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Breaking polymer chains by dynamic plowing lithography

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Abstract

The adhesion of poly(methyl methacrylate) (PMMA) and polystyrene (PS) films, whose surface has been previously structured by dynamic plowing lithography (DPL), has been measured by means of force–displacement curves. The different adhesion of modified and unmodified PS leads to the assumption that polymer chains are broken during DPL. After measuring the energy dissipated by the tip during DPL, in order to check that the transferred energy is sufficient to break covalent bonds, the polymer chain scission caused by the lithographic process has been definitely confirmed by size exclusion chromatography measurements of the lithographed films. © 2002 Published by Elsevier Science Ltd.

Keywords: AFM; Nanolithography; Chain scission

1. Introduction

Dynamic lithography modes have been introduced in the past few years in order to overcome the drawbacks and difficulties of *static plowing*, i.e. the scratching of sample surfaces in contact mode [1–6]. Performing contact mode lithography on Langmuir–Blodgett films or on thin polymer films [7–17] presents mainly three problems. First of all only thin films (5–25 nm) on hard substrates can be modified. In addition the acquisition of the topography, following the modification, can be performed only with small forces, and hence with a reduced resolution. If the force is too large (the threshold depends on the polymer and on its thickness), the polymer will be further modified. The only solution is to scan the surface after the modification in a different mode. The third drawback is the torsion of the cantilever during lithography that causes irregularities in the profile of the edges. Because of the torsion the direction of the lithographed lines is limited in a certain range around the axis of the cantilever [18,19].

Due to the numerous problems of contact mode lithography, several researchers have tried to modify this technique and to perform lithography by modulating the force between tip and sample [19–23]. Dynamic plowing lithography (DPL), i.e. the lithography technique in Tapping Mode, in which the force between tip and sample is increased by suddenly increasing the amplitude of the cantilever oscillations, has been found by Klehn and Kunze [24–27]. Tapping mode enables to image soft samples with relatively low forces, i.e. without dragging, and to eliminate the irregularities of the topography of the modified structures due to the torsion of the cantilever.

In previous articles [28–30] we have proved that DPL, unlike other lithography techniques, engenders changes in the density of the lithographed material. The structuring of a polymer surface by means of DPL produces carved surfaces surrounded by large border walls, whose volume is bigger than the volume of the carved out regions. Such a ‘creation’ of volume is due to changes in the density of the polymer, caused by a loosening of the structure of the polymer provoked by the fast indentation of the AFM tip. The analysis of the border walls by means of force–displacement curves has shown that the changes of the physical structure of the polymer are accompanied by changes in its chemical structure.

In the present article we report the analysis of border walls by means of force–displacement curves and we confirm the results of such an analysis through size exclusion chromatography (SEC) measurements.

2. Experimental

The AFM used in the present experiments is made up by a commercial microscope head (Topometrix TMX 2000

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Explorer, Santa Clara, CA), a piezoelectric scanning table for XY scanning, and a Z piezoactuator for Z positioning (P-517.2CL and P-753.11C, respectively, Physik Instrumente GmbH and Co., Waldbronn, Germany). Both the PI table and the PI Z piezoactuator are equipped with integrated capacitive displacement sensors.

The experimental set-up for DPL has been discussed in a previous article [31]. During DPL, the microscope is operated in tapping mode. In tapping mode, the cantilever driven by a dither piezoactuator vibrates near its resonance frequency. The vibration amplitude is kept constant by a feedback loop that changes the distance between the sample surface and the cantilever. The changes of the Z piezo extension are used to reconstruct the topography of the sample. In order to perform a modification of the sample surface, the modulation amplitude given to the dither piezo is suddenly increased (within 100 μs), passing from the value V_r (reading) to the value V_w (writing). Without discussing the complicated response of the cantilever in details [31], when the oscillation amplitude is suddenly increased, the sample is further approached to the tip and the tip indents the sample surface. When the force is large enough, plastic deformations are obtained.

In our experiments, DPL is performed in *scanning mode*, that is a synchronisation of the raster scan mode with the desired pattern, contained in a pixel image called mask, where white and black pixels correspond to V_w and V_r, respectively. Uniformly deep surfaces cannot be carved with uniform white surfaces in the mask, and a pulse train of alternated white and black pixels, producing overlapping holes in the sample surface, has been used in the experiments discussed in this article [28]. The result is shown in Fig. 1, where a single hole (a) and three squares (b), written through overlapping of single holes, can be seen. Each hole inside the square is surrounded by a border wall, like the single hole in Fig. 1(a). These ‘border walls of the single holes’ are partially moved by the tip outside the carved surface. The structure obtained with such masks is a carved surface surrounded by large border walls that are made of the border walls of the single holes. Not all the material of the border walls of the single holes can be carried outside by the tip, and some residues stay inside the square. Only a small portion of the surface of the square, i.e. the left and the bottom side, is free from residues of the border walls of the single holes. For large amplitudes DPL provokes always an increase of the total volume, as it is evident in Fig. 1 for the single hole as well as for the three squares.

The same program has been used to perform lithography, to acquire topography in tapping mode, and to carry out force–displacement curves measurements. The scanning method employed for the acquisition of force–displacement curves is described in details in Refs. [29,32].

The poly(methyl methacrylate) (PMMA) films (thickness about 1 mm) have been prepared by depositing droplets of a solution of 10 g of PMMA (Röhm GmbH, type 7N, $M_\mathrm{w} \cong 120$ kDa) in 15 g of acetone on a glass slide and letting the acetone slowly evaporate within some days. The polystyrene (PS) films (thickness about 1 mm) have been prepared by depositing droplets of a solution of high concentrated PS in cyclohexanone on a glass slide and letting the cyclohexanone slowly evaporate within some days. In the experiments presented in this paper only the glass side of the films has been used.

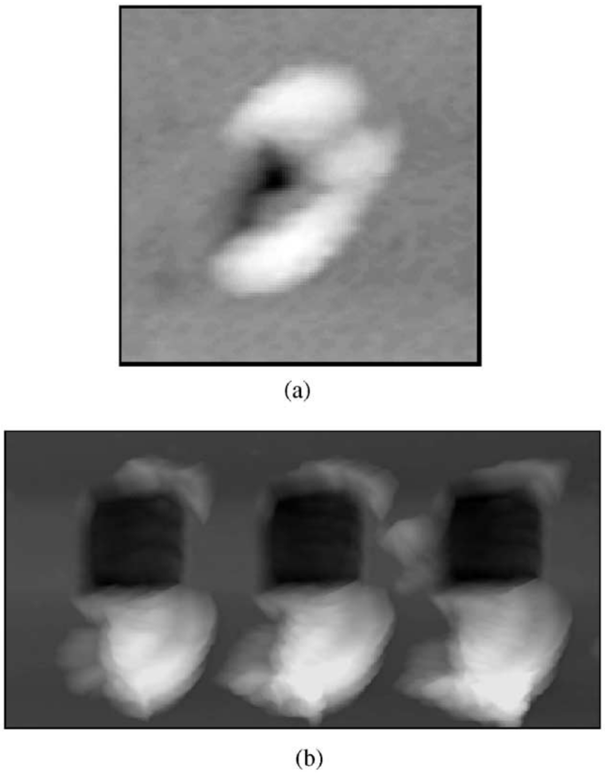

Fig. 1. (a) A single hole carved in PMMA through DPL ($\Delta Z = 100$ nm, $\Delta XY = 700 \times 700$ nm^2). The hole is surrounded by a border wall, called ‘border wall of the single hole’. The volume of the hole is about 2×10^5 nm^3, while the volume of the surrounding border wall is about 25×10^5 nm^3. (b) Three squares carved on PS through overlapping of single holes ($\Delta Z = 970$ nm, $\Delta X = 8$ μm, $\Delta Y = 4$ μm). The squares are partly surrounded by large border walls, resulting from the deposition of the border walls of the single holes. The amplitude used to carve the squares is smaller than in the case of the single hole shown in (a). The volume of the squares is 2.2×10^8, 1.9×10^8, and 1.8×10^8 nm^3; the volume of the surrounding large border walls is 7.3×10^8, 8.3×10^8, and 7.9×10^8 nm^3.

Since our interest was focused on the properties of border walls, we have chosen films whose thickness is very much larger than the performed indentation. In this case, it is not necessary to take into account the effect of the substrate [33] and very large border walls can be obtained.

Commercially available cantilevers (Pointprobe NCL, Nanosensors, Wetzlar-Blankenfeld, Germany) with length $L = 225$ μm, width $W = 38$ μm, thickness $T = 7$ μm, resonance frequency $F = 156$ kHz, and spring constant $k_\mathrm{c} = 30$ N/m were used for DPL, Tapping Mode images, and nanoindentation. V-shaped cantilevers (Park Scientific Instruments, Sunnyvale, CA) with $L = 180$ μm,

$W = 18\ \mu m$, $T = 0.6\ \mu m$, and $k_c = 0.064$ N/m were used for force–displacement measurements in water.

3. Results and discussion

By acquiring force–displacement curves information about the chemical nature of the sample can be gained. Even if it is not possible, in most cases, to determine the chemical composition of the sample, it is possible to highlight differences in some quantities influenced by the chemical composition [34]. The most important quantity is in this case the adhesion or the jump-off-contact force.

Force–displacement curves have been acquired at first on PMMA in air. Sixteen squares, similar to the ones in Fig. 1(b), had been previously written on the sample, with the method already described in Section 1. The side of the squares is 1 μm, and they are 70 ± 10 nm deep. They are surrounded by a border wall of irregular shape, whose volume is much bigger than the volume of the carved square.

The adhesion on unmodified PMMA is lesser than 5×10^{-6} nJ. On the residues of the border walls inside the squares the adhesion is between 4×10^{-5} and 6×10^{-5} nJ. On the tall border walls outside the squares the adhesion is between 1×10^{-4} and 1.4×10^{-4} nJ. The differences in adhesion do not depend on the different chemical composition of the sample. They depend rather on the different stiffness and density of the sample. It has been proved [29, 30] that border walls and residues inside the squares are very much softer and looser than unmodified PMMA. As a consequence, the tip indents them very much deeper than unmodified PMMA. This leads to an increase of the contact area that is proportional to the adhesion, and to the fact that the loose polymer on the surface of the border walls can follow the tip during the withdrawal, increasing again the adhesion. Most of all, the meniscus force exerted by adsorbed water layers exceed the Van der Waals force. So, even if the cross-talk with the density were not present, information about tip and sample surface energies could not be gained through measurements in air [34].

Measurements of the adhesion in deionised water are necessary in order to overcome the problem of meniscus force. However, the measured adhesion of modified and unmodified PMMA in water is the same. This confirms that the cross-talk with the density can be eliminated by acquiring force–displacement curves with smaller maximum force. This is possible only in water, where no meniscus force is present.

The fact that modified and unmodified PMMA have the same adhesion does not exclude chemical modifications of the sample. As a matter of fact, force–displacement curves are not sensible to all changes of the molecular composition of the sample. The most effective change for measurements in water would be a change in the hydrophilicity of the surface.

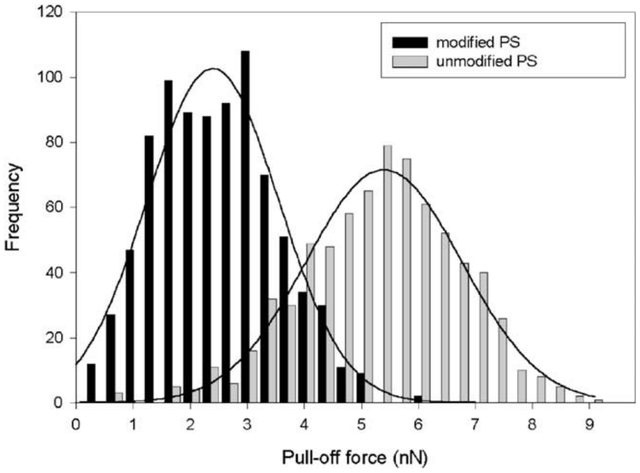

Fig. 2. Histograms of the measured pull-off force on modified (black bars) and unmodified (grey bars) PS, collecting the pull-off force of 1600 force–displacement curves. The mean force is 5.4 nN for unmodified PS and 2.4 nN for modified PS.

In order to have the possibility of measuring an hydrophilicity contrast, the same kind of squares has been written on hydrophobic PS. The obtained structure is essentially the same, except the depth of the squares (170 ± 20 nm). Fig. 2 shows the histogram of the measured pull-off force. It is evident that the adhesion on modified PS in smaller than on unmodified PS. Both histograms have been fitted with a gaussian function. The mean force is 5.4 nN for unmodified PS and 2.4 nN for modified PS.

This difference cannot be due to a cross-talk with the density and the stiffness, because

1. the dimensions of the structures written on PMMA and on PS are essentially the same, and no cross-talk between adhesion and stiffness has been observed in the case of PMMA in water, so there cannot be any cross-talk in the case of PS;
2. if such an effect was present, the adhesion on modified PS, that is softer than unmodified PS, would be larger than on unmodified PS, as already seen in the measurement in air.

The difference in adhesion between modified and unmodified PS can be explained only through a change in the hydrophilicity of the polymer. Changes in the hydrophilicity of the polymer are in turn due to chain scission followed by oxidation. The capability of the AFM tip to break the chains during DPL will be discussed in the following. Let us at first assume that the tip has enough energy to cause such a chain scission. The new end groups oxidize and become hydrophilic. Since PMMA is already hydrophilic, the number of new hydrophilic groups created by DPL does not change considerably the total hydrophilicity of the sample. The adhesion of PMMA to the tip in water, that is already very small, becomes certainly smaller, but the slight difference between the histograms on modified and on unmodified PMMA does not permit to distinguish them. On the other hand, PS is hydrophobic, and the effect of the small number of new hydrophilic groups created by DPL on the adhesion with the tip is very much stronger. This effect is further enhanced by the fact that, when the sample is dipped in water, the new hydrophilic end-groups will dispose themselves in contact with water, i.e. on the surface. This means that on unmodified PS the tip comes in contact with a hydrophobic surface, leading to a particularly large adhesion [34–38], but on modified PS it contacts mainly hydrophilic end-groups exerting a smaller adhesive force. The difference between the mainly hydrophilic and the hydrophobic part of the surface is large enough to separate the two histograms, as it can be seen in Fig. 2.

A determination of the force per unit surface is unfortunately not possible. Even if the increase of the contact area on border walls and in general on soft PS is not so large as in air, it cannot be neglected, and the contact area cannot be assumed as constant. As a consequence, our AFM measurements cannot provide an estimation of the amount of hydrophilic groups created through DPL. For this reason, only a rough comparison with the data in other works is possible. The ratio of the pull-off force between two hydrophilic systems, i.e. –COOH terminated, and the pull-off force between an hydrophobic, i.e. $-CH_3$ terminated, and an hydrophilic system in deionised water is in Ref. [38] about 22. This ratio is very much larger than the ratio given by our measurements, i.e. 2.25. This discrepancy can be explained taking into account the fact that in our experiment not the whole surface of modified PS has become hydrophilic. The agreement with the data in Ref. [36] is very much better. In this article the ratio between hydrophilic–hydrophilic pull-off force and hydrophilic–hydrophobic pull-off force is 3.

Modelling the cantilever as an harmonic oscillator, the power $\bar{P}$ dissipated by the tip oscillating at its resonance frequency ω_0 is given by Refs. [39,40]:

$$\bar{P} = \frac{1}{2}\frac{k_c A^2 \omega_0}{Q}\left[\left(\frac{A_0}{A}\sin\varphi - 1\right)\right], \qquad (1)$$

where k_c is the elastic constant of the cantilever, A the amplitude of the oscillations near the sample surface, A_0 the amplitude of the oscillations of the free cantilever, Q the quality factor, and φ is the phase of the cantilever relative to the driver.

The dissipated power can be calculated by measuring A and φ while performing DPL. Vertical stripes, i.e. parallel to the slow scan direction, have been written on PMMA with different V_w/V_r. (V_r is 100 mV, corresponding to an amplitude A of about 60 nm and a 'free' amplitude A_0 of about 100 nm). Fig. 3 shows the dissipated power and the energy per tap as a function of X, i.e. the fast scan direction, while writing three stripes with $V_w/V_r = 49$, 25 and 9.

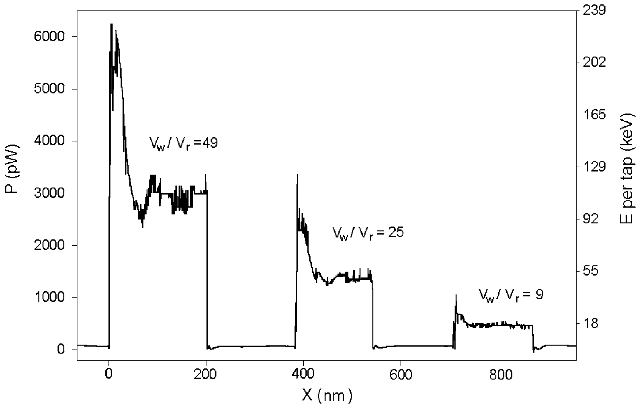

Fig. 3. Dissipated power and energy per tap as a function of the position along the fast scan direction, while writing three stripes with $V_w/V_r = 49$, 25 and 9. The stripes are 200 nm wide and have been written with a scan frequency of 1.5 Hz. The dissipated power shows a peak at the beginning of the stripes, when the feedback has not tried to compensate the increase of the oscillation amplitude.

There are a couple of features in Fig. 3 that are immediately interesting. During the 'reading' the dissipated power is about 55 pW, corresponding to an energy of about 2 keV per tap. This is a rather large value, when compared with the values of Ref. [40], but it has been proved, that the dissipated power depends also on the stiffness of the sample and can reach some keV when the elastic constant of the sample is smaller than 10 N/m [41]. Such an energy is enough to break several covalent bonds, but it is distributed over the whole contact area, so that the energy per atom is a fraction of an electron volt. For this reason it is possible to image the polymer surface non-destructively.

During the writing the dissipated power becomes very much larger, e.g. a 30-fold factor for $V_w/V_r = 49$. The dissipated power is not constant inside the stripe. It is more or less the double at the beginning of the stripe, where P presents a peak. This peak is due to the interplay between lithography and feedback [31]. The feedback tries to react to the perturbation in the amplitude, i.e. the sudden increase of the cantilever oscillations, and when the perturbation is continuous, as in this case, it tries to reduce the amplitude to its prefixed value (set-point). When the perturbation is relatively small, the feedback is able to compensate the increase of the amplitude, and the surface is carved only at the beginning of the stripe. On the other hand, when the increase of amplitude is rather large, the stripe is uniformly carved, but the first line is deeper than the rest. The dependence of the power dissipation inside the stripe reflects the depth of the carved structure.

Since the tip is close to the surface, when A_0 is increased from the 'reading' value to the 'writing' value, A cannot increase more than about 30%, and φ, passing from about 120° to about 160°, cannot compensate the increase of the ratio A_0/A. Hence, the increase of the dissipated power depends substantially on A_0. Fig. 4 shows the dependence of the mean of the dissipated power inside the stripes on V_w/V_r, confirming the proportionality between the dissipated power and A_0.

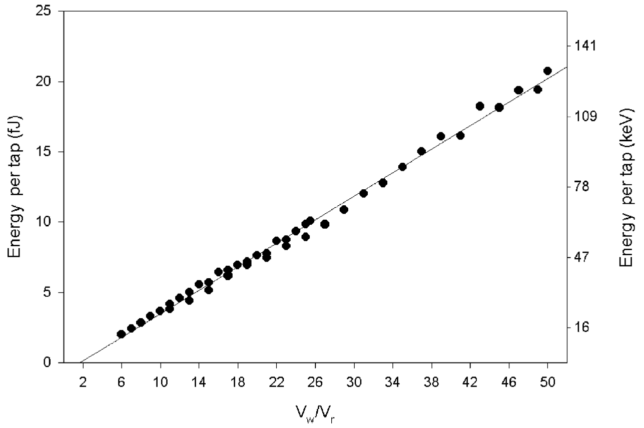

Fig. 4. Dependence of the mean of the dissipated power inside the stripes on V_w/V_r. The dissipated power is proportional to A_0.

Also the energy per tap during the writing is distributed over the contact area, and in this case the contact area is very much larger than during the reading, due to the indentation of the tip. It has been proved [28] that the tip indents gradually the polymer during the first 40–50 contacts. During the following contacts, the hole carved by the tip does not increase considerably its width, and the action of the tip is limited to a slight further compression of the polymer, leading to a slight increase of the depth. Assuming that, during the writing, the tip always comes in contact with the whole surface of the hole, the energy per tap and per atom exceeds 1 eV only at the beginning of the carving. Also the largest dissipated energy per tap obtained in our experiment (250 keV at the beginning of the stripe with $V_w/V_r = 49$), when distributed over a surface of 2500 nm^2, gives a dissipated energy of 1 eV per atom.

The results of the experiments on the dissipated energy can be summarized as follows:

1. during the writing the tip has enough energy (> 1–2 eV) to break the polymer chains;
2. the amount of energy per atom necessary to break chains is available only at the beginning of the writing, when the tip has not indented completely the polymer and the contact area is still relatively small;
3. assuming a contact area of 20 × 20 nm^2, when V_w/V_r is smaller than 10, the dissipated energy per atom is a fraction of eV and is too small, not sufficient to break chains.

In order to verify the assumption that polymer chains are broken by the tip during DPL, it is necessary to measure the length of the polymer chains. To this purpose, squares have been written on a PS standard ($M_w = 130$ kD, corresponding to 1250 monomers) and the lithographed sample has been dissolved in a solution of cyclohexanone. Even if a relatively large surface has been lithographed, the percentage of modified PS in the solution is rather small, since unmodified PS was still present between the squares and since only the surface of the about 200 nm thick film had been modified.

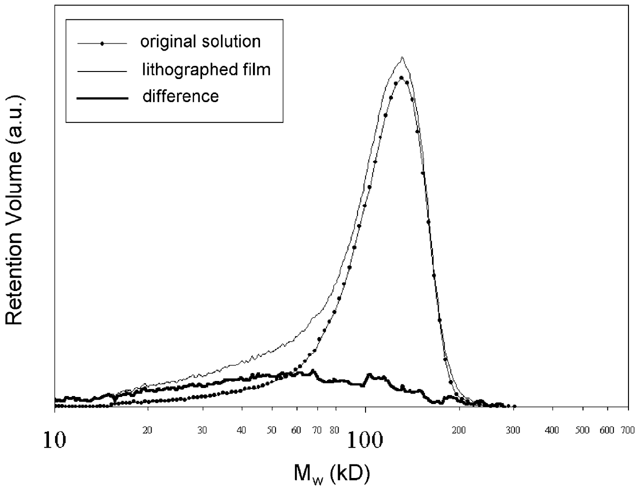

Fig. 5. Size exclusion chromatography measurements on the original solution, i.e. completely unmodified PS (thin line with black points) and on the lithographed film (thin line). The second measurement has been normalized to the first one. The thick black line puts in evidence the difference between the two measurements.

Fig. 5 shows the result of SEC measurements on the original polymer, i.e. completely unmodified PS (thin line with black circles) in tetrahydrofurane, and on the lithographed film (thin line). The second measurement has been normalized to the first one. The solution of the lithographed film shows a large peak in correspondence of 130 kD (unmodified polymer and unbroken chains), but also a large shoulder between 130 and 20 kD (broken chains). The thick black line puts in evidence the difference between the two measurements. The large percentage of unbroken chains is due not only to the fact that the solution of the lithographed film contains large amounts of unmodified polymer, but also to the fact that not all the chains of the modified PS are broken during DPL. Since it is not possible to determine the ratio between lithographed and not-lithographed polymer, it is impossible to calculate the ratio between broken and unbroken chains.

The distribution $p(L)$ resulting from a random scission of the gaussian distribution $g(\lambda)$ of polymer chains is given by:

$$p(L) = \int_L^{\infty} a \exp\left[-\frac{1}{2}\left(\frac{\lambda - \lambda_0}{\sigma}\right)^2\right]\frac{1}{\lambda}\mathrm{d}\lambda. \tag{2}$$

This expression is obtained with the assumption that all ‘new’ lengths L are equiprobable and hence the chains of length between λ and $\lambda + \mathrm{d}\lambda$ generate all the possible ‘new’ lengths, i.e. all lengths between 0 and λ. The number of chains of length L is given, accordingly to this assumption, by the number of chains of length λ, i.e. $g(\lambda)$, divided by λ.

The integral in Eq. (2) cannot be calculated, but it is well approximated by the following function:

$$p(L) = \frac{a\sqrt{2\pi\sigma^2}}{\lambda_0} \frac{1}{1 + \exp\left(\frac{L - \lambda_0}{\sigma}\right)}. \quad (3)$$

This function is constant for $L < \lambda - 3\sigma$ and is zero for $L > \lambda + 3\sigma$. The distribution obtained with our SEC measurements is not constant and decreases with L. Several reasons may be responsible for this dependence of $p(L)$. First of all, the shorter and lighter chains might stick to the tip. As a matter of fact, SEM images of the cantilever after DPL show that the tip is contaminated with polymer. Due to the small amount of material collected by the tip, it is not possible to perform SEC measurements of the material sticking to the tip. In addition, the shorter chains could re-polymerise after the chain scission, so that, after the re-polymerisation, there are more longer chains and less shorter chains than immediately after the chain scission.

4. Conclusions

PMMA and PS films have been structured by means of DPL. Measurements of the adhesion of the structured films by AFM force–displacement curves show that the modified surface of PS has a smaller adhesion than the unmodified surface. This result leads to the assumption that the hydrophobic PS chains have been broken during DPL and, due to oxidation, have become hydrophilic.

The energy dissipated by the tip during DPL has been measured. During the first 20–30 contacts between tip and sample, the energy per tap and per atom has been found to be sufficient to break covalent bonds (>1–2 eV). After these first contacts, the energy becomes smaller and is distributed over a larger contact area.

In order to definitely prove that polymer chains are broken by the tip during DPL, the lithographed films have been analysed by SEC. The measurements show that a small percentage of the polymer chains has been broken, generating pairs of shorter chains.

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