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Nano-mechanical behaviour of the 3rd body generated in dry friction—Feedback effect of the 3rd body and influence of the surrounding environment on the tribology of graphite

Philippe Stempflé [a,*], Jürgen von Stebut [b]

[a] Laboratoire Génie de Production, Ecole Nationale d'Ingénieurs de Tarbes 47, Avenue d'Azereix, BP 1629, 65016 Tarbes Cedex, France
[b] Laboratoire de Science et Génie des Surfaces, Ecole des Mines, Parc de Saurupt, 54042 Nancy Cedex, France

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Abstract

Wear particles often agglomerate into cohesive transfer films (so-called 3rd body) during dry friction. Although described in terms of load carrying capacity and velocity accommodation, the 3rd body finally became a kind of *black box* where the comprehension of the real interactions, which occur in the tribocontact, can be unknown. We study the behaviour and the mechanisms of action of the 3rd body in a graphite/steel dry contact. Our experimental approach simultaneously involves the determination of the mechanical properties, the topography and the microstructure of transfer films in order connecting them to the fundamental tribological parameters (surrounding environment, load carrying capacity, friction and wear). In this paper, a methodology combining nano-indentation and AFM is developed. The results explain the various links between the effect of the environment on 3rd body generation and related friction and wear by taking of account the detachment mechanisms, the agglomeration mechanisms and their evolution during friction.

Keywords: Dry friction; Graphite; 3rd body; Environment; Nano-indentation; Wear debris

1. Introduction

During dry friction, wear particles are detached from surfaces and form more or less cohesive transfer films by agglomeration (so-called 3rd body) [1]. In the contact, these films naturally contribute to the load carrying capacity and/or the velocity accommodation phenomena often associated with the natural protector effect of the 3rd body [2,3]. Although the influence of the 3rd body on the tribological behaviour is frequently observed [4], its mechanisms still remain obscure. Most authors just observe that the significant variations of friction and wear with the experimental conditions are related to the changes in the amount and topography of the 3rd body, without determining their real origins [5]. However, these studies reveal the great difficulties to quantify all of the mechanical properties of the 3rd body required to establish any correlations between the 3rd body behaviour and the evolution of friction and wear.

At this scale, only instrumented nano-indentation [6,7] provides the mechanical properties of films whose thickness rarely exceeds some micrometers [8,9]. In this approach, the normal load, P, applied by the indenter, is plotted versus the corresponding penetration depth, h. The elastic (Young's modulus), the plastic (hardness, yield stress) and/or the fracture properties (toughness) [7,10] of the sample are deduced from the load–displacement plot. However, the determination of the properties of thin and soft tribochemical films still remains very difficult. Actually, many superimposed effects can strongly modify the measured mechanical characteristics (influence of the substrate, piling-up around the indenter, tip rounding) [11–15].

Furthermore, the amount of 3rd body in the contact and its morphological and microstructural evolution must be evaluated.

* Corresponding author. Tel.: +33 5 62 44 27 24; fax: +33 5 62 44 27 08.
E-mail address: philippe.stempfle@enit.fr (P. Stempflé).

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Nomenclature

A_c	contact area below the indenter (μm^2)
a	contact radius
E	Young's modulus (GPa)
E_e	sample modulus (GPa)
E_{eff}	effective modulus of film and indenter combination (GPa)
E_f	film modulus (GPa)
E_i	indenter modulus (1141 GPa)
E_s	substrate modulus (GPa)
E_{sf}	modulus of film–substrate combination (GPa)
F_N	normal force (N)
H	hardness (MPa)
H_f	hardness of the film (MPa)
H_s	hardness of the substrate (MPa)
H_{sf}	hardness of the film–substrate combination (MPa)
h	indentation depth (nm)
h_c	plastic depth (nm), contact depth beneath the indenter
h_e	normalized volume of the 3rd body ($\mu m^3\ \mu m^{-2}$), thickness of the 3rd body (μm)
h_f	depth of residual impression (nm)
h_m	total indentation depth measured from specimen free surface (nm)
L_c	crystallites size (nm)
P	indenter load (mN)
R_a	arithmetic roughness (nm)
R	radius of the spherical indenter (μm)
RH	relative humidity (%)
S	contact stiffness ($mN\ nm^{-1}$)
t	film thickness, thickness of indented agglomerates (nm)
U_d	dissipative component of deformation energy (J)
U_e	elastic component of the deformation energy (J)
U_t	whole deformation energy (J)
v	sliding speed ($mm\ s^{-1}$)
X	area covered by the film (%)
Y_f	yield stress of the film (MPa)
Y_s	yield stress of the substrate (MPa)
ε	size of the nano-particles (nm); constant of the Vickers indenter: 0.75
ν_i	Poisson ratio of the diamond indenter 0.07
ν_e	Poisson ratio of the sample
μ	coefficient of friction

To approach such problem, a simple tribological system consisting of a graphite pin rubbing against polished steel is proposed. This model system leads to discontinuous transfer films easily quantified by 3D profilometry [16] and AFM [17]. As this chosen system is often studied, so it lends itself to a comparison with the literature [25–59].

This paper addresses the mechanical properties of the 3rd body generated under tribological conditions by means of nano-indentation and relocation AFM. Friction properties are tracked as a function of the sliding distance and environment implying modifications of the amount, the morphology and the microstructure of the 3rd body. We will thus address the mechanisms of action of the 3rd body which control the load carrying capacity and the velocity accommodation in a graphite/steel dry contact.

2. Experimental

2.1. Tribometer

The transfer films examined in this study are generated under controlled atmosphere at room temperature (20 °C) by the repeated friction of a compacted graphite pin against the surface of a polished steel disk [18]. The normal load (F_N) and the sliding speed (v) were, respectively, 40.4 N (corresponding to a contact pressure of 2 MPa) and 15 mm s^{-1}. The sliding distance varied from 4.5 to 135 m (corresponding to 50–1500 rotating cycles on a friction track with 15 mm radius). The environment is controlled during the test by the circulation of air or dry nitrogen (1 L min^{-1}) in a confined space of 17 cm^3. The relative humidity (RH) is maintained at 10 and 30% and controlled during overall the test with a saturated aqueous solution of $CaCl_2$ maintained, respectively, at 5 and 20 °C. The friction force is detected by the flexion of two parallel spring blades whose stiffness is 151 kN m^{-1} and measured with a LVDT sensor.

2.2. Samples

The pins are made of powder of natural *Madagascar* graphite with granulometry of 15 μm compacted at slow speed, without any additive, under a pressure of 0.5 GPa. Before the tests, they are preserved safe from moisture in a desiccator at room temperature. The steel discs have 0.38% carbon content. In order to reduce the mechanical interactions caused by the roughness of metal (abrasion), they are polished with a final polishing diamond (3 μm) followed by an OPS polishing ($R_a < 10$ nm) [17,18] and a cleaning with ethanol.

2.3. Morphological characterization of the 3rd body

The morphology of the transfer films is assessed by 3D optical profilometry. The morphological characteristics are the area covered by the film (X in %) and the volume per unit area (h_e in $\mu m^3\ \mu m^{-2}$). These characteristics are estimated by image analysis of the topographic pictures using the *DigitalSurf Mountainsmaps*™ software [18]. In practice, a vertical threshold of 80 nm is performed to reduce the