

Article

Effects of Additives Adsorbed to Wet Clutch Disks on Their Tribological Performance Found in a Comparative Investigation

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Abstract

Tribolayer build-up was investigated on wet clutch steel disks with experiments in three different test levels of the tribological verification chain via X-ray photoelectron spectroscopy. A new characteristic value of the tribolayers was developed, i.e., the lateral elemental distribution uniformity. This value together with the parameters of additive elemental concentration as well as the thickness and the vertical composition of the layers were the basis for the subsequent correlation of the tribolayer properties with the tribological performance of clutch systems from different tests. One important finding was a non-zero threshold concentration of the additive elements calcium and phosphorous below which the clutch performance was unsatisfactory, even with a uniformly covered disk, concluding that the presence of some slightly higher additive element concentration seemed to stabilize the clutch performance. The importance of representative investigation area selection was demonstrated.

Keywords: wet clutch; X-ray photoelectron spectroscopy; tribolayer composition; shudder behavior



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1. Introduction

It is well known and established state of the art that the behavior of a tribosystem is influenced not only by material properties and surface geometry (both micro and macro) of the contacting tribo-elements, but also by tribologically functional chemical layers (tribolayers) that are formed during operation, e.g., by the lubricant under sufficient power and energy input. The influence of material properties and surface geometry in dependence of the loading conditions of wet clutches has already been investigated and described in numerous works [1–6]. But, in real application a better understanding of the impact of tribolayers on the wet clutch performance is needed, because some of the observed tribological phenomena cannot be explained only by the material or micro geometrical property change in the surface or the properties of the oil. To better understand that influence, more detailed descriptions of the mechanisms that occur in the tribological contact are needed. For this purpose, it is necessary to define appropriate properties that can be described quantitatively, and to achieve a more thorough picture of the structure of the tribolayers.

For the formation of a stable tribolayer, the appropriate lubricant is crucial. In wet clutches, automatic transmission fluids (ATFs) are often applied. However, they also have additional tasks such as lubricating the friction surfaces, rapid heat dissipation for sufficient cooling of the clutch, keeping the system components clean and, of course, adjusting the desired tribological performance [7–10]. To meet all requirements imposed, ATFs must possess specific properties. These include, among others, anti-shudder characteristics, excellent antiwear properties, low viscosity in combination with a high viscosity index, and a high oxidative stability [8,11]. Thus, they mainly consist of mineral or synthetic base oils with additive packages, which are specifically designed according to the requirements of clutch applications. Additives contained in such additive packages are, e.g., friction modifiers (e.g., amide, amine, acids, alcohols), detergents (e.g., sulfonates), dispersants (e.g., succinimide), antiwear compounds (e.g., ZDDP, phosphates, phosphites), viscosity index modifiers (e.g., olefin, poly-methacrylate), and antioxidants [12,13]. However, current trends in the formulation of ATFs are moving in the direction of replacing fossil components with sustainable, environmentally friendly alternatives. In this context, vegetable oils have turned out highly promising due to special advantages like biodegradability, renewability, non-toxicity, or improved interaction with metallic surfaces resulting in strengthened tribolayers [9,14].

To guarantee a smooth operation of the clutches, it is of great importance not only to know the system components already applied [15], such as mechanical elements or ATFs, but also to analytically assess the tribolayer that only forms during operation. In an early example of investigating wet clutches that takes the lubricant and specific additives into account, Heilenkötter et al. [16] focused on the micro slip occurring in closed clutches as an undesirable phenomenon. In an extensive test bench experiment series, parameters like temperature, compressive stress, and difference in rotational speed, as well as geometrical aspects of construction were systematically tested for their influence on the tribological behavior of the clutch. The main factors determining the slip characteristic that were subsequently identified are misalignment of drive shaft and output shaft, surface structure, and mechanical stability of the paper disk, as well as the lubricant properties, in particular friction modifiers and detergents.

A thorough consideration of the shudder behavior of wet clutches can be found in [17,18]. In systematic experimental investigations, the influences of friction pad material, steel disk surface finish, and lubricant composition and state in dependence of the operational parameters were examined. It was found that shudder behavior can be avoided by careful optimization of the composition of the lubricant. In particular, the effects and interactions of different friction modifiers, antiwear additives, and detergents were considered. Accompanying surface analyses allowed further correlations with the tribolayer properties. Moreover, deterioration of the clutch behavior with respect to the shudder caused by lubricant aging and contamination was observed [19]. Zhou et al. transferred the insights acquired at the component level up to the system level and investigated gear rattle in dual-clutch transmissions [20]. Further studies focused on the influence of oil temperature on friction characteristics and wear behavior [10,21,22]. It was discovered that, due to lower oil viscosity at higher temperatures, a smoother worn surface is developed affecting the tribolayer formation and stability.

Zhao et al. investigated the influence of lubricants on shudder behavior in wet clutches [23,24]. Synthetic oils additivated with friction modifiers and dispersants were used in a model test setup with clutch materials, varying temperatures and rotational speed differences, and constant compressive stress. The authors found correlations between the chemical properties of the oil and the physical properties of the disk materials, with different consequences for the friction behavior depending on the rotational speed difference.

Moreover, the adsorption of lubricant on the involved surfaces as well as the clutch material porosity were identified as significant factors. In particular, detergents gathered in the pores and thereby worsened the shudder behavior of the clutch [25]. Further investigations used ToF-SIMS to inspect tribolayers after thermal and mechanical stress and analyze the correlation to base oils and oil additives [26–30]. This influence coheres with friction characteristics observed from test-rig based analyses [29].

Experimental studies dedicated to the influence of water contamination demonstrated that appropriate tribometer experiments are able to reproduce the tribolayers formed in wet clutches in vehicle operation [12,31–33]. Analyses of the steel disks of both origins with ToF-SIMS showed that the influence of water added to the lubricant, in this case responsible for the undesired shudder behavior of the clutch in the vehicle, was likewise reflected in the structure of the tribolayers from the tribometer.

Zhao et al. investigated the correlation between ATF chemistry, tribolayer composition and shudder properties of friction pairings based on laboratory tests with different oil formulations [34,35]. They concentrated their research, including ATR-FTIR, SEM/EDX, XPS and ToF-SIMS analyses, on various organic functional groups of the tested oil additives, verifying that these groups could also be detected on the disk surfaces after the tests. The respective additives were accordingly linked to the quality of the shudder performance. Ca and P, likewise, originating from oil additives, were also detected on the sample surface, but not or only very loosely correlated with the shudder properties.

Li et al. also investigated laboratory scale tests of friction pairings, using oils with various additive packages and employing SEM/EDX, XPS and ToF-SIMS for post-test surface analysis [36–38]. The focus of this work was the Ca and P chemistry, initially provided by detergents and friction modifiers, respectively. They conclude that Ca and P react in the contact, forming a hydroxyapatite tribolayer that improves the shudder performance.

However, information on lateral distribution of additive components and its impact on clutch performance is missing in the literature. In the present paper, tribolayers on steel disks from long-time component, system, and vehicle tests are investigated by large-area XPS mappings of significant elements and depth profile measurements. Correlations between characteristics and distribution of the tribolayers and shudder performance of the samples are discussed.

2. Materials and Methods

2.1. Gear Oils

Within this study, two different transmission oils designed for wet clutch applications were applied. Table 1 gives an overview of oil technology, the viscosity range and the elemental composition of oil A and B.

Table 1. Lubricant properties.

Designation	A	B
Base oil	Mixture of group IV/III (PAO/Mineral oil)	Group III (Mineral oil)
Viscosity in cst @40 °C	28	17.5
Viscosity in cst @100 °C	6	4.4
Ca in mg/kg	530	220
Mg in mg/kg	100	-
B in mg/kg	130	140
P in mg/kg	350	280
S in mg/kg	1600	1400
Zn in mg/kg	<5	<5

2.2. Investigated Disks

In the current study, steel disks and friction disks with paper material with the same specification each were kept constant during all performed tests. For this purpose, commercially available steel disks with an inner diameter of 99 mm, an outer diameter of 140 mm and a thickness of 1.3 mm were applied. An exemplary photographic documentation of both paper (a) and steel (b) friction disks as well as a zoom on the selected area for further surface investigation (c) is presented in Figure 1. It has to be noted that the measuring area was chosen to scan the total radial width of the disk ($y = 17.5$ mm). The length of the measuring area was selected regarding to reasonable feasibility in accordance with scientific added value and lay between 5 and 20 mm.

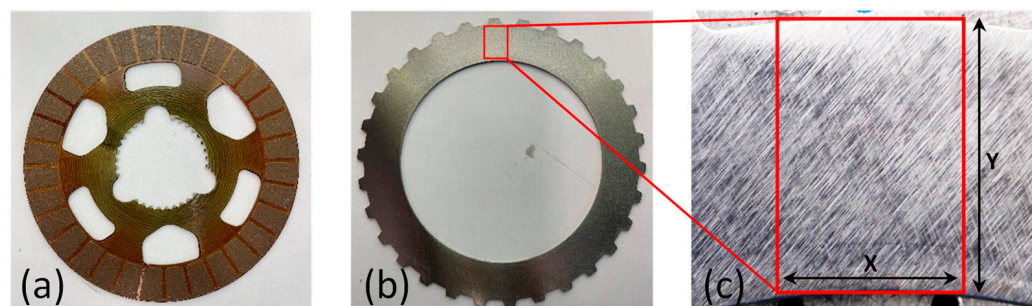


Figure 1. Photographic documentation of paper friction disk (a) and steel disk (b) as well as approximate area of surface investigation marked by red rectangle with x as measuring length and y as measuring width (c).

In this study, the results of all performed tests were compared related to the specific energy input. To calculate this specific comparison value, the nominal contact area was used which was defined as the area of the paper friction disk that is in contact with the mating body of the steel disk. Thus, it corresponded to the sum of the 32 friction pad areas without the respective grooves, as depicted in Figure 1a. The nominal contact area was defined according to the construction drawing.

2.3. Tribological Exposure of the Steel Disks

The disk samples used in the context of this paper have been obtained from durability tests of different test levels, in particular, from component tests, system tests, and vehicle tests.

Component tests exhibit the highest degree of simplification compared to system and vehicle tests, because only the clutch pack together with the oil is evaluated. They can be performed at lower costs, enabled by easier production and assembly of samples and less test complexity. Additionally, component tests can run under more controlled conditions and provide detailed information from various measurement sensors, which are more complicated to be integrated in system or vehicle tests.

System tests include the impact of lubrication effects in the clutch influenced by the unit design as well as other factors, such as geometrical deviations of the architecture or contaminations from other wearing design parts. Nonetheless, they represent an important link between real-life application and laboratory setups.

In general, vehicle tests possess the highest real-life relevance as no simplifications in system designs and system integration into the powertrain are made and, hence, environmental influences are reconsidered. However, vehicle tests result in comparable high costs, which is why durability tests in the vehicle are usually performed in later development phases. Before, the functionality, performance, and lifetime of the clutch application needs to be proven on component and system test level as well as with functional tests in the ve-

hicle based on an extensive design verification plan (DVP), the functionality, performance, and lifetime of the clutch application.

Figure 2 shows the test levels, which were used for clutch durability tests and their differences in influencing parameters. Vehicle tests thereby were performed in real passenger cars, operated in common road traffic, and system tests were performed in 3 e-machine test benches, and the component tester is described in the following section.

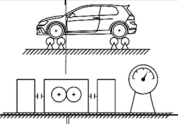
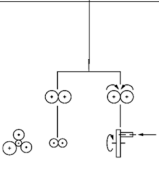
Category	Test level	Principle sketch on the example of gear test in vehicles	Cost level / Complexity / Testing Time	Represent tribocontact conditions	Tribosystem Specification	Lubrication Conditions	Further Design Influences	System Integration
I	Vehicle test		very high	other influences next to the tribocontact properties given	origin	origin	origin	origin
II	Vehicle test on test bench		↑	↓	test level not reconsidered in current investigations			
III	System tests				origin	origin	origin	- vehicle geometrical integration, powertrain stiffness and environmental influences (e.g. surrounding air humidity, dirt) not reflected.
IV	Component tests				origin	- lubrication influence as parameter excluded (only tribological behavior in the friction contact shall be investigated).	- in durability tests: no consideration of wear or liquid media influence - ok/nok rating: used clutch plates and used oil consisting deposits from system design reconsidered	- same deviation from vehicle test as system test - drivetrain stiffness in component test designed comparably sensitive to detect tribological failures with evidence
V	Model tests with similar sample geometry and similar conditions				test level not reconsidered in current investigations			
VI	Model tests with simplified sample geometry				test level not reconsidered in current investigations			
			low	very good				

Figure 2. Tribological test chain as applicable to the current investigation (adapted from [39]).

A detailed description of the component test rig is given in [40] as well as [41]. In this rig, four steel disks and three paper friction disks were used for one test with the given lubricant. Rotational frequency is applied to paper friction disks by a torsion-sensitive shaft, which includes a torque measurement cell. This shaft is connected to the motor with a belt. Load is applied hydraulically from the bottom of the test cell. The oil is pumped in a circuit. To gain a parameter of specific energy input, the energy input introduced to the

system was divided by the total nominal contact area of one disk surface times the number of contacting surfaces.

2.4. Evaluation of the Friction Acceptance Criteria

In general, the friction performance of the tested setups was categorized into “okay” (o.k.) and “not okay” (n.o.k.), namely the friction acceptance criteria, using well-established evaluation methods described in the following section.

The component tests used a torque measurement cell to measure the friction torque. In case of vibrations, the friction torque oscillates about its mean value with a noticeable amplitude. To compare different systems and/or load conditions, an equivalent coefficient of friction is computed according to the following equation:

$$\text{COF} = \text{torque} / (\text{load} \times \text{mean radius of paper friction disk} \times \text{number of contacting surfaces})$$

Figure 3 shows the coefficient of friction of all three component tests at 2 MPa nominal pressure.

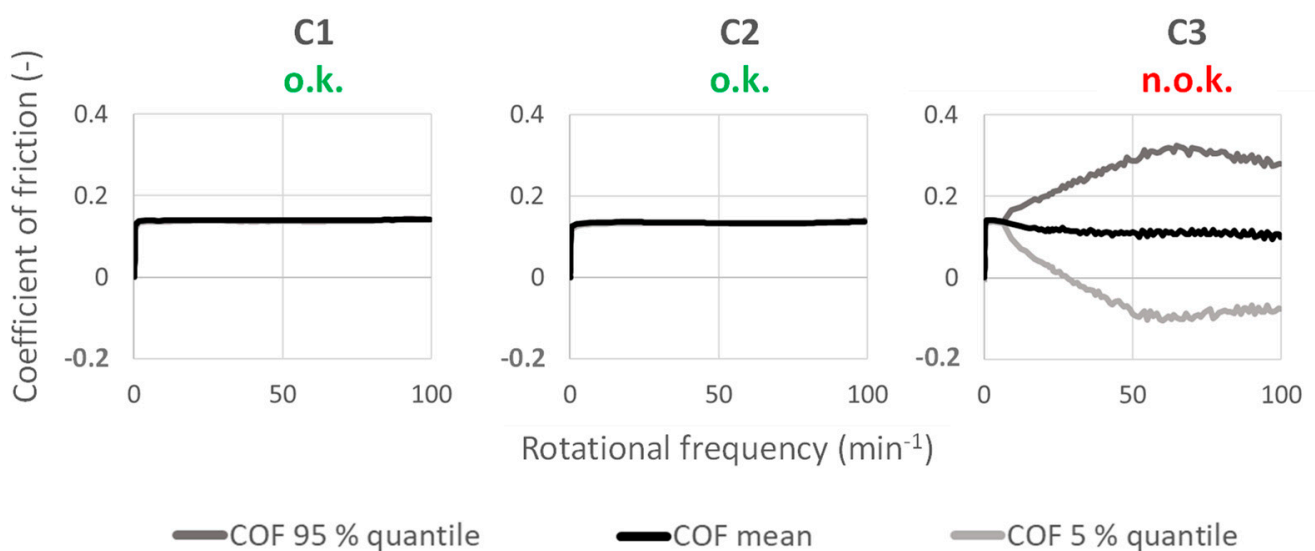


Figure 3. Coefficient of friction (COF) in component tests at 2 MPa nominal pressure and rotational frequency linearly increased from 0 to 100 min⁻¹ in 10 s at 30 °C oil temperature.

The vibration amplitude is shown via the 5 % quantile and 95 % quantile of the COF. For this purpose, the measured signal is sampled at 1000 Hz and aggregated over 10 Hz. Consequently, the quantiles and the mean of the COF are computed over a block size of 100 samples. In system C1 and C2, COF 5 % quantile and 95 % quantile are very close to COF mean (arithmetic mean), which is classified as “okay” (o.k.). In contrast, in system C3, the COF 5 % quantile and 95 % quantile show strong deviations to COF mean and therefore strong vibrations. This state is also accompanied by strong noises and vibrations. This system is classified as “not okay” (n.o.k.).

In general, the “o.k.” and “n.o.k.” ratings from component tests correlate with the observations regarding clutch noise, vibration, and harshness (NVH) ratings performed in system and vehicle tests during endurance testing at defined checkpoints and with specific, state-of-the-art evaluation procedures. Within this study, such correlations could be identified with validation tests performed on two test rigs: (1) the component test rig described in [41] and (2) the component test rig KLP-260 described in [19]. Selected used clutch disks and related used oils from vehicle and system tests were evaluated regarding

their friction acceptance criteria with a defined procedure on component test level to prove a tribological “o.k.” or “n.o.k.” behavior.

2.5. Sample Overview and Classification of Behavior

Table 2 summarizes the samples investigated and displays their respective tribological origin and history.

Table 2. Sample overview and classification of behavior.

	Test Type	Lubricant	Temperature	Specific Energy Input (kWh/dm ²)	Friction Acceptance Criteria
C1	Component test	A	low	94.4	o.k.
C2	Component test	B	mid	69.9	o.k.
C3	Component test	B	high	35.0	n.o.k.
S1	System test	A	low	3.7	o.k.
S2	System test	A	mid	2.8	o.k.
S3	System test	A	low	2.7	n.o.k.
S4	System test	A	low	2.7	n.o.k.
V1	Vehicle test	A	low	1.0	n.o.k.
V2	Vehicle test	A	low	1.0	n.o.k.
V3	Vehicle test	A	low	2.7	o.k.

2.6. Surface Analysis

2.6.1. XPS Analysis

Prior to analysis, the steel disks were cleaned in an ultrasonic bath with petroleum ether.

X-ray photoelectron spectroscopy (XPS) was performed with a Thermo Fisher Scientific Theta Probe (Waltham, MA, USA) equipped with a monochromatic Al K α X-ray source (1486.6 eV). The base pressure in the analysis chamber was base pressure 2×10^{-8} Pa. An X-ray spot size of 400 μ m resolution was chosen for all analyses. Survey spectra were acquired at a pass energy of 200 eV. Elemental detail spectra were acquired at a pass energy of 200 eV during mappings and 50 eV for depth profiles.

XPS mappings extended from the outer to the inner diameter of the steel disk (see Figure 1c), with a lateral step size of 500 μ m. Spectrum evaluation was performed with the Thermo Fisher Scientific Avantage Data System software Version 6, using Gaussian/Lorentzian curve fitting. The C1s peak for adventitious carbon at 284.6 eV was used as binding energy reference.

XPS sputter depth profiles were acquired using 3 keV Ar⁺ ions for sputtering. Prior to the mappings, the samples were briefly cleaned by sputtering. Sputter time to depth conversion was estimated at 0.05 nm/s, related to sputter values of SiO₂ on Si.

2.6.2. Evaluation of Lateral Elemental Distribution Uniformity

Exemplarily, the evaluation of the lateral elemental distribution uniformity of phosphorus on three different disks is displayed in Figure 4.

Elemental maps of the samples were acquired to investigate the distribution uniformity of elements over the steel disk surface. A distinction was made between the following surface appearances: (a) sharp uniform distribution, (b) wider range of concentrations, but still distributed uniformly, and (c) distinct areas of differing concentrations (i.e., stains). To evaluate the significance of the distribution uniformity, the resulting maps were converted into histograms and afterwards fitted with one or, in the case of stains, two Gaussian functions. This resulted in numerical values for the peak concentration(s) and the spread of the distribution over the respective concentration range.

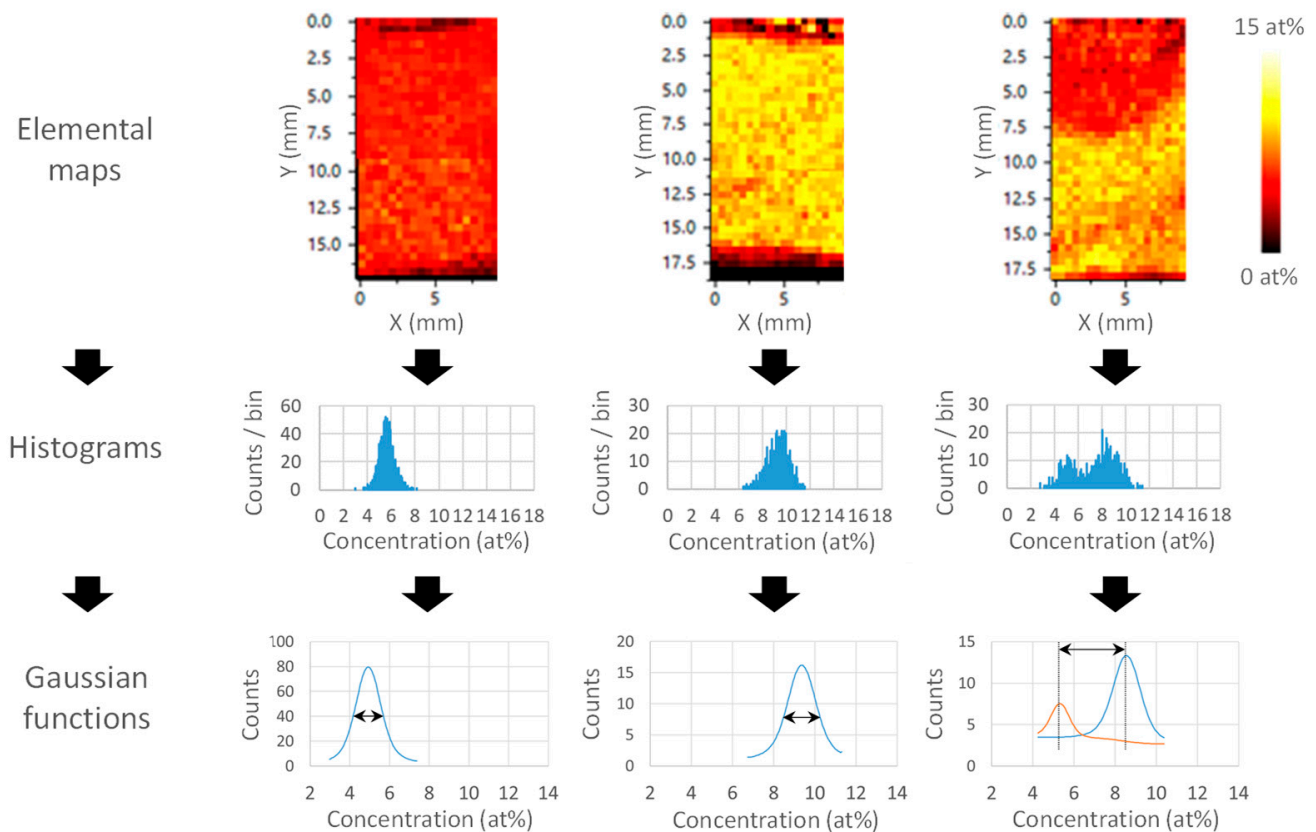


Figure 4. Evaluation of lateral elemental distribution uniformity via elemental maps, histograms, and Gaussian functions. Note: Displayed results are examples of sharp uniform distribution, wider range of concentrations, but still distributed uniformly, and distinct areas of differing concentrations. They intend to illustrate the scientific approach.

From the fitted histogram curves, a single “uniformity value” was defined for each elemental map to enable comparing the uniformity of the elemental distributions with each other. In the first step, for the width of the histogram, the full width at half maximum (FWHM) of the Gaussian function was taken in the case of single peak distributions and the distance between peaks for dual peak distributions (see marked by arrows in Figure 4). As the second step, this value was subsequently normalized by dividing it by the concentration range of the respective element found for all investigated samples to enable comparison of the uniformity of all investigated elements to each other.

3. Results and Discussion

All commonly known tribological quantitative values of the tribological experiments were investigated. However, results thereof, e.g., the friction and wear behavior, are not part of the present manuscript as it focuses on the investigation of the developed tribolayers. Moreover, influences of the running-in processes were not explicitly considered in Confoir correlation analyses in the paper at hand.

Figure 5 presents the mapping images of all investigated disks where elements are displayed individually in their respective distribution. Moreover, the positions for depth profile measurements are depicted for each disk. This figure is intended to enable a general overview of performed experiments supporting the reader. A detailed discussion of results regarding elemental distribution and depth profile measurements is presented in Sections 3.1 and 3.2.

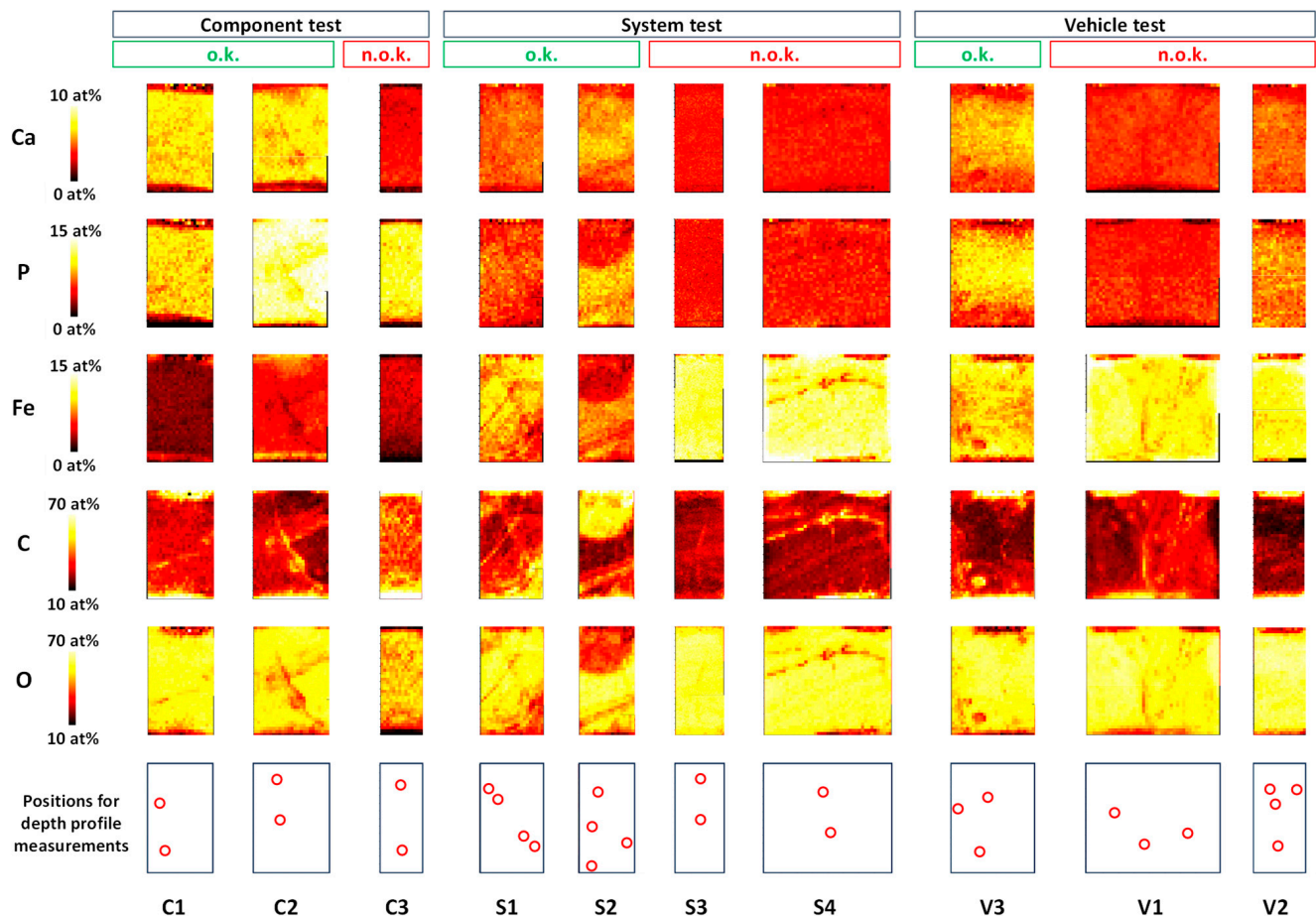


Figure 5. XPS maps from all investigated disks and depiction of position for depth profile measurements. Note that the measuring area was chosen to scan the total radial width of the disk ($y = 17.5$ mm); the length lay between 5 and 20 mm.

3.1. XPS Mapping

In the 3D analysis of the tribolayer on the disks using XPS, the focus was laid on typical additive elements of the oil such as Ca and P, the general oil elements C and O as well as Fe, the substrate beneath. Zn and S, both also typical for oil additives, were omitted for the reasons that, on the one hand, oil analysis proved that the two applied oils did not contain any Zn and, on the other hand, preliminary tests using XPS showed only marginal amounts of S present on the disk surfaces.

Overall, Figure 5 demonstrates that the main components on the surface are C and O, both with a concentration of up to 70 at%. Fe and P with up to 15 at% as well as Ca with up to 10 at% are far below this level. The especially comparatively low ratio of Fe indicates the presence of a clearly developed tribolayer over the entire radial extent. Moreover, it is obvious that there are differences in the distribution of the individual additives. Therefore, the corresponding uniformity value was calculated for each image of each element and subsequently correlated with the test level of the tribological verification chain (i.e., component test, system test, or vehicle test), the concentration of the respective element, as well as the specific energy input and the performance assessment in terms of o.k. or n.o.k. by each individual tribological experiment. Results thereof are presented in Figures 6 and 7.

Figure 6 categorizes the uniformity values of each investigated element according to the respective test level of the tribological verification chain.

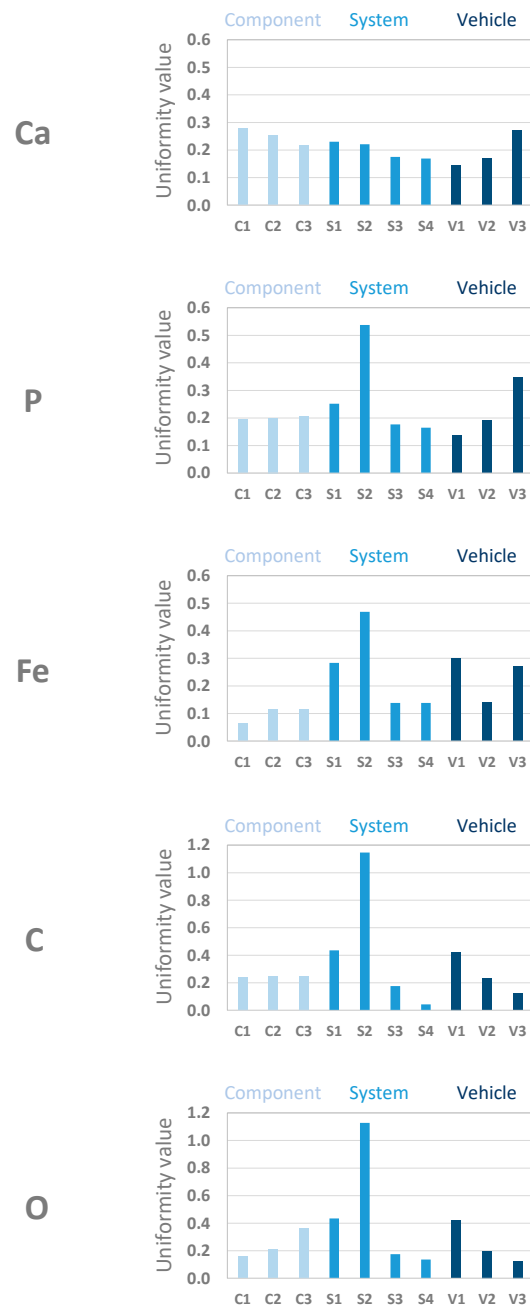


Figure 6. Uniformity values of all acquired elements arranged by the test level of the tribological verification chain. Note: y-axes are scaled differently.

As depicted in Figure 6, no significant correlation between uniformity of elemental distribution and the tribological exposure of the steel disks, i.e., the three different test levels of the tribological verification chain, can be determined. All values are quite low (<0.5) with only the exception of system test S2 showing a very high uniformity value for all the investigated elements and, hence, indicating a very uneven distribution of the elements. One possible explanation for the behavior of S2 is that this test is the only test run at high temperature and therefore other surface interactions may have been involved. This hypothesis has to be verified by further investigations with an enlarged test matrix. For all other cases, no significant impact of test type or of temperature on the uniformity values was found. Analogously, no obvious correlation was found for the lubricant type. Thus, for further graphic processing, these three parameters were not taken into account anymore. For this purpose, Figure 7 displays the uniformity values and concentrations

for all acquired elements arranged by the height of uniformity value and categorized by performance assessment o.k. or n.o.k.



Figure 7. Left: Uniformity values and concentrations for all acquired elements arranged by height of uniformity value and categorized by performance assessment (o.k./n.o.k.); Note: The arrows indicate the respective y-axis of the value. Middle: Uniformity values for all acquired elements in correlation with specific energy input. Right: Concentration values for all acquired elements in correlation with specific energy input. Note: y-axes are scaled differently.

In principle, the smaller the uniformity value is, the more even the distribution of the element appears. If the uniformity values are now considered in connection with the performance assessment during the tribological experiment (see Figure 7; full-colored columns in the left of the diagram), no obvious correlation can be seen for the general oil elements C and O as well as for the substrate element Fe. This implies that a positive (o.k.) or negative (n.o.k.) tribological performance cannot be concluded on the basis of the uniformity values of these elements alone. The situation is different with the additive elements Ca and P. Here it can be noted that a tendency can be seen to put more uniform distributions (lower uniformity values) into the n.o.k. category. This may seem contradictory at first, however, it should not be considered in isolation. An important parameter in this context is the respective concentration in which the individual elements are present on the surface. The histograms were consequently used to obtain information about the concentration distribution of the elements. The values of the respective highest concentration peaks in the histograms are shown as striped columns in the diagram in Figure 7 on the left. Again,

no trends can be seen for C and O. These two elements are present in similar, very high concentrations on all disks after all tribological experiments. On the contrary, it can be stated for Fe that higher Fe concentrations tend to be present during experiments with n.o.k. performances and lower ones at o.k. experiments. This is in line with expectations, as higher Fe indicates that a lower, possibly insufficient tribolayer was formed and therefore the tribological performance is also poorer. A clear trend can also be seen for Ca and P, indicative of additive coverage on the steel surface. Ca shows a clear tendency toward higher peak concentrations for o.k. samples, although it is remarkable that this tendency occurs within a concentration range as small as roughly 3 at%, from 3 at% to 6 at%. P peak concentrations somewhat parallel the Ca tendency, if not with such a clear distinction between o.k. and n.o.k. samples. While it is obvious that a higher coverage of the surface with P- and Ca-containing compounds is beneficial for the test performance, as shown in [34], two less obvious facts can be remarked. First, there seems to be a non-zero threshold concentration below which the performance is not satisfactory anymore, even though the surface is still uniformly covered. Secondly, even a non-uniform distribution with peaks at a slightly higher concentration, i.e., some stains, seems to be sufficient to achieve a good test performance.

Another interesting correlation is the comparison of uniformity values and the concentrations with the specific energy input during the tribological experiments as shown in Figure 7 in the middle column and on the right-hand side. A clear trend can be identified by displaying the uniformity values as function of the specific energy input. It can be stated that tribological experiments with either high uniformity values and, hence, less uniformity or with high specific energy input, result in an o.k. performance assessment. Conversely, experiments with n.o.k. performance are those with low uniformity values and/or low specific energy input. This applies equally to all analyzed elements and corroborates the statement made above that a non-uniform distribution is not necessarily a disadvantage. Analog, although not as pronounced, trends can be found for the correlation of element concentration and input of specific energy. In addition to the respective concentration in which the element is present on the surface, the amount of energy introduced also has a certain influence on whether the experiment has an o.k. or n.o.k. performance.

3.2. XPS Sputter Depth Profiles

In order to acquire information about the thickness, structure, and composition of the tribolayer, the samples were examined by sputter depth profiling after the mapping investigation. Since depth profiling of the mapped surface areas in their entirety would have been prohibitively time-consuming, between two and four spots per sample were selected for that purpose. Figure 5 displays the positions for depth profile measurement in detail for each disk. Referring to the previously acquired maps, spots were chosen that represented different regions of elemental concentration within the map areas. For the samples with the most uniform elemental distributions, i.e., with no discernible distinctly different concentration regions, two random spots were chosen.

The depth profiles were obtained by acquiring elemental detail spectra, which had to be selected in advance. Comparatively to the mapping procedure, Ca, P, Fe, C, and O were selected for this investigation. Acquisition of the detail spectra allowed for further distinguishing between different binding states of the elements. C was differentiated into carbidic (binding energies 284 eV or lower) and other states (binding energies 284.5 eV or higher), Fe was differentiated into metallic state (binding energies around 707 eV) and bonded states (binding energies 709 eV or higher). Ca and P were each present in only one state. O was not further differentiated because states like metal oxides, hydroxides, phosphates, or carbonates could not be unambiguously distinguished quantitatively (even

if it was qualitatively clear that various of those states are present). Thus, O is omitted from the depth profile diagrams. Moreover, in the presented depth profile diagrams, metallic Fe and carbide C were added together to give the substrate bulk.

In the first step, evaluation focus was laid on the interpretation of differences in the spots found during mapping in order to clarify whether the superficial elemental variation correlates with the thickness of the layers or their composition at depth. For this purpose, the depth profiles of the detected elements in all spots (spot position see Figure 5, bottom row) of each test were compared with each other.

Figure 8 exemplarily shows the depth profiles of two tests, C1 and S2. These were selected because C1 has a very uniform surface and is therefore representative of all very homogeneous surfaces, whereas S2 is the steel disk with the greatest differences according to mapping.

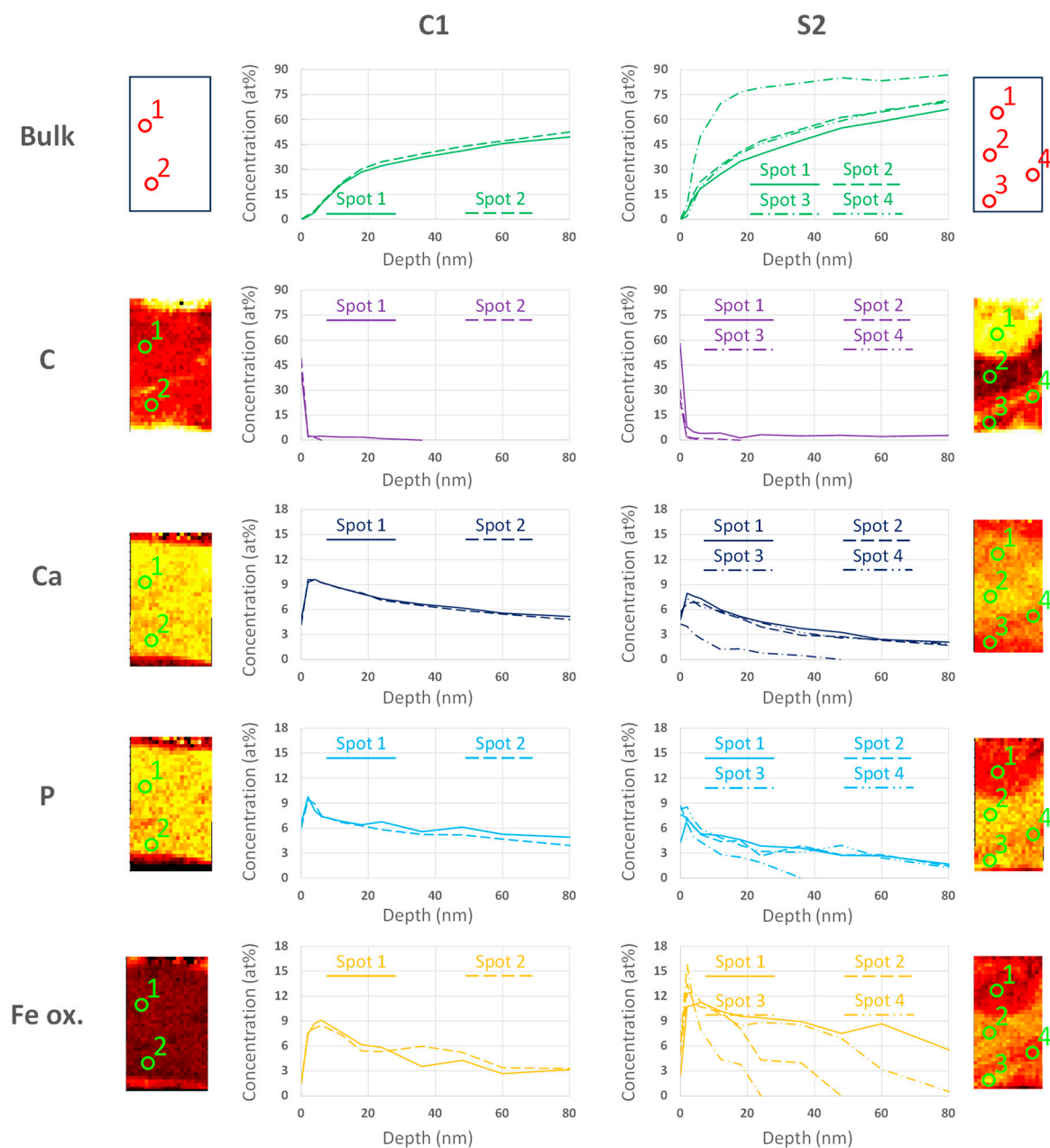


Figure 8. Depth profiles of detected elements after experiments C1 and S2. Note: Mapping graphs document the spot positions (in green) for depth profile measurement within the investigated steel disk area.

In general, it can be stated that disks that appear very homogeneous on the surface also have relatively similar depth profiles. For test C1 in Figure 8 on the left side, there were no significant differences found between spot 1 and spot 2 down to a depth of 80 nm for all elements and also for the bulk.

The situation is different for disks where areas with different additive or organic layers were found on the surface. The best example of this is test S2, which is shown in Figure 8 on the right side. Here, variations in the depth profiles can also be detected. Spots with a significantly lower layer thickness were found, clearly indicated by respective bulk values. It is interesting to note that positions with a high concentration at the surface did not automatically form particularly deep layers. In the case of P, for example, the spot with the lowest surface concentration has formed a very deep layer comparable to other spots with a higher surface concentration, while the spot with the highest concentration on the surface has by far the thinnest layer. Similarly, for Fe ox., the spot with the highest concentration at the surface has the thinnest layer thickness. Thus, it has to be emphasized although superficial mapping of the elements provides excellent information about the distribution of the elements on the disk surface, no compelling conclusions can be drawn about the layer depths.

Another important aspect that was addressed in the course of this work is the correlation of the layer thickness information of the individual elements with the performance o.k. or n.o.k. during the tribological usage. For this purpose, Figure 9 shows a comparison of the depth profiles of all detected elements divided according to the respective test level of tribological verification chain and colored in green for o.k. and red for n.o.k.

This approach reveals a different picture for the three test levels of the tribological verification chain examined which will be addressed within the next section.

The values for the bulk can be used as an indicator for the thickness of the layers. If the bulk values are low for a comparably great depth, this is a sign of a thick layer. If the bulk concentration quickly approaches 100%, only relatively thin layers adhere at this point. Figure 9 generally shows that during system tests, bulk values rise considerably within the first 20 nm which is much faster than during component and vehicle tests, where this happens within the first 40 nm. Hence, it can be assumed that during component and vehicle tests thicker layers are built by trend. If this layer thickness is now correlated with the respective tribological performance, it appears for the component tests that o.k. experiments tend to possess thinner layers and n.o.k. experiments thicker layers by trend. This would suggest that thicker layers are rather harmful for the tribological behavior of the disks. However, this result should not be over-interpreted, as oxygen is also present in the resulting layers. It is therefore possible that the layers formed are counterproductive deposits rather than effective tribolayers built-up by oil additives. For both system and vehicle tests no correlation was found between the tribological performance of the disks and the layer thicknesses.

In connection with the bulk, Fe ox. is also of great interest, as this corresponds to an oxidation of the substrate surface. During the vehicle tests, the thickest layers with the highest concentrations were developed, in system tests similarly high concentrations were detected but with lower depth. During component tests, Fe ox. can also be found even at lower depths but it is always present in comparatively low concentrations. In general, it has to be stated for all three test types by trend that higher concentrations of Fe ox. cause a n.o.k. rather than an o.k., but the thickness of the layer does not seem to have such a great influence.

There were also differences found for C during the three experiment types. Vehicle and system tests exhibit relatively similar results with very thin layers of C with a comparable concentration to additive elements Ca and P already after a few nm (exception: one system

test with o.k. performance displayed disproportionately high C concentrations). Moreover, no correlation between concentration and performance was detected. On the other hand, component tests show a clearly different trend. Here, the C depth profiles for tests with o.k. performance are analogous to those found in system and vehicle tests, but the profiles of tests with n.o.k. performance demonstrate an elevated C concentration of approximately 30 At% throughout the whole investigation depth. This was measured in the same way for both investigated spots. The bulk values already proved the presence of the thickest layers during the component tests. The C profiles now indicate that these are also very carbon-rich in the case of n.o.k. performance.

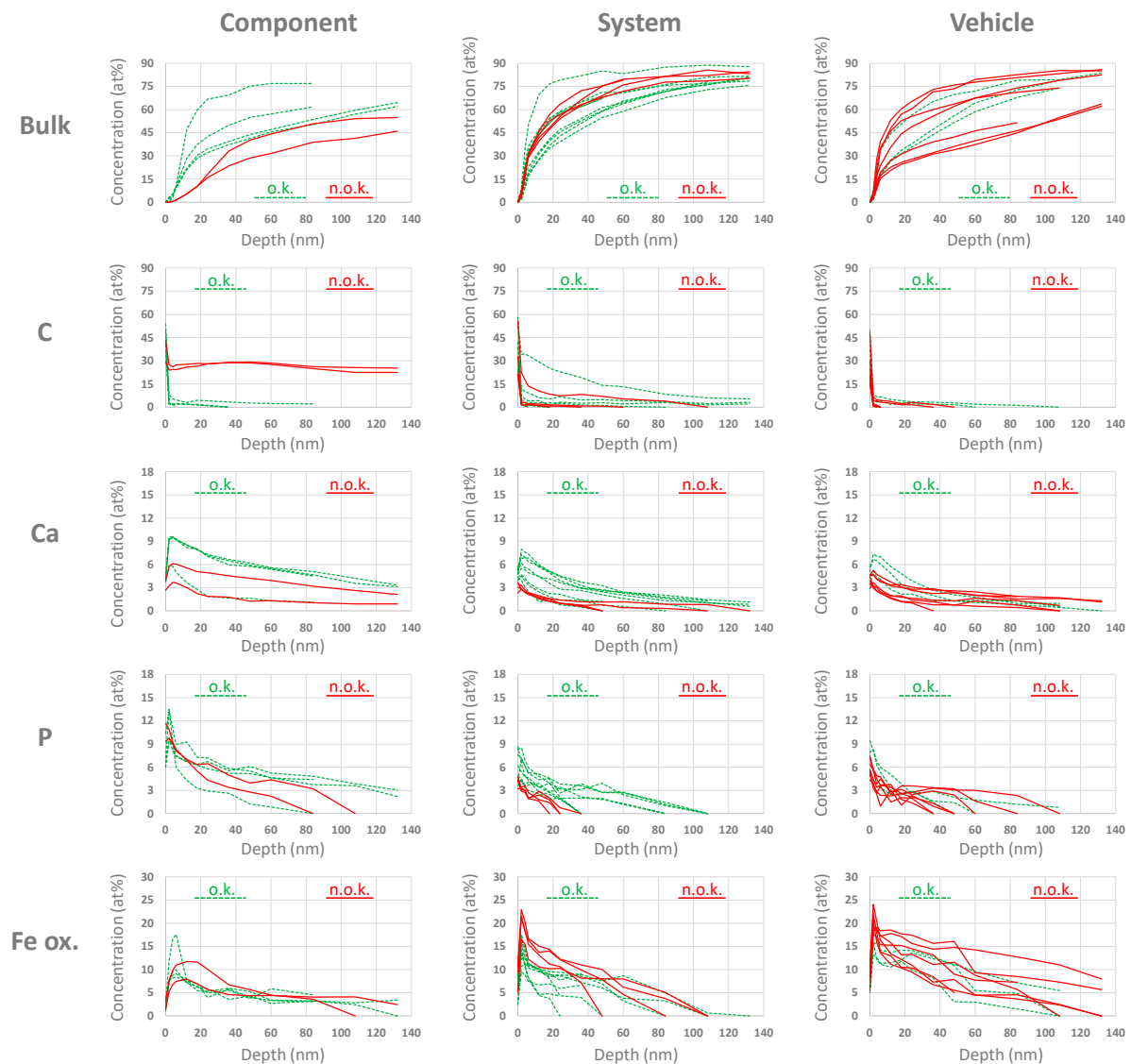


Figure 9. Depth profiles of detected elements categorized by test level of tribological verification chain as well as performance assessment (o.k. ... dashed green lines; n.o.k. ... solid red lines). Note: y-axes are scaled differently.

The occurrence of the two additive elements Ca and P is also extremely important. It was shown that they are present in a considerably lower but comparable concentration range than all other components examined (see different scales for the y-axis in Figure 9). During system and vehicle tests, the peak concentrations for both elements are comparable, during component tests slightly higher by trend. Overall, Ca has a slightly higher layer thickness than P. Again, the results for system and vehicle tests are very comparable, but the

layers for component tests tend to be slightly deeper. A correlation of the layer thickness and concentration of Ca and P with the performance in terms of o.k. and n.o.k. generally shows the trend that higher concentrations of Ca and P lead to an o.k. result rather than the lower concentrations, which is in accordance with findings in [34]. This can also be observed for the layer depth. Experiments in which Ca and P are still found in deeper layers tend to be o.k.; n.o.k. experiments more probably show thinner layers of Ca and P. This effect is most pronounced with system tests.

4. Summary

The presented study investigated the tribolayer build-up on wet clutch steel disks during three different tribological experiments representing three test levels of tribological verification chain, i.e., component tests, system tests, and vehicle tests. For this purpose, XPS was applied to assess the lateral elemental distribution uniformity by converting the resulting maps into histograms, fitting them with Gaussian functions, and subsequently calculating a single uniformity value. Moreover, XPS sputter depth profiles were used to elucidate the tribolayer thickness and its vertical composition. The uniformity values together with the peak concentration of various elements as well as their vertical distribution were correlated with the tribological performance of the investigated clutch system in terms of shudder tendency.

XPS mapping gave the following results:

- Clear differences in the distribution of individual additives were found. Tribolayers mainly consisted of C and O (up to 70 at%), Fe, and P (to 15 at%) as well as Ca (up to 10 at%).
- No obvious correlation between uniformity of elemental distribution and test level of tribological verification chain, i.e., component, system, or vehicle test, was determined.
- For the elements C and O, no obvious correlation was observed between uniformity value and clutch NVH rating as well as between concentration and clutch performance.
- On the contrary, Ca and P showed the tendency that a more uniform distribution rather led to n.o.k. performance. However, a considerable trend toward o.k. performance with higher elemental concentrations was detected. This leads to the conclusion of a non-zero threshold concentration below which the clutch performance even with a uniformly covered disk is not satisfactory anymore. Moreover, some stains with slightly higher concentration seem to achieve good test performance even with a non-uniform elemental distribution on the surface.
- An influence of the amount of specific energy input was determined corroborating that a non-uniform distribution is not necessarily a disadvantage for clutch performance.

The following findings were made by XPS sputter depth profiling:

- Homogeneous surfaces showed relatively similar depth profiles by trend.
- Positions with high concentration detected via surface mapping did not automatically result in deep layers emphasizing that superficial distribution of elements does not give information on tribolayer thickness.
- During component and vehicle tests, thicker layers were built by trend. During component tests, o.k. experiments tended to possess thinner layers than n.o.k. experiments. For system and vehicle tests, no correlation was found between the tribological performance of the disks and the layer thickness.
- For all three test levels of tribological verification chain, it was detected that higher concentrations of Fe ox. caused n.o.k. rather than o.k. performance. The thickness of the layer seemed to have inferior influence.

- Overall, Ca layers were found to be slightly thicker than P layers. It was shown by trend that higher concentrations of Ca and P as well as thicker Ca and P layers rather lead to an o.k. result.

5. Conclusions

Clutch development is subject to the ongoing trend that cost- and goal-oriented design plays a major role in the development of wet clutches in drive trains. From a tribological perspective, the drive train imposes friction and wear requirements on clutches that must be met over the entire clutch service life and depending on operating conditions and the influences of the overall system. The tribolayer characteristics presented in this publication enable the targeted development of new additive and clutch surface technologies, as they enable assessing the functionality of such additives and the interaction of the disk surfaces with the oil. Furthermore, the developed method can be used to investigate the influence of clutch-specific requirements, e.g., regarding different clutch power and energy, fresh oil supply, wear ingress, or contamination, on the functioning of additives or on the interaction with clutch materials. An important question arises for future work regarding the investigation of tribolayers: is the change in the developed characteristic values and the associated supposed deterioration of the properties of the tribolayers a condition that is reversible by oil change and/or power input? Answering this question is essential for the service life design of wet clutches, as only with this information can it be scientifically determined whether tribolayers are permanently damaged or can be reconditioned. Permanent damage would mean that the clutch disks can no longer be operated to their originally defined design limit, despite an oil change. If reconditioning is possible, the question of a changed service life limit no longer arises. Looking ahead, further method and parameter verification and validation will also open up new opportunities for addressing tribological issues: if it can be proven that the behavior of the clutch friction contact in system and vehicle tests can be replicated through component tests, i.e., if it can be demonstrated that, on the one hand, the widely known tribosystem parameters (e.g., roughness, material properties, etc.) and, on the other hand, the chemically functional layers, are comparable at all levels of the tribological verification chain, this will create the opportunity to reduce system tests and thus costs. Furthermore, in the future, parameters of the chemically functional tribolayers will also be able to serve as a descriptive parameter for describing tribosystems and tribosystem behavior in tribological databases. Ultimately, however, it is necessary to make a move toward standardizing tribolayer parameters.

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Abbreviations

The following abbreviations are used in this manuscript:

ATF	Automatic Transmission Fluid
ZDDP	Zinc Dialkyldithiophosphate
ToF-SIMS	Time-of-Flight Secondary Ion Mass Spectrometry
ATR-FTIR	Attenuated Total Reflectance–Fourier Transform Infrared Spectroscopy
SEM/EDX	Scanning Electron Microscope/Energy Dispersive X-ray Spectroscopy
XPS	X-ray Photoelectron Spectroscopy
o.k.	Okay
n.o.k.	Not Okay
NVH	Noise Vibration Harshness
FWHM	Full Width at Half Maximum

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