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Determination of adhesive properties of lubricants on surfaces

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Summary
There are various approaches to describe the physicochemical dependencies in the case of total wetting or nonwetting and the conditions in between, as well as measurements which directly or indirectly describe indications of the adhesive and dispersive energy of the liquids and surfaces. The interpretation of results achieved through laboratory tests in terms of function prediction of real components however is challenging and needs some experience. Within the scope of a development project a test method has been developed which allows one to dynamically measure the adhesive force of a lubricant drop directly on the respective materials and surfaces with the technology of a centrifuge. The detection of adhesion forces under dynamic conditions extends the range of laboratory test methods for the characterisation of surface wetting properties. In particular, the possibility of testing on real component surfaces and with small oil volumes opens up new aspects for the function prediction of lifetime lubricated systems in precision engineering.

Introduction
Choosing the correct lubricant for micromechanical parts is, amongst other things, hindered by the fact that the parts to be lubricated are very small and the life time of components sometimes is decades. The demands onto the lubricants, especially in terms of forlife lubrication, minimum quantity lubrication and single application lubrication therefore are very high. The evaluation of the adhesive properties of the lubricants is a critical aspect, since due to long-term wetting and spreading effects a lack of lubricant may occur, which results in a raise of friction and wear up to a failure of the component.

Modelling of Tests
There are various approaches to describe the physicochemical dependencies in the case of total wetting or nonwetting and the conditions in between, as well as measurements which directly or indirectly describe indications of the adhesive and dispersive energy of the liquids and surfaces[1]. These include e.g. the surface tension of the liquids or the surface

energy of the material surfaces. However, in the direct contact of lubricant and surface, the interfacial tension dominates, which cannot be measured directly, and which only indirectly can be determined via the contact angle. If no equilibrium occurs at the interface, the measurement is made more difficult because the contact angle decreases as a function of time. Furthermore, in the field of precision mechanics, capillary forces are increasingly superimposing the wetting effects in narrow gaps, on rough surfaces and small drop volumes, and thus the transferability of measuring results into practical application is difficult.

Another point to consider is that under practical running conditions of lubricated sliding bearings, frictional heat is created, which results in an acceleration of wetting processes due to Marangoni convection effects[2]. The oil actively creeps out of the bearing (and lubrication zone respectively) onto colder surfaces.

In order to better assess long term wetting effects in real parts, a method may be used, that allows one to visualize even thin oil films on surfaces. With the reflection-contrast-equipment, oil droplets which, for example, have been placed directly on the parts to be lubricated, may be monitored. A camera placed over the test setup makes time lapse recordings to visualise the effects which occur. Additionally, by raising the temperature of the test specimen, wetting and creeping effects can be accelerated, too. Results obtained give important information on the influence of oil type, surface roughness, material, etc. In Figure 1 four different situations are given of two oils, a polyglycol with high surface tension of 36mN/m (upper line) and a silicone oil with low surface tension (21mN/m, below), where droplets with oil volumes of 2µl have been placed on a glass slide, the left side with a smooth surface and the right side with a rough surface. Only on the smooth surface, the polyglycol droplet stays with a stable contact angle, on the rough surface even the polyglycol begins to creep through the roughness. The silicone oil immediately starts to wet the surfaces, on the rough surface to a much higher extent.

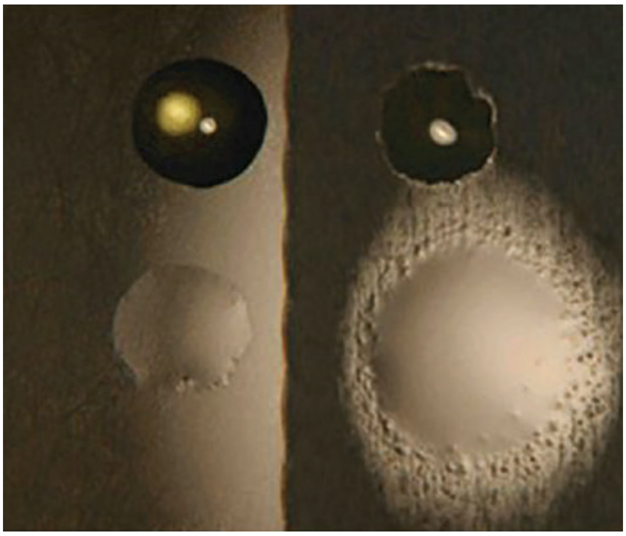

Figure 1. *Spreading behaviour of polyglycol (above) and silicone droplets (below) on glass with smooth (left) and rough surface (right)*

A typical exposure for lubricated fine mechanic components is given in Figure 2. Here olephobic coatings are used to realise forlife lubrication by a purposeful design of the contacting surfaces. A silicone oil on a rough plastic surface remains there with a stable contact angle, if a certain oleophobic layer has been applied (lower part). On the nontreated area (upper part) the silicone oil wets completely.

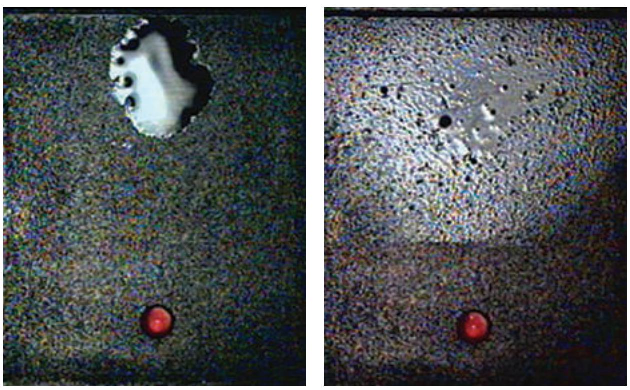

Figure 2. *Spreading behaviour of silicone oil on a rough plastic surface with (zone below) and without olephobic coating (upper zone)*

This test method gives a very good presentation of dynamic wetting and creep effects, but if there are stable conditions at the interface between lubricant and surface, a further differentiation is difficult. Questions like "Is adhesion strong enough?", or "Is there even an oil repelling effect?", "Is oil removed due to gravity or centrifugal forces?", cannot be answered.

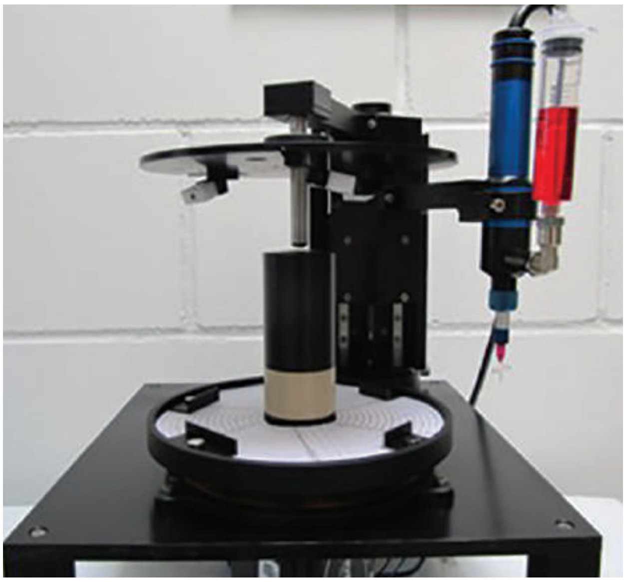

Figure 3. *APG Adhesion tester with rotating disk and dosing unit (swivelling)*

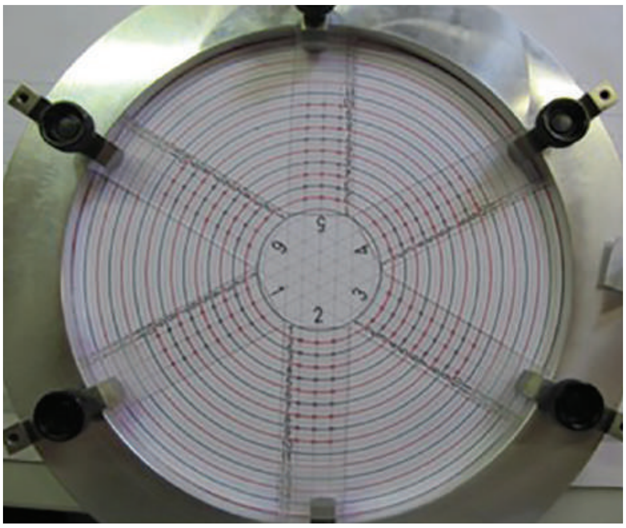

Figure 4. *Rotating disk with test samples containing oil droplets on different radii*

Dynamic Adhesion Measurement

Within the scope of a development project[3] a test method has been developed which allows one to dynamically measure the adhesive force of a lubricant drop directly on the respective materials and surfaces. The developed APG Adhesion Tester (Figure 3) is based on the technology of a centrifuge.

On a rotating disk maximum six standardised plates (size 76 x 26mm, Figure 4) out of any material (i.e. glass, metals, plastics) can be easily positioned with clamping claws. A pattern on the base of the disk with different radii from 30 to 95 mm allows us to find the correct position for the oil drops. Normally we work with three drops per radius.

It is also possible to make tests on other components or parts by fixing them anywhere on the disk with shiftable magnetic strips (Figure 5). The shifting forces of the magnets are higher than the maximum centrifugal acceleration.

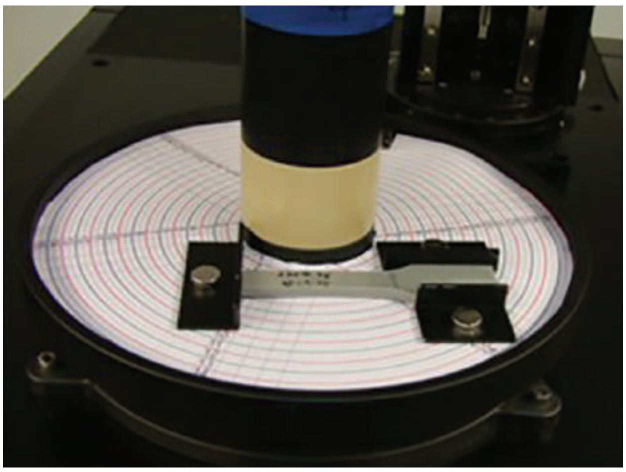

Figure 5. *Easy fixing of varying forms of test specimen with magnetic strips*

Now oil droplets with a defined volume are selectively positioned on the material surfaces in different radii, and are subjected to different graduated centrifugal accelerations (a_n varying from 1.1 to 41.7 m/s²) when the disk rotates. From the specific gravity, the drop volume, the distance from the axis of rotation and the rotational speed, the centrifugal force can now be precisely determined, at which acceleration force the drops are moving. At that point the adhesion force must be overcome respectively.

To allow for easy comparative evaluation of series tests independent of the mass of the drops, the so called transition factor is calculated, which is the normal acceleration when drops start to move, divided by the specific gravity: transit $a_n = a_n/g$.

The movement of the drops can be determined optically. If olephobic coatings with high concentration are tested, the droplets move in total (Figure 6), the red dots represent the initial position of the drops. If uncoated surfaces are tested, the droplets leave traces of smaller drops (Figure 7).

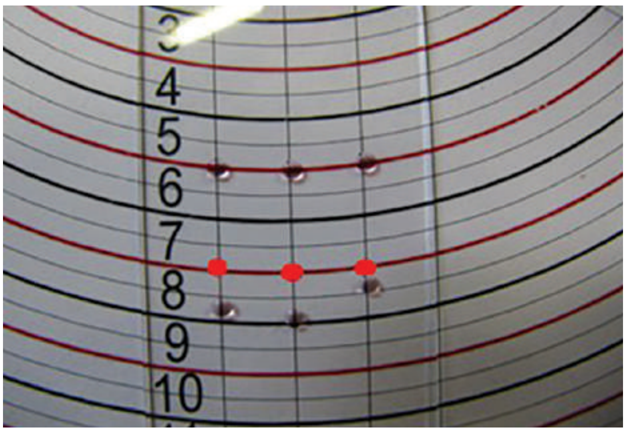

Figure 6. *Transition point of droplets on oleophobic surfaces*

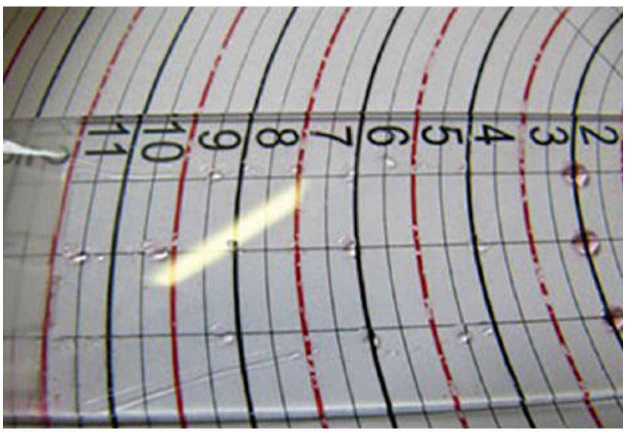

Figure 7. *Transition point of droplets on non-coated surfaces*

Test Plan and Results

The test plan for the comparative assessment comprised three base oils used as lubricants in fine mechanics, a polyalphaolefin (surface tension σ=30 mN/m), an ester oil (σ=32 mN/m), and a silicone oil (σ=21 mN/m). The materials used are glass with surface roughnesses "smooth" (R_a 0,04µm, R_z 0,45µm) and "rough" (R_a 0,91µm, R_z 7,91µm), a polyamide 66, a polybutylenetherephtalate and a polyacetale.

In addition, the influence of an oleophobic surface coating in varying concentrations (10-high, 50-medium high, 200-medium, 500-low) as used in forlife lubricated fine precision components on the adhesiveness of the lubricants has been tested. Drop volumes varied from 0.2 to 1.5µl (Figure 8).

Figure 8. *Varying drop volumes for adhesion tests*

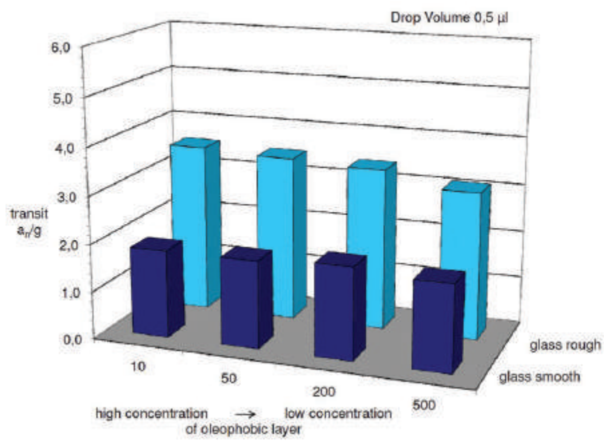

Figure 9. *Influence of surface roughness on transit factor*

The influence of surface roughness and concentration of oleophobic layers on the transit factor of silicone oil on glass is given in Figure 9.

On smooth surfaces the adhesion of the drops is not affected by the concentration of the oleophobic layer, whereas on rough surfaces the efficiency of the oleophobic layer decreases with the concentration. But whatever the concentration of the oleophobic layer, the adhesion of droplets on rough surfaces is twice as high as on smooth surfaces.

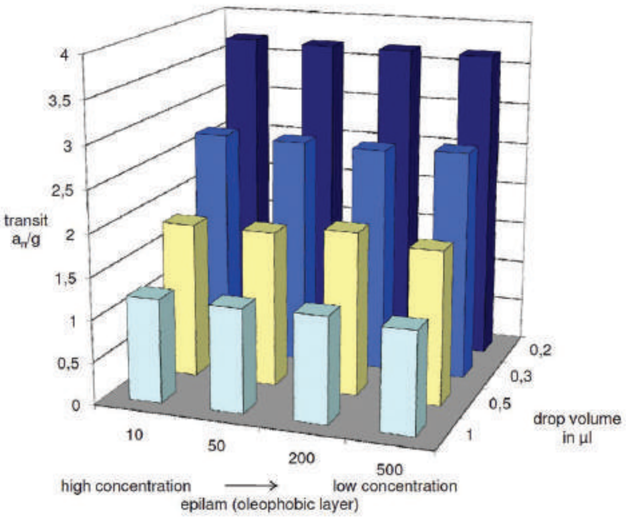

Figure 10. *Influence of drop volume on transit factor*

In Figure 10 the influence of drop volume and concentration of oleophobic layers on the transit factor of silicone oil on glass (smooth surface) is given. With reducing the drop volumes from 1µl to 0.2µl the transit factors raise by a factor of 3.5.

Another quite interesting aspect of using oleophobic layers is to apply them as a creeping barrier for fluids. Here considerable volumes of fluids can be hindered to step onto coated surfaces. In the test array of the APG a silicone oil drop of 1.5µl was set exactly on the uncoated glass surface at the borderline to the coated area (Figure 11). When determining the transit factors, higher values are observed compared to tests when placing the droplets directly onto the oleophobic layer. Additionally a strong influence of the concentration of the coating can be observed (Figure 12): higher concentrations give higher barrier effect.

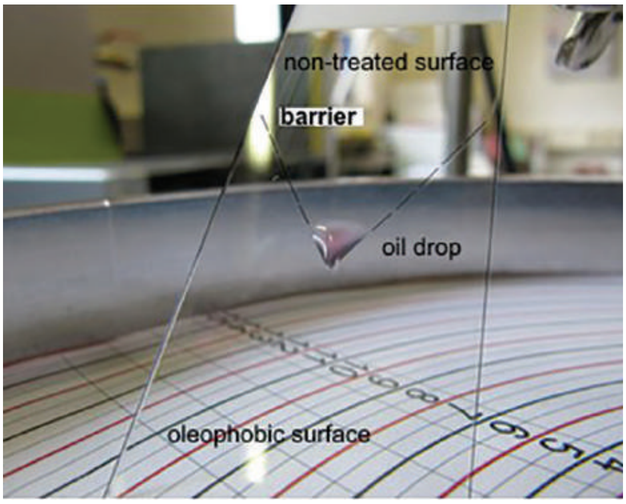

Figure 11. *Test specimen prepared for creeping barrier effect*

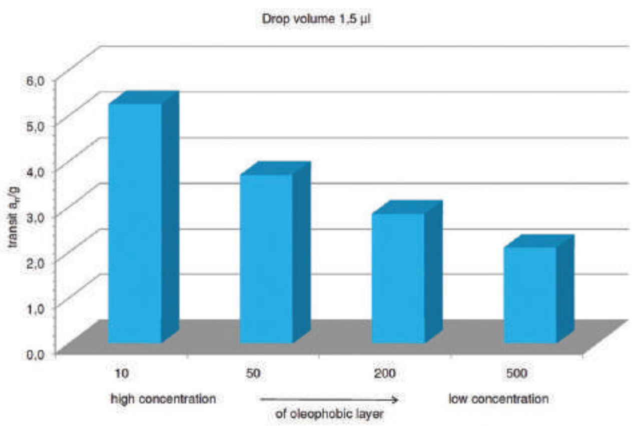

Figure 12. *Influence of concentration of olephobic layer on transit factor*

On the surfaces of plastic materials the adhesion of lubricants is quite different. Each base material and olephobic coating concentration acts specifically with the respective lubricants. Trends may be observed, but each combination shows individual behaviour. So, for example, for each lubricant type used with a certain plastic material the optimum concentration of the oleophobic layer can be determined having the highest adhesion. The transit factors of tests with a high concentration of oleophobic layer (10) and a medium concentration of oleophobic layer (200) examples are given in Figures 13 and 14.

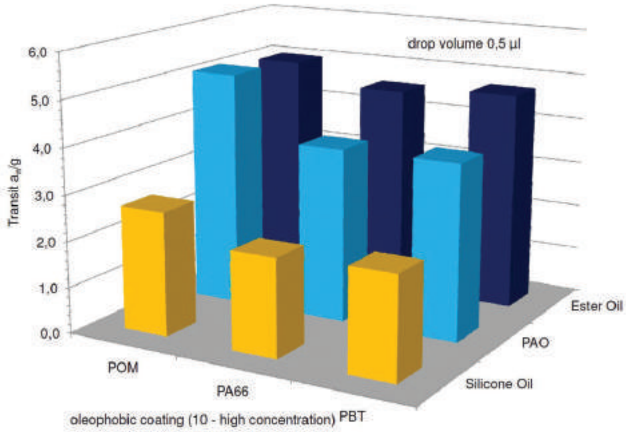

Figure 13. Influence of type of lubricant and type of plastic material (oleophobic coating 10-high concentration) on transit factor

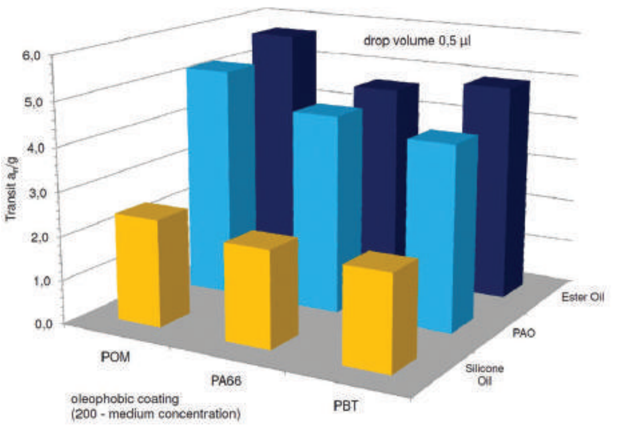

Figure 14. Influence of type of lubricant and type of plastic material (oleophobic coating 200-medium concentration) on transit factor

Conclusion and Outlook

The determination of adhesion forces under dynamic conditions extends the range of laboratory test methods for the characterisation of surface wetting properties. In particular, the possibility of testing on real component surfaces and with small oil volumes opens up new aspects for the function prediction of lifetime lubricated systems in precision engineering.

Current works now focus on the interfacial tension between lubricants and material surfaces and so to extend the information that can be retrieved of the test method. This includes the evaluation of the centrifugal forces in relation to the circumference of drops, expressed as force per length and the evaluation of the adhesive forces based on the surface tension of fluids and the circumference of drops. Materials to be evaluated are injection moulded plastic bars with different surface qualities (i.e. polished, grounded or eroded with various roughnesses), commonly used for industrially produced functional parts.

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Acknowledgements

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