer

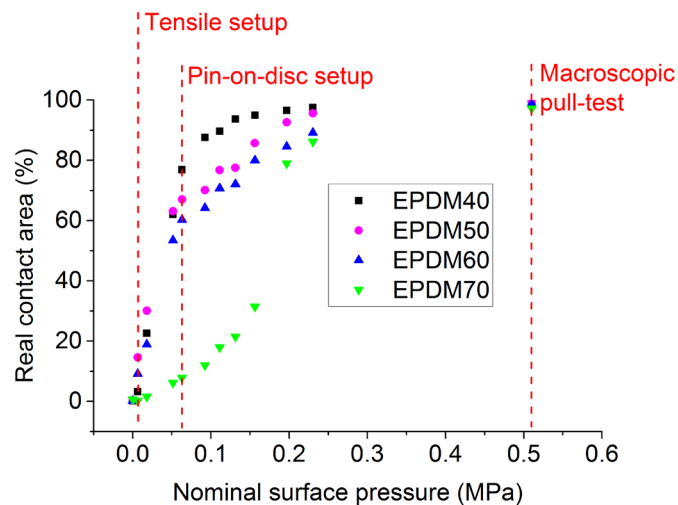


Figure 13. Contact area growth for EPDM materials, with different surface pressures.

EPDM material reveal a higher gradient in the increase of the contact area, whereas the EPDM70 has a lower one. This leads to tremendous differences in the contact area for most surface pressures, although all materials have a similar roughness.

When looking at the used surface pressures of the different test setups, there are obviously big differences in the contact areas. Most likely these differences led to the mismatch between the tribological and the macroscopic adhesion tests. For the tribological setups the investigation was that the friction force decreases with increasing hardness. As shown in **Figure 13**, the real contact area decreases also with increasing hardness for the chosen nominal surface pressures. The decreasing friction force correlates most likely with the decreasing real contact area. The lower real contact area led to lower friction. This explains also, why the friction force for the pin-on-disc setup was higher when comparing it with the tensile setup, although the tensile setup was performed with a greater nominal contact area (see **Figure 7** & **Figure 8**). For the applied nominal surface pressures, the real contact area is nevertheless smaller on the tensile setup.

In contrast to that, the macroscopic pull-test was performed with a nominal surface pressure, in which the contact areas of the different materials are nearly the same. The outcome was that the adhesion force increases with increasing Shore hardness. That means without restrictions, that the adhesion force is increasing with increasing Shore hardness.

Furthermore, it can be assumed that if the tribological tests were performed with a surface pressure at which the contact areas are approximately equal, the friction force should increase with increasing hardness. This would also fit to the existing models from literature [4] [10].

These results emphasize that the friction force is mainly dependent from the real contact area. This real contact area cannot be deduced from roughness values of the elastomers. The deformed state has to be taken into account. For fric-

tional tests with similar contact areas, it has to be clarified whether the bulk properties (hardness, shear strength, viscosity etc.) or the surface energies of the elastomers are mainly affecting the friction.

4. Conclusion

Overall it can be said, that the real contact area has the main influence on the friction of elastomers, at least for the observed low velocity friction tests. This real contact area depends on the hardness of the materials and it can be assumed, that it also depends on other bulk properties such as shear strength, viscosity etc. Although the investigated EPDM materials have equal roughness values, the contact areas showed differences. Same substrate roughness does not suggest equal real contact surfaces. The deformation of the contact area has to be taken into account. To compare different friction or adhesion experiments, at first comparable real contact areas must be ensured by adjusting the surface pressure. Beyond that, further experiments have to clarify, whether the bulk properties or the surface energies of elastomers are mainly affecting the friction when having equal real contact areas. Also, the influence of the counterpart roughness on the real contact area has to be investigated in further experiments.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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