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Influence of load on the dry frictional performance of alkyl acrylate copolymer elastomers coated with diamond-like carbon films

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In this work, the influence of applied load on the frictional behavior of alkyl acrylate copolymer elastomers coated with diamond-like carbon films is studied at dry conditions. The performance of two coatings with very different microstructure (patched vs. continuous film) is compared with the uncoated substrate. A wide range of applied loads is explored, from 1 mN to 1 N, which is achieved by using a specific tribometer. The variation of 3 orders of magnitude in the applied load leads to a strong variation of the observed frictional phenomena. The different behavior of both samples at various loads is explained using a model that considers two contributions to the friction coefficient, namely, an adhesive and a rubber hysteresis part. The constraints and applicability of such model are critically evaluated. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4934854>]

I. INTRODUCTION

Protection of elastomers with diamond-like carbon (DLC) coatings has several industrial applications, like in windshield wipers,¹ ball bearings,² and others.^{3–5} Such approach allows a reduction of the energetic losses due to friction and improves the lifetime of products. In addition, the use of liquid lubricants, which degrade the elastomer and which are often harmful for the environment, can be reduced or even eliminated. However, the systems' DLC-elastomer show a complex frictional behavior that needs to be understood further in order to optimize their performance depending on the operating conditions.

Figure 1 shows a scheme of the two main contributions to the Coefficient of Friction (CoF) that can be observed in DLC-coated elastomers. The first component (blue arrow) consists of the adhesive interaction between the counterpart and the DLC surface. The second component (red arrows) is due to the energetic losses caused by the hysteresis behavior of the elastomer. Thus, the energy used by the front part of the counterpart to deform the region of the rubber, which opposes the movement, is not fully recovered at the back side of the counterpart. This dissipation of energy, which is schematically indicated by the different thickness of the red arrows in Figure 1, is denoted as a frictional loss.

In addition, to make the situation even more complex, the behavior of the rubber depends strongly on the operating conditions. Rubber can be interpreted as a collection of different “dumping units,” which are characterized by reaction time and intensity.^{6,7} Thus, the velocity and frequency of the relative movement leads to the activation of different “dumping modes.”⁸ If the velocity of operation is too slow for a given unit, it may still have time to deform totally, and it will behave as perfectly elastic. On the contrary, the unit

will not have enough time to deform at all, and it will perform as perfectly stiff. A certain “dumping mode” is activated only when the conditions are adequate, and it will be inactive in any other situation. Studies show that the higher test velocity leads to similar^{9,10} or higher CoFs.^{11,12} However, the confluence of two test parameters (velocity and frequency) make these observations difficult to interpret.

The other external parameter that influences the response of the rubber is the contact force with the counterpart, which is known as “load.” In the earlier publications,^{3,11–17} it has been demonstrated that the load has a strong influence on the frictional behavior of rubber. Only in the case of the publication of Martínez *et al.*,¹¹ a reduction of CoF is observed with increasing load, probably due to the large load employed (10 and 40 N) and a different test configuration. In all the other cases,¹⁸ the CoF increased with load, which has been explained in terms of the relative weighted contributions, adhesive and hysteresis, to the frictional performance.^{15,16} However, the range of variation of load was too small (between 1 and 5 N) to observe any big variations in the frictional mechanism. The aim of this work is to explore a wider range of load (3 decades, from 1 mN to 1 N) during tribotesting, using a specific device to perform this task.

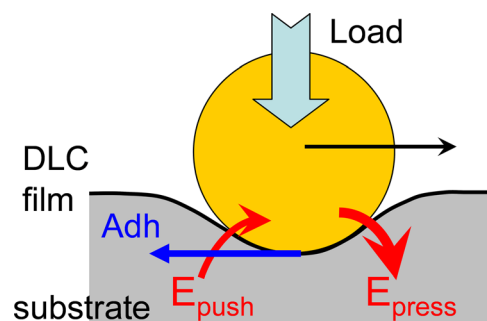


FIG. 1. Scheme of the different contributions to CoF on frictional experiments on DLC coated elastomers.¹⁸

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II. EXPERIMENTAL DETAILS

A. Sample preparation

Before deposition, the alkyl acrylate copolymer (ACM) rubber substrates were cleaned by two subsequent wash procedures in order to achieve good film adhesion. The first treatment comprised five cycles of ultrasonic washing in a 10 vol. % solution of detergent (Superdecontamine 33 from N.V. Intersciences S.A. in Brussels) in demineralized water at 60 °C for 15 min; the second treatment comprised five cycles of ultrasonic washing in boiling demineralized water for 15 min in each cycle.

The DLC thin films were deposited by plasma-assisted chemical vapor deposition (PACVD) apparatus with the magnetron heads powered off.^{12,19,20} A pulsed DC (Advanced Energy) power unit was used as a substrate bias source, operating at 250 kHz with a pulse off time of 500 ns and voltages between 300 V and 600 V. The deposition process comprised two steps. First, the ACM samples were etched for ~30 min in Argon plasma in order to further clean the surface from contaminations, followed by second treatment in ~10 min in a plasma mixture of argon and hydrogen, which was used in order to further improve the adhesion of the subsequent deposited DLC film. In the second treatment, hydrogen was replaced by acetylene and deposition took place. The deposition time was adjusted to obtain DLC coatings with a thickness of ~300 nm.

By tuning the voltages applied during the pretreatment and the depositions, the temperature variation during the film deposition could be controlled. The microstructure of the films depends on the temperature variations of the films, since rubber substrates shrink or expand during film growth, causing the DLC coating to break during film growth.^{19,21} For this study, two coatings with varied microstructures^{15,19,20} were selected (see Figure 2); on the one hand, a

continuous DLC film (see Fig. 2, left), which was deposited at constant temperature during deposition; on the other hand, a patched DLC film (see Figure 2, right), which was deposited with a large and negative temperature variation during film growth. Therefore, in the following, both samples will be referred to as “patched” and “continuous.” An uncoated piece of ACM rubber was also measured on the same experimental conditions for the sake of comparison.

B. Sample characterization

The frictional behavior of DLC coated ACM rubbers has been measured using an Anton Paar pin-on-disk NanotriboMeter (NTR²). The NTR² is a unique instrument allowing tests at such a large range of loads and contact pressures. The NTR² uses an active force feedback to ensure a precise control of normal load under various conditions. Its concept with easily exchangeable double cantilevers allows maintaining excellent force and displacement resolution. The counterbody in all experiments was 100Cr6 stainless steel ball with a diameter of 2 mm, which was cleaned with ethanol before each measurement. The stainless steel ball can be easily fixed on a specially designed support shaft, which also allows attachment of other customer made counter bodies.

All tests were performed at room temperature and a humidity of 40%, and the linear speed and the test length were set to 5 cm/s and 10 000 laps, respectively. Four loads were employed on each specimen (1, 10, 100, and 1000 mN), which were carried out at wear track radii of 4, 6, 8, and 10 mm, respectively. Both the coefficient of friction (CoF) and pin depth were recorded. Here, it is worth mentioning that the sequence of the tests was from low to high load (i.e., 1 mN was measured first, then 10 mN, 100 mN, and finally 1000 mN). This procedure was followed in order to reduce the

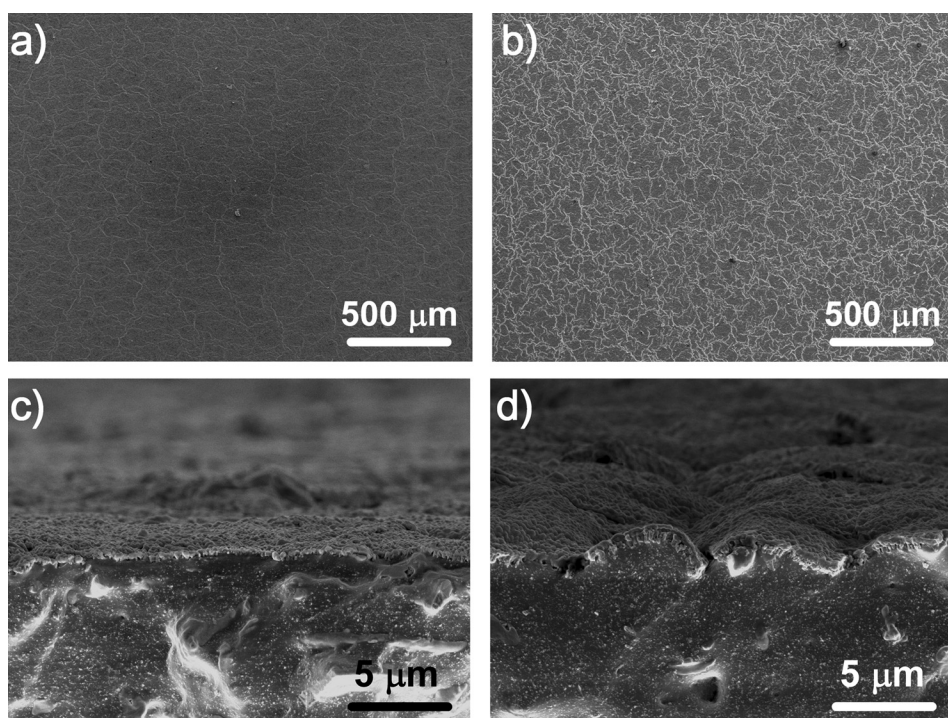


FIG. 2. SEM images of the continuous and patched DLC coatings deposited on ACM rubber.¹⁸ Top: top view ((a) and (b)). Bottom: x-sections ((c) and (d)). Left: continuous film ((a) and (c)). Right: patched film ((b) and (d)).

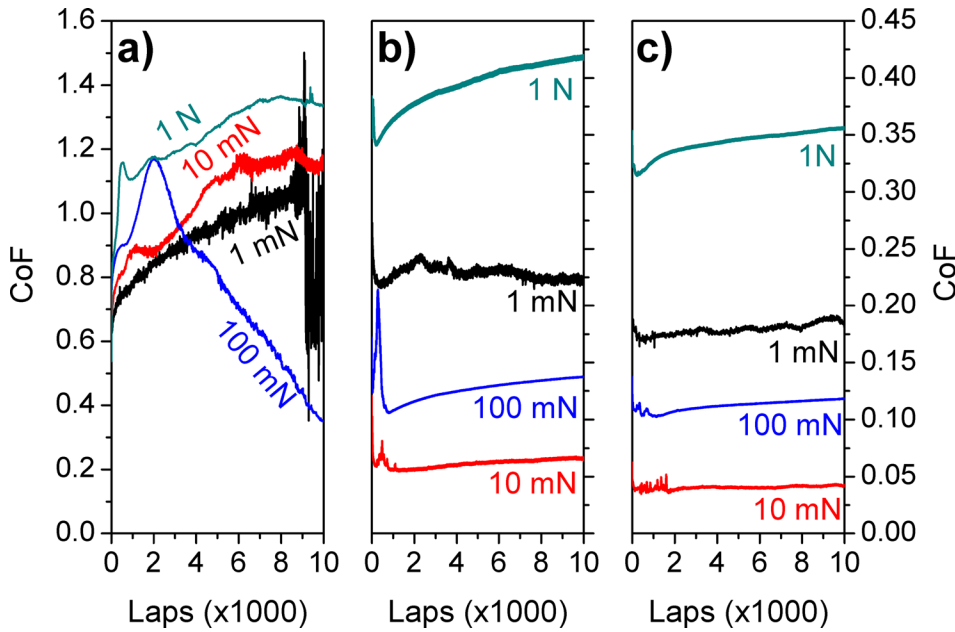


FIG. 3. CoF of the uncoated ACM substrate (a), continuous DLC film (b), and patched DLC film (c) under different loads. The y scale of the graph on (b) is the one of (c).

influence of a previous test (e.g., substrate deformation) in the results of the subsequent measurement.

III. RESULTS AND DISCUSSION

Figures 3 and 4 summarize the results of CoF and pin depth obtained in the three samples under study: uncoated ACM, ACM coated with a continuous DLC film, and ACM coated with a patched DLC film. Three main observations can be made from these figures.

The first observation deals with the frictional behavior of the different specimens. Therefore, the CoFs can be written as

$$CoF(\text{uncoated ACM}) \gg CoF(\text{continuous DLC}) > CoF(\text{patched DLC}). \quad (1)$$

The first part in the inequality in Eq. (1) is because the presence of the DLC on rubber avoids the contact between rubber and steel ball. The adhesion between the rubber and the ball is very high because of the tackiness of the rubber.^{22,23}

In fact, the frictional mechanisms operating on the tribotests of the uncoated rubber is different, which can be inferred from the comparison of the CoF curves in the uncoated rubber (wavy and with wide changes) vs. coated rubber. In addition, the pin depth during tribotest is much larger than that observed in the coated pieces (cf. Figure 4). The differences between the coated and the uncoated specimens are reflected in the wear tracks of both types of coatings. As shown in Figure 5, the damage in the uncoated rubber is much larger than that observed in any of the DLC coated samples. The second inequality in Eq. (1) is related to the microstructure of the DLC films. In dry conditions, films with better

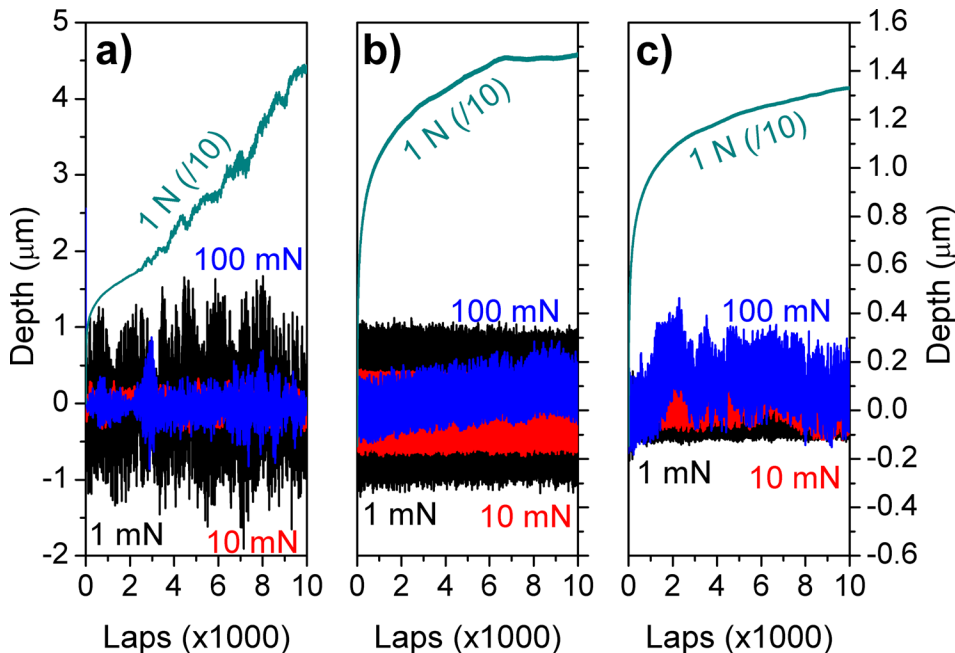


FIG. 4. Depth variation during tribotest of the uncoated ACM substrate (a), continuous DLC film (b), and patched DLC film (c) under different loads. The y scale of the graph on (b) is the one of (c).

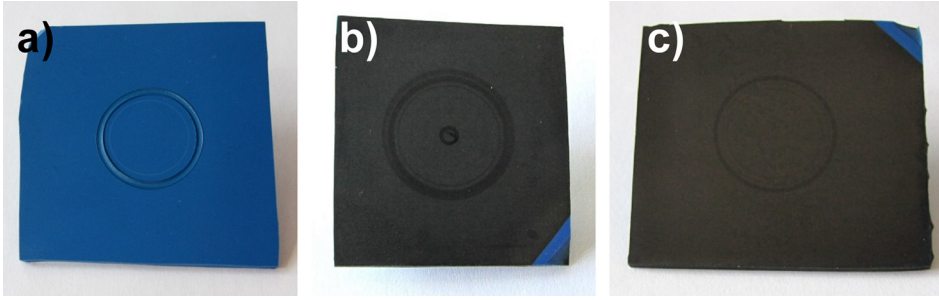


FIG. 5. Images of the wear tracks of the uncoated ACM substrate (a), continuous DLC film (b), and patched DLC film (c) under different loads.

flexibility tend to show lower CoF than coatings with worse flexibility.^{9,19,21} The presence of cracks in a patched structure facilitates the flexibility of the DLC coatings, leading to lower CoFs. Such results reproduce what has been observed earlier in a conventional tribometer.^{12,15,19}

The second observation is about the influence of load in the overall frictional behavior of each individual sample. The uncoated rubber is excluded from this point on, because the tribological mechanism is totally different from the DLC-coated samples. In this latter type of samples, there are two contributions to the CoF, adhesive and rubber hysteresis. The adhesive contribution appears in any frictional circumstance, and it is due to the interaction between surfaces in contact. It can be expressed as follows:¹⁸

$$\mu_{Adh} = S \frac{A_c}{L}, \quad (2)$$

where A_c is the contact area, S is the shear strength between the DLC film and the counterpart, and L is the applied load. In case of elastic contact, the contact area can be expressed as follows:

$$A_c = KL^x, \quad (3)$$

where the load exponent x depends on the geometry of contact. Thus, for a cylinder on a plane $x = 1/2$, while for a ball on a plane (present case) $x = 2/3$.²⁴ Therefore, the relation between friction and load for the adhesive contribution can be written as

$$\mu_{Adh} = K_{Adh}L^a, \quad (4)$$

where $-1 < a < 0$.

The second contribution to friction is due to the hysteresis of the rubber, which is a consequence of the viscoelastic behavior of the rubber during the tribotest. This causes the front and back parts of the contact area to have non-equal contributions to the CoF, and thus they do not cancel each other (which is what happens in case of a perfectly elastic response, i.e., without viscous component). This contribution is difficult to calculate, but it can be written as¹⁸

$$\mu_{Hyst} = K_{Hyst}L^h, \quad (5)$$

where the exponent h is always positive: $0 < h < 1$. Depending on the model, different values for this parameter are obtained: $z = 1/3$ with an elastic model,⁶ $z = 1/2$ in case of using a mattress approach,⁶ and $z = 2/3$ if a cyclic frictional model is used.^{8,18}

In any case, it can be observed that both the contributions to friction show an opposite behavior with load. When load increases, the adhesive contribution decreases, while the hysteresis contribution increases. Therefore, both contributions can be separated by fitting the values of CoF using an equation that combines Eqs. (4) and (5) as

$$\mu = \mu_{Adh} + \mu_{Hyst} = K_{Adh}L^a + K_{Hyst}L^h. \quad (6)$$

The third observation is about the shape of each CoF curve for the DLC-coated rubber pieces. It can be seen that all CoF curves show a growing trend, whose amplitude increases depending on the load. Thus, at a load of 1 mN, the CoF is barely constant, while at 1 N a great CoF variation can be observed (cf. Figure 3). The same trend is seen for the penetration depth of the ball in the rubber (cf. Figure 4). Thus, tests carried out at 1 and 10 mN almost show no variation of ball depth. At 100 mN, a slight increase can be observed (easily seen in the sample coated with a continuous DLC film), and at 1 N a clear increase is appreciated. It can also be observed that the noise in the depth evaluation decreases with the load. This is probably because the depth sensor is less sensitive to sample topography at larger loads. This latter result (larger changes of ball depth are correlated with larger changes in CoF) suggests an adhesive control, since larger ball penetration is connected with larger contact areas. This view is in agreement with conclusions reached in the previous papers;^{8,15,16} on the one hand, simulations show that the contact area increases with the number of laps (i.e., the adhesive contribution increases). In parallel, the shape of the contact area changes from asymmetric (larger in the front than in the back) to more symmetric (similar in the front than in the back), which leads to a reduction of the hysteresis contribution during a tribotest.⁸ On the other hand, different tribotests were carried out under constant experimental conditions, only varying the shear strength of the samples (S in Eq. (2)) by adding lubricants and/or changing the nature of the counterpart.¹⁵ On the contrary, the hysteresis contribution was kept unchanged. The strong variation observed on the frictional behavior indicated that the shape of the frictional curves is controlled by the adhesive contributions to the CoF.

In our previous papers, the evolution of the CoF with the number of laps has been correlated with an exponential behavior.^{7,8,16} This is because the deformation of the rubber in a given pass is not fully recovered during the next lap, and therefore the contact area is slightly larger in the following pass. This behavior is controlled by the test conditions

(velocity and frequency) and the viscoelastic properties of the rubber.⁸ For this particular case, more than one damping unit of the rubber is active, and the frictional behavior during a tribotest can be described by the following equation:

$$\mu = \mu_{\infty} - \Delta\mu_1 \exp\left(-\frac{l}{K_1}\right) - \Delta\mu_2 \exp\left(-\frac{l}{K_2}\right), \quad (7)$$

where l is the number of laps, μ_{∞} is the CoF when the equilibrium condition is reached, K_1 and K_2 represent the response rate of both the damping units, and $\Delta\mu_1$ and $\Delta\mu_2$ represent the “frictional intensity” of both the damping units.

To consider the whole picture, Eqs. (6) and (7) should be used together, so as to explain the frictional behavior. Both expressions just represent separate views of the same phenomenon from the perspective of the “influence of load” and “influence of number of laps,” respectively. As a result, the behavior of CoF can be expressed in two expressions derived from Eqs. (6) and (7)

$$\mu = \mu(l, L) = \mu_{\infty}(L) - \Delta\mu_1(L) \exp\left(-\frac{l}{K_1}\right) - \Delta\mu_2(L) \exp\left(-\frac{l}{K_2}\right), \quad (8a)$$

$$\mu = \mu(l, L) = K_{Adh}(l)L^a + K_{Hyst}(l)L^h, \quad (8b)$$

which means that μ_{∞} , $\Delta\mu_1$, and $\Delta\mu_2$ are the constant for a given tribotest (i.e., do not vary with the lap), but depend on the load. Likewise, K_{Adh} and K_{Hyst} are the constant, i.e., independent of the load, but depend on the lap. The exponents a and h depend uniquely on the geometry of the system, while K_1 and K_2 depend only on the mechanical performance of the elastomeric substrate. As a result, in order to make a proper data analysis, we have fitted the frictional data of both samples to Eq. (8) at the same time. The fitting to Eq. (8a) is performed while keeping the values of K_1 and K_2 equal in all curves. In these fittings, the tests performed at 1 mN are not considered, since the noise and oscillations are too large to detect any underlying exponential trend. In addition, the first laps are excluded because of the presence of some instabilities at the beginning of the tests. All the CoF curves have been re-scaled prior to the fitting in order to ensure that all the curves have the same weight.

The fitting to Eq. (8b) is performed on both samples at the same time as fitting to Eq. (8a). This procedure is carried out only at some selected laps, since we observed that just a few values of l are enough to introduce the effect of Eq. (8b) in the resulting parameters. We have selected the values of $l = 3000$ and $l = 9000$, because the experimental CoF is reliable at these laps in all the datasets. We have also included the values of $l = 0$ and $l \rightarrow \infty$ (i.e., the beginning and the steady state of the tribotests). In these points, the CoF corresponds to $\mu_0 = \mu_{\infty} - \Delta\mu_1 - \Delta\mu_2$ and μ_{∞} in Eq. (8a), respectively. The inclusion of these extreme values improves the quality and stability of the results obtained. The values of the exponents a and h are kept constant in all cases.

It is worth mentioning that, for the tribotest performed at 1 mN, the average value of CoF was used regardless of the

lap under consideration. The other three points used to fit to Eq. (8b) (i.e., CoFs at loads of 10, 100, and 1000 mN) were taken from the fittings of the corresponding curves to Eq. (8a) instead using the experimental values directly. This approach is preferred to improve the consistency of the fittings, particularly at $l = 0$ (the experimental CoFs are not stable) and at $l \rightarrow \infty$ (none of the experiments reaches the steady state condition).

The results of the fitting procedure are displayed in Figure 6 and in overall 3D plots in Figure 7. The fitting parameters are listed in Tables I and II. In general, the fittings to Eqs. (8) reproduce successfully the results observed experimentally. The fittings of CoF of both samples to Eq. (8a) are displayed in Figures 6(a) and 6(b). The fitting lines (in red) overlap the experimental results (in black), except at the beginning of the tribotest, where some oscillations are registered. In all the cases, the same values of K_1 and K_2 can be used, which differ in an order of magnitude (see Table I). This means that there are 2 damping factors activated in these test conditions, one of them faster (mode 1, lower K) and the other slower (mode 2, higher K). The frictional influence of the mode 2 is always larger than the mode 1, since $\Delta\mu_2 > \Delta\mu_1$ in all cases. These fitting parameters also contain information about the variation of the adhesive component to CoF over the laps, which is a consequence of the increase in contact area during the tribotests (see Eq. (2)). In that regard, the values of $\Delta\mu_1$ and $\Delta\mu_2$ increase with load, as expected for larger variations of the contact area. Finally, the continuous DLC film shows a larger CoF than the patched DLC film under all conditions, which is represented by lower values of $\Delta\mu_1$ and $\Delta\mu_2$ for the patched film. This could be explained in terms of a lower shear strength in Eq. (2).

Figures 6(c) and 6(d) show the evolution of CoF as a function of load for both samples. Only examples at 3000 and 9000 laps are shown, although similar plots are obtained for any lap from the beginning to the steady state (in fact, the fittings at lap 0 are included in Figure 7). It can be seen that the adhesive components (blue lines) dominate at low loads, and then the hysteresis contributions (green lines) take over

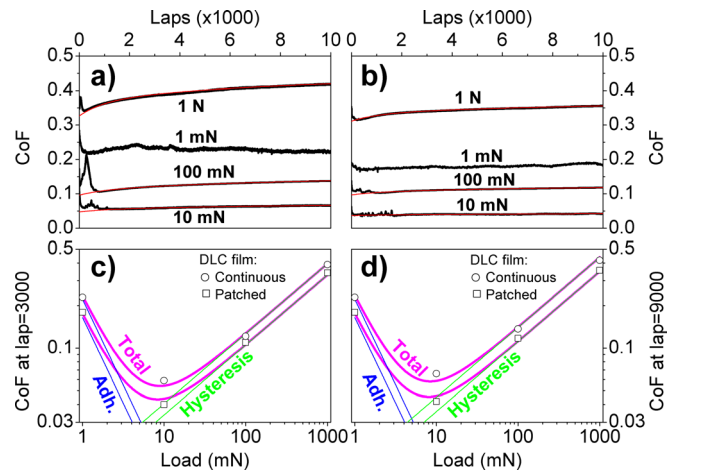


FIG. 6. Fittings of the CoF data to Eqs. (8). Top: fittings to Eq. (8a) of continuous DLC film (a) and patched DLC film (b). Bottom: fittings to Eq. (8b) at lap = 3000 (c) and lap = 9000 (d).

geometrical constraints should be the same. We can recast Eq. (8b) considering the coating and the substrate as

$$\mu(l, L)^C = K_{Adh}^C(l)L^a + K_{Hyst}^{ACM}(l)L^h, \quad (9a)$$

$$\mu(l, L)^P = K_{Adh}^P(l)L^a + K_{Hyst}^{ACM}(l)L^h. \quad (9b)$$

In these expressions, the superscripts *ACM*, *C*, and *P* represent the ACM rubber, continuous films, and patched films. For the same values of load *L* and lap *l*, we can calculate the difference. As explained earlier, since the hysteresis contributions only depend on the substrate, they will annihilate. In other words, whatever the hysteresis behavior of the rubber would be, it should be the same for both samples at the same load. Therefore, we arrive at

$$\mu(l, L)^C - \mu(l, L)^P = (K_{Adh}^C(l) - K_{Adh}^P(l))L^a. \quad (10)$$

The meaning of Eq. (10) is relevant. If we focus on a particular lap, the variation of the difference between the CoF of both samples scales with the load. Since the value of the exponent *a* is negative, this would mean that the difference between CoF of both samples decreases with increasing load. However, this is not the experimental observation in Figures 3 and 6. As a consequence, the attempt to fit the experimental data including this additional constraint failed. The reason for the failure of the model is that apparently the equations used are too simple to capture the full complexity of the phenomenon. This is true, in particular, for Eq. (4). On the one hand, the assumption that K_{Adh} is invariant with load is based on the assumption that the shear strength *S* does not depend on load (Eqs. (2)–(4)), which could be a too strong assumption for a range of loads that covers three orders of magnitude. On the other hand, Eq. (4) does not clearly include the variation of contact area with the number of laps. Therefore, additional development of models is needed, particularly for the adhesive frictional behavior with load.

IV. CONCLUSIONS

The influence of load on the frictional performance of DLC-coated elastomers has been studied. To do so, one uncoated and two coated ACM rubbers with different DLC films (continuous and patched film) have been tribo-tested at four different loads on a range of three decades (from 1 mN to 1000 mN). Four main observations are made: (i) the CoF of the uncoated rubber is much larger than the coated rubbers due to its high tackiness (and consequent wear) in comparison with the DLC films; (ii) the patched DLC coating shows lower CoF than continuous film, which is attributed to its better flexibility; (iii) the frictional behavior with load shows a minimum at ca. 10 mN, which is a consequence of the

tradeoff between adhesive and hysteresis contributions to friction; (iv) CoF increases with the number of laps in each tribotest, which is interpreted in terms of increase in the adhesive contribution due to a increasing contact area originated by the viscoelastic response of the substrate. These latter observations can be explained semi-quantitatively by using simple 2 fitting equations. Nevertheless, the model does not explain all details in the observations because of the complexity and hence additional research is needed.

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