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Tribology of polymer composites for elevated temperature applications

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Abstract

Polymers as construction material are common in the industry. Although more recently the use of polymer composites in more demanding applications has increased, requiring more of them mechanically, tribologically and thermally. To enhance the properties various fillers are used, from common glass fibers to more advanced nanoparticles. For this study three types of base polymers have been studied: poly-amide (PA), poly-phenylene-sulphide (PPS) and poly-ether-ether-ketone (PEEK). They have been filled with glass fibers, carbon fibers, poly-tetra-fluoro-ethylene (PTFE), graphite and thermally conductive modifier in various combinations. Fibers are used to increase the mechanical properties, PTFE and graphite are added as lubricating additives to reduce the friction, and the thermally conductive modifier to increase the thermal conductivity. Five general groups of polymer composites were studied.

- Pure PEEK
- PPS, PA and PEEK filled with fibers
- PPS, PA and PEEK filled with fibers and lubricating additives
- PA filled with lubricating additives
- PEEK filled with fibers and additives for lubrication and thermal conductivity

The polymer composites have been tribologically tested in a reciprocating sliding test set-up. Friction, wear and surface damage have been studied. Three types of counter surfaces have been used: ball bearing steel balls, stainless steel cylinders and anodized aluminum cylinders. Load, surface temperature of the polymer composites and number of cycles were varied to study any changes in friction and wear. The wear marks on the polymer composites were studied using an SEM. Cross sections of some tested samples were prepared to study any subsurface damage.

From the tests the polymer composites showed similarities in friction. Lubricating additives gave lower friction, often around 0.05-0.15, while pure and only reinforced gave higher, often around 0.4-0.5. The wear was also less for polymer composites with lubricating additives. There was no clear influence of temperature but for most tests an increase in temperature gave lower friction. The only influence of load was that higher load gave wider wear tracks. Since no cross sections were prepared to compare subsurface damage due to different loads there might be a possibility that there were some differences below the surface as well. Otherwise cross sections showed that polymer composites with only fibers had cracks and cracked fibers below the surface due to the high stresses the polymer composite had been subjected to. With lubricating additives there was no large subsurface damage and it seems as if the lubricating additives formed a protective tribofilm in the wear track, giving both lower friction and wear. The presence of such a tribofilm was confirmed by XPS analysis that showed a surface layer containing F from PTFE.

The conclusions are that the tribological properties of a polymer composite are strongly dependent on its fillers. Lubricating additives form a tribofilm that lowers friction and wear. Elevated temperatures might drastically change the tribological behavior of a polymer composite why it is important to do tests at higher temperatures. Cross sections can give information about subsurface damage and might help to understand the wear mechanisms and deformation of polymer composites better. More microscopy and mechanism studies are required in order to further understand the tribological behavior of polymer composites.

List of Papers

This thesis is based on the following papers, which are referred to in the text by their Roman numerals.

I. **Friction and wear studies of some PEEK materials**
J. Lind, P. Lindholm, J. Qin, Å. Kassman Rudolphi
Tribologia – Finnish J. Tribol., 33 (2015) 20-28

My contributions: All experimental work, part of planning, evaluation and writing.

II. **Cross section microscopy studies of wear tested polymer composites**
J. Lind, Å. Kassman Rudolphi
Submitted to Tribology International

My contributions: All experimental work, major part of planning, evaluation and writing.

III. **Surface and subsurface deformation of polyamide and poly-ether-ether-ketone composites sliding against steel cylinders**
J. Lind, Å. Kassman Rudolphi
Manuscript

My contributions: All experimental work, major part of planning, evaluation and writing.

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Results presented at conferences

This thesis is also based on the following conference proceedings/contributions, which are referred to in text by their letters.

A. Effect of PTFE on the tribological behaviour of PPS with glass fiber
J. Lind, Å. Kassman Rudolphi
Extended abstract and oral presentation at The 1st International conference on Polymer Tribology, PolyTrib 2014

B. Friction and wear of PA66-PPA composites sliding against anodized aluminium cylinders
J. Lind, Å. Kassman Rudolphi
Poster presentation at The 20th International conference on Wear of Materials, WOM 2015

C. Effect of temperature on the performance of some polymer composites
J. Lind, Å. Kassman Rudolphi
Poster presentation at The International Tribology Conference, ITC 2015

D. Cross section studies of polymer composites
J. Lind, Å. Kassman Rudolphi
Extended abstract and oral presentation at The 2nd International conference on Polymer Tribology, PolyTrib 2016

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1 Introduction

Polymers have been used within the industry for a long time, with their low weight as one of the benefits. Reinforcement, with fibers for example, widens the area of use. The development of more high temperature stable polymers has made it possible to use polymer composites as machine components even in more demanding environments. Using polymer composites instead of traditional metals can give a decrease in weight, cost and waste of material during manufacturing.

When it comes to tribology different microscopy and analysis techniques can provide information about friction and wear mechanisms. Combining several techniques gives us an understanding of what the material has been subjected to in regards of friction, wear and deformation. For polymer composites the field of tribology has mostly been focused on friction coefficients, wear rates and the effects of different kinds of fillers. There is also some knowledge about adhesive and abrasive wear but not so much about the mechanisms behind the deformation of the material.

The major part of the work presented in this thesis was done in collaboration with Kongsberg Automotive. Kongsberg Automotive has used polymer composite products for a long time and sees a value in increasing the use of them. One study was done together with ABB Corporate Research, Paper I.

The focus for this thesis has been materials for applications and components subjected to temperatures between -40 °C and 150 °C, with only room temperature and above studied due to limitations of our testing equipment. The aim has been to gain a deeper understanding of polymer composites and their tribological behavior.

2 Background

2.1 Polymer composites

Polymers are interesting for the industry due to their low weight, cost and manufacturing processes. But pure polymers have low strength and it is therefore common to add reinforcement to the material. The reinforcement is often made with fibers, for example glass or carbon fibers, or particles of, for example, titanium and aluminum oxide [1-6]. The added strength widens the field of use and polymer composites can even replace metal in some applications.

There is a wide variety of fillers for polymer composites and recently nanoparticles have become more common within the field of research [6-10]. It is not only the type of filler that is important for the function of the composite, but also the amount. For example, too little reinforcement does not provide enough strength, while too much makes the material brittle. It is as important to choose right type and amount of filler as it is choosing polymer matrix.

It is important to consider what working environment when choosing polymer matrix. Different polymers have various resistances to degradation by air, sun or other compounds and/or swelling when exposed to water or other liquids. Temperature is another important parameter to take into consideration. A component that is used at temperatures around -10 °C might not function at 100 °C. It is important that the polymer matrix has the desired properties at the current temperature. Temperature properties are often connected to the type of polymer chain. Aromatic rings, large side groups and stronger interactions between the chains are a few features that can give a more stable polymer at elevated temperatures. An important temperature property for polymers is the glass transition temperature, T_g, which is well under the melting temperature, T_m. Above T_g the molecular mobility increases and leads to a loss in elastic modulus. The molecular mobility occurs in the amorphous regions in the polymer material and increasing crystallinity is an example of T_g modifier [11]. Fillers can also contribute to a change in the polymer properties at T_g, for instance can the drop in elastic modulus be greater for a specific filler [12,13]. Molecular weight, large side groups and aromatic rings on the polymer chain all have an effect on the T_g [11-16].

It is important to know that this decrease is not sudden but gradual. This makes the definition of T_g as a specific temperature a bit misleading since the decrease in elastic modulus can begin below the given T_g. Knowing T_g and its behavior is important when choosing polymer matrix for components used at any temperature. Depending on desired properties and working temperature for the polymer composite it can be used either below or above its T_g. Often the important factor is to have stable mechanical properties for the material at the working temperature. Examples are polymer composites used at room temperature, some are used above T_g because they are too brittle below, and others are used below T_g because the elastic modulus is too low above. In many industrial applications it is desired to maintain the mechanical properties at elevated temperatures, which is why higher T_g is requested. In general all factors that constrain the polymer chains to move relative each other have an effect on the polymers properties, such as elastic modulus and T_g.

Even though the working temperature might be well below T_g the friction can lead to an increase in temperature and change the conditions for the material in contact [17-19]. In these cases the polymer composite can be modified for better thermal conductivity to draw the heat away from the contact [20,21].

2.1.1 PA, PPS and PEEK

The materials chosen in this thesis are commercially available polymer composites with matrices of poly-amide (PA), poly-phenylene-sulphide (PPS) and poly-ether-ether-ketone (PEEK), which have a T_g of 65-130 °C, 90 °C and 143 °C respectively, see Table 1. All have relatively high T_g, which is important because these materials are meant to be used at temperatures of 80-120 °C. That is why other common polymers, such as POM with a T_g of -30 °C and T_m of 183 °C, are not suitable.

In this thesis different kinds of PA materials with different T_g are used. PA can be modified in various ways, where the differences might be the placement of the amide group on the chain, chain length between amide groups and to what extent the chain is aromatic. High performance polyamide (PPA) has aromatic polymer chains, which for instance increases T_g, chemical resistance and elastic modulus. PA can therefore be designed, to a certain degree, to fit desired properties, such as T_g, T_m or elastic modulus.

Injection molding is used when producing components from these materials. This gives a certain type of surface, with a thin surface layer containing only polymer. For comparison, a machined part will have exposed fillers at the surface. When designing and testing new components machined parts are often used, for example because the cost is too high to produce a form for injection molding. This is important to keep in mind when testing new components. The surfaces in this thesis are produced by injection molding and are therefore similar to those of injections molded components.

2.2 Polymer tribology

When reading papers studying polymer tribology a vast majority says that polymers are favorable for their low friction, but that is not true for all polymers. Many polymer materials give low friction contacts, but they are often lubricated, either with grease or oil or with lubricating additives within the polymer composite itself. Polymers can have quite high friction coefficient, especially rubbers, which in some cases is desirable. It is therefore often unfortunate to refer to low friction as a desired property within the field of polymer tribology since the field is so wide. Therefore, it is important to distinguish between a specific composites' low friction and the polymer material groups' varying friction, as well as the fact that the friction is also dependent on other parameters in the tribological system. This thesis focuses on polymer composites for machine components with low friction as a desirable property. Polymer composites can have quite high friction coefficient, around 0.4, and the benefits of lubricating additives as low friction additives can be great. PTFE, graphite, MoS_2 and WS_2 are a few examples of lubricating additives in polymer composites [6,7,9,10,22-25].

Polymers are often subjected to adhesive wear [26]. When the polymer adheres to the counter surface the sliding interface is more polymer-polymer than polymer-metal, if the counter surface is e.g. steel. This also means that the friction is mostly dependent on the polymer's resistance to shear. The adhesion during sliding gives rise to a transfer layer, which is important in polymer tribology [6,15,23,24,27]. The transfer layer, and the mechanisms behind it, is important for the tribological behavior of polymers and their composites. No general conclusion can be drawn about the effect of a transfer layer, since it seems to depend on type of polymer composite if a thin or thick layer is required to reduce friction and wear [6,26,27]. The addition of reinforcement to a polymer matrix affects the transfer layer and also the wear. So reinforcement might also improve the tribological properties of a polymer composite. Transfer of material back and forth as well as repeated sliding of the same area can lead to deformation hardening,

which creates a harder surface layer in the wear track, characterized by surface cracks [28]. This surface layer might affect the tribology and hence performance of the component.

In tribology generally, it is important to study both friction and wear since low friction doesn't always correlate to low wear and vice versa. This seems to be particularly true for polymers, with PTFE as a good example of very low friction but high wear. Reinforcing PTFE with hard particles can increase the wear resistance. It is also possible to add PTFE particles as filler material to other polymer matrices to gain some of its low friction properties while using the base polymer's properties for the bulk. Using a lubricating additive such as PTFE, or graphite, MoS_2, WS_2, carbon nanotubes, can in some cases replace oil or grease as lubricant. In most cases the desired effect of a lubricating additive is to form a thin tribofilm at the interface, lowering both friction and wear [24,29]. The use of lubricants is on the other hand common and the development of lubricants has given greases and oils that are suitable for polymer composites. But the large variety of polymer composites might cause failure for the component if the wrong lubricant is used. Not only must the lubricants give low friction and wear, it is also important that the lubricants do not lead to degradation. Degrading the polymer might lead to loss of mechanical strength or failure of the fillers to function as intended. But using a lubricating additive might not be effective for all polymer composites. For example, filled PTFE performs different with different fillers and it seems as if the tribochemistry is important for the tribofilm formation [29]. To get the best protecting and low friction tribofilm from lubricating additives factors such as polymer matrix and fillers seem important. If the lubricating additive is easily transferred to the counter surface and over the entire contact area the benefits of its tribofilm are the greatest. PTFE for instance has been proven to spread to parts of the contact area not directly in contact with the PTFE, spreading the benefits of low friction tribofilms [30].

Reinforcing the polymer increases the elastic modulus and widens the materials field of use. This enables it to be used as machine components and replace metal in some cases. A common reinforcement is by fibers, for instance glass (GF) or carbon (CF) [1,3,15,28,31,32]. The orientation of these fibers affects the tribological performance [3,33,34]. Polymer composites with reinforcement might cause abrasion of the counter surface [1-3,28,31,33,34]. Either by exposed reinforcement fillers, such as fibers or particles, or by three-body abrasion. Reinforcement is therefore not only positive in a tribological contact with polymer composites.

A lot of papers on polymer composites and their tribological performance focus on comparing a pure polymer or a polymer composite to another with other fillers. Friction coefficients and wear rates are presented and one composite often performs the best. But in general the field of polymer tribology lacks an understanding of the deformation of the polymer composite during testing. There is no way of determining how much deformation the material has been subjected to aside from the removal and displacement of material. In this thesis some cross sections of tested polymer composites have been prepared and studied, hoping to give more information about the deformation of polymer composites in sliding contacts.

3 Experimental methods

3.1 Polymer composites studied in this thesis

The polymer composites were delivered injection molded into discs with thicknesses of 2-5 mm and cut into samples of approximately 20x60 mm. Polymer matrices, fillers and some properties are presented in Table 1.

One important thing to think about with commercial polymer composites is that glass fibers in different composites might vary in size, shape and composition. The percentage given in Table 1 is for total filler amount, which means that two composites containing 30 wt% glass fibers + PTFE might have different ratios between the two fillers. Often there is a reason for the use of certain fiber types and/or ratios between fillers, but these reasons are rarely given to the customer. In addition, controlled fiber orientation has been proven to give different tribological performance depending on fiber orientation relative sliding direction. For the tested polymer composites the preferred fiber orientation is parallel to the surface, coming from the flow during injection molding, see Figure 1. This should give a somewhat better performance than if the fibers are perpendicular to the surface. The choice of fillers is important for the tribology and since all tests are performed with commercially available polymer composites only general conclusions can be drawn about the effect of reinforcement and lubricating additives.

Elastic modulus is a material property that correlates to the stiffness of the material. Stiffness is to what extent the material resists deformation under an applied load. Stiffness should not be confused with strength or hardness. The elastic modulus of the polymer materials in this thesis varies from 2.7 GPa, without any reinforcement, to 18 GPa with only reinforcement. For polymer composites the elastic modulus is given for the whole composite but the fact is that the polymer matrix has a much lower elastic modulus and the material will therefore not behave as a homogenous material. The polymer matrix can still be sheared quite easily at the surface but the fibers prevent the deformation well below the surface. For this thesis the elastic modulus is only available for the whole composite but for further studies it would be interesting to do some nanoindentation in order to better understand the material.

Table 1. The polymer materials studied in this thesis, with polymer matrix and fillers as well as some properties specified. M1 and M2 are abbrevations for manufacturer 1 and 2, which means that the PEEK composites are made by two different maufacturers.

| Polymer matrix	Fillers (wt%)	E-mod (GPa)	T_g (°C)	T_m (°C)	Study
PPS	GF, 40%	15.5	90	280	Paper II, A, C, D
PPS	GF + PTFE, 30%	12.5	90	280	Paper II, A, C
PA66	PTFE + silicone oil, 20%	2.7	65	260	C
PA66 + PA6I/X	GF, 50%	18	70	260	B, C
PA6T/66	GF + PTFE, 30%	11	100	310	Paper III, B
PA6T/6I	GF, 50%	18	130	325	Paper III, B, C
PEEK (M1)	None	3.5	143	343	Paper I
PEEK (M1)	CF, 30%	27	143	343	Paper I
PEEK (M1)	CF + PTFE + graphite, 30%	12.5	143	343	Paper II, III, C, D
PEEK (M2)	CF, 30%	32	143	343	Paper I
PEEK (M2)	CF, lubricant, thermally conductive modifier	22	143	343	Paper I
PEEK (M2)	CF, PTFE, other lubricating additive	24	143	343	Paper I
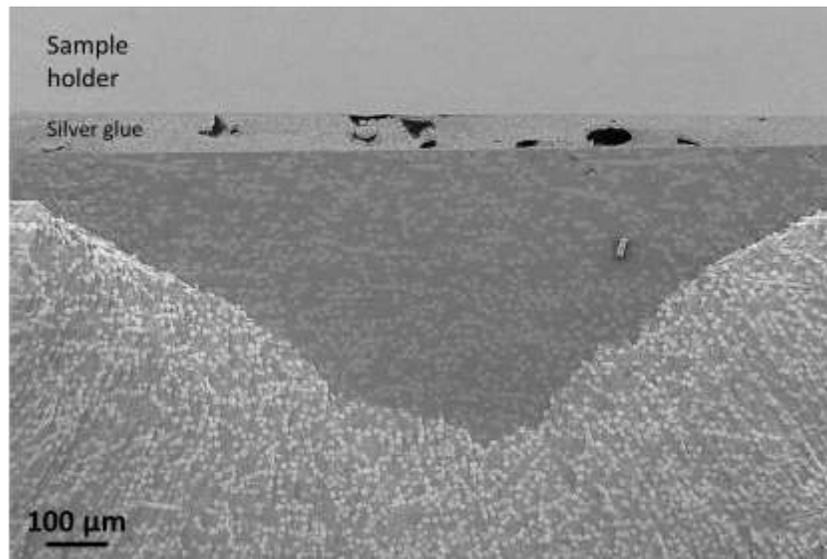
Figure 1. Cross section of PA6T/6I with GF, approximately 1 mm wide and 400 µm deep. The brighter areas are the glass fibers in the polymer matrix. Information about fiber distribution and orientation can be obtained.

3.2 Tribological test set-ups

As always the tribology is dependent on the whole system and not only one part. The large variety in polymer composites also reflects in their tribological performance. The friction can be very low but also quite high, as well as the wear. The many possible combinations of test parameters, such as load, sliding speed, counter surface material and geometry, makes it possible to produce a wide range of friction and wear results for the same polymer composite. It is therefore important to design a test that as close as possible simulates the type of environment and parameters that the composite is meant to be subjected to.

To ensure the relevance, the test parameters were chosen together with Kongsberg Automotive, and ABB for Paper I.

3.2.1 Reciprocating sliding test set-up

All tests where performed using a reciprocating sliding test set-up, applying a sliding distance of 5 mm and frequency of 3 Hz. The duration of the tests varied between 2000, 10 000 and 20 000 cycles.

The contact geometry was ball-on-disc or cylinder-on-disc, with polymer discs. The ball-on-disc was chosen due to the simple set-up and small contact area. The cylinder-on disc geometry provided contact conditions more similar to the application. In order to ensure an even pressure distribution over the entire contact area the cylinder sample holder was designed to tilt, see Figure 2.

Contact loads of 5 N and 15 N were used. The friction was measured using strain gauges, put together in a Wheatstone bridge, on the counter surface sample holder. The sample holder was then calibrated to measure the friction force.
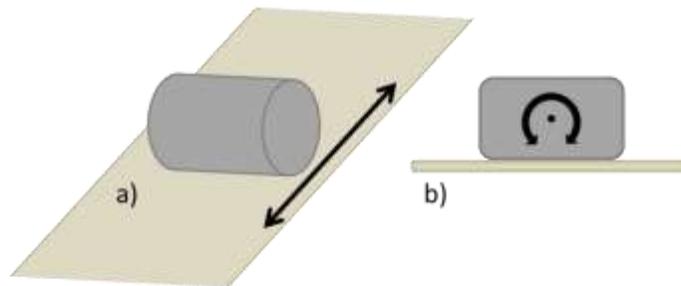

Figure 2. The set-up for cylinder-on-disc tests. a) The sliding direction perpendicular to the cylinder axis, b) the cylinder tilt axis parallel to the sliding direction. The arrow indicates sliding direction.
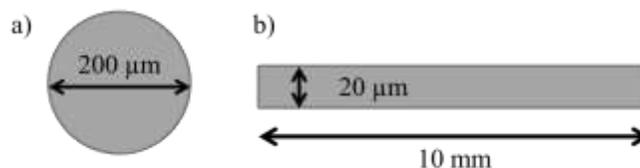

Figure 3. Contact widths for a) steel ball and b) steel cylinder against PPS with GF under 5 N contact load.

3.2.2   Counter surface materials

Ball bearing steel balls, 100Cr6, with the diameter of 10 mm, where used in Paper I and II and also for conference presentation A, C and D.

In Paper III machined stainless steel cylinders, AISI 431, with a diameter of 10 mm and length of 10 mm were used. For conference presentation B aluminum cylinders, with a diameter of 10 mm and length of 10 mm, were used. These were made from cast aluminum, machined and anodized, to ensure a surface layer of aluminum oxide.

The maximum Hertzian contact pressure at 5 N for ball and cylinder in contact with PPS with glass fibers was 210 MPa and 20 MPa, respectively. The corresponding contact diameter was 220 μm for the ball and the contact width was 20 μm for the cylinder, see Figure 3.

3.2.3   Test temperature

Tests were performed at four surface temperatures of the polymer composites, room temperature (RT), 80 °C, 120 °C and 150 °C. The temperature was achieved by thermo elements mounted in the polymer sample holder, i.e. the whole disc was heated. The counter surface was in contact with the disc for five minutes before the test to ensure heating of the counter surface as well. In Paper I and conference presentation B all four surface temperatures were studied. In conference presentation D only room temperature tests were performed.

3.3   Characterization methods

3.3.1   Light optical microscopy (LOM)

Light optical microscopy was used to study the counter surfaces and wear tracks in Paper I, this to get a first overview of the materials and their differences. Width and surface characteristics of the wear tracks for the polymer composites as well as transfer of material to and wear of counter surface were studied.

3.3.2   Scanning electron microscopy (SEM)

The polymer composites were studied using a scanning electron microscope (SEM) (Zeiss 1550, Zeiss 1530 and Zeiss Merlin). In order to avoid charge up of the surfaces a thin layer of conductive Au/Pd was deposited on the samples. An acceleration voltage of 5 kV was mainly used when studying the wear tracks, this to get a surface sensitive analysis in order to see exposed fibers and fillers in the wear track instead of also getting signal from fillers below the surface. For the study of the cross sections of PEEK with carbon fibers, PTFE and graphite 10 kV was used in order to see the graphite flakes.

3.3.3   Energy dispersive X-ray analysis (EDS) in SEM

EDS analysis was done in connection to SEM microscopy (Zeiss 1550). EDS was used to get information about polymer matrix and the different fillers as well as for detecting any wear particles in the wear tracks. Accelerating voltages between 5 kV and 20 kV were used depending on what elements were interesting to detect. For instance, if information about PTFE was interesting 5 kV was used, to get a higher probability of detecting F, while 20 kV was used when wear particles from the counter surface, containing Fe, were more interesting. .

3.3.4   X-ray photoelectron spectroscopy (XPS)

XPS was used to detect possible formation of PTFE surface layers. This was done by mapping the wear tracks with respect to the elements C, N, O and F, where the F signal was used to specifically track PTFE.

In order to get as detailed map as possible for mapping the wear tracks a beam diameter of 20 μm was used. Since XPS is a very surface sensitive method the samples were not studied in the SEM, because that would have required deposition of a conductive surface layer.

3.3.5 Cross section preparation and microscopy

Cross sections were prepared using an ion polisher (Gatan Ilion+ II). Prior to the ion polishing the samples was attached to a sample holder using silver glue. Polishing was done at 6 kV for 2 hours and 4 kV for 20 hours directly afterwards. This produces cross sections approximately 700 μm wide and 300-500 μm deep. For the tests in Paper II, with ball bearing steel balls as counter surface, the cross sections were done perpendicular to the wear track. For Paper III, with steel cylinders as counter surface, they were done parallel to the wear tracks. See Figure 4 for a schematic view of the process.
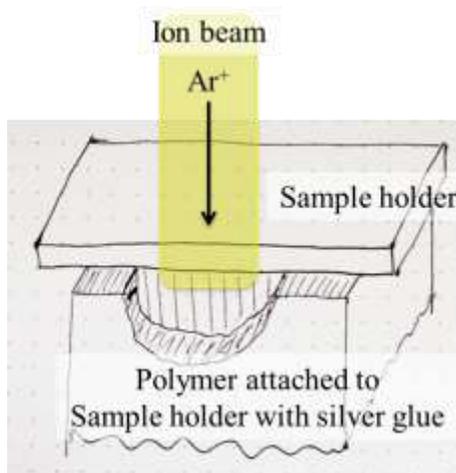

Figure 4. Schematic view of the process of producing cross sections. The sample holder protects the sample and ensures a flat surface of the cross section.

4 Present findings

In this chapter the results from all papers and conference proceedings are presented and discussed.

4.1 Influence of load and temperature

For the contact load interval used in our tests there is no larger influence of load on the friction and wear of the polymer composites. The only visible difference is that higher load gives wider wear tracks, i.e. more deformation, for the tests against ball bearing steel balls, see Figure 5. The load might also affect the subsurface damage but no cross sections were made that compares two samples tested with different loads. Therefore no conclusions can be drawn about subsurface damage at different loads. This is something to be studied further on.

For most of the polymer composites, a test temperature of 80 °C gives a somewhat better performance, i.e. lower friction and less surface damage. This might be due to the polymer being easily sheared but still maintaining the bulks elastic modulus at this temperature, giving an advantageous transfer layer.

For most polymer composites the friction and wear behavior changed above its T_g. The loss in elastic modulus might be a reason. But it also seems as if surface damage and wear can be prohibited by low friction tribofilms. The low friction prevents high stresses in the material, hence less deformation.
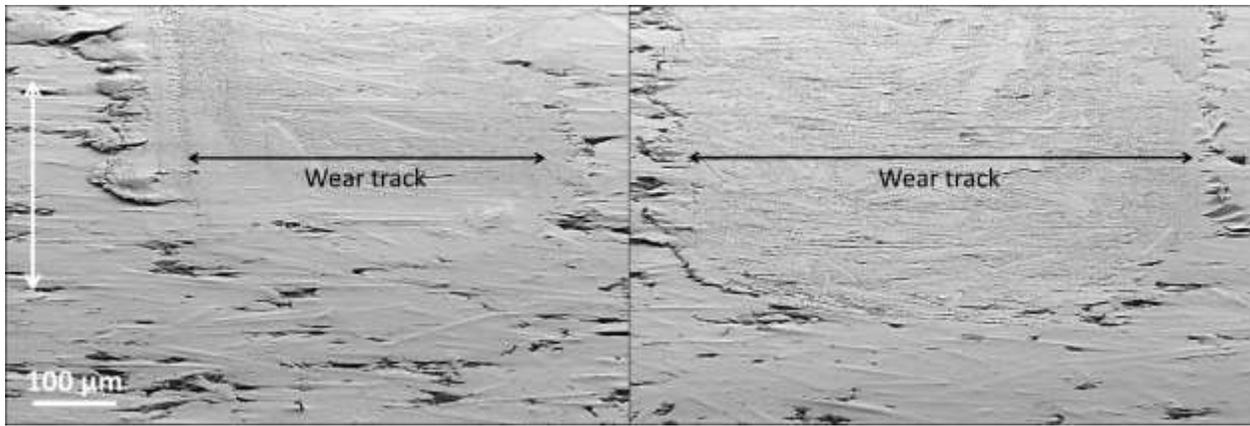

Figure 5. Wear marks for PA6T/6I with GF tested against a ball bearing steel ball at RT with a) 5 N and b) 15 N load. At 15 N the wear track is wider. The white arrow indicates sliding direction.

4.2 Influence of reinforcement

In Paper I pure PEEK shows lower friction than PEEK with carbon fibers at room temperature, about the same at 80 °C and higher friction at higher temperatures. It is also shown that carbon fiber reinforcement prevents deformation of the wear track and increases the wear resistance. Both wear and friction are higher for pure PEEK than PEEK with carbon fibers at 120 °C and 150 °C. This is an indication that, in this case, the higher friction might be due to more deformation, and thus more material to be sheared, for the pure PEEK. Although, a polymer composite without reinforcement might have both low friction and wear above its T_g if the sliding forms a tribofilm. For PA66 with PTFE and silicone oil in conference proceeding C there is almost no wear or surface deformation, in fact some wear marks were hard to even find in the microscope.

From Paper I and II it is clear that glass fibers acts more abrasive on the counter surface than carbon fibers, see Figure 6. Glass fibers are harder than carbon fibers and also more brittle and cracks more easily, which results in abrasive particles. Exposed and/or cracked glass fibers that have been pushed back into the polymer, causes abrasive wear of the counter surface. This leads to steel wear particles in the wear track of the polymer composite. In Paper III it is shown how wear particles from the steel cylinders are imbedded in the wear track. There is a higher probability to find worn steel in contact or close to a fiber, see Figure 7. This is confirmed by the results in conference proceeding D.
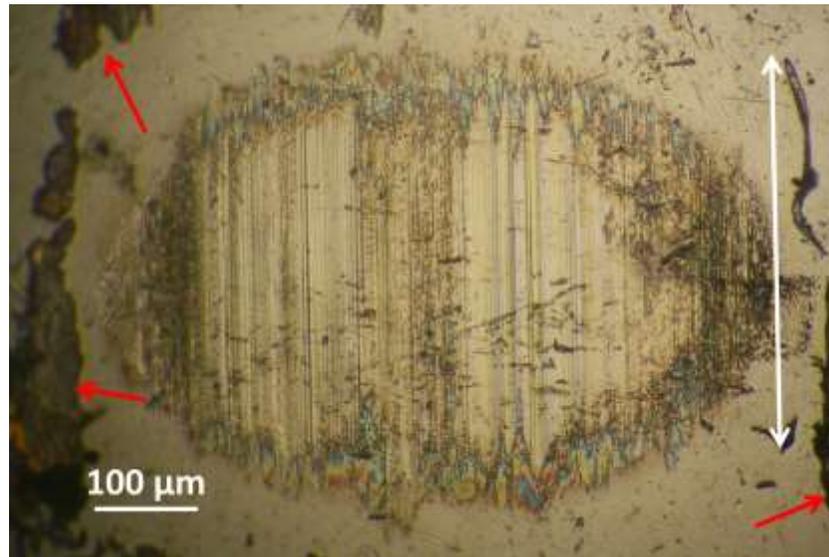

Figure 6. The ball bearing steel ball tested against PPS with GF at RT with 15 N load for 10 000 cycles. The ball has abrasive wear marks from the glass fibers. Red arrows indicates worn material from the polymer composite outside of the wear track. The white arrow indicates sliding direction.
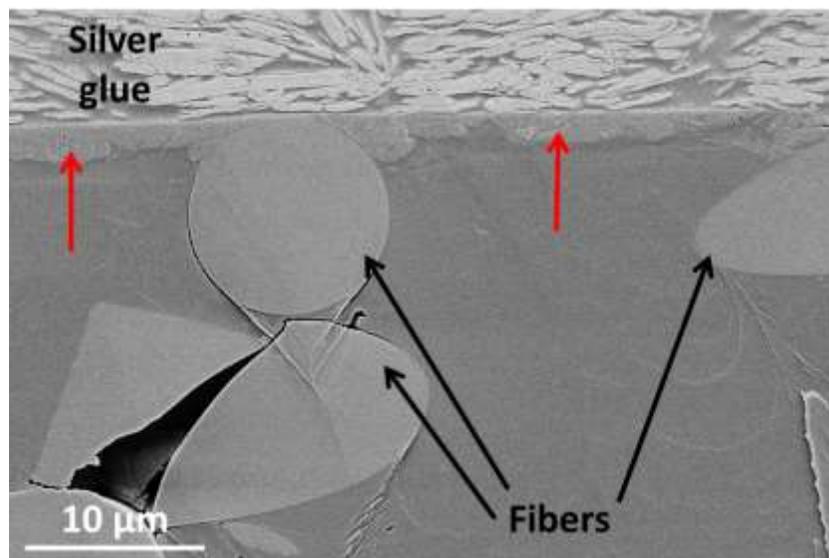

Figure 7. Cross section of PA6T/6I with GF tested against a steel cylinder at 80 °C with 15 N load for 20 000 cycles. Red arrows indicate worn steel material in the wear track of the polymer. The worn material is often located near a fiber.

4.3 Influence of lubricating additive

Adding lubricating additives fillers to the composites lowers both friction and wear, see Figure 8 and 9. Since the type of lubricating additives varies for the different tested polymer composites it is hard to draw any conclusions of the performance for the specific lubricants.
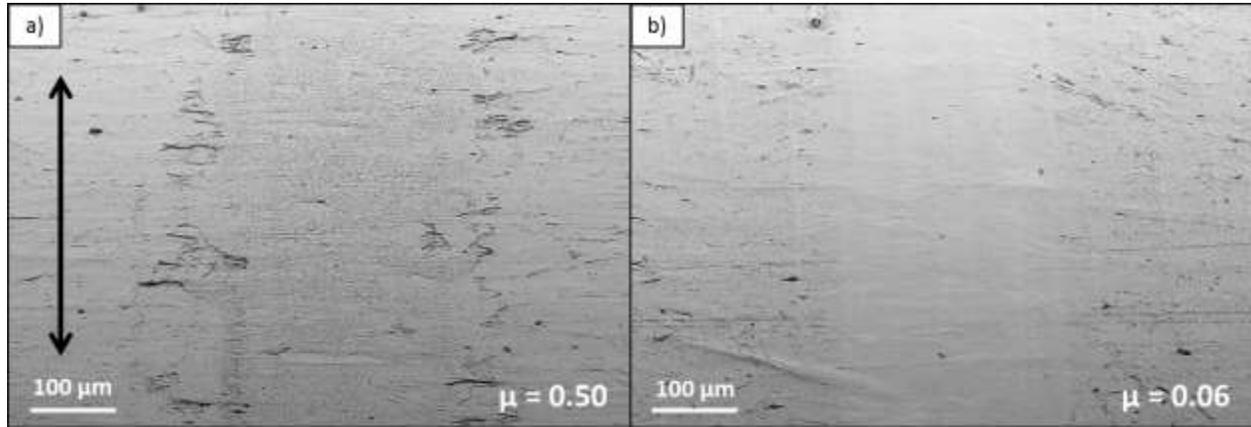

Figure 8. Wear marks and mean value of friction coefficients for a) PPS with GF and b) PPS with GF and PTFE tested against a ball bearing steel ball at RT with 5 N load for 2000 cycles. The friction is lower and the surface damage is much smaller for b) than a). The arrow indicates sliding direction.
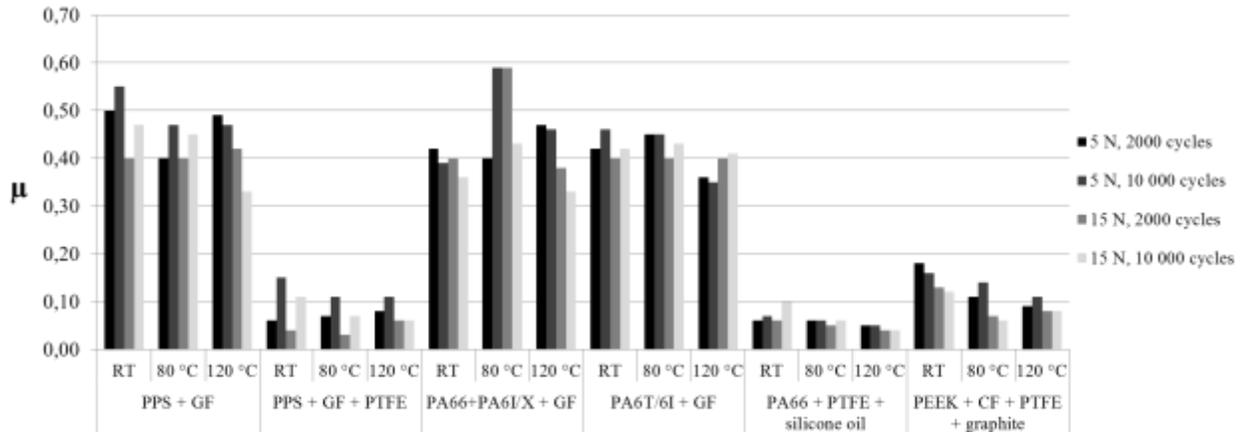

Figure 9. Friction coefficients from tests against ball bearing steel balls with different load, temperature and number of cycles. Mean values over the last 100 cycles. Lubricating additive reduces the friction drastically.

When studying the polymer composites in Paper II the difference is clear, lubricating additive does not only lower friction but also seems to protect the surfaces from wear, both the polymer composite and the counter surface. PTFE is known for its low shear resistance and seems to form a protective tribofilm at the contact interface. Comparing cross sections of PPS with glass fibers with PPS with glass fibers and PTFE it is clear that lubricating additive also protects the material from deformation below the surface. PPS with glass fibers shows cracks, as well as cracked fibers, below the surface, see Figure 10 and 11. At 120 °C a large crack is visible under the whole wear track and there is also a deformed surface layer, which can be discerned even when studying the surface from above, see Figure 11 and 12. But also at RT, when the surface is not as worn, cracks can be seen in the cross section, see Figure 10. Thus, high friction correlates both to surface wear and deformation and subsurface crack initiation. At higher temperatures, when the

polymer composite has exceeded T_g in this case, the material cannot withstand the stress as well as at lower temperatures. This results in fatigue of the material. Adding PTFE to the material, as for PPS with glass fibers and PTFE, lowers the friction, thus resulting in lower stress and no cracks or cracked fibers can be found below the surface. Besides the cracks and cracked fibers, also wear debris from the counter surface are detected at the surface of PPS with glass fibers at 120 °C, comparable to the results seen in Figure 7.

In Paper III, on the other hand, PTFE in PA6T/66 does lower the friction about 20 % at RT and 80 °C but at 120 °C the friction is the same as for PA6T/6I. The reason might be that 120 °C is higher than T_g for PA6T/66 and the mechanical properties have therefore changed resulting in this friction behavior. The wear tracks of PA6T/66 with glass fibers and PTFE are somewhat smoother than those of PA6T/6I with glass fibers, but there are clear signs of deformation, and patches of material seem to have been replaced within the wear track. PEEK with carbon fibers, PTFE and graphite shows lower friction than both PA composites, as well as smoother wear tracks. In Paper III there seems to be an influence of lubricating additive on the wear particles. For PA6T/6I with glass fibers the wear particles are roll-like, which is typical for PA, but for PA6T/66 with glass fibers and PTFE they are more flake-like, see Figure 13.

PTFE has shown to lower friction and wear and the reason is probably the formation of a tribofilm, which gives low friction and protects the surfaces from wear. When studying wear tracks of PEEK with carbon fibers, PTFE and graphite in Paper III, fine threads can be seen, see Figure 14. These threads indicate that PTFE can be spread over the wear track, another sign of a surface tribofilm of PTFE. No surface layer of PTFE can be detected by EDS in the cross sections, probably due to the fact that this tribofilm is too thin to give any clear results when mapping with EDS. The fact that the analysis takes place close to an edge makes any results questionable. In Paper III, where the wear tracks are mapped by XPS with respect to the signal for fluorine (F), it is clearly shown that there is some sort of surface layer containing F, see Figure 15. This comes from the PTFE since no other F containing material is present. This surface layer is also present for PA6T/66 with glass fibers and PTFE even though it had a relatively high friction coefficient, 0.3. The fact that the tribochemistry can affect the performance of reinforced PTFE might be an explanation for this deformation. Within PA6T/66 with glass fibers and PTFE the formation of a protective tribofilm might not be favorable. It may also be that the deformation depends not only on the transferred material to the counter surface, which the tribofilm can reduce, but that the deformation takes place further down in the matrix, which the tribofilm cannot protect against. The high friction is a sign of this. But comparing these results with the ones for PA66 with PTFE and silicone oil, which has a much lower elastic modulus than PA6T/66 with glass fibers and PTFE, it is hardly deformed at all. PA66 with PTFE and silicone oil had a much lower friction, about 0.05. Maybe the insufficient adhesion between polymer matrix and fibers leads to the higher friction and deformation or maybe silicone oil is a much better lubricant, forming an even better tribofilm. No cross sections or XPS analysis have been done of PA66 with PTFE and silicone oil so no conclusion can be drawn.

These studies are just the first and more studies, with a deeper analysis combining SEM, cross sections and XPS, are required to understand this tribofilm better.
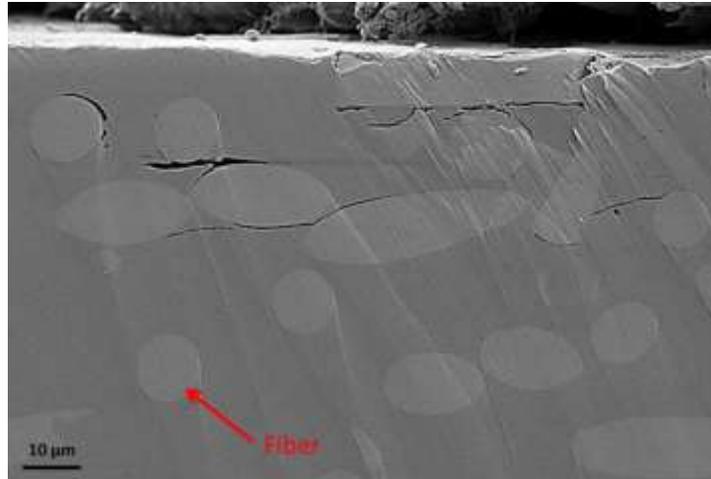
Figure 10. Cross section perpendicular to the wear track of PPS with GF tested against ball bearing steel ball at RT, 15 N contact load and 10 000 cycles. Small cracks are visible under the surface.
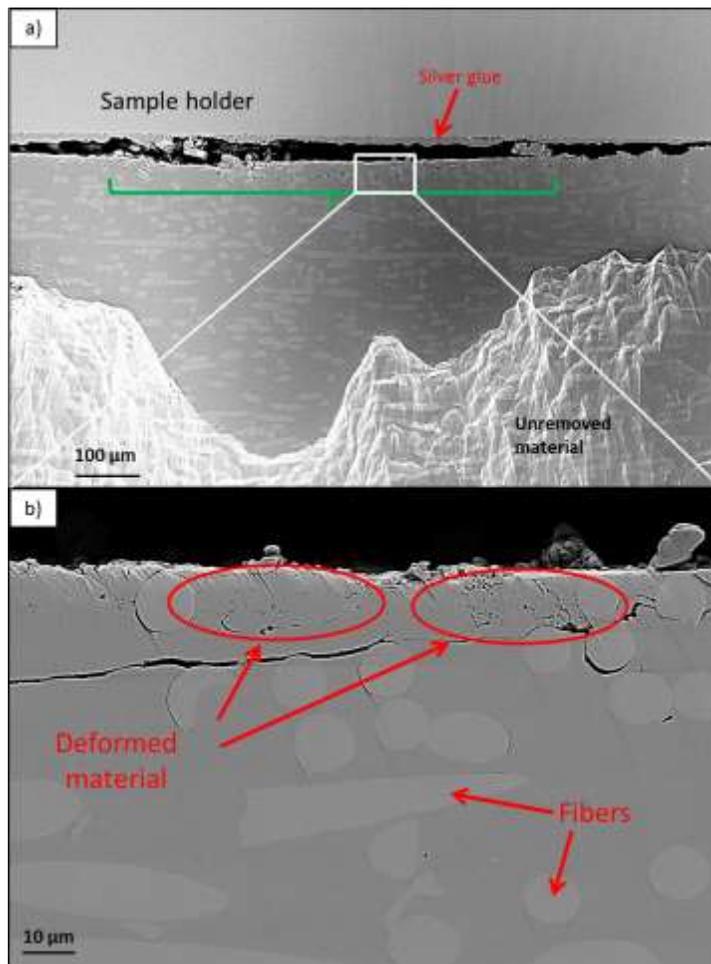

Figure 11. Cross section perpendicular to the wear track of PPS with GF tested against ball bearing steel ball at 120 °C, 15 N contact load and 10 000 cycles. a) An overview. The approximate width of the wear track is indicated with the green line. A crack below the surface is seen almost as wide as the wear track. In the lower part of the image residual ion milled material is visible. b) Fibers are visible and deformed material can be seen at the surface.
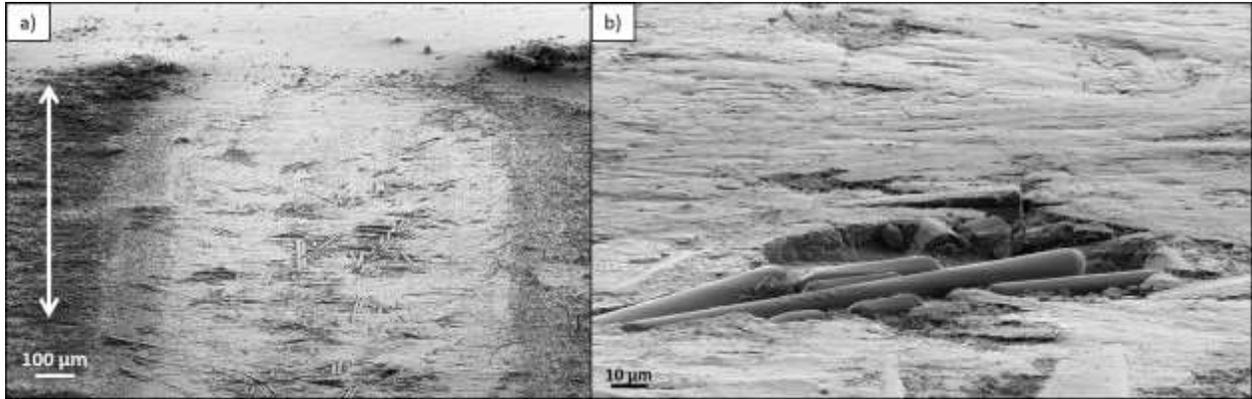
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Figure 12. a) An overview and b) a close up on PPS with GF tested against a ball bearing steel ball at 120 °C, 15 N contact load and 10 000 cycles. Exposed fibers, pits, cracks, wear debris and delamination of material can be seen. The arrow indicates sliding direction.
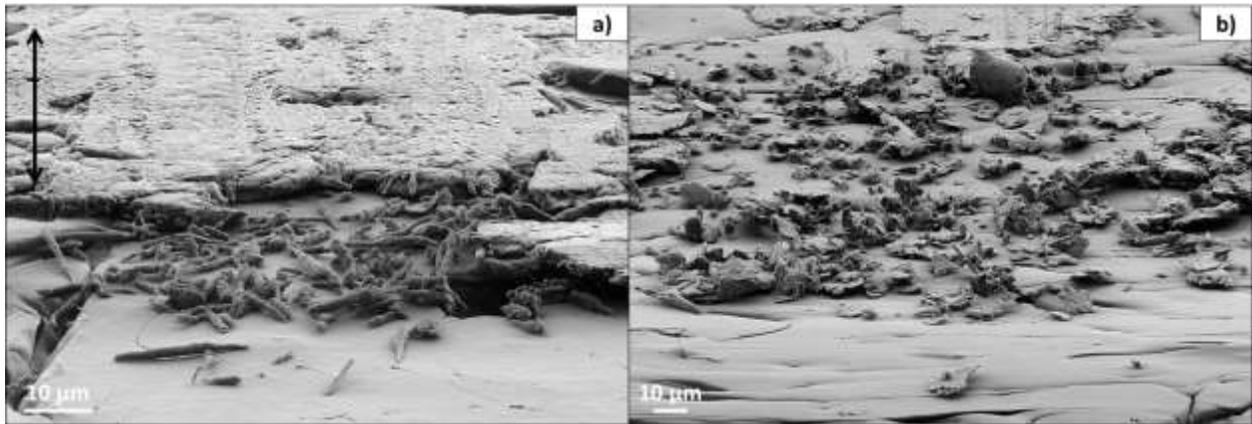

Figure 13. Wear particles at the turning position of a) PA6T/6I with GF at 80 °C and b) PA6T/66 with GF and PTFE at RT tested against steel cylinders with 15 N contact load for 20 000 cycles. The wear debris differs and is roll-like in a) and flake-like in b). The arrow indicates sliding direction.
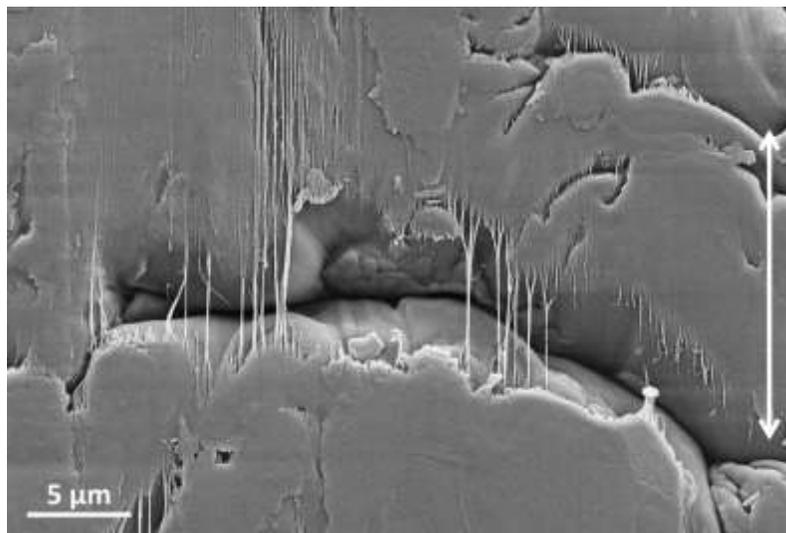

Figure 14. PEEK with CF, PTFE and graphite tested against steel cylinders at 80 °C, 15 N contact load and 20 000 cycles. Fine threads of PTFE are seen in the wear track. The arrow indicates sliding direction.
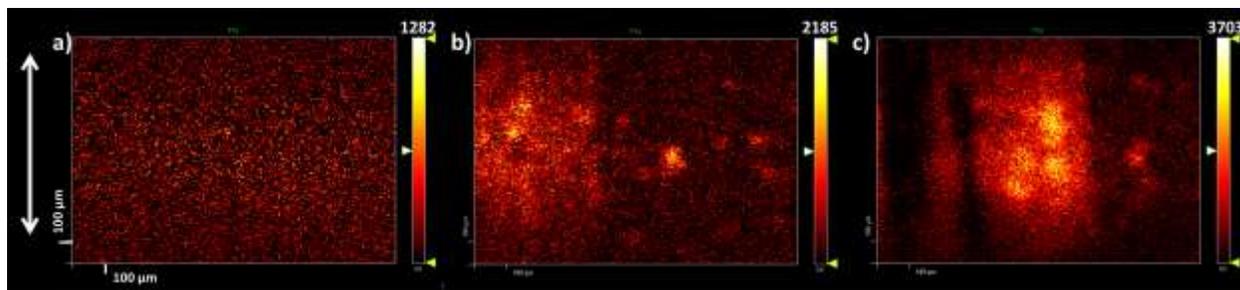
Figure 15. XPS results of a) PA6T/6I with GF, b) PA6T/66 with GF and PTFE and c) PEEK with CF, PTFE and graphite tested against steel cylinders at 80 °C, 15 N contact load and 20 000 cycles. The left hand side of the map is within the wear track and the right hand side outside of it. The exact position of the wear track differs a bit between the three maps. The mapped signal is for F and indicates a tribofilm of PTFE in the wear track in a) and b) while no such surface layer is visible in c). The arrow indicates sliding direction.

4.4 Influence of polymer matrix

As already described the polymer matrix seems to influence the effect of PTFE on the friction and wear. Adding PTFE to a composite is expected to lower friction and wear, but in Paper III the difference between PA6T/66 with glass fibers and PTFE and PA6T/6I with glass fibers is small. This might be due to the polymer matrix not being able to withstand the stress from the tribological testing. The PA6T/66 with glass fibers and PTFE material shows clear signs of deformation. Extrusion of material into tongues at the edges of the wear tracks is seen. The deformation is also clear when studying the cross section of the wear track, see Figure 16. In this case it seems as if the adhesion between polymer matrix and fillers is insufficient. Poor adhesion between polymer matrix and fibers might result in fibers coming loose from the surface leading to more wear for instance. The resulting void in between fiber and polymer matrix can also worsen the performance since these voids will grow when subjected to stress from the tribological testing. This might be the reason for large deformation in our case. Another reason could be that this specific polymer matrix has low strength and/or resistance to deformation.

When studying the different polymer composites they show both similarities and differences. The friction coefficient can be about the same but the deformation of the wear track can be very different, see Figure 17.

One important parameter to take into consideration when choosing polymer composites for elevated temperature applications is the glass transition temperature T_g. The results show that when the polymer composites are tested at temperatures higher than their T_g the surface characteristics of the wear tracks changes. No definitive conclusion can be drawn from the friction since it decreases for some and increases for other polymer composites above their respective T_g.

The different polymer matrices also seem to have a variation in adhesion to the fillers. But as said before it is hard to draw any conclusion from this since the fillers might differ from each other for the different polymer composites. As for PA6T/66 with glass fibers and PTFE no conclusion can be drawn about the cause for the large difference in performance compared to the other polymer composites containing lubricating additive additives. It might be due to the polymer matrix's deformation under stress or that PTFE does not form a sufficient tribofilm to protect the surface. Further studies are needed to try to understand this effect.
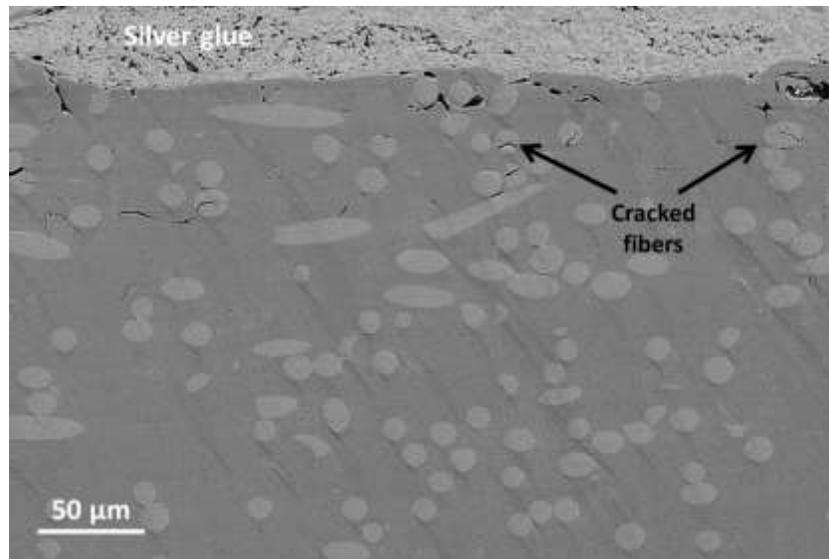
Figure 16. Cross section parallel to the wear track of PA6T/66 with GF and PTFE tested against steel cylinder at 80 °C, 15 N contact load and 20 000 cycles. Deformation at as well as below the surface is seen. Cracked fibers are present no more than about 60 um below the surface.
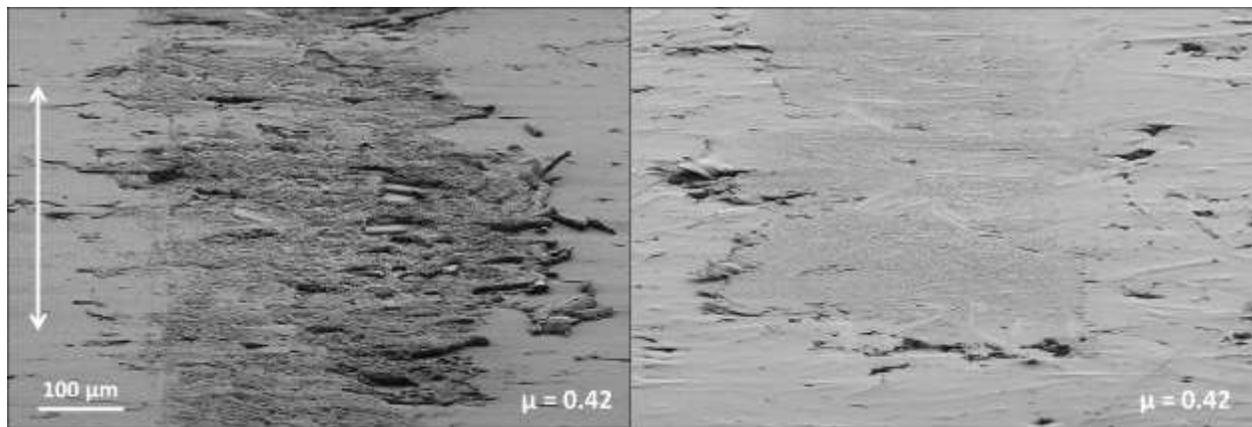

Figure 17. Wear tracks of a) PA66+PA6I/X with GF and b) PA6T/6I with GF tested against ball bearing steel balls at RT, 5 N contact load and 2000 cycles. The mean value of the friction coefficient is 0.42 for both a) and b) but the deformation in the wear tracks is distinctly different. The arrow indicates sliding direction.

4.5   Concluding discussion

The combination of fiber-reinforcement and lubricating additive, such as PTFE, is favorable when using polymer composites as mechanical components. Fibers add strength to the material and the lubricating additive can lower both friction and wear. The tribofilm formation is an important property for these polymer composites and the mechanisms behind it need to be further studied.

In this thesis the main focus has been the wear and deformation of the polymer composites. Although there are clear signs of transferred material to the counter surfaces in Paper I no further studies have been done. Studies of the mechanisms behind material transfer and transfer layer formation could give more information about the deformation and wear of the polymer composites.

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In Paper I cracks in the wear tracks of PEEK with carbon fibers tested at 150 °C can be seen, see Figure 18, which are a sign of deformation hardening. This process might create a harder surface layer which might change the transfer layer and surface properties and affect the tribological performance. If this only occurs for PEEK with carbon fibers, or if it happens for all materials sooner or later, is not known but something that might affect the performance over time for these polymer composites.

One of the tested polymer composites contains a thermally conductive modifier as filler. The main purpose of such modifiers is that any rise in temperature in the contact should be drawn away from the contact and thus enhances the performance of the polymer composite and the possible rise in contact temperature it can be subjected to. This polymer composite showed the highest wear and surface damage as well as high and the most unstable friction (Paper I). Since thermal conductivity has not been the main focus of this thesis, this material has not been studied further.

In general the deformation of the surfaces has some characteristics in common for all material. There are wear particles in the turning positions as well as tongues of material that seems to have been extruded from the wear track due to the deformation. If this is due to viscous plowing or gradually extrusion of material is not certain. More studies are required in order to understand these mechanisms better. What is clear is that there are more wear particles and material removed from the wear tracks for the polymer composites with only fibers. This might be due to the lack of a low friction tribofilm at the surface, which means that the polymer is more deformed.

The cross sections in Paper II and III give initial information about subsurface deformation and crack formation, which is important to further understand the wear mechanisms for polymer composites. To get a deeper understanding of these mechanisms more cross sections need to be studied of tribologically tested polymer composites.
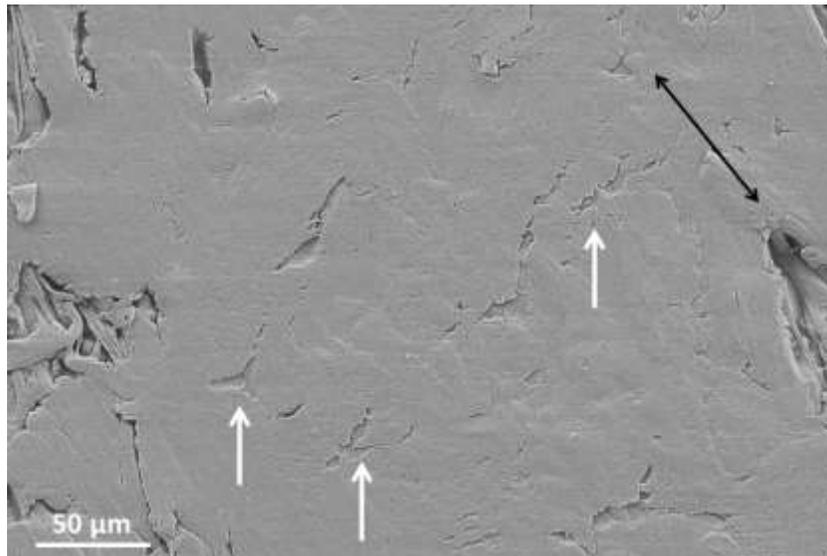

Figure 18. Wear track of PEEK with CF (M1) tested against ball bearing steel ball at 150 °C, 5 N contact load and 2000 cycles. White arrows indicates surface cracks due to deformation hardening by repeated deformation. The black arrow indicates sliding direction.

5 Conclusions

The work presented in this thesis has been focused on tribology of polymer composites. From the results obtained using different tribological test set-ups and analysis methods the following conclusions can be drawn:

- The formation of low friction tribofilms lowers both friction and wear. Not only wear of the polymer composite itself but also wear of the counter surface.

- PTFE as lubricating additive forms a protective tribofilm at the surface, resulting in lower friction and less surface and subsurface damages. All except for one test where the damages are comparable to unlubricated. This is thought to depend on an insufficient tribofilm, giving too high friction to prevent deformation.

- Cross sections reveal distribution and orientation of fillers in the polymer matrix.

- Cross sections give information about surface deformation and subsurface damage, both about the polymer itself but also about worn material from the counter surface embedded in the wear track of the polymer composite.

- Subsurface crack initiation occurs before failure. These subsurface cracks grow in size and finally lead to delamination and removal of material from the wear track. This exposes fibers that can abrade the counter surface causing worn material from the counter surface material in the wear track.

- Glass fibers seem to wear the counter surface more than carbon fibers.

- A slight increase in temperature can give better tribological performance of the polymer composites than at room temperature. Probably due to a lower shear resistance at the surface while the bulk's elastic modulus in unaffected.

- T_g seems to be an important parameter when using polymer composites at elevated temperatures. The friction behavior is often changed above T_g and the wear tracks changed appearance. A sufficient low friction tribofilm can prevent deformation and wear even above T_g.

- The counter surface roughness and characteristics seemed to be more important than type of material for the surface damage of the polymer composites.

6 Future work

Much has been done within the field of polymer tribology but in general more knowledge about the mechanisms behind friction and wear phenomenon is needed to get a better understanding.

The deformation of polymer materials and the mechanisms behind it would be interesting to do deeper analysis of. For the tongues of material exiting the wear track as well as wear particle formation and subsurface deformation are mechanisms to study further. Studying the wear tracks in more detail might give information about deformation hardening, transfer layers, tribofilms and other surface characteristics, which are also something to study further. It would also be interesting to study the counter surfaces more closely. Is this transfer layer present on the counter surface as well, how thick is it and what about the adhesion? Tribofilm formation and the presence of a tribofilm on the counter surface is also something for the future. How well the tribofilm performs compared to traditional greases is an interesting question. Tests comparing lubricated polymer composites with only reinforcement and dry tests of polymer composites with fibers and lubricating additives would be interesting. Do they behave the same and can they maintain this low friction and wear for the same length of time. Here it might be useful to use fretting conditions, to see if the tribofilm eventually gets pushed out of the contact or if it manages to reform and keep protecting the surfaces.

For the future it would also be desirable to compare the composites to pure polymer material to further understand the role of reinforcement and lubricating additive within polymer tribology.

7   Sammanfattning på svenska (Summary in Swedish)

Inom industrin används polymera material till många komponenter och applikationer. På senare år har högre krav börjat ställas på materialen då de i större grad används i mer påfrestande situationer. Detta kräver bättre mekaniska, termiska och tribologiska egenskaper hos de polymera materialen. För att förbättra dessa egenskaper används olika fyllnadsmedel, från vanliga glasfibrer till mer avancerade nanopartiklar. Dessa fyllnadsmedel är t.ex. fibrer eller andra hårda partiklar för att öka den mekaniska hållfastheten, poly-tetra-fluoro-etylen (PTFE) eller grafit som fasta smörjmedel, eller partiklar med bra värmeledningsförmåga för att förbättra denna hos den polymera kompositen. I denna studie har tre typer av baspolymerer studerats: poly-amid (PA), poly-fenylen-sulfid (PPS) och poly-eter-eter-keton (PEEK). Dessa är fyllda med glasfiber, kolfibrer, PTFE, grafit och värmeledande partiklar i olika kombinationer.

Fem generella grupper av polymera kompositer har studerats:

- Ren PEEK
- PPS, PA och PEEK fyllda med fibrer
- PPS, PA och PEEK fyllda med fibrer och fasta smörjmedel
- PA fylld med fasta smörjmedel
- PEEK fylld med fibrer, fasta smörjmedel och värmeledande partiklar

Dessa material har testats tribologiskt i en uppställning med glidande kontakt med fram -och återgående rörelse. Friktionen har mätts och nötning, deformation och andra skador har studerats. Tre typer av motytor har använts: kullagersstålskulor, cylindrar av rostfritt stål samt cylindrar av anodiserat aluminium. Pålagd last, yttemperatur hos de polymera kompositerna och antalet cykler har varierats. Detta för att studera och jämföra friktionen och nötningen hos de olika materialen. Nötningsmärkena hos de polymera kompositerna har studerats med svepelektronmikroskopi (SEM). Tvärsnitt har gjorts med hjälp av jonpolering för att studera eventuella skador och deformationer under nötningsytan.

När det kommer till friktion så visade de polymera kompositerna med fasta smörjmedel en lägre friktionskoefficient, ofta runt 0.05-0.15, än de som bara var fiberförstärkta, som ofta låg omkring 0.4-0.5. Även nötningen och deformationen av materialen var mindre för de polymera kompositerna med fasta smörjmedel. Det fanns ingen entydig inverkan av temperatur, men vanligen gav en viss ökning i temperatur lägre friktion. Dock fanns det ofta en kritisk temperatur för materialen, över vilken materialens beteende förändrades vad gäller både friktion och nötning. Med ökande last visade resultaten jämförbar friktion men bredare nötningsspår. Det kan finnas skillnader i deformation under ytan mellan de olika lasterna men då inga tvärsnitt gjorts för att studera detta går det inte att bekräfta. Tvärsnitten visade att de polymera kompositerna med endast fiberförstärkning hade sprickor och skadade fibrer under ytan. Ofta fanns också nötningspartiklar från motytan inbäddade i nötningsytan. Att det enbart var fiberförstärkta polymerer som visade detta kan bero på de höga spänningar som uppstått i materialet i kontakten på grund av den höga friktionen. Med fasta smörjmedel som gav låg friktion fanns inga ytskador och inte heller några sprickor eller spruckna fibrer under ytan. Det verkar som att de fasta smörjmedlen bildar en skyddande tribofilm i nötningsspåret, vilket ger både låg friktion och lägre nötning och deformation. För vissa nötningsspår kunde en sådan ytfilm bekräftas genom elektronspektroskopi för kemisk analys (XPS) som visade att en skikt innehållandes F, från PTFE, fanns i nötningsspåret.

Till slutsatserna hör att de tribologiska egenskaperna hos polymera kompositer är starkt beroende av dess fyllnadsmedel, t.ex. kan fasta smörjmedel bilda en tribofilm som ger låg friktion och nötning. En ökning i temperatur kan drastiskt förändra det tribologiska beteendet hos polymera kompositer och därför är det viktigt att testa materialet vid högre temperaturer om den är tänkt att används vid sådana. Tvärsnitt av nötningsspår kan ge information om ytskador och kan bidra till att förstå de mekanismer som ligger bakom friktion och deformation av polymera material. Mer ingående studier bör göras för att bättre förstå det tribologiska beteendet hos polymera kompositmaterial.

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