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## Investigation of laminated fabric cages used in rolling bearings by ToF-SIMS

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#### ABSTRACT

Properties of textile-laminated phenolic composition cages were studied by ToF-SIMS: (i) by monitoring the *di-octyl-sebacate* (DOS) content of the lubricant film within the elastohydrodynamic (EHD) contact region of the inner and outer rings of tested high-speed rolling element bearings, and (ii) by the investigation of boundary layers of cages in the region of the cage-ball-contact. Portions of the DOS amount stored in the cage by the bearing conservation process are released vs. the operation time via the cage surfaces into the EHD contact regions.

It was revealed that the amount of the diester DOS separated from the cage into lubricating film of the raceway, is influenced by the lubricant additive formulations used in the tribological tests via their attributes concerning the acting tribological system processes like wear, boundary layer formation, adsorption, etc. ToF-SIMS research [1,2] of both the lubricant surface and the boundary layer in the EHD contact region showed that the primary antioxidants used as additives do not only act as oxidation inhibitors, but that they also do influence the wear processes of the counteracting bodies and the formation processes of boundary layers on rolling bearing steel SAE 52100.

With the amounts of DOS, found in dependence on six different lubricant additive formulations A–F investigated, also an influence on the boundary layer formation processes is detected for the surface of the textile-laminated phenolic composition cage. ToF-SIMS imaging and depth profiling of cage boundary layers prove this dependence of the boundary layer formation process.

The secondary ion (SI) intensities of DOS correspond with the lubricant dependence of the  $^{56}\text{Fe}^+/C_4H_8^+$  ratios obtained from the used lubricant films in the EHD contact regions [1,3] as well as with the ratios  $^{56}\text{Fe}^+/C_4H_8^+$  of the boundary layers in the raceways and of the cages [3]. Both the  $^{56}\text{Fe}^+/C_4H_8^+$  ratios and the DOS intensity values show an indirect dependence to the lubricant service life of the lubricants A–F.

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#### 1. Introduction

Most of the rolling bearings used today consist of an inner and outer ring, rolling bearing elements and a cage. The cage is used to separate the rolling elements from each other during operation to avoid interaction between them. Apart from polymer and metal cages, laminated fabric cages are often used. Such cages are usually made of a porous textile-laminated phenolic material, which is saturated with a lubricant by the bearing conservation process or by vacuum impregnation of the cage.

The operational performance of grease-lubricated high-speed rolling element bearings depends on the lubricant additive formulations used. The intention of this research framework [3–5] was to perform basic research on the influence of primary antioxidants on the investigated tribological systems. This contribution focuses on ToF-SIMS results with regard to the chemical composition of tribological system surfaces in relation to: (i) one of the bearing components – the laminated fabric cage – and (ii) the used lubricant additive formulations under investigation.

#### 2. Experimental details

#### 2.1. Tribological tests and lubricants

The tribological tests were performed with six different lubricant additive formulations (lithium soap greases A–F). In 5 cases, primary antioxidants were added to the composition of a base lubricant in order to improve the lubricant properties [1,2]. The tribological short- and long-term tests were performed with a machinery spindle arrangement under almost realistic operation

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conditions [4] with two new commercial angular contact ball bearings (type: FAG B7008C.T.4PS.UL, Fig. 1) for each test run. The long-term tests were terminated by passing of failure criteria – either a temperature of the outer ring T=373 K or a frictional momentum of  $400\,\mathrm{N}\,\mathrm{mm}$  – or otherwise if a value of  $\tau$ =5000 h was exceeded for the operation time  $\tau$ . For a set of short-term A–F tests a fixed operation time of  $\tau$ =150 h was used. A more detailed description of the tribological short- and long-term experiments is represented elsewhere [4]. Specific results of indentation experiments are included in [5,6].

The base stock of the lubricant grease consists of poly-alphaolefins (PAOs) and steric hindered polyol esters TMP(C8/C10) a esterification of trimethylolpropanol (TMP) and the carboxylic acids caprylic acid (C8) and caprinic acid (C10). As a dispersed phase of the colloidal suspension, the soap Li-12-hydroxystearate was used. The primary antioxidants used were in the case of the lubricant additive formulation A: no additive; B: phenyl-alphanaphthylamine (PANA); C: dioctyl-diphenyl-amine (DODPA); D: 2,2'-methylen-bis-(4-methyl-6-tert-butyl-phenol) (AO2246); E: polymeric tri-methyl-quinoline (TMQ); and F: a blend of the additives B and D.

A well-defined cleaning and greasing procedure was applied to the commercial rolling bearings before the tribological tests [4] to ensure optimal and in particular reproducible surface activation/passivation states with respect to surface-active components of the lubricant additive formulations used. Didziulis et al. [7] studied effects on the lubricant service life due to varied cleaning procedures of rolling bearings.

#### 2.2. Time-of-flight secondary ion mass spectrometry

A comprehensive overview about the capabilities and potentialities of this surface analytical method is given in [8,9]. For the ToF-SIMS characterization performed here, a reflectron-type time-of-flight mass spectrometer built up at the Institute of Physics (University of Münster) was used. The basic instrument design was equivalent to the TOF-SIMS IV type (ION-TOF GmbH). As analysis primary ion gun, a pulsed electron impact ion source (10 keV, <sup>40</sup>Ar<sup>+</sup>) was used for various operational modes such as static ToF-SIMS, imaging and depth profiling. In the case of the static ToF-SIMS investigations, the analysis area at the sample surface was  $200 \,\mu\text{m} \times 200 \,\mu\text{m}$  and measured typically  $750\,\mu m \times 750\,\mu m$  in the case of ToF-SIMS imaging. Sputter depth profiling was performed in the non-interlaced dual beam mode using a pulsed <sup>40</sup>Ar<sup>+</sup> sputter beam at energies of 3 keV, 2 keV and 750 eV. During sputter depth profiling oxygen flooding was applied to sample surface by using an <sup>18</sup>O<sub>2</sub> molecular beam jet in order to stabilize and to enhance secondary ion yields. A pulsed low-energy electron flood gun was applied for charge compensation purposes.

#### 2.3. Double focusing magnetic sector SIMS

The double focusing magnetic sector SIMS (DF-SIMS) instrument used here for depth profiling of cage boundary layers was a modified Cameca IMS 6f (Cameca IMS LAM) at the CRP 'Gabriel Lippmann'. A continuous  $^{16}\mathrm{O}^-$  sputter beam (8 keV impact energy) was used for the sputter depth profiling. It was scanned across an area of  $250\,\mu\mathrm{m}\times250\,\mu\mathrm{m}$  and only secondary ions coming from a circular area limited to a diameter of 60  $\mu\mathrm{m}$  in the middle of the scanned sputter area were analyzed. Such analytical conditions using  $^{16}\mathrm{O}^-$  as primary ions allow performing analyses without the use of the  $90^\circ$  off axis electron gun. In contrast to the ToF-SIMS sputter depth profiling, 80-times higher sputter ion current densities have been applied to the sample surface in order to obtain analytical information from deeper boundary layer regions.

### 2.4. Sample preparation for SIMS

After the tribological tests, the rolling bearings were disassembled (Fig. 1). Inner and outer bearing rings were mechanically broken in order to get access to the lubricant surface and boundary layers of the EHD contact regions. Cages were cut into pieces to obtain an analyzable shape for the investigation of the ball–cage-interface.

For the investigation of the lubricant surface in the EHD contact regions of inner and outer rings unchanged lubricant layers between final ball localizations were selected.

The analytical information depth of static ToF-SIMS is in the order of 1-3 monolayers. Therefore, it is possible to investigate either the lubricant surface or the boundary layer surface. For the investigation of the boundary layers of the bearing components the attached lubricant layer was removed by ultrasonic cleaning procedures ( $t=15\,\mathrm{min}$ , three times) with the organic solvents n-hexane and acetone, respectively.

The ultrasonic cleaning procedures were applied only for the preparation of the boundary layers. The boundary layer here is defined by the tribologically modified boundary layer of bearing material including reaction layers and molecular adsorbates. All nonadsorbed portions of the lubricant film (adsorptive) are removed by the cleaning procedures used.

#### 3. Results and discussion

#### 3.1. Tribological tests

For each of the six lubricant additive formulations A–F, four to five pairs of rolling bearings were tested in the long-term tests and one pair in the short-term tests. The results show that the primary antioxidants used affect the operational performance and







Fig. 1. Tested bearing (left) and disassembled test-bearing (middle-left) with the main components: the inner and outer ring, the rolling elements (balls:  $\emptyset$ =7.93 mm) and the laminated fabric cage. Cage and inner ring of an untested reference bearing (middle-right), schematic cross-section of the angular contact ball bearings used for the tribological tests (right).

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