Experimental Researches of the Grease Durability via Microscopic Investigation

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Abstract. The working life of a bearing depends on the aging characteristics of the lubricant. Aging produces physical, structural and rheological modifications in the grease that have rarely been studied from an overall stand-point. Also, analysis of used greases can be a valuable source of information on the mechanisms involved when a bearing is lubricated with grease. From this point of view, it is very useful to establish a few criteria for the grease life time, as follows: modification of the physical and chemical properties of the greases, modification of the rheological parameters, modification of the global electrical resistance of the grease film and modification of the microscopically grease structure and apparition of the wear particles. The paper proposes a comparative study of greases in the fresh state and after use in ball bearings, analysing the modification of the rheological and structural properties. The mechanical aging test was carried out on an experimental stand and the grease structure was studied using a scanning electron microscope. The rheological properties were studied on a cone and plate Brookfield viscometer.

1. Introduction
It is necessary to carry out a comparative study of greases in the fresh state and after use in bearings for two main reasons. First, the working life of a bearing depends to a great extent on the aging characteristics of the lubricant. Aging produces physical, structural and rheological modifications in the grease that have rarely been studied from an overall stand-point. Secondly, analysis of used greases can be a valuable source of information on the mechanisms involved when a bearing is lubricated with grease. From this point of view, in the literature are presented a few criteria for the grease life time, as follows:
- modification of the physical and chemical properties of the greases (drop point, penetration, neutralization number etc.), [1-3];
- modification of the rheological parameters (yield stress and viscosity), [4-7];
- modification of the global tribological properties of the bearings, [8-11];
- modification of the global electrical resistance of the grease film, [12, 13];
- modification of the grease structure (microscopical analysis or X-ray diffractionmetry) and apparition of the wear particles, [14-16].

The present study was undertaken to understand the net effect on the change of structure of greases used in ball bearings after being operated in centrifugal fans. Electronic microscopically technique has been applied in this work to diagnose the changes in chemical composition of the soap residues of the used greases.
2. Experimental facilities
The experimental test machine used for the researches is a centrifugal fan (Figure 1) especially equipped with transducers, which was integrated in an industrial installation.

![Figure 1. General view of the experimental stand.](image)

The principal dynamic elements measured were: the torsion moment, the radial force in the bearing, the load variation of the fan and the temperature in the bearings. Figure 2 presents the electronically part of the experimental stand, which is used in data acquisition and measurement for the characteristic parameters. It was used a computerized system for the data acquisition, with specifically signal, an data acquisition board and a computer. The LABVIEW software has been used for the data acquisition and numerical processing.

![Figure 2. Schematic of the experimental stand](image)

The principal technical characteristics of the experimental stand are:
- the measurement of the loads on the mainstays with the one-directional force transducers, produced by the Hottinger Baldwin Messtechnik firm (U9B type - the force is measured until 50 kN);
- the measurement of the torque with a torque transducer placed on the fan shaft (the transducer is produced by the Hottinger Baldwin Messtechnik firm and his values are until 200 Nm);
- the measurement of the temperature, with the aid of temperature transducers (temperature range 35…170 °C).

The ball bearings assembly analyzed and tested is presented in Figure 3.
Figure 3. Ball bearings assembly

Several types of physical and chemical analysis were carried out to determine the composition of the greases and the main characteristic parameters: penetration at 25°C, dropping point, colloidal stability, acidity and alkalinity, oil separation, oxidation stability and evaporation loss. The rheological properties of the greases were studied on a cone and plate Brookfield viscometer, assuming the validity of Bingham rheological model.

In order to analyse the microscopically structure of the greases, a few stages have been performed:

- extraction of the oil from the grease, using successive applications of a suitable nonpolar solvent (hexane);
- weighing of the oil to determine the concentration in the grease;
- examination of the structure of the metal soap after extraction using a scanning electron microscope;
- identification of the possible wear particles found in the lubricant.

3. Experimental investigations

To observe the influence of the temperature on the grease durability and its microscopically structure, two types of greases were used: one thickened with lithium (UM 185 Li 2) and the other with a calcium complex (UM 170 LiCa 2). Their NLGI grade is 2. Table 1 summarizes the known components of the greases and the information relating to the bearing tests that produced the used greases.

| Parameter / Grease | UM 185 Li 2 | UM 170 LiCa 2 |
|--------------------|-------------|---------------|
| Thickener          | lithium     | lithium - calcium |
| Base oil           | Mineral oil (naphtenic) | |
| Aspect             | Unctuous and homogenous |
| Colour             | Yellow green |
| Usual application  | ball bearings, mechanisms | ball bearings, journal bearings |
| Diameter of the inner ring, mm | 35 |
| Diameter of the outer ring, mm | 80 |
| Bearing load capacity, kN | 30.5 |
| Operating speed, rev/min | 1500 |
| Radial load, N | 2500 |
| Temperature, °C | 60…80 |
| Test time, hours | 480 |
The tested ball bearings are 2307K, according to the ISO/R15 standard. The bearings assembly is presented in Figure 3. The grease quantity that fill the ball bearing is approx. 0.7 cm$^3$ (5% of bearings free volume).

4. Results and discussions
The test was carried out in two stages. At the initial moment, the fresh grease was analysed in order to obtain the physical – chemical and rheological properties and also the microscopically structure. After 480 hours of testing, samples of used grease were taken at various points from a bearing: cage ribs, middle and roller joints and surfaces. The same analysis were repeated and the comparatively results are presented in Table 2. At different periods of time, data acquisition for the radial force in the ball bearing, for the torque and for the temperature were realized. A typical result for the measurement of the radial force is shown in Figure 4.

Table 2. Physico-chemical characteristics of the grease samples before and after test.

| Physical – chemical and rheological characteristics | UM 185 Li 2 | UM 170 LiCa 2 |
|-----------------------------------------------------|------------|---------------|
| Dropping point, °C (ASTM D 566)                      | Fresh      | Used          |
| Penetration at 25°C, 1/10 mm (ASTM D 217)            | 185        | 168           |
| Alkalinity (NaOH), % (ASTM D 128, par. 18 and 20)   | 0.14       | -             |
| Acidity, mg KOH/g (ASTM D 128, par. 18 and 20)       | -          | -             |
| Oxidation stability, % (ASTM D 942)                  | 0.35       | 0.39          |
| Oil separation, % (ASTM D 1742)                      | 15         | -             |
| Evaporation loss (ASTM D 2595)                       | 0.45       | -             |
| Yield stress, Pa                                     | 399        | 197           |
| Viscosity, Pa·s                                      | 0.798      | 0.413         |

Figure 4. Typical variation of the load during the experiments
Concerning the lithium grease, Table 2 shows small variations of the main physico-chemical parameters between fresh and used grease. The same observation is valid also from the point of view of the microscopically structure. From the rheological point of view, it can be observed an important reduction of the values of the rheological parameters.

Figure 5 illustrates the fiber network of the fresh lithium grease after extraction of the metal soap. The fibers are formed by arrangements of soap molecules linked by hydrogen and semi polar interactions. The characteristic dimensions of an elementary fibre are about 0.2 x 0.3 x 12 μm, which is the average value measured on isolated samples. The fibers appear to be twisted, which may be a consequence of the asymmetric shape of the soap molecule resulting from the lateral hydroxyl group.

Figure 6 shows the fiber network of the used grease lithium grease. There is no perceptible difference, except for the presence of colloidal aggregates (with dimensions of order of 2…3 μm). These aggregates are thus formed mainly from particles produced by wear. A typical image of an wear particle is presented in Figure 7.

In conclusion, in the specific case of the test on a roller bearing lubricated with lithium grease, comparative microscopy analysis shows that only a minority of the fibers are used, by cutting them. In addition, metal particles produced by wear become mixed with the grease. Transmission microscopy reveals the size of the particles that form the aggregates observed with the scanning electron microscope.
Figure 9. Used calcium complex grease thickener

Figure 10. Impurities particles found in the calcium complex grease from bearings

In the case of the calcium complex grease, Table 2 shows important differences between fresh and used grease, much more significant than for the lithium grease. This observation is directly connected with microscopically structure of the calcium complex grease. Microscope examinations carried out on samples of fresh calcium complex grease after cleaning with hexane show that the thickener is in form of spherical particles (Figure 8), with mean dimension of 1…3 μm.

Figure 9 shows that the thickener in the used grease is formed mostly of elongate ellipsoids, with mean dimension of 2…5 μm. Similar to the case of the lithium grease, in this case also have been identified metal wear particles (Figure 10), with dimensions of order of 3…4 μm.

A structural change therefore occurs in the calcium complex grease during use. The structural transformation of the thickener is accompanied by the chemical transformation, which occurs with certain kinetics increased by the temperature of the bearing.

5. Conclusions

The physico-chemical, rheological and structural studies of fresh and used lubricating greases conduce to the following conclusions:

- the mechanical properties of the used grease can be very different from those of the fresh grease and depend on the chemical composition of the grease;
- the modifications on the physico-chemical and rheological properties of the grease during lubrication may have a few causes: a variation in oil concentration, the introduction of the wear particles in the network structure of the grease, working temperature etc;
- these modifications provide information on the nature of the flow in the bearings and the qualities required of the grease for the lubrication of the bearing;
- comparatively analysis of these two greases shows that the destruction degree of the lithium grease is less important than the complex calcium grease;
- microscopically study of used greases taken after different periods of use could provide useful information on the state of the ball bearings, in order to make more appropriate choices.
- no individual technique is capable of providing complete characterization of a sample, and a combination of several techniques is usually required if all the parameters must be determined.

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