Quantitative assessment of nano wear of DLC coated samples using AFM and optical confocal microscopy

G. Dai[1], F. Pohlenz[1], H. Bosse[1], A. Kovalev[2], D. Spaltmann[2], M. Woydt[2]
[1]*Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, D-38116 Braunschweig, Germany*
[2] *Federal Institute for Materials Research and Testing (BAM), D - 12203 Berlin, Germany*

Gaoliang.dai@ptb.de

Abstract

Nano wear of high-performance DLC-based thin film coatings, which had undergone tribological tests under rolling/slip-rolling conditions, was quantitatively assessed using calibrated AFM and confocal microscopy. Different areal S-parameters such as arithmetic average roughness S_a, root mean square roughness S_q, surface skewness S_{sk}, as well as areal V-parameters (related to Abbott-Firestone curve) have been evaluated from the AFM images measured from the worn and unworn areas. The cumulative distributions of surface area, projected area and material volume (loss) were analyzed in addition. The study shows that for the analyzed S-parameters the skewness *Ssk* was the most sensitive and reliable parameter to indicate the very small wear-related changes of the DLC-coated surface.

1 Introduction

The amorphous diamond-like carbon (DLC) coating is well known for its high hardness and wear resistance as well as low friction coefficients. It has been increasingly applied, in particular, in the automotive industry to increase the lifetime of car components and to reduce fuel consumption. Coating of rolling/slip-rolling parts is subjected to cyclic fatigue and furthermore under deficient lubricant is rarely reported and remains nowadays an attractive challenge [1].

Until today there is no single approach which can provide a complete and simple description of the surface topography to reveal the fine changes on its asperity peaks due to friction. In particular, it remains a challenging task for so-called “zero-wear” processes, where the material loss due to wear is within the height range of the original topography [2]. Consequently, a reliable quantitative analysis of such wear characteristics becomes crucially important to understand the "zero-wear" conditions, which may significantly increase the wear resistance and longevity of friction components. Quantitative assessment of nano wear of a DLC coating, which had undergone rolling/slip-rolling wear testing, is studied in this paper.

2 Workpieces used for wear tests

The substrates were made of the quenched and tempered steels 100Cr6H (OVAKO 'PBQ'), whose hardness is in the range of 66 HRC (Rockwell hardness C). Such steels are typically used in bearings and serve as a reference for the investigations of DLC-coated and uncoated novel steel grades. The DLC coating used for this study was deposited using a pulsed vacuum arc deposition system. The steel substrates were treated by an ultrasonic cleaning in an alkali water solution. The sample was sputter-cleaned prior to deposition. The thickness of DLC coating was 2-3 µm, coating type is a-C:H. The operating parameters of the friction test were as follows; slip-rolling scheme with a difference of rolling velocity of 10%, the average pressure was 1.5 GPa, the number of revolutions over the duration of the test were 10 million cycles, the lubricant VP1 SAE 0W-40 was applied to the contact zone.

3 Surface characterisation

An optical confocal laser scanning microscope ("LEXT OLS 4000" of the company Olympus) with a short light wavelength of 405 nm was applied for fast overview measurements, and an atomic force microscope (AFM) ("Dimension Icon" of the company Bruker) was employed for detailed quantitative measurements at the local areas of interests in this study. For both instruments, the amplification and linearity of the scales was traceably calibrated by applying a set of step heights and lateral gratings calibrated by a metrological AFM. Fig.1 presents two AFM images of the DLC surface at the worn and unworn areas. The general surface structure (morphology) seems to be similar, but the surface of the worn area (Fig. 1b) has some flat areas (marked with circles) on the top of asperities that are not present on the unworn surface shown in Fig. 1a.

These flat areas are the result of friction on the top of asperities without significant destruction of the latter. It should be noted here that most of asperities have not undergone such changes on their asperity tips during friction.

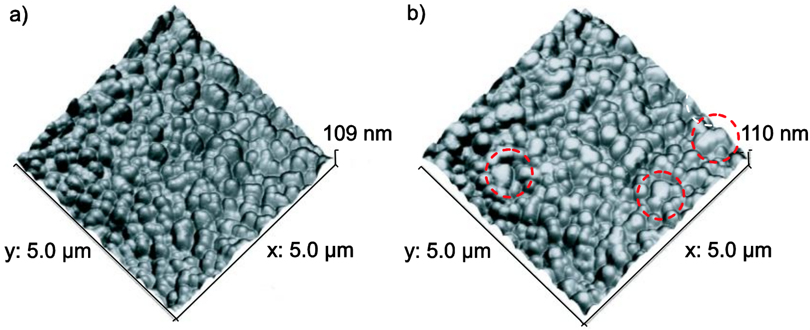

Figure 1. AFM images of unworn (a) and worn surface (b) of the tested DLC coating. Some flat areas on the top of asperities of the worn surface are marked.

4 Analysis of AFM measurement data

Different areal S-parameters such as arithmetic average roughness S_a, root mean square roughness S_q and surface skewness S_{sk} were evaluated from the AFM images measured on the worn and unworn areas [3]. In order to reduce the influence of the surface waviness on the evaluation, a 2D Gaussian filter with a cutoff wavelength of about 1.25 µm along the x and y axes was applied to pre-process the raw AFM images. Surface parameters characterised at 21 different measurement locations at the worn and unworn areas were compared. The result suggests that the skewness *Ssk* was the most sensitive and reliable parameter for assessing the nano wear. As shown in Fig. 2, the *Ssk* value is clearly reduced from the unworn areas to the worn areas, which indicates the loss of peak structures of the asperities during the friction test, agreeing well with the physical understanding of the "zero-wear" process. The reliability of the measurements has been investigated and confirmed. The standard deviation of the *Ssk* values of 8 repeat measurements on the same area is only 0.0003, much smaller than the wear induced *Ssk* change, 0.49. Moreover, the relationship between *Ssk* and the material volume loss is simulated by a wear model which assumes the removal of the top of asperities of worn surface during the wear test, based on which the volume loss of about 2.5×10^7 nm^3 per measured surface area of 5×5 µm^2 is estimated.

However, areal S-parameters mentioned above cannot directly reveal the tribological behaviour and wear phenomena of the surface, which should be better interpreted by surface features such as local contact spots, the distribution of real areas of contact between the rough surfaces, mean curvature radius of asperities, and root-mean-square (*Rq*) of asperity peaks. To overcome this limitation, other surface characteristics such as bearing projected area, bearing surface area and bearing material volume were proposed and investigated. The bearing projected area curve is the cumulative distribution of areas of material, intersected by a virtual cutting plane along the height range of surface structures. It has a strong relation to the important functional property of the surface such as the real contact area. In addition, the bearing surface area curve is calculated as the sum of surface areas which exceed the virtual cutting planes. The curve is valuable in tribology for calculating the adhesion interaction of rough surfaces. The bearing material volume curve is calculated as the sum of material volumes which exceed the virtual cutting planes. The curve is crucial for estimating the volume loss of surface due to wear. As an example, the bearing material volume curves calculated from a worn and unworn area are shown in Fig. 3. Their difference especially in shape at the "peak zone" is clearly visible.

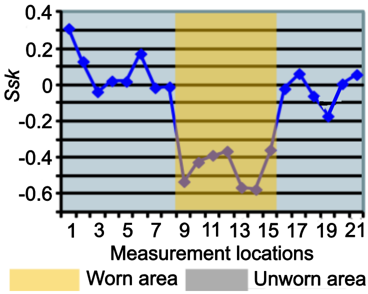

Figure 2. Characterised surface parameter *Ssk* calculated at 21 different locations selected at the worn and unworn surface

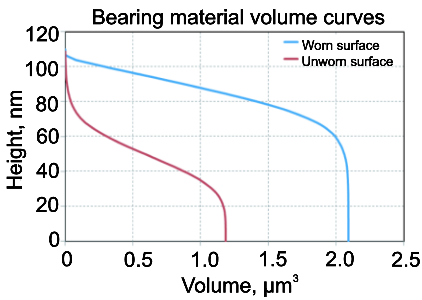

Figure 3. Bearing material volume curves evaluated from the worn and unworn surface area of 5 x 5 μm^2

Further studies will be carried out to quantitatively assess nano wear from these curves. The work was performed within the joint research project "JRP MADES" funded by the European Metrology Research Programme (EMRP).

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