COMBINED DEVELOPMENT OF ECOLOGICALLY ACCEPTABLE HYDRAULIC FLUIDS AND COATINGS FOR FLUIDPOWER APPLICATIONS

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SUMMARY
Fluid power is a firm constituent of machine tools. It is used to realize main, secondary and peripheral tasks. The application of fluid-technical systems is always combined with the use of fluids. Environmental aspects have a major impact on the application of technical products and decide about their success. Especially, with regard to hydraulic systems, these aspects can be fulfilled by the use of environmentally friendly fluids, based on synthetic esters. This is the reason why within a collaborative research centre at the University of Aachen new environmentally friendly fluids are developed and tested in tribosystems applying new surface coated materials. The aim is to transfer important tribological features from fluids onto these base materials. Taking this route certain additives are no longer required and the missing characteristics have to be taken on by specially coated materials. This paper presents the most important developed results with respect to the application of the new fluids and coatings for hydraulic systems.

Keywords: Hydraulik fluids, fluid-coating interaction, Siebel/Kehl, carbon

1 INTRODUCTION
Hydraulic systems are used in many technical applications. For example in agricultural and forestry machines as well as in aeroplanes and ships [1, 8]. Hydrostatic pumps and engines are essential components of hydraulic mechanisms. They transform mechanical energy into hydraulic energy and vice versa. Hydrostatic machines and their standard fluids based on mineral oils have been developed to a high technical standard. In order to withstand the operating loads, engineers have made considerable efforts to find the best combination of additives. The fluids in this case are not ecologically acceptable and often toxic. Fluids that are ecologically acceptable cannot include additives that are toxic. Consequently, the problem of having ecologically acceptable fluids which show a worse tribological and ageing behaviour than standard fluids have to be solved. Therefore, a new strategy of developing tribosystems has been established within a research program at the university of Aachen (Figure 1).

There, new ecologically acceptable fluids based on esters are produced and combined with new coatings. The goal is to transfer important tribological qualities of the fluid to the materials. Generally, the properties of the new ecologically acceptable esters strongly differ from the ones of the well known mineral oil based fluids. Moreover, the new fluids are in interaction with the coatings. This is why, on the one hand, analysis of the properties of esters, their change due to usage and the interaction with hydraulic components have been carried out. On the other hand, in order to gain basic information about the quality of the coatings, experiments have to be performed using a tribometer (Figure 2).

An experimental setup according to Siebel/Kehl is used for this.

As a result of the experiments, coatings such as TiAlN/Al₂O₃ or CrAlN, which can be used for milling and drilling applications, have worse results than
conventional materials such as bronze/steel [4]. Improvements are achieved by adding carbon, however, the results are highly dependent upon the application method. Coatings that are graduated like HfCg or ZrCg show a low friction coefficient and wear rate. In addition to the tribological qualities of the coatings, the durability as well as the scratch resistance is important for the application. These considerations are especially important in the case of thin PVD coating which are used in applications with insufficient hydrodynamic lubrication.

2 AGEING INVESTIGATIONS

2.1 Strategy

According to the goal of the collaborative research centre tribological functions of the lubricants have to be substituted by the materials. This leads to a decrease of the technical requirements on lubricants. Consequently, the fluid’s ageing behaviour becomes more important. Especially, when having in mind that a short period of usage of a fluid leads to increased costs. To reach the goal first of all the development of new fluids based on oleochemicals is started. By chemical modification of the fluid’s properties, especially the oxidation stability shall be improved. Since the new developed fluids are used in combination with the new developed surface coatings, the interaction between the fluids on the one hand and the coatings on the other hand has to be analysed. Since aged fluids have different physical and chemical properties compared with new ones, the influence of ageing on the fluid’s properties is also assessed to be of great importance. Therefore, wear analysis has been carried out with a specially designed test rig. Figure 3 shows a summary of the investigation strategy.

Since the research program is focused on tribosystems as being used in standard machine tools ageing investigations have to take into account the specific structures of the different tribosystems. To reach the goal of ageing investigations which incorporates fluids of high ageing stability for usage in environmentally acceptable and powerful tribosystems ageing investigations have to be very sophisticated. Method is firstly to analyse the ageing behaviour of synthetic esters which are state of the art. These ones are the reference oils. Besides, oxidation stability of the new developed fluids is analysed. Aim is to come as close as possible to the oxidation stability of the reference fluids. Secondly, the influence of typical base materials and surface coatings on reference fluids is tested. As reference fluids not only hydraulic fluids are considered but also gear and cutting fluids since they are used in machine tools. Each fluid is combined with such catalysts so that specific structures of tribosystems are simulated. This means that gear and hydraulic fluids are combined with standard materials as iron, copper, chromium and titanium and further with new developed coatings as zirconium carbide whereas the cutting fluid is combined with standard materials and titanium hafnium carbide. This analysis is carried out with the Rotary Bomb Test according to ASTM D2272. Thirdly, rig tests which are long endurance tests are carried out. There, the fluid is aged in a hydraulic system. Since a combination of different loads like temperature, pressure and material combinations is put on the fluid, a more realistic ageing of the fluid can be simulated.

2.2 Impact of Base Materials and Surface Coatings on Oxidation Stability

Since the new developed fluids will be used in combination with surface coatings, further investigations have been done. Aim is to generally evaluate the interaction between lubricants and base materials and surface coatings respectively. Thus, fluids and coated rings of an area of about 7000 mm² have been tested in the rotary bomb test in different combinations.
Combinations were chosen such that specific structures of tribosystems were simulated. Figure 4 shows the oxidation stability of tested reference fluids in dependence of base materials and surface coatings.

From the picture you can see that standard base material iron decreases the oxidation stability of the hydraulic and gear fluid most. Compared to the influence of the base materials iron, chromium and titanium the influence of the new developed surface coatings zirconium carbide and titanium hafnium carbide is less. When comparing the oxidation stability of the reference fluids tested without any catalyst and tested with zirconium carbide there is hardly any difference at all. To sum up, you can say that base materials as well as coatings influence the oxidation stability. The impact can be decreased by the use of specially coated materials. The function of additives which influence the passivity of surfaces can be transferred onto the surface coatings. They are passive by themselves. The fact that oxidation stability of the cutting fluid is hardly affected by catalysts can be explained by special additives which only this fluid contains.

Since these investigations show a significant impact of catalysts on oxidation stability of fluids further analysis have been carried out. Aim was to find a correlation between the amount of catalyst and decrease of oxidation stability. So, oxidation stability of hydraulic fluid H.L.P.ES which contains additives and the corresponding base oil H.0.E.S was tested in dependence of the size of the catalyst surface. As catalyst iron and copper coils in different lengths were chosen. Always the same amount of oil was used. The coefficient best describing the catalyst size is the relation surface of catalyst [m²] versus amount of oil [m³]. Since the two fluids were tested with copper and iron catalyst Figure 5 shows 4 curves. Each curve shows the dependence between the relative decrease of oxidation stability and an increase of catalyst surface size. Hydraulic fluid H.L.P.ES is tested at 150 °C whereas its base oil H.0.E.S is tested at 110 °C so that oxidation stability of these two fluids is in the same range. From test results, being shown in figure 7, two conclusions can be drawn. Firstly, by constantly increasing the catalyst surface the relative decrease of oxidation stability depressively increases. Secondly, this general behaviour is not dependent on additives and catalyst, even though iron less influences oxidation stability than copper.

However, when regarding technical hydraulic systems, this value is far smaller. This is the reason why rig tests have been carried out, using a specially designed hydraulic system shown in Figure 3. Compared to the rotary bomb test facility, a more realistic ageing of fluids can be simulated by the use of this test rig. On the one hand, several loads are put on the fluid like temperature, pressure, shear stress and catalysts. On the other hand, the surface size of catalysts compared to the amount of fluid is smaller due to the chosen test disc geometry. By the use of 6,5 litre oil in the circuit the relation catalyst surface/amount of oil is 0,26 [1/m]. Tests are carried out at a temperature at the suction side of the pump (1) of 70 °C. The pump is working against a pressure relief valve (4) which leads to a pressure of 300 bar. Behind the pressure relief valve temperature is about 85 °C.

The fluid which flows at a rate of 18 litres per minute now enters the tribosystem (5). Two discs are rotating against each other under normal load which can be adjusted by a throttel (3) and a pressure relief valve (4). Tests have been done with hydraulic fluids H.L.P.ES and its base oil H.0.E.S. Each fluid has been tested twice. One time tests without a material combination in the tribosystem have been carried out in order to characterise the ageing influence of the hydraulic circuit itself. The other time tests have been done with a material combination steel/bronze since this is an often used material combination in hydraulic components. Further, rotary bomb tests have shown that copper influences the oxidation stability of fluids most. Test time for all runs was 408 hours. Results can be seen in Figure 6.
The relative change of TAN and viscosity respectively was chosen to describe the ageing of fluids. Again, test results show a significant influence of additives like the rotary bomb tests have shown. Moreover, when regarding the ageing behaviour of hydraulic fluid H.I.P.E.S it can be seen that the impact of the material combination is marginal. This is caused by the small surface size of the catalyst relative to the amount of fluid. Base fluid H.0.E.S shows a similar ageing behaviour. Altogether, these rig tests show that catalysts still have an impact on the ageing of fluids. However, it could be proven that the influence is not as significant as shown in the rotary bomb tests. Figure 7 shows that rather loads like temperature, water and oxygen as well as additives influence ageing stability. Comparing the different loads and the corresponding relative change of TAN and viscosity a ranking can be made up. This ranking shows that additives and high temperature in combination with oxygen mostly influence oxidation stability whereas the impact of catalysts and temperature without the presence of oxygen is only marginal. To evaluate the influence of pressure which induces shear stress is difficult. This results from the fact that a pressure decrease correlates to a temperature decrease behind the pressure relief valve. So, changing pressure always induces a change of temperature in the same direction. However, the small change of TAN and viscosity of fluid H.I.P.E.S tested at 90°C without oxygen, shown in figure 10, indicates a shear stress influence which is smaller than the one of oxidation [2].

3 FRICITION & WEAR INVESTIGATIONS

3.1 Tribological Contacts of an Axial Piston Pump

In a hydraulic system axial piston pumps are mainly used. Major tribological contacts (Figure 8) suited for the use of coatings in these hydraulic displacement units are [5]:

- swash plate / slipper
- ball joint / slipper
- plunger / cylinder bore
- cylinder / valve plate

which are mainly stressed by two-body and three-body abrasion.

For the above mentioned tribological systems new types of PVD coatings are necessary, which provide low wear of test piece and counterpart in combination with low friction.

3.1.1 Bench Test

For the evaluation of new coatings a simplified test is performed. By this test a preselection of coatings with a minimum of time and cost efforts becomes possible. The test is based on a model by Siebel/Kehl, which is shown schematically in Figure 9.
to rotate the two test parts relative to each other a torque must be applied, from which the friction coefficient can be determined as:

\[
\mu = \frac{12M}{\pi p(d_1^2 - d_2^2)}
\]

where

- \(\mu\) : coefficient of friction[\(\text{\textdegree}\)]
- \(M\) : torsional moment[Nm]
- \(p\) : surface pressure[N/m²]
- \(d\) : diameter[m]

An evaluation of the coating characteristics is based on the Striбbeck curve derived from the above mentioned tests in combination with the amount of wear and changes in the coating surface which are determined by surface measurements. Parallel to the definition of investigation method a basic load cycle is to be defined. At that it is necessary to imitate the critical loads of tribological contacts of a displacement pump. Critical loads occur if system works in mixed friction state. This is reached also in the experiment. All materials were loaded with the values represented in Table 1. If further studies are necessary, this test procedure can be enlarged.

<table>
<thead>
<tr>
<th>Surface pressure (p)</th>
<th>2.5 N/mm²</th>
<th>2.5 N/mm²</th>
<th>2.5 N/mm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Running speed (v)</td>
<td>2.3 m/s</td>
<td>0.2 m/s</td>
<td>2.3 m/s</td>
</tr>
<tr>
<td>Duration (t)</td>
<td>15 min</td>
<td>4 h</td>
<td>15 min</td>
</tr>
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**Table 1: Basic testing method**

### 3.2 Graduated Carbon Coatings

#### 3.2.1 Fundamentals

For the improvement of wear and friction behaviour metal/carbon coatings are tested. The coatings “HfCg” and “ZrCg”, developed at the ‘Material Science Institute, RWTH Aachen, Germany’, have no or a non-relevant amount of carbon on the substrate level which continuously increases to the coating surface [9]. Coating characteristics as coating hardness and elastic modulus vary widely, depending on the carbon content. As a consequence of the graduation, the coatings offer on the substrate level the same hardness as the metal component. In this case the metal components are zircon (Zr) and hafnium (Hf). Consequently, the hardness increases due to the occurrence of hard phases until the maximum of these phases is reached. Succeeding, the hardness decreases, since an increasing amount of carbon is inserted into the coating. In Figure 10 the different coating structures over its height are visible. A theoretical course of carbon content and hardness is also shown in figure 5.

By the coating structure and its resulting characteristics, three major goals are aspirated. The outer, relatively soft layer of the coating allows a reduction in surface roughness. By this means the tribological qualities of the coated parts are self-improving. This effect increases the percentage contact area, which can be made visible by an analysis of the part’s surface. A closer examination of these effect will follow later on in this paper. Due to the described changes in the test piece surfaces, the height of the hydrodynamic surface film required for a complete separation of the tribologically stressed surfaces and therefore also the operation range with high wear and friction are reduced.

### 3.2.2 Investigations with constant loads

In a first step a general characterization of the selected coatings was aspirated. Thus they were investigated with a constant pressure load of \(p = 2.5\ N/mm²\) and \(v = 0.2\ m/s\) for a time period of four hours. A comparison of the Striбbeck-curve before and after the test already shows significant changes of the tribological behaviour of the HfCg-material. The diagram in Figure 11 shows the friction coefficient versus the average sliding speed. Compared to a combination of bronze and steel, also shown in figure 6, these new coating materials offer a 40% reduction in the static friction.

![Image of HfCg coating](image-url)

**Figure 10: HfCg coating**

Additionally there is a chance that carbon particles of the outer coating area are transferred to the tribological counterpart, which leads to improved tribological behaviour of the counterpart and thus also to a tribological improvement of the system in general. Furthermore, the changes in coating properties (hardness, elastic modulus) over its height allow an adaptation of the coating to a desired application. This adaptation process is carried out by a minimal wear of the coating in the range of \(\mu\) or nm. The results achieved with the new coatings are described subsequently.

![Image of Striбbeck curve](image-url)

**Figure 11: Striбbeck curve**

Both coating systems, HfCg and ZrCg, show a running in or changes in their properties, which can be seen easily in a comparison of their characteristics before and
after the 4 hour tests. General characteristic values as static friction and coefficient of perfect lubrication stay unchanged. Major differences occur in the area of mixed friction. The transitional speed of the area of mixed friction is shifted toward lower sliding speeds and the friction level within the area of mixed friction is decreased. For the shown example of HfCg, the transitional speed changes from n₀=300 1/min to 100 1/min. As the tribological behaviour in a running in condition are of high relevance for practical application, most of the subsequently described investigations focus on coatings that are already running-in. At this time studies onto discrete ZrC coatings (instead of graduated) show also a good friction-behaviour. With these results the following generation of ZrCg will receive similar behaviour as HfCg.

Compared to other coating systems HfCg offers the advantage that it provides lower friction coefficients over the total range of the mixed friction area than the reference of steel and bronze. As a consequence, the power loss of tribological contacts, which are operated in the area of mixed friction, can be reduced and the overall efficiency can be increased. This is especially true for the tribological system plunger/cylinder bore, since a loss of the supporting fluid film is unavoidable.

A major reason for the changes of the friction behaviour during the tests and for the differences between the different coatings is the structure of the coating surfaces. For clarification, Figure 12 and 13 show profile measurements of the HfCg surface and ZrCg, before and after the tests.

ZrCg shows only little changes of the surface due to the load. Only the percentage contact area increases from Tₚ₀,₅ = 45 % to Tₚ₀,₅ = 60 %, which can be explained by a levelling of the highest roughness. Since shown values represent average value of different measurements, deviations from shown values of several percent can occur. The increase of percentage contact area is one reason for improvement of the stribbeck curve of ZrCg. Influences of tribooxydation have not been investigated, yet. A look on HfCg coating system gives a different impression. Besides an increase of the percentage contact area also a significant decrease of the characteristic values of the roughness occurs. These effects are especially strong in contrast with the coated test piece. The percentage contact area Tₚ₀,₅ increases from 30 % to 100 % and the characteristic value of the roughness Rₚ decreases from 0.16 to 0.06. Improvement in surface quality can be regarded as a major reason for the reduction of friction coefficient.

Besides considered friction coefficients, which mainly influence efficiency of the applied components, consideration of the wear is important to evaluate of tribological systems. Figure 14 shows a radial cut of the test-piece with the greatest wear amount, which have been caused by a four hour test. In this context HfCg coating has a wear height of about 1.3 µm. This is still low compared to the reference material combination. In this case results can be interpreted more as a levelling of the roughness than as a real abrasive wear. In contrast to this, ZrCg coating shows almost no wear phenomena. Only a levelling of the maximum peeks of roughness can be observed.

In general, it is difficult to reveal all causes for changes in a tribological behaviour, since it is determined by a high variety of different influences, which are not only difficult to measure but which also influence each other. In this context the term tribological mutation [7] was introduced. Although not all causes for the tribological behaviour of investigated coatings have been determined, it is already obvious that improvements in surface condition have a crucial influence on friction and wear. Especially in the case of new HfCg coating the influence on friction level is clearly visible. The respective surface plots and scanning electron microscope photos are shown in Figure 15. On the coated sample, the tool marks from machining are clearly visible. After load tests significant changes in the surface structure can been. After load is applied, an extremely flat and regular surface structure occurs. Tool marks are completely levelled out in this phase. Wear of different investigated coatings is shown in Figure 16. In this direct comparison, advantages of the carbon
coatings compared to the standard materials bronze or CrN are visible. Exclusive insertion of carbon into coatings alone provides no guarantee for an improved tribological behaviour. Early investigations showed, that the insertion process has a crucial influence on coating characteristics [4, 6].

![Figure 15: variation of the surface structure of HfCg](image)

**Figure 15: variation of the surface structure of HfCg**

![Figure 16: abrasive wear of different materials](image)

**Figure 16: abrasive wear of different materials**

### 3.2.3 Increase of test duration

Based on the finding that investigated coating systems improve their tribological properties during load, the question arises, whether this self-adjusting mechanism comes to an end after a limited time. If this should not be true, the functionality of the tribological system would break down after the coating is completely worn out. In order to avoid this effect, graduated coatings are designed in a way that ensures a steady increase of the hardness and therefore also of the wear resistance over the coating height. In order to prove that the applied coating systems fulfil the demands duration of tests was increased and characteristic wear and friction coefficients were determined in defined time periods. These are the current amount of wear as well as the change of the percentage contact area $t_{p0.5}$ and $t_{p0.25}$. In Figure 17 results are shown for a test duration of 120 hours. A striking aspect is the high rate of wear during the first hour which continuously decreases and comes to an end after about 48 hours. Thus, a limitation in terms of endurance is of no concern. Similar results are represented in the percentage contact area. After a test duration of four hours no more significant changes can be detected. Only the percentage contact area $t_{p0.25}$ of the test piece shows changes until a test duration of $t = 48$ hours. Thus it can be stated that the whole tribological system has adapted itself to the test load after a time period of 48 hours. Identical investigations on ZrCg coatings, which are not presented here, show similar results. For those coatings, also no continuous increase in wear could be observed for longer test duration.

![Figure 17: abrasive wear and percentage contact area for a test duration of 120 hours](image)

**Figure 17: abrasive wear and percentage contact area for a test duration of 120 hours**

In an analogy to wear characteristics, significant changes in friction behaviour of graduated coatings can only be observed during the first test hours. Between 0, ½ and 2 hours test times coefficient of friction as well as transitional rotational speed are decreasing distinctively. Subsequently (2 to 24 h), the decrease is relatively small (Figure 18).

![Figure 18: stribeck curves for a test duration of 24 h](image)

**Figure 18: stribeck curves for a test duration of 24 h**

### 3.2.4 Variation of the surface load

Investigations presented so far have been carried out with a fixed surface pressure. In real components such constant loads occur very rarely. Thus research on the influence of variable surface pressure is required. A new HfCg(2) test piece is now loaded with a surface pressure of 1.25, 2.5, 3.5, 7.5 and 10 N/mm². Figure 19 shows total wear after running-in.

![Figure 19: abrasive wear through increase of load](image)

**Figure 19: abrasive wear through increase of load**
From a pressure of $p = 2.5 \text{ N/mm}^2$ no enlargement of wear is detectable.

The system has adapted itself and no destruction of the coating is to be expected.

**4 OUTLOOK**

On the one hand, in this paper the oxidation stability of the general impact of catalysts on oxidation stability is presented. It could be proven that the influence of specially coated materials on oxidation stability is less than the influence of standard materials like copper, iron, bronze, chromium and titanium. Further, investigations show that the significant impact of catalysts on the oxidation stability of fluids as shown in the Rotary Bomb Test is strongly dependent on the relation catalyst surface size / amount of fluid. This is the reason why results from rig tests did not show a strong impact of the material combinations on the fluid’s oxidation stability. There, the size of catalyst surface in relation to the amount of fluid is much smaller. To further assess the influence of catalysts considering real operating conditions oxidation tests in gears are planned. Moreover, a new test bench has to be built in order to investigate the oxidation stability of cutting fluids.

On the other hand, in order to gain basic information about the quality of the coatings with respect to wear and friction, experiments have been performed using a tribometer. The investigations performed so far have proved clearly feasibility of the chosen approach to achieve the desired goals in the collaborative research centre 442. Especially the investigated graduated coatings offer extraordinary characteristics. By their properties a reduction of the friction and wear in displacement units becomes possible. The successes gained so far can be interpreted as a hint towards the high potential of these coatings for the application in displacement units and other technical systems. In subsequent steps it is necessary, to enlarge the knowledge about the composite materials in order to amplify the positive qualities and to combine them with others, especially with new ZrC coatings. This requires further research with simplified test samples as well as with industrially used components. On the other hand, various parameters which influence coating properties should not be overlooked. In this context, aspects such as coating material or process temperature during the coating process should be considered. Required number of investigations necessary for an optimal application of coatings are numerous. However it already became obvious that the chosen way is appropriate. Already now it is possible to produce coatings, which offer superior characteristics compared to standard materials.

**5 REFERENCES**


**6 ACKNOWLEDGEMENT**

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**7 CONTACT**

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