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# Characterisation of cohesion, adhesion, and tackiness of lubricating greases using approach–retraction experiments

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#### ABSTRACT

Greases are widely used in machinery and automotive components to protect components from frictional and wear losses. As a result, interaction properties like adhesion to the substrate, cohesion or consistency, and tackiness become crucial factors and often dictate their performance. All these properties are related to microstructural aspects of grease like thickener network, wetting agents, and additives. The aim of this paper is to use approach–retraction experiments for qualitative/quantitative determination of the above mentioned properties and also understand the influence of grease constituents on these properties. It was found that among all the grease constituents, the thickener in particular dictates the cohesiveness and tackiness of a grease with less influence on the adhesion. The effect of thickener on the lubricating properties of the greases was not clear, which supports the notion that oil in the grease provides the lubrication and thickener to be a carrier. The data on cohesiveness/consistency obtained from this approach–retraction experimental method correlate with the traditional cone penetration tests thus validating this methodology.

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#### 1. Introduction

Lubricating greases are widely used in many applications ranging from electrical contacts [1], wire-rope applications [2], and largely on machinery where lubricants cannot be successfully applied, e.g. on unsealed or non-enclosed components. The main constituents of lubricating grease are the oil and the thickener which in most cases is metal based soaps [3]. The function of lubricating grease is analogous to a wet sponge. The oil retained in the thickener network is released based on several equilibrium conditions such as temperature, shear stress, and mechanical pressures [4]. Some lubricating greases contain additives for friction reduction, anti-wear properties, temperature stability, etc. [5]. One of the desired properties of greases is stickiness to the surface, which is determined by factors such as cohesiveness, adhesion to a surface, and tackiness or formation of thread like structures. By varying the composition of the constituents the properties of greases can be adjusted. For example, high viscous polymers (called tackifiers) are added to increase tackiness or thread formation.

Most experimental methods to characterize lubricating greases with respect to their cohesive and adhesive properties are rather empirical in nature. The cohesiveness historically has been

qualitatively measured by cone penetration [6] and oil separation measurements [7], while more recently rheological measurements provide some scientific insight [8]. In cone penetration the consistency or cohesiveness of grease is measured by dropping a cone of known mass into grease filled trough and recording the penetration depth of the cone. For instance, a low depth of penetration means firm grease with good consistency. The adherence of greases to the substrate is characterised by water spray off or water wash out measurements [8]. In water spray test, the greased metallic surface is subjected to direct water jet and the adherence is measured from the mass of grease lost as a function of time [9,10]. Another method used for quantifying adherence is by subjecting greased cylinders to centrifugal forces. Depending on the amount of grease mass lost, the adhesion strength is ranked [11]. In most cases, failure occurs within the grease, which technically is cohesive failure. A further improvement in adhesion measurement is done by simultaneous conduction current measurements during centrifuging. With this technique the adhesion of grease to the substrate is defined in terms of coverage area or ratio of metallic to greased area after centrifuging [11]. The above mentioned test methods suffer from issues like poor precision and limited correlation to industrial practise [12]. Another interesting aspect of grease is tackiness (ability to form strings), which is desirable in some applications and unwanted in some others (like gears, wire ropes, etc.). The measurement of tackiness is still very subjective.

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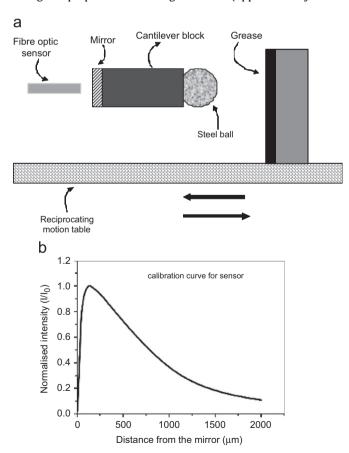
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In this paper, we explore the possibility of using approachretraction experiments to characterise properties of greases like adherence, cohesion, and tackiness (ability to forms threads before separation). The importance of various constituents of grease like oil, thickener, and additives on the above properties will be explored. Moreover, the illustrated method can be an useful tool for the selection of appropriate grease or in the research and development.

#### 2. Experimental

The approach–retraction test method is simple in which a greased substrate is moved towards a ball attached to a flexible cantilever. On establishing the contact, the greased substrate is moved further until a certain contact load is achieved. On reaching the target contact load, the greased surface is moved away from the ball (retraction) until complete physical separation. The basic technique is analogous to pull-off force experiments done with an atomic force microscope for studying physical interactions [13]. After this approach–retraction cycle, when the deflection force is plotted as a function of moved distance, important information on the properties of greases can be obtained.

A schema of the experimental set-up is shown in Fig. 1a, which consists of steel ball attached to a sensitive cantilever with a low spring constant of 80 N/m and can deflect in the direction parallel to the motion table. The deflection on the cantilever is measured using fibre optic sensor technology (microtribometer [14], Falex NV, Belgium). The sensor used in this study has bundle of emitters and receivers arranged randomly and has characteristic intensity to distance relationship as shown in Fig. 1b. The optical fibre is aligned perpendicular facing a mirror (approximately 1 mm



**Fig. 1.** (a) Experimental set-up for approach-retraction experiments; (b) characteristic normalised intensity to distance curve for a fibre optic sensor. (LST curve).

away) attached to the cantilever deflection axis (see Fig. 1a). The optical fibre emits certain intensity of light, which is reflected back from the mirror,  $I_0$ . When there is a deflection, the reflected light intensity changes and the deflection can be determined from the calibration curve shown in Fig. 1b. The obtained deflection in  $\mu$ m is then multiplied with the spring stiffness to precisely determine the deflection forces. The desired load between the ball and the greased surface can be controlled by constantly monitoring the deflection force on the cantilever through a closed feedback loop system. The same set-up can also be used to measure frictional forces by simply changing the cantilever which can deflect in both perpendicular and parallel to the motion table.

#### 2.1. Materials

The greases used in this work were supplied by Dow Corning GmbH, Wiesbaden, Germany. The commercial names of the greases are not revealed due to confidentiality. The microstructure of the greases is shown in Fig. 2. The investigated greases are

Grease 1 (Fig. 2a) is silicone oil based containing lithium soap thickener (15–20 w%) with a specific gravity of 1.05.

Grease 2 is mineral oil based with 3-7~w% lithium soap thickener along with solid lubricant micro-particles ( $2-10~\mu\text{m}$  in size) and has a specific gravity of 0.93 (Fig. 2b). Solid lubricant particles are commonly used as additives and remain dispersed structure.

Grease 3 is mineral oil based with only solid lubricant particles in the range of 2–10  $\mu$ m (minimum 30% by weight) with a specific gravity of 1.4 (Fig. 2c). Micro-particles in Grease 3 were polarised. The polarisation of micro-particles improves their adhesion to the substrate.

It can be seen that grease 1 has a spongy structure and appears distinct from other two greases. Grease 2 and 3 bear some similarity because of similar type of oil and solid lubricant particles.

A polyoxymethylene (POM) polymer substrate with a 200 μm depression was chosen for this investigation. To have a consistent and repeatable layer, grease was first applied on this depression and the excess was scrapped off leaving behind 200 µm thick layer. A bearing steel ball 3 mm in diameter (Stainless steel ISO 3290 grade with HRc 60) was chosen as a counterbody. Prior to the experiments, the surface of the steel counterbody was cleaned with acetone and ethanol. A cantilever with a stiffness of 80 N/m but extremely rigid in normal direction was used. A fresh substrate was used for each variety of grease in order to avoid contamination. A contact load of 14 mN was applied as target load corresponding to a maximum Hertzian stress of 20 MPa [15]. On each grease, 25 approach–retraction cycles were done, i.e. the motion table (LMS in Fig. 1a) is moved 25 times towards and away from the steel ball. The approach-retraction speed was kept constant at 500 µm/s.

#### 3. Results and discussion

A typical plot of cantilever deflection vs. distance moved during approach–retraction cycle for greased and ungreased POM substrate against a steel ball is shown in Figs. 3 and 4, respectively. For an ungreased POM substrate in Fig. 4, both approach (dashed line) and retraction (black) sections of the curve overlap whereas for a greased substrate the two sections of the curve are clearly different. The curve on the greased contact appears like a hysteresis loop, which has to be further analysed to extract useful information (Fig. 3). A schema of events taking

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