EUROPHYSICS LETTERS 15 March 2005

Europhys. Lett., **69** (6), pp. 971–977 (2005)
DOI: 10.1209/epl/i2004-10451-7

Sliding friction between an elastomer network and a grafted polymer layer: The role of cooperative effects

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received 7 September 2004; accepted in final form 24 January 2005
published online 23 February 2005

PACS. 61.41.+e – Polymers, elastomers, and plastics.
PACS. 68.08.-p – Liquid-solid interfaces.

Abstract. – We study the friction between a flat solid surface where polymer chains have been end-grafted and a cross-linked elastomer at low sliding velocity. The contribution of isolated grafted chains' penetration in the sliding elastomer has been early identified as a weakly velocity-dependent pull-out force. Recent experiments have shown that the interactions between the grafted chains at high grafting density modify the friction force by grafted chain. We develop here a simple model that takes into account those interactions and gives a limit grafting density σ_l beyond which the friction no longer increases with the grafting density, in good agreement with the experimental data.

Introduction. – The interface between a solid surface and a crosslinked elastomer network can be strengthened by the addition of chains that are tethered by one end to the solid surface [1]. As a crack grows along the interface, these coupling chains are progressively pulled out from the elastomer leading to significant energy dissipation [2,3]. The presence of end-tethered chains plays also an important role in friction [2–5]. Very recently, Bureau and Léger [6] studied the friction of a poly(dimethylsiloxane) (PDMS) elastomer network sliding, at low velocity, on a substrate on which PDMS chains are end-tethered and clearly evidenced the contribution to friction of the pull-out mechanism of chain-ends that penetrate into the network. This study, while confirming semi-quantitatively the picture of arm retraction relaxation of the grafted chains proposed by Rubinstein *et al.* [4,5], also reveals the unexpected feature that the friction stress, after increasing with the grafting density of tethered chains, reaches a plateau. In this letter we proposed a simple model, based on the role of cooperative effects, that is able to explain this result. In the whole paper we will consider a cross-linked elastomer of reticulation number P (the mesh size is $\lambda_0 = aP^{\frac{1}{2}}$, where a is the monomer size), in contact with a flat neutral surface with N-mer grafted chains ($N > P$) of the same chemical constitution. The starting point of our study is the description of the partial penetration of a single grafted chain in a static rubber, made by O'Connor and McLeish [7]. They considered the case where the end of a grafted chain penetrates the elastomer at a distance d from the

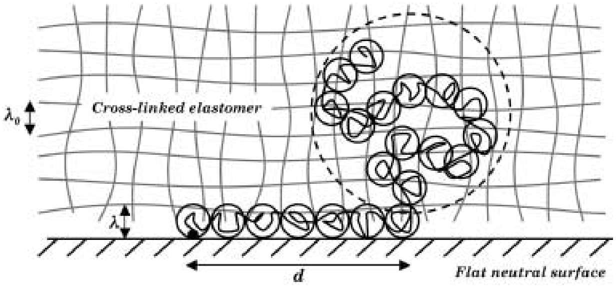

Fig. 1 – Grafted chain that has partially entered the elastomer (λ_0 is the distance between cross-links, λ the slab width, and d is the stretching length of the n_{eq} confined monomers).

grafting point (see fig. 1), leading to a partial penetration on only m monomers. The remaining n monomers ($n = N - m$) are assumed to be simply confined in a slab (of width λ) which should correspond to the first mesh of the elastomer (λ of the order of λ_0).

As the swelling energy of the elastomer can easily be shown to be negligible for a single chain (and even for higher grafting densities [8], see the last section), the m monomers "feel" like in a melt of longer chains, and the free energy of the chain only contains the stretching and the confinement energies of the n monomers [9]:

$$\frac{F(n)}{kT} \simeq \frac{3}{2}\frac{d^2}{a^2 n} + \frac{3}{2}\frac{a^2 n}{\lambda^2} \tag{1}$$

when $d > \lambda$. Minimizing this expression with respect to n, we get the equilibrium value of n: $n_{\text{eq}} = \lambda d/a^2$ if d is inferior to $d_{\max} = a^2 N/\lambda$, and the minimum free energy $F_{\text{in}}/kT = 3d/\lambda$. The n_{eq} monomers constitute a stretched string of blobs of size λ (see figs. 1 and 2a), which exerts a strong horizontal force $f_0 = 3kT/\lambda$ on the elastomer. If $d < \lambda$, the stretching force is not horizontal, and its projection on the horizontal axis is $f \simeq 3kTd/\lambda^2$. This partial penetration state is metastable as long as $d \geq 0$, but its lifetime is very long, for the chain has to go through a high-energy state in order to reach another state of smaller d. In this high-energy state the whole chain is confined in the slab, it forms a string of flat blobs of size $l = a^2N/d$ if $d > aN^{\frac{1}{2}}$, and a blob of size $aN^{\frac{1}{2}}$ otherwise (see fig. 2b), and the free energy is $F_{\text{out}} = F(N)$. Then, the time it takes for the chains to relax to another state is proportional to $\exp[(F_{\text{out}} - F_{\text{in}})/kT]$.

When the elastomer slides on the grafted surface, the chain cannot relax to the state where $d = 0$. The competition between the relaxation and the pull-out due to the elastomer sliding is

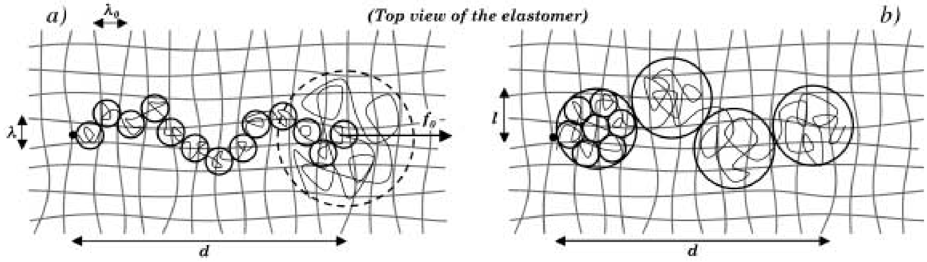

Fig. 2 – a) Top view of a grafted chain that has partially entered the elastomer. b) The same chain confined in the slab when relaxing from the stretched conformation a).

thus key to the understanding of how a grafted chain can enhance friction. In the next section we will reconsider the calculation of the relaxation time for a given d, initiated in [7] and completed in [4]. Then, we will see how a sliding velocity $\boldsymbol{v}$ imposes a mean extension d and a pull-out friction force $\boldsymbol{f}$. In the following sections we will consider interactions between several grafted chains, and show how it can modify the average friction force by grafted chain. Lastly, we will compare our results with recent experiments carried out by Bureau and Léger [6].

Relaxation time. – When the chain partially penetrates the elastomer and has n monomers in the slab, the difference between its free energy and the minimum free energy is $(F(n)-F_{\mathrm{in}})/kT = 3a^2(n-n_{\mathrm{eq}})^2/(2n\lambda^2)$. Then, setting $l = a^2 n/\lambda$, we can consider the chain free end as a random walker diffusing in the potential $U(l) = 3(l-d)^2/(2l\lambda)$ $(0 < l < d_{\max})$ with a diffusion coefficient $D_{\mathrm{eff}}(l)$. The relaxation time we are looking for is the mean first passage time at $l = d_{\max}$ (that is when the chain retracts entirely in the slab, $n = N$), from the initial position $l = d_{\max} - \lambda_0$ (that is the chain just penetrates the elastomer on a mesh size, $n = N - P$). This mean first passage time τ_{in} can be shown [10, 11] to be

$$\tau_{\mathrm{in}}(d) = \int_{d_{\max}-\lambda_0}^{d_{\max}} \mathrm{d}x \exp[U(x)] \int_0^x \mathrm{d}x' \frac{e^{-U(x')}}{D_{\mathrm{eff}}(x')} \,. \tag{2}$$

The inner integral is dominated by the region near $x' = d$ and can be approximated by $(D_{\mathrm{eff}}(d)(2U''(d)/\pi)^{\frac{1}{2}})^{-1}$, where $U''(d) = 3/\lambda d$, and $D_{\mathrm{eff}}(d) = 2a^2/\tau_0 N$ (τ_0 is the monomer relaxation time) if $\lambda = \lambda_0/2$, which we will assume hereafter. The outer integral can be approximated by $\lambda_0 \exp[U(d_{\max})]$, as $(U'(d_{\max}))^{-1} > \lambda_0$. Then, the mean relaxation time at a given d is

$$\tau_{\mathrm{in}}(d) \simeq \begin{cases} \tau_0 N \frac{\lambda^2}{a^2} \sqrt{\frac{\pi}{6}} \exp\left[\frac{3}{2}\frac{(d_{\max}-\lambda)^2}{a^2 N}\right], & \text{if} \quad d < \lambda, \\ \tau_0 N \frac{\lambda^2}{a^2} \sqrt{\frac{\pi d}{6\lambda}} \exp\left[\frac{3}{2}\frac{(d_{\max}-d)^2}{a^2 N}\right], & \text{if} \quad \lambda < d < d_{\max} - \frac{1}{2}\sqrt{\frac{\pi}{6}} a N^{\frac{1}{2}}, \\ \tau_0 N \frac{\lambda}{a^2} \left(\frac{1}{2}\sqrt{\frac{\pi}{6}} a N^{\frac{1}{2}} + d_{\max} - d\right), & \text{if} \quad d_{\max} - \frac{1}{2}\sqrt{\frac{\pi}{6}} a N^{\frac{1}{2}} < d < d_{\max} \,. \end{cases} \tag{3}$$

Notice that τ_{in} does not vanish at $d = d_{\max}$, as the chain end can still explore the inside of the elastomer on the curvilinear distance $\sqrt{\pi/24} a N^{\frac{1}{2}}$. This calculation of the relaxation time notably differs from the one of Rubinstein *et al.* [4, 5], who overestimated τ_{in} by a factor $(N/P)^{\frac{1}{2}}$ to N/P for $d < d_{\max} - \sqrt{\pi/24} a N^{\frac{1}{2}}$, and underestimated it for larger values of d.

Another interesting time is the mean first-passage time at $n = N - P$ from the initial position $n = N$, which is the time τ_{out} the chain spends entirely in the slab before to hop inside the elastomer. It corresponds to the diffusion time of the chain free end through the first mesh of the elastomer: $\tau_{\mathrm{out}} = \tau_0 P^2$. During this short time, the free end is driven back toward the grafting point as a result of the elastic shrink of the chain on the mean distance $6d\tau_{\mathrm{out}}/\tau_0 N^2$, but it also diffuses horizontally on the distance λ_0 which is much bigger than this mean distance. Therefore, diffusion dominates.

Sliding friction. – If the elastomer slides on the grafted surface at the velocity $\boldsymbol{v}$, a fully penetrating chain would be pulled out of the elastomer and stretched in the sliding direction. But if $v\tau_{\mathrm{in}} < d_{\max}$, the chain will spontaneously relax and hop out of the elastomer before its complete stretching. Then, a permanent regime will settle, corresponding to cycles of hopping-in and -out, fixing an average value for d. Every hopping-out, the free end of the chain diffuses in the slab on the distance λ_0, and every hopping-in this free end is shifted of $v\tau_{\mathrm{in}}(d)$ in the sliding direction. Then, the smallest possible value $d_{\min}$ for d is given by the relation $v\tau_{\rm in}(d_{\rm min}) = \lambda_0$. All the values between $d_{\rm min}$ and $d_{\rm max}$ can be explored by diffusion, but as $\tau_{\rm in}(d)$ is a strongly decreasing function of d, the time-averaged value of d is approximately $d_{\rm min} - \lambda_0(v\tau'_{\rm in}(d_{\rm min}))^{-1}$, with the standard deviation $\delta d = -\lambda_0(v\tau'_{\rm in}(d_{\rm min}))^{-1}$. The value of $\delta d \simeq a^2N/3(d_{\rm max} - d_{\rm min})$ varies from $\lambda_0/6$ when $d_{\rm min} = \lambda_0$, to $aN^{\frac{1}{2}}/\sqrt{3}$ when $d_{\rm min} = d_{\rm max} - aN^{\frac{1}{2}}/\sqrt{3}$, and is thus always much smaller than $d_{\rm min}$. Then, we can approximate the average value of d by $d_{\rm min}$, which gives

$$v\tau_{\rm in}(d) \simeq \lambda_0\,. \tag{4}$$

Equation (4) has already been proposed by Rubinstein *et al.* [4], but with different predictions for $\tau_{\rm in}(d)$ (see eq. (3)). Now that we get d as a function of v, we can express the force f applied by a grafted chain on the elastomer for different velocities. Four velocity regimes can be brought out of this result:

– If $v < v_1 \simeq \lambda_0 e^{-\frac{3}{2}\frac{a^2N}{\lambda^2}}/(\tau_0 NP)$, then $d < \lambda$, the chain is almost fully relaxed, and $f \simeq f_0\frac{v}{v_1}$. For we consider cases where $a^2N \gg \lambda^2$, v_1 is extremely small.

– If $v_1 < v < v_2 \simeq a/(\tau_0 N^{\frac{3}{2}})$, then the chain is partially stretched, and as $\lambda < d < d_{\rm max}$, $f \simeq f_0$. In addition to this stretching force we shall take into account the Rouse friction of the elastomer on the whole chain([1]), which is approximately $kT\tau_0 Nv/a^2$, much smaller than f_0 in this regime and the latter one. We note $v'_1 \simeq v_2 e^{-\frac{3}{2}\frac{a^2N}{\lambda^2}}$ the velocity for which $d = aN^{\frac{1}{2}}$. Below v'_1 the chain relaxes forming a flat blob of size $aN^{\frac{1}{2}}$. The velocities v_1 and v'_1 are extremely small. When $v < v_2/2$, as $d < d_{\rm max} - \frac{1}{2}\sqrt{\frac{\pi}{6}}aN^{\frac{1}{2}}$, d is given by the relation

$$\frac{v}{v_2} \simeq \frac{1}{2}\sqrt{\frac{d_{\rm max}}{d}}\,e^{-\frac{3}{2}\frac{a^2N}{\lambda^2}\left(1-\frac{d}{d_{\rm max}}\right)^2}. \tag{5}$$

– If $v_2 < v < v_3 = a/(\tau_0 P^{\frac{3}{2}})$, then the chain is pulled out faster than it can relax, $d = d_{\rm max}$, and the friction force on the chain is $f_0 + kT\tau_0 Nv/a^2$, where $kT\tau_0 Nv/a^2$ is no longer much smaller than f_0.

– Finally, if $v_3 < v$, the Rouse friction dominates, and $d > d_{\rm max}$.

The existence of these regimes has been depicted by Rubinstein *et al.*, but their misevaluation of $\tau_{\rm in}$ led them to underestimate v_1 and overestimate v_2. Notice that the friction of the elastomer on the substrate is to be added to the friction of the chains, and that it could be partially screened out by the grafted layer [2].

At this point it is important to notice that nothing prevents the chain orientation from fluctuating around the sliding direction([2]). If the angle between the chain orientation and $\boldsymbol{v}$ is θ, one should replace v by $v\cos\theta$ in eq. (4), which really changes d only for angles close to $\pi/2$ or $-\pi/2$. These fluctuations lower the effective pull-out friction force by grafted chains of a factor $2/\pi$. Considering more than one grafted chain, one can foresee that a more important consequence of these fluctuations is that it allows the chains to entangle one with the other.

Cooperative effects at higher grafting densities. – If two chains are grafted at a distance D smaller than d, they can cross, and possibly entangle one with the other. Two situations can be distinguished. First, if the distance $D_\perp$ between the two grafting points perpendicularly to $\boldsymbol{v}$ (see fig. 3a) is bigger than the size l of the flat blobs, the entanglement can only form at the end of one of the chains, and will untie within approximately $(l/\lambda_0)^4$ hopping-in and -out cycles

([1])Even if the part of the chain that is in the elastomer is relaxed, it is pulled out at the velocity $v\lambda/\lambda_0$ on average.

([2])This was not possible in the situation Rubinstein *et al.* studied, as the chain was dragged inside the elastomer.

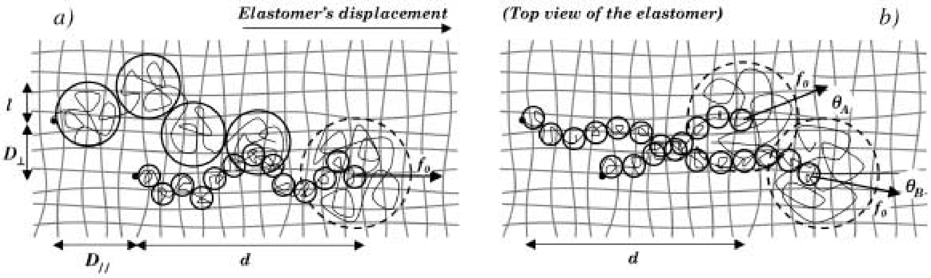

Fig. 3 – Entanglement process between two grafted chains: a) The free end of the relaxed chain B passes over and underneath the stretched part of the chain A. b) The chain B recovers its plume conformation.

(the time for the end of the chain to diffuse on the size l), whereas it took $(D_\perp/\lambda_0)^4$ cycles to form. Thus, that kind of entanglement is fleeting and irrelevant. If $D_\perp$ is smaller than l, then an entanglement can form relatively quickly and let the chains recover the same orientation as $\boldsymbol{v}$. Then, the stretching forces of the two chains equilibrate, and the entanglement slides toward the middle of the chains (see fig. 3b). The effective friction force applied on the elastomer by those two chains is then $f_A + f_B = f_0(\cos\theta_A + \cos\theta_B) < 2f_0$. This entanglement can untie only if one of the chains free end reaches it. But as d cannot be smaller than $d_{\min}$, this kind of entanglement has a quasi-infinite lifetime if the distance $D_\parallel$ between the two grafting points parallel to $\boldsymbol{v}$ is smaller than $d_{\min}$. Therefore, we can assume that a chain entangle with all the chains that are grafted within the area $2l \times d_{\min} \simeq 2ld = 2a^2N$ if $v_1' < v < v_2$. Then, if σ is the grafting density scaled by a^2, we can roughly evaluate the average value of $\cos\theta$ as

$$\langle\cos\theta\rangle \simeq \frac{\frac{d}{2\sigma N}}{\sqrt{\left(\frac{d}{2\sigma N}\right)^2 + \left(\frac{l}{2}\right)^2}} = \frac{1}{\sqrt{1+\sigma^2\left(\frac{aN}{d}\right)^4}}\,. \tag{6}$$

This gives the pull-out friction by surface unit as a function of σ:

$$\Sigma \simeq \frac{\sigma f_0}{a^2\sqrt{1+\sigma^2\left(\frac{aN}{d}\right)^4}} \simeq \begin{cases} f_0\frac{\sigma}{a^2}\,, & \text{if} \quad \sigma < \left(\frac{d}{aN}\right)^2, \\ f_0\frac{d^2}{a^4N^2}\,, & \text{if} \quad \sigma > \left(\frac{d}{aN}\right)^2. \end{cases} \tag{7}$$

So, this rough model exhibits an interesting feature of elastomer-grafted surface friction: at low grafting densities ($\sigma < \sigma_l \simeq (d/aN)^2$) the friction force by surface unit increases linearly with σ, there is no interaction between grafted chains. At higher grafting densities ($\sigma > \sigma_l$) the friction force by surface unit saturates at the value $\Sigma_l = f_0 d/aN$ (we do not consider here the friction of the elastomer on the substrate). Note that our rough estimate of $\langle\cos\theta\rangle$ only allows us to get an expression of σ_l without the precise numerical prefactor. Nevertheless, $\sigma_l \simeq (d/aN)^2 = a^2/l^2$ is the grafting density beyond which the mean distance between grafted chains is smaller than l, and we can understand that entanglements produce an important orientation disorder beyond this limit.

The general tendencies for σ_l correspond to the four velocity regimes we developed in the previous section. If $v < v_1'$, then $l = aN^{\frac{1}{2}}$ and $\sigma_l = \sigma_{\min} \simeq 1/N$, whereas if $v_2 < v < v_3$, then $l = \lambda$ and $\sigma_l = \sigma_{\max} \simeq 4/P$. When $v > v_3$, the Rouse friction dominates and should not be sensitive to entanglements. Using eq. (5) we can establish the relation between v and σ_l

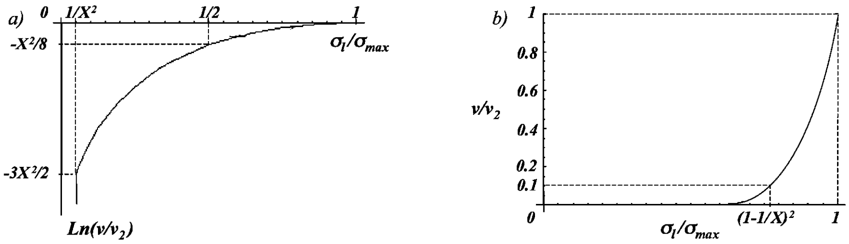

Fig. 4 – Characteristic plots of the evolution of $\sigma_l/\sigma_{\max}$ with v/v_2 ($X^2 = a^2N/\lambda^2$).

between v_1' and $v_2/2$:

$$\frac{v}{v_2} \simeq \frac{1}{2}\left(\frac{\sigma_{\max}}{\sigma_l}\right)^{\frac{1}{4}} e^{-\frac{3}{2}\frac{a^2N}{\lambda^2}\left(1-\left(\frac{\sigma_l}{\sigma_{\max}}\right)^{\frac{1}{2}}\right)^2}. \tag{8}$$

The ratio $X^2 = a^2N/\lambda^2 \sim N/P$ is an important parameter characterizing the system; it gives the range of saturation grafting densities $\sigma_{\max}/\sigma_{\min} = X^2$. The parameter X also drives the way σ_l evolves within $(\sigma_{\min}, \sigma_{\max})$ while v varies from v_1' to v_2 (see fig. 4). Indeed, the range of velocities for which σ_l is bigger than $\sigma_{\max}/2$ is given by $v(1/2)/v_2 \simeq \exp[-X^2/8]$, which represents several decades when $X \gg 1$ (see fig. 4a). Another way to see the role played here by X is to write the saturation grafting density corresponding to $v = v_2/10$: $\sigma_l(1/10)/\sigma_{\max} \simeq (1-1/X)^2$ (all logarithmic factor being ignored, see fig. 4b), which correspond to $d \simeq d_{\max} - N^{\frac{1}{2}}$.

It is interesting to note that $\sigma_{\max}$ and $\Sigma_{\max}$ are independent of the chains length N; $\Sigma_{\max}$ being the maximum friction enhancement one can obtain grafting polymer chains on the flat surface, and $\sigma_{\max}$ the minimum grafting density one should use in order to reach $\Sigma_{\max}$. The grafted-chains length, though, is an important parameter as it fixes the velocity range over which $\sigma_l \sim \sigma_{\max}$. One should use long chains ($a^2N \gg \lambda^2$) in order to have Σ_l close to $\Sigma_{\max}$ at very low velocities. Nevertheless, the maximum grafting density one can experimentally reach is $1/N^{\frac{1}{2}}$, and one can show that beyond $\sigma^* \simeq P^{\frac{1}{10}}N^{-\frac{3}{5}}$ the grafted layer no longer interdigitates with the elastomer [8]; thus the maximum chain length one should use is $N \simeq P^{\frac{11}{6}}$.

Results and experiment comparison. – Comparison with experiments is made harder by the fact that λ is an unknown parameter assumed to be approximately $\lambda_0/2$. Nevertheless, it can give insights into the validity of the model. Using a PDMS elastomer with reticulation number $P = 100$ and a grafted surface of $N = 1540$ [6] and 380 (unpublished studies) PDMS chains, Bureau *et al.* systematically studied $\Sigma = f(\sigma)$ for sliding velocities from 0.3 to 250 μms^{-1}, each time observing a friction force by grafted chain on the order of kT/λ_0 at low grafting densities, and a saturation of the friction at high grafting densities [6].

For $N = 1540$, they studied saturation at $v = 0.3$, 10, and 100 μms^{-1}, while $v_2 \simeq 300\,\mu$ms^{-1}. For $N = 380$, they studied saturation at $v = 10$, 100, and 250 μms^{-1}, while $v_2 \simeq 2000\,\mu$ms^{-1}.

a)

$v(\mu m.s^{-1})$	0.3	10	100
σ_{lexp}	0.025	0.035	0.04
σ_l	0.03	0.04	0.055

b)

$v(\mu m.s^{-1})$	10	100	250
σ_{lexp}	0.015	0.025	0.035
σ_l	0.01	0.02	0.03

Fig. 5 – Comparison of experimental saturation grafting density $\sigma_{l\exp}$ with the present theoretical prediction σ_l. a) $N = 1540$. b) $N = 380$.

As $1/a^2P = 0.04\,\mathrm{nm}^{-2}$, $\sigma_{\max} \simeq 0.08\,\mathrm{nm}^{-2}$ would fit with $\lambda \simeq 0.7\lambda_0$. Then, for $N = 1540$, $X^2 = a^2N/\lambda^2 \simeq 30$, and for $N = 380$, $X^2 \simeq 8$. The saturation grafting densities $\sigma_{l\exp}$ obtained experimentally and the theoretical predictions are gathered together in fig. 5. As we can see, the model reasonably captures those experimental data, even if a slight misevaluation of λ would induce consequent errors on X^2 and $\sigma_l(v)$.

Although more data are needed to confirm the model, we think that it is the best candidate explaining the saturation of the friction: The first other possibility comes from the fact that increasing the grafting density can induce an increase of λ, and a decrease of $f_0 \simeq kT/\lambda$. Indeed, we can estimate that $\lambda \simeq \lambda_0/2 + \sigma n_{\mathrm{eq}}a$. Then, $\sigma f_0 \simeq \sigma kT/(\lambda_0/2 + \sigma n_{\mathrm{eq}}a)$, which would give a saturation grafting density equal to $P^{\frac{1}{2}}/n_{\mathrm{eq}}$. This overestimates σ_l, and would give σ_l as a decreasing function of v and N, which is not the case experimentally. The second possibility comes from the fact that when $\sigma > \sigma^* \simeq P^{\frac{1}{10}}N^{-\frac{3}{5}}$, the grafted chains no longer penetrate the elastomer because the swelling of the elastomer would not be negligible any more. But, again, this overestimates σ_l, and would give σ_l as a decreasing function of N.

Conclusion. – We have described here a model for the sliding friction of an elastomer on a flat grafted surface that allows us to understand the participation of grafted chains on friction at low and high grafting densities. This model describes the pull-out process of the grafted chains and the formation of entanglements between grafted chains at high grafting densities. The general feature that follows from this is a linear relation between the pull-out friction force by surface unit and the surface grafting density when σ is low, and a saturation of the pull-out friction force by surface unit beyond a limit grafting density σ_l, which is an increasing function of v and N. The predictions for σ_l are in good agreement with experimental results of Bureau *et al.* which have been mainly conducted in the range $v < v_2$. For $v_2 < v < v_3$, our model predicts that σ_l simply varies like $1/P$; it would be very interesting to experimentally check this prediction in the future.

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We thank L. BUREAU and L. LÉGER for very interesting discussions and for bringing to our knowledge unpublished results.

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