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Characterization of wear debris of systems operated under low wear-rate conditions

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Abstract

Wear particles found in ultra-low wear rate experiments were analyzed in order to understand the wear mechanism. Using a sophisticated particle separation method, the debris were separated from the oil and then analyzed using transmission electron microscopy accompanied by electron diffraction for chemical characterization. The majority of the wear particles have a size of about 250 nm, only very few are larger than 1 μm. The particles appear mostly amorphous and contain chemical elements of both interacting solids as well as the oil. In addition, almost all particles have a thickness of 30 nm or less. The proposed particle generation mechanism is a squeezing process of flowed material under pressure.

Keywords: Particle analysis; Wear mechanisms; Nanomechanics

1. Introduction

Wear particle analysis is a widespread method to characterize the tribological condition of mechanical systems [1,2]. In most of the cases debris with a size of some micrometers up to hundreds of micrometers are analyzed as summarized in Table 1.

In contrast, sliding bearings and other moving parts of state-of-the-art mechanical systems usually show wear rates in the range of a few nanometers per hour. Table 2 provides wear rates of engine components.

The specific machine part was labeled with nuclides. Due to wear also the debris coming from this location carry nuclides that are registered in order to obtain the amount of wear averaged over the labeled area. Taking these low values, it is therefore improbable that large wear particles, as shown in Table 1, lead to ultra-low wear rates. Wear particles must be significantly smaller and wear mechanisms must be different than proposed in the literature [5,6]. Thus, a new procedure has to be found to analyze smaller and extremely small particles. To analyze the wear mechanism, knowledge about the chemical composition of the particles is also necessary.

2. Experimental details

2.1. The tribological experiment

The oil samples were taken from a pin-on-disk test described in detail elsewhere [4,7]. The tribological pairing consisted of a chromium-plated steel pin and a gray cast iron disk. The system was run fully lubricated with additivated engine oil (Fuchs Titan 5W40). Both wear rate and coefficient of friction were recorded continuously with high precision. To measure wear, the pin was labeled with radionuclides induced by the bombardment of thermal neutrons. The nuclide density is about 10^{+15} cm^{-3} and does not measurably change the mechanical properties of the material. The generated wear

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Table 1
Particle analysis methods and sizes [3]

Method	Particle size range	Type of particle
Direct reading ferrography	1–100 μm	Ferromagnetic particles
Magnetic filter plug	25–1000 μm	Ferromagnetic particles
Patch test	3–100 μm	All types
Acid digestion	No limits	All types
SEM-EDX	From submicron to macro range	All types

Table 2
Typical average wear rates of engine components as determined by radionuclide technique [4]

Engine component	Wear-rate
Piston ring	5–15 nm/h
Small conrod bearing	Maximum 8 nm/h
Large conrod bearing	2–10 nm/h
Tappet	10 nm/h
Cam	5–10 nm/h

particles carry nuclides that are used to detect the concentration of wear particles in the oil. After proper calibration and calculation, the wear rate was measured with a resolution of a few nanometers per hour.

The oil samples were taken after the end of test at 68 h. To obtain realistic stressing conditions, the tribometer as the model system was operated to show wear rates as in real applications. Thus, model and real system were made similar with respect to dissipated power [8].

2.2. Transmission electron microscopy

At the end of the test, oil containing wear particles was taken with care from the oil bath. The particles were extracted from the oil by centrifugation, rinsed in *n*-heptane and deposited on a copper grid, covered by a very thin carbon film (approximately 5 nm thick) for transmission electron microscopy (TEM) observations. The wear particles were observed in an analytical transmission electron microscope (PHILIPS 420EM) equipped with electron energy loss spectroscopy (EELS) and EDS spectroscopy. This microscope produces also energy loss energy spectra giving information on the elementary composition of the wear particles which is related to the near-surface material composition, where it originated from.

3. Results

3.1. Morphology

The obtained TEM images show a whole variety of different shapes and sizes. A common feature of all particles is their very small thickness, mainly less than 30 nm, since they are translucent. Besides round particles (see arrow A in Fig. 1), also flaky particles exist as shown in the upper right corner (arrow B). These particles have similarity to fractions of a layer. Arrow C points to particles showing crystalline features. This particle has irregular shape and resembles a piece of metal that was rapidly quenched. Finally, arrow D highlights small and uniformly shaped overbased detergent particles, partially coagulated to clusters. Using image processing software, the particle size was determined and is shown in Fig. 2. Accordingly, the majority of the particles has a size of about 250 nm. The probability of particles with a size of larger than 1000 nm is almost zero.

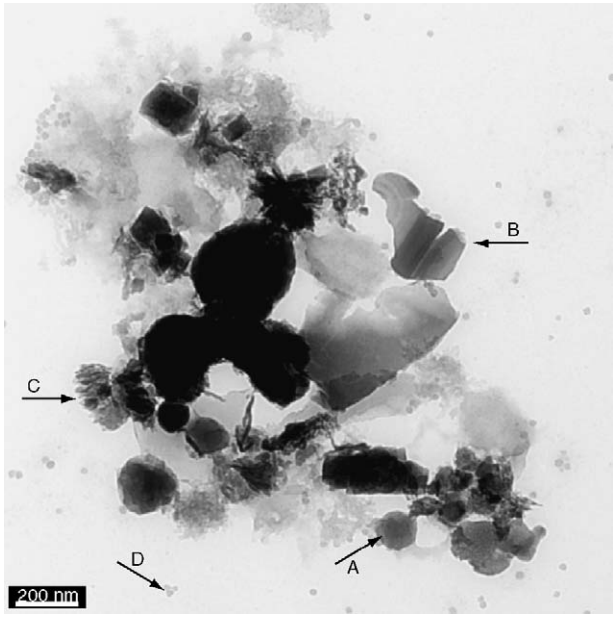

Fig. 1. Wear particles.

3.2. Structure

Transmission electron microscopy showed at large magnification that the structure of a flaky wear particle is basically amorphous, see Fig. 3. The particle has a very high carbon content and shows no crystalline clusters of metals or other elements.

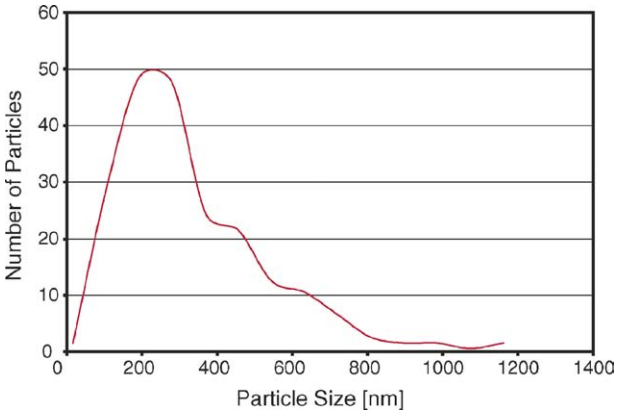

Fig. 2. Particle size distribution.

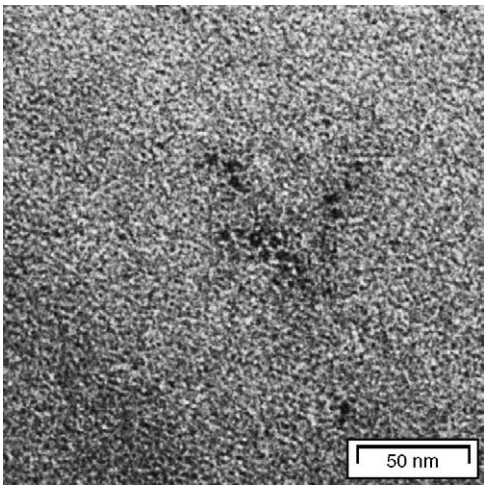

Fig. 3. High resolution TEM image of a wear particle.

3.3. Chemistry

The chemical analysis shows that nearly all elements occurring in either the two interacting solids or the oil are present within single wear particles, see Fig. 4. The element spectrum shows a high content of carbon and oxygen as the result of a contact with lubricant and/or air. P, S and Zn are the fingerprints of additives, especially of antiwear additives, whereas the high concentration of Ca originates from a detergent. The concentration of iron and chromium is extremely low. The copper peaks have its origin in the grid used to hold the wear particles and do not originate from the experiment.

Besides the randomly round shape of most of the particles, very small particles showing features of crystals exist. Fig. 5 shows in the upper part many 20 nm small crystals with a high concentration of Ca. These are overbased detergent particles. They consist of an amorphous $CaCO_3$ (calcium carbonate) core surrounded by a shell of detergent molecules, most probably calcium sulfonate or salycilate. At the left side of the figure, a flaky particle was analyzed. It turned out that this particle showed zero intensity of iron and chromium. In this case, the particle only contained elements of environment and lubricant (P, S, O, Zn and Ca).

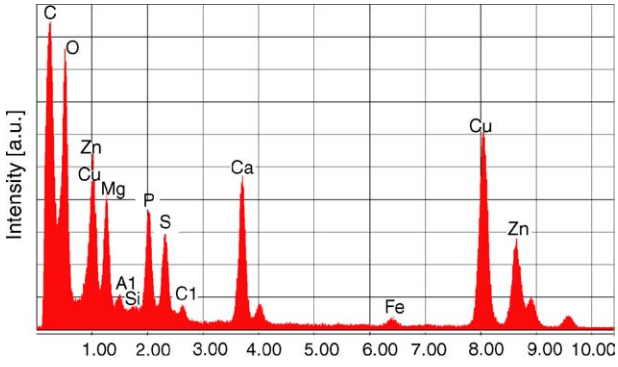

Fig. 4. Chemical composition of wear particles.

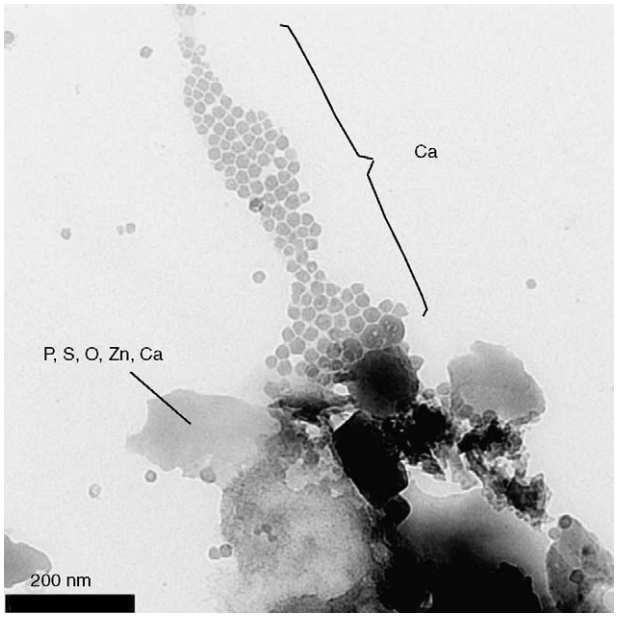

Fig. 5. Chemical composition of wear particles at selected locations.

4. Discussion

Due to mechanical intermixing, the near-surface zone of the solids becomes alloyed with elements of lubricant, counterbody and environment. Since the interaction of both solids has the greatest intensity right at the interface and decreases towards the bulk, also the chemical depth profiles show gradients in concentration. At the surface usually carbon shows the highest concentration. For the case of cast iron, the iron concentration gradually increases from almost zero to bulk concentration. The depth of the modified zone is a function of the dissipated power. Wear particles originating from this near-surface zone show a low concentration of the base metal (either chromium or iron).

The fact that wear particles contain a certain variety of elements suggests that besides intensive mechanical intermixing chemical reactions also take place, leading to new products as, for example, $CaCO_3$.

In 1973, Suh proposed a new theory for wear of metals. The theory focuses on plastic deformation and dislocations at the surface, subsurface cracks and void formation. The subsequent joining of cracks by shear deformation as well as the crack propagation lead to particle generation. The theory predicts flakelike wear particles [9]. In continuous wear measurement using radionuclide technique (RNT), wear curves show a strong increase of total wear during running-in. For optimized running-in conditions [7], the increase in total wear becomes gradually smaller and adopts constant increments (stationary conditions). After the running-in, the wear rate is often low but not zero. Assuming that the interaction of both solids with the additivated oil forms a protective film and that the tribological interaction is confined to that film, then the RNT should not be able to detect any wear, i.e., the wear curve

should become horizontal. Interestingly, this effect has never been observed so far. Therefore, the delamination theory applied to the formed additive layer alone does not hold. Only when we assume that, due to delamination, also base material (carrying nuclides) is removed, then low but increasing total wear can be achieved. The TEM images have shown that the majority of the wear particles have a thickness in the range of typically less than 30 nm suggesting that the formed film should not be thicker.

Calculations of the mechanical response of both solids suggest that, due to plastic flow and intermixing, a third body is formed as proposed by Kragelski and Dobycin [10] and Godet [11]. This third body has completely different mechanical properties than both solids. Nanomechanical testing showed that the third body has lower hardness [12] suggesting that plastic flow can be initiated easier and that therefore the dissipated power is lower. During the process of plastic flow, it is highly probable that wear particles become squeezed out of the asperity contact. This process occurs parallel at thousands of asperity sites. The reason for the low wear rate after running-in can be caused by re-insertion of the particles into the solid surface.

5. Summary

Wear particles derived from fully formulated motor oil were analyzed after realistic tribological stressing. The majority of the particles have a size of about 250 nm. The particles contain all the elements present in both solids, environment and oil. Most of the particles are amorphous. However, crystalline particles exist originating from chemical reactions that lead to new compounds, as for instance $CaCO_3$, but do not originate from crystalline metallic surfaces. TEM analysis suggests that when a tribological film is formed its thickness should be less than 30 nm.

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