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Optimum topographical and morphological information in AFM tapping mode investigation of multicomponent polyethylene

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Abstract

For systematic AFM tapping mode studies of a binary blend of multicomponent polyethylene height and phase images were recorded at different set-point amplitude ratios r_{sp} ranging from 0.95 to 0.1. Force-probe mode measurements were carried out both in stiff and soft regions of the blend. Based on the resulting amplitude/phase versus distance curves a contrast interpretation of the height and phase images at different r_{sp} was carried out. In agreement with the results of several authors it was demonstrated that only at light tapping, with r_{sp} close to 1, does the profile in the height image present a true surface topography, whereas with $r_{sp} \approx 0.5$ much harder tapping is necessary, so that maximum phase contrast is observed. Therefore, interleave scanning with lighter and harder tapping in the main and interleaved scans, respectively, was used to simultaneously record height and phase images with optimum topographical and morphological information, respectively. Furthermore, interleave scanning combined with negative lift mode was used to make local changes in indentation visible.
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1. Introduction

In recent years, atomic force microscopy (AFM) and, in particular, tapping mode AFM (TMAFM) [1] has emerged as an important analytical tool for characterization of the structure and the properties of heterogeneous polymers, as for example, reviewed in [2–4]. Generally, in the TMAFM a cantilever is forced to oscillate with the probing tip at a given amplitude of A_0, i.e. free vibration, typically at or near its resonance frequency ω_0. The cantilever is then brought close to the specimen and made to tap the surface with a given reduced set-point amplitude A_{sp}. The probe–sample interaction also involves a shift in the resonance frequency and a phase shift $\Delta\Phi$ in the vibration with respect to that of the freely oscillating cantilever. Both the resonance frequency and the phase shift are sensitive measures of the forces acting on the probe. Attractive forces acting on the AFM probe cause a negative shift in its resonance frequency, while repulsive forces lead to a positive shift. In the usual TMAFM imaging the specimen surface is scanned at a set-point amplitude A_{sp} kept constant by a feedback loop. During the scan the vertical displacements Δz needed to keep the amplitude constant are displayed as "height image" and the locally varying phase shift $\Delta\Phi$ is displayed as "phase image". In principle, the height image should reflect the sample topography, while phase images are well-known

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to show morphological structures of heterogeneous polymers. However, the contrast of height and phase images strongly depends on experimental conditions. Factors significantly affecting height and phase images in TMAFM of multicomponent polymers include the cantilever force constant, the tip shape, the amplitude A_0 of free vibration and, in particular, the set-point amplitude ratio $r_{sp} = A_{sp}/A_0$, as described in [2,5–9]. To examine the dynamics of the tip–sample interaction causing the image contrast, additional TMAFM experiments can be carried out in a force-probe mode [5]. In this mode the lateral position of the tip is fixed, and the amplitude and phase shift $\Delta\Phi$ of the tapping cantilever are measured as a function of the varied tip–sample distance Δz. Generally, the resulting amplitude versus distance and phase shift versus distance curves (see Fig. 2a) show the following typical features: When an amplitude drop is small, a negative phase shift indicates that the overall tip–sample interactions are attractive. With a further amplitude drop, repulsive interactions become dominant as seen by the switch in the phase shift from negative to positive. Finally, the phase shift drops to zero when the amplitude is reduced to zero and the vibration has ceased. These interactions also depend on the magnitude of the initial amplitude A_0. After varying A_0, the following results were typically obtained on surfaces of polyethylene samples [2]: When A_0 is small (1–5 nm), phase shifts are negative at all amplitudes. Under these conditions, tip–sample interactions are always attractive. At large amplitudes (above 60 nm), the phase shifts are always positive owing to the dominant repulsive force interactions.

The results from force-probe mode investigations of the components of heterogeneous polymers yield information concerning the contrast interpretation of phase images including contrast inversions which could take place when r_{sp} is varied between 1 and 0. On the other hand, the indentation δ in dependence on r_{sp} can be determined from the amplitude versus distance plots by comparing them with corresponding plots of hard materials such as silicon [2,5,10]. Local variations of δ influence the height image contrast and falsify the topography information. Based on results of amplitude/phase versus distance curves, quantitative contrast interpretations of TMAFM height and phase images concerning different blends [2,5,8] and block copolymers [10–12] were reported.

In the present paper, TMAFM investigations at different set-point amplitude ratios r_{sp} and force-probe experiments on an HDPE/EOC blend are described. These experiments were carried out in order to find the necessary conditions for obtaining optimum topographical and morphological information. In this context, optimum is defined as achieving both maximum image contrast and clear interpretation of the image structures. Maximum image contrast is not very helpful if a clear interpretation of the image structures is not possible. For example, a high contrast height image with merged topographical and morphological image structures is difficult to interpret, when there is no additional information as to whether a selected image structure is caused by the topography or by the morphology of the sample. Therefore, a clear interpretation of the image contrast is also essential for optimum information. Furthermore, results described in this paper are particularly focused on TMAFM investigations for which both topographical and morphological information are of importance. This is of particular interest when, in combination with a special deformation device, the deformation behaviour of a polymer sample is studied with the aid of TMAFM during an in situ tensile test. Morphological structures as well as the surface relief are significantly changed during such a test, for example, in blends of ethene/1-hexene copolymers, as reported in [13–15]. Concurring with the published results of several authors concerning various samples, specifically multicomponent polyethylenes as reported in [2], our investigations reveal that only when r_{sp} is close to 1, does the profile in the height image present a true surface topography. This image regime is known as "light tapping". However, a much harder tapping with $r_{sp} \approx 0.5$ is necessary to observe maximum phase shift contrast between stiff and soft regions of the blend. Thus, using TMAFM in the usual manner a single scan cannot yield optimum topographical and morphological information. Therefore, in a TMAFM investigation of multicomponent polyethylene, interleave scanning was used with light and harder tapping in the main and interleaved scans, respectively, for a direct differentiation between topographical and morphological information. Furthermore, local differences of δ can be made visible when interleave scanning at light tapping in the main scan is combined with tapping without feedback loop in the interleaved scan at a reduced tip–sample distance using negative lift mode.

2. Experimental

2.1. Material

The material used in this study was a blend consisting of 25 wt.% high density polyethylene (HDPE) and 75 wt.% ethylene/1-octene copolymer (EOC). Both the components HDPE 53050E (density 0.952 g/cm^3) and EOC AFFINITY* EG 8150 (density 0.868 g/cm^3) are commercial products from the Dow Chemical Company. Plates 1 mm in thickness of the binary blend were produced by extrusion. Finally, the blend morphology of the melt-crystallized samples was mainly influenced by the sample preparation described in the next section. Owing to phase separation of the components the blend demonstrates a matrix with a morphology resembling that of the pure EOC. The formation of regions with ordered crystalline lamellae, as found in the pure HDPE, is hindered in the blend. However, heaps and bundles of crystalline lamellae are embedded in the matrix. The pure EOC consists of disordered, worm-like crystalline domains ca. 10 nm in size, dispersed in amorphous material. Following the classification suggested in [16] and taking into account the density of 0.868 g/cm^3 the EOC is a copolymer of type I and the granular, non-lamellar morphology with worm-like crystalline regions can be described as fringed micelles.

2.2. Sample preparation

Systematic TMAFM investigations were carried out on samples with very flat surfaces. To prepare these samples a piece of the blend was melted at 160 °C, pressed on a polished glass slide and cooled down at a rate of 10 K per min. Finally, the glass support was removed from the sample to uncover the surface to be investigated.

Additionally, samples with a meander-like surface relief were used to test the use of interleave scanning. These samples were prepared in the same way. However, a silicon calibration grating (TGZ01, Silicon-MDT Ltd., Moscow) with a step height of 22 nm was used instead of the glass support in order to impress the desired relief into the sample surface.

2.3. AFM investigation

All investigations were performed under ambient conditions using a commercial scanning probe microscope (Digital Instruments Dimension 3000 with a Nanoscope IV controller). Commercial silicon cantilevers were used (Nanosensors, Type NCL-100) with resonance frequencies between 163 and 169 kHz and a nominal cantilever spring constant of between 31 and 71 N/m. All TMAFM experiments were carried out by driving the cantilever at its resonance frequency. It is important to find the resonance frequency in the immediate vicinity of the sample surface, as far away from the surface, the resonance frequency of the cantilever will be higher due to reduced damping of the cantilever motion in a near-surface confined environment [2]. Therefore, the amplitude versus frequency curves, from which the resonance frequency was determined, were measured at a distance of about 200 nm above the sample surface. As described in [10], this distance is controlled by disabling the feedback-loop of the AFM, which is in light tapping mode, and subsequently retracting the tip and performing the frequency sweep. For systematic TMAFM studies of the blend, the height and phase images presented were recorded simultaneously using a free amplitude $A_0 \approx 40$ nm and set-point amplitude ratios, $r_{sp} = A_{sp}/A_0$, ranging from 0.95 to 0.1. Force-probe mode measurements were also carried out at $A_0 \approx 40$ nm both in a matrix area of the blend and at a site of a crystalline lamellae bundle. The measured amplitude and phase shift versus distance curves were transformed into corresponding phase shift $\Delta\Phi$ versus r_{sp} plots. Differences in these plots corresponding to matrix and lamellae regions directly reveal the contrast of phase images recorded at different r_{sp}.

Additionally, force-probe mode measurements under identical conditions were carried out on a silicon wafer. On the one hand, this was done in order to calibrate the RMS amplitude signal of cantilever vibration, enabling cantilever amplitudes to be measured in nm instead of V units. On the other hand, the slope of the amplitude versus distance curve on silicon was used as a reference point to determine the indentation δ of the tip in polymeric materials.

The complete series of investigations was also carried out at $A_0 = 80$ nm to test the influence of amplitude variation. Essentially, the results are similar. Therefore, only results concerning a free amplitude of $A_0 = 40$ nm are presented.

Interleave mode is a feature of the scanning software which allows the simultaneous acquisition of two data types, i.e. height and phase images, as the scan parameters for the main and interleaved scan lines can be set independently. This enables topographical and morphological data to be simultaneously recorded resulting in optimum information, i.e. maximum contrast and clear interpretation. After each main scan line trace and retrace, a second (interleave) trace and retrace is made with data acquired to produce an image concurrently with the main scan. Based on the results of systematic TMAFM investigation and force probe mode experiments of the blend the main scan was used to record a height image at light tapping with r_{sp} close to 1. This presented the true surface topography, while the interleaved scan, performed at harder tapping with $r_{sp} \approx 0.5$, produced a phase image with maximum phase shift contrast. All the other scan parameters remained unchanged in the main and in the interleaved scans.

Furthermore, interleave scanning was used in combination with negative lift mode to make local changes in indentation visible. This combination resembles the technique introduced to carry out experiments in a force modulation mode [17–20]. This combination was applied in the following manner: The main scan was used to record a height image at light tapping with r_{sp} close to 1, presenting the true surface topography. To obtain the image revealing local changes in indentation, a feature called negative lift mode was employed during the interleaved scan. In the negative lift mode the feedback loop for the interleaved scan line was disabled and the oscillating tip tracked the surface at a user specified distance (the negative lift height) below the topography acquired during the previous main scan line. Corresponding to the negative lift height the measured RMS amplitude signal of the tapping cantilever was reduced and a corresponding indentation of the tip took place in soft samples. Locally different material properties, such as local stiffness variations, caused corresponding changes in indentation. The latter were equal to the changes of the measured amplitude signal. However, it has to be mentioned that owing to Nanoscope software, brighter regions correspond to smaller amplitude and darker regions to larger amplitude, when amplitude data was monitored in lift mode.

All investigations using interleave scanning were carried out with a free amplitude $A_0 \approx 85$ nm.

3. Results

3.1. TMAFM at varied r_{sp}

Fig. 1 shows a series of TMAFM height (a,c,e) and phase (b,d,f) images of nearly the same specimen area recorded at an amplitude $A_0 = 40$ nm and three different set-point ratios r_{sp} of 0.95 (a,b), 0.5 (c,d) and 0.1 (e,f). Due to the variation of r_{sp} small changes in contrast are observed in the height images (a,c,e). Phase image (b) recorded at light tapping with $r_{sp} = 0.95$ shows no contrast. However, lamellar structures and matrix regions can be clearly differentiated in the phase images (d) and (f) recorded at harder tapping with set-point amplitude ratios of 0.5 and 0.1, respectively. On the other hand, the contrast inversion illustrates the problem of contrast interpretation of TMAFM images, as is discussed in detail, for example, in [5,8].

3.2. Force-probe mode experiments

To determine the influence of r_{sp} on image contrast force-probe mode experiments were carried out, on the one hand, in a soft matrix area of the blend, and on the other, at a specimen site where a crystalline lamellae bundle was located. The resulting amplitude versus distance and phase shift versus distance curves are shown in Fig. 2a. The full and dotted lines correspond to lamellar and matrix regions, respectively. Taking into account the initial amplitude and the correlation of the phase shift $\Delta\Phi$ at an arbitrary point of the abscissa Δz with the corresponding reduced amplitude of the amplitude versus distance curve, the $\Delta\Phi$ versus Δz curves were transformed into the $\Delta\Phi$ versus r_{sp} plots, shown in Fig. 2b. Differences of the two plots presented in Fig. 2b recorded on the lamellar and the matrix regions of the blend, respectively, correlate directly with the contrast of phase images at different r_{sp}. At light tapping with r_{sp} close to 1, both plots nearly coincide, thus phase shift contrast is not observable under this condition, as shown by Fig. 1b. Concurring with Fig. 1d the greatest difference in the plots in Fig. 2b at about $r_{sp} = 0.5$ causes a maximum phase shift contrast. Lamellae appear bright in these conditions as the corresponding curve in Fig. 2b exceeds that of the matrix region. A cross-over of the curves takes place at about $r_{sp} = 0.2$, where phase shift contrast vanishes again. A further decrease in r_{sp} results in a contrast inversion of phase images as shown in Fig. 1f.

The interpretation of contrast in height images is more complicated. The interpretation takes advantage of an evaluation of the tip indentation δ in soft materials. Due to indentation δ amplitude versus distance curves recorded on polymer samples deviate from the straight line plot, which describes the amplitude drop when the sample is hard, such as is the case for silicon wafer. In the latter case, the amplitude drops linearly with the varied vertical distance and vanishes totally when the sample surface crosses the cantilever baseline. Fig. 3a shows the amplitude versus distance curves of Fig. 2a together with the corresponding curve found on a silicon wafer (thin full line marked by Si). As already mentioned above and described in detail in [21], the indentation δ is estimated at an arbitrary point of the amplitude versus distance curve for the polymer by its difference from the corresponding reduced amplitude of the hard silicon wafer. As the slope of the Si amplitude curve is 45° either the difference in vertical direction or in horizontal direction can be used to obtain δ, as illustrated in Fig. 3a. The estimation of δ as the difference of ordinate values of the amplitude curves of polymer and Si corresponds with the force-probe mode experiment where, due to the indentation in soft materials, the amplitude drop in dependence on Δz is weaker than that on hard materials, where no indentation takes place. On the other hand, the indentation δ expressed as the difference of abscissa values of the two amplitude versus distance curves at the same reduced amplitude corresponds with variations of δ monitored in TMAFM height images where the set-point amplitude is kept constant by changing Δz via the feedback loop. Fig. 3a also shows the estimated indentation curves, where full and dotted lines again correspond to lamellar and matrix regions of the blend, respectively. From the same procedure used on phase shift versus distance curves a useful transformation into plots of δ versus r_{sp} (presented in Fig. 3b) was carried out. At light tapping with r_{sp} smaller than about 0.85, neither plot differs significantly. Thus, height images monitored under this condition, as for example shown in Fig. 1a, show the actual surface topography. Using harder tapping with $r_{sp} < 0.8$ the indentation in lamellar and matrix regions of the blend differ significantly, thus height images recorded under this condition (Fig. 1c,e) show a superposition of topographical and morphological information.

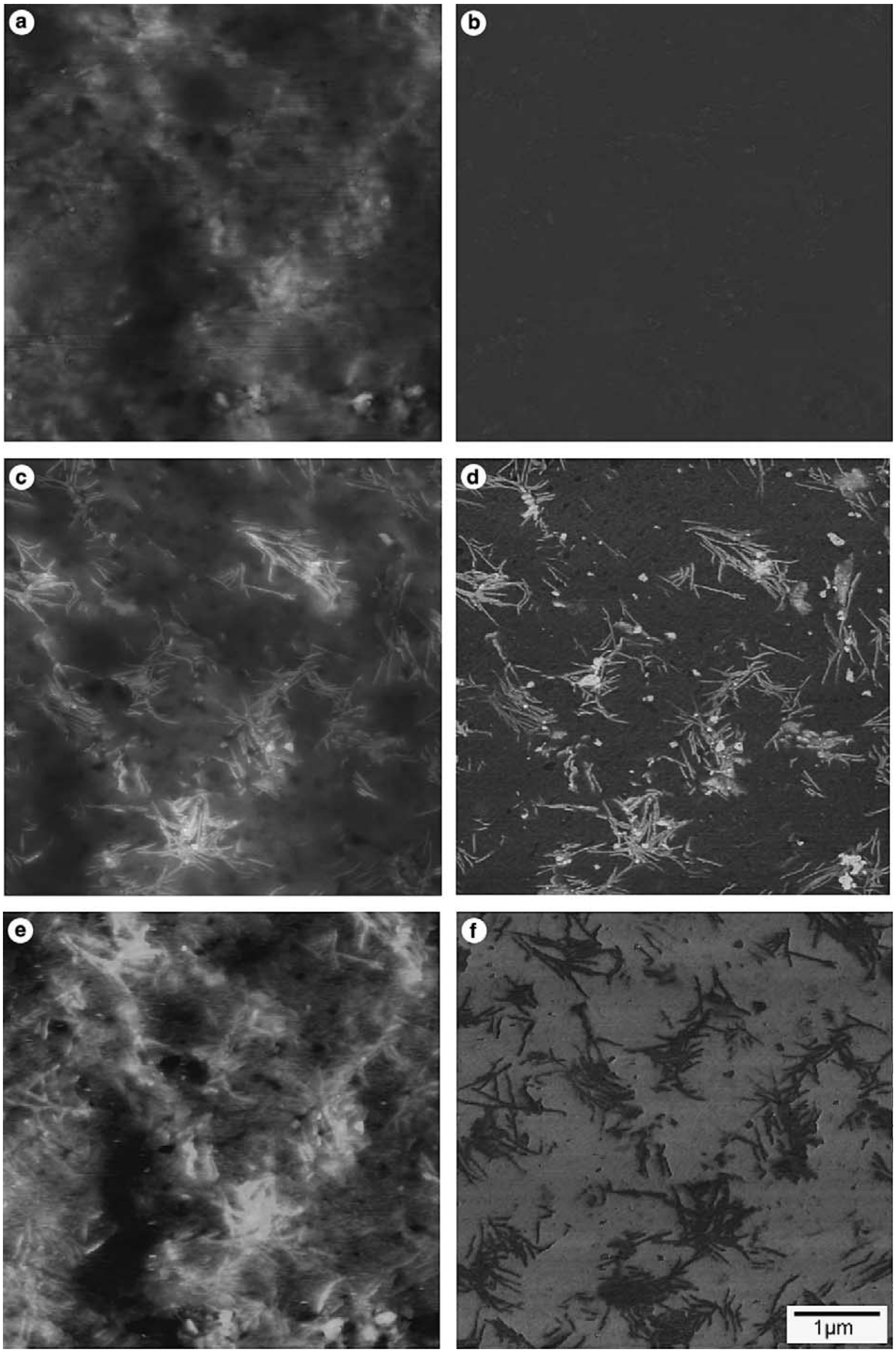

Fig. 1. TMAFM height (a,c,e) and phase (b,d,f) images of the same area of the HDPE/EOC blend recorded at three different amplitude set-point ratios r_{sp} of 0.95 (a,b), 0.5 (c,d) and 0.1 (e,f). The contrast covers height variations in the 120 nm range (a,c,e) and phase shift variations in the 100° range (b,d,f).

3.3. Application of interleave scanning

Fig. 4 shows results of interleave scanning on a flat sample surface. The TMAFM images (a–c) were recorded simultaneously with unchanged scan parameters apart from r_{sp} which was different in the main and interleaved scan. In accordance with the results of the previous section the main scan was used to record the

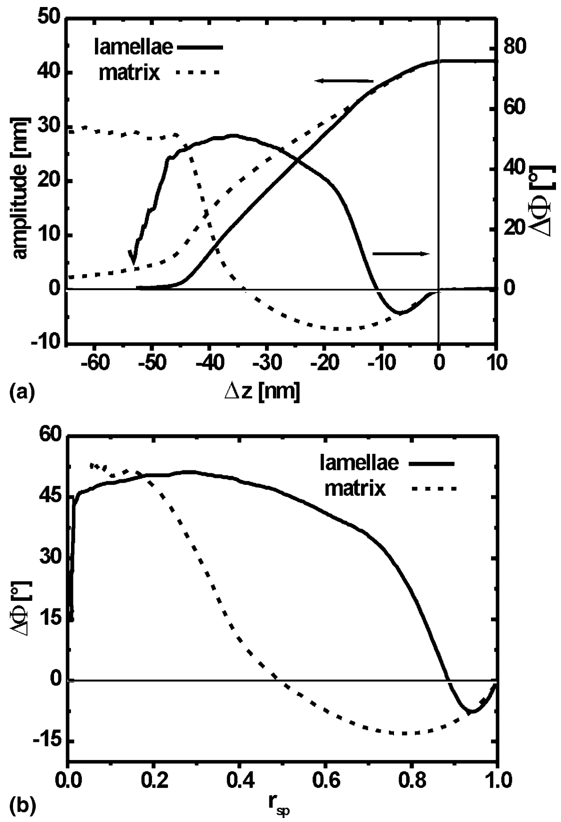

Fig. 2. Results of force-probe mode experiments: (a) Amplitude versus distance Δz curves and phase shift $\Delta\Phi$ versus distance Δz curves of lamellar (full lines) and matrix (dotted lines) regions of the blend. (b) Phase shift $\Delta\Phi$ versus r_{sp} plots of lamellar (full line) and matrix (dotted line) regions of the blend, estimated by a transformation (see text) of the $\Delta\Phi(\Delta z)$ of (a).

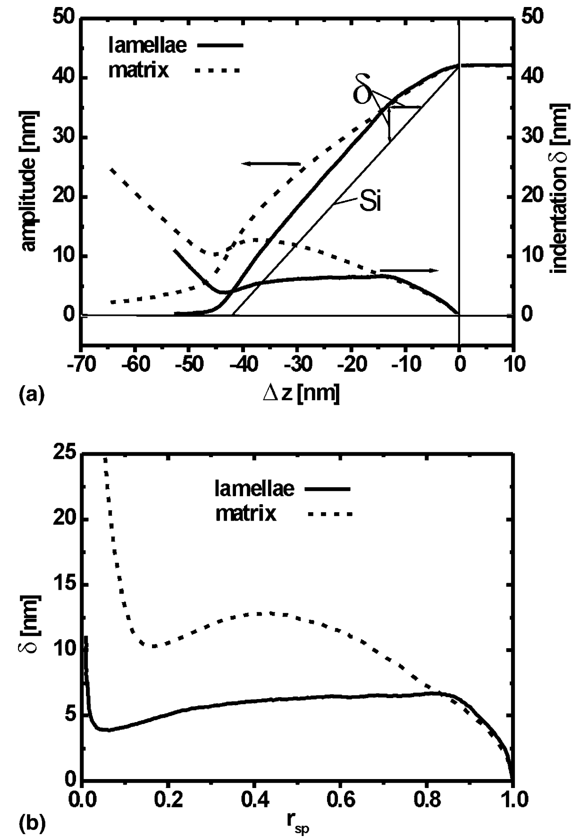

Fig. 3. (a) Amplitude reference curve of a silicon wafer (Si), amplitude versus distance Δz curves of Fig. 2a and estimated indentation δ versus distance Δz curves of lamellar (full lines) and matrix (dotted lines) regions of the blend. The $\delta(\Delta z)$ curves were estimated using the ordinate or abscissa differences marked by δ (see text). (b) Indentation δ versus r_{sp} plots of lamellar (full line) and matrix (dotted line) regions of the blend, estimated by a transformation of the $\delta(\Delta z)$ curves of (a).

height image (a) at light tapping with r_{sp} close to 1, which presents the true surface topography, while the interleaved scan, performed at harder tapping with $r_{sp} \approx 0.5$, produced the phase image (b) with maximum phase shift contrast. The height image (c) recorded simultaneously with the phase image (b) during the interleaved scan illustrates the superposition of topographical and morphological information, caused by differences in indentation at harder tapping. These differences in indentation in lamellar and matrix regions are made visible in the amplitude image (d), which is a result of the second application of the interleave scanning combined with the negative lift mode. The contrast of this amplitude image resembles that of the phase image (b) as owing to the software a smaller amplitude signal, and hence also a smaller indentation, appears brighter. A corresponding image series of a sample with an impressed meander-like surface relief, presented in Fig. 5, shows that the investigations based on interleave scanning can also be successfully applied to samples of greater roughness.

4. Discussion

The morphology and micromechanical behaviour of multicomponent polymers such as the blend investigated were extensively studied in our group by means of electron microscopy and scanning force microscopy. Within the scope of these studies the TMAFM experiments described above were preliminary investigations with the goal of finding the ideal experimental conditions for using TMAFM when the interpretation of image structures needs both topographical and morphological information. TMAFM investigations of polyethylene blends during an in situ tensile test are a typical example. Morphological structure as well as the roughness of an originally very flat sample changed significantly with increasing deformation, as shown in [13–15]. Both morphological and topographical information are also of particular interest when changes in

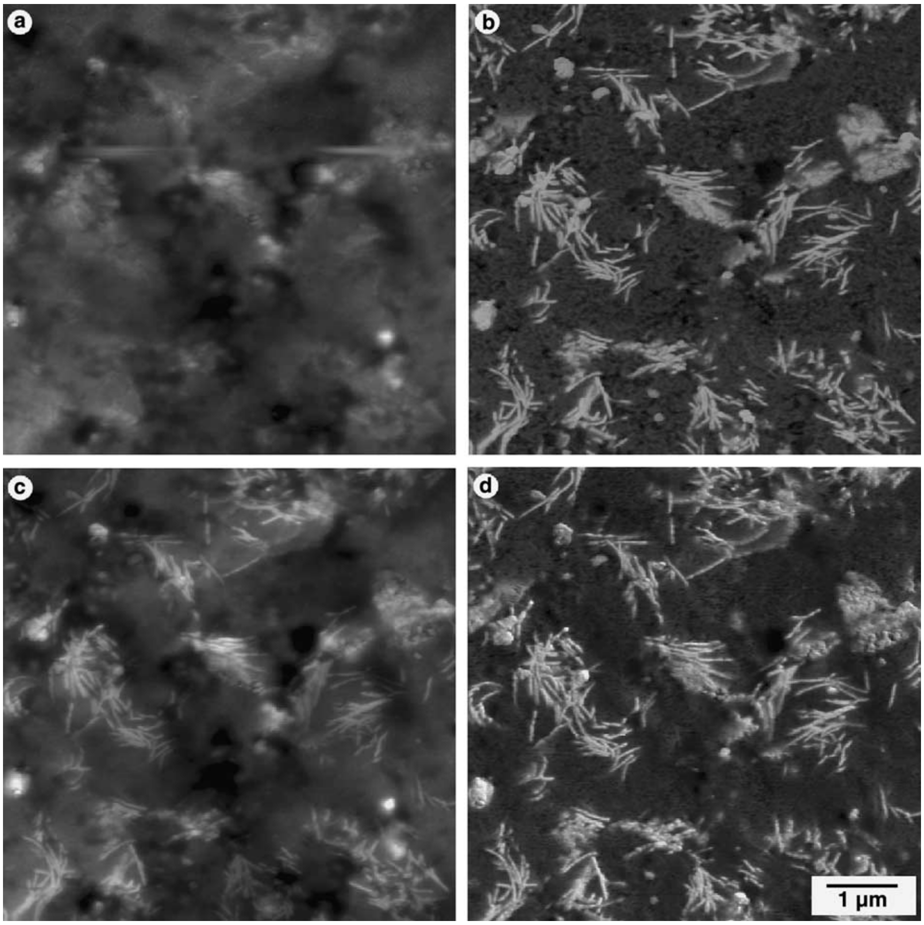

Fig. 4. Results of interleave scanning on a flat blend surface: Main scan height image recorded at $r_{sp} \approx 1$ (a), interleaved scan phase (b) and height (c) images recorded at $r_{sp} \approx 0.5$; amplitude image (d) of another application of interleave scanning presenting indentation differences. The contrast covers height variations in the 80 nm range (a,c), phase shift variations in the 60° range (b) and variations in indentation in the 30 nm range (d).

blend surfaces caused, e.g., by selective chemical etching, plasma etching or electron beam irradiation, are investigated with the aid of TMAFM.

The results presented above demonstrate that TMAFM investigations with different scan parameters are necessary to get the desired morphological and topographical information. Thus, two different scans have to be carried out if conventional TMAFM is used. A match of those two images requires very stable imaging conditions. If, however, a deformed polymer sample is investigated, a specimen drift due to relaxation often cannot be avoided. In such a case, a better matching of the images is possible when interleave scanning is used. Owing to the acquisition of the pair of images line by line the local drift between corresponding pixels in the two images is significantly reduced. On the other hand, it has to be taken into account that owing to the doubled scan time for interleave scanning the distortions of the images due to specimen drift are correspondingly increased.

Fig. 2b was used to define scan parameters for TMAFM investigations with optimum morphological information. According to the largest difference between the $\Delta\Phi(r_{sp})$ plots maximum phase shift contrast between lamellae and matrix should be found for $0.4 < r_{sp} < 0.6$. This condition also allows a clear contrast interpretation of phase images as lamellae appear bright in a wide window of $0.2 < r_{sp} < 0.9$. Fig. 1d recorded under those conditions for $A_0 \approx 40$ nm confirms this conclusion.

A complete series of images at different r_{sp} were also recorded and force-probe experiments were carried out at $A_0 \approx 80$ nm. Detailed results are not shown here. It was found that compared with Fig. 2b, the corresponding $\Delta\Phi(r_{sp})$ plots for $A_0 \approx 80$ nm reveal a crossover at $r_{sp} \approx 0.1$ and generally smaller differences between the plots. Furthermore, the matrix also shows a

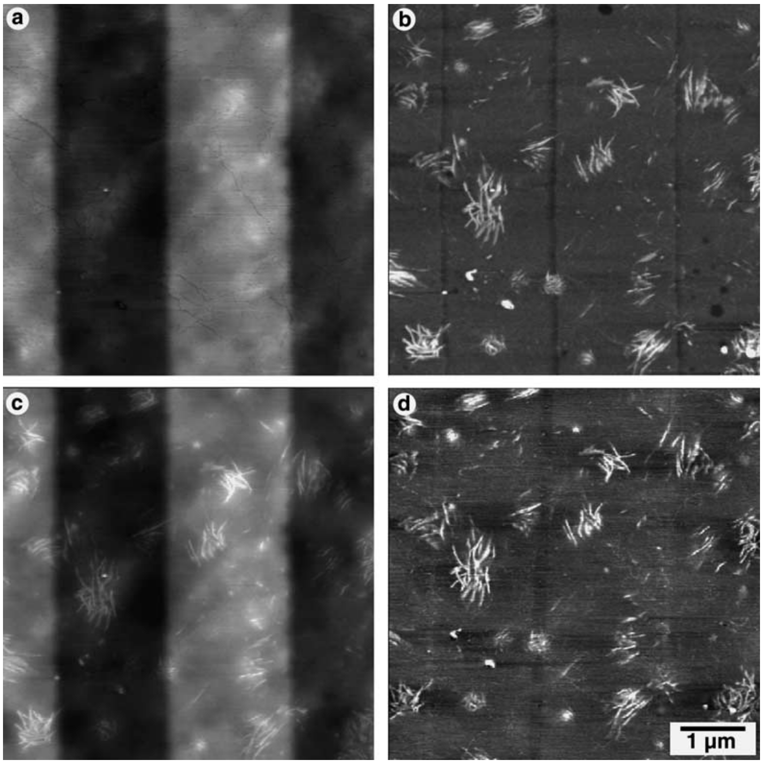

Fig. 5. Results of interleave scanning on a blend surface with a meander-like relief: Main scan height image recorded at $r_{sp} \approx 1$ (a), interleaved scan phase (b) and height (c) images recorded at $r_{sp} \approx 0.5$; amplitude image (d) of another application of interleave scanning presenting indentation differences. The contrast covers height variations in the 60 nm range (a,c), phase shift variations in the 60° range (b) and variations in indentation in the 15 nm range (d). Alternating dark/bright stripes in the height images a and c are caused by the meander-like surface relief with steps of 22 nm.

positive phase shift for $r_{sp} < 0.85$. Nevertheless, these plots also reveal that $0.4 < r_{sp} < 0.6$ is a good choice for maximum phase shift contrast between lamellae and matrix. Fig. 3b shows that for $A_0 \approx 40$ nm under these conditions the tip indentation in lamellae and matrix areas is 6.5 and 13 nm, respectively. An increase in the corresponding values to 18 and 29 nm, respectively, was found for $A_0 \approx 80$ nm.

As demonstrated by Figs. 4d and 5d, the significantly different indentations in areas of the matrix and that of the lamellae can also be used to reveal the morphology of the sample when the particular combination of interleave scanning and lift mode is used. To the best of our knowledge, the present work is the first to report the direct observation of local differences in indentation. The deep indentation at moderate tapping with $0.4 < r_{sp} < 0.6$ reveals that the morphology information is a result of intensive interaction of the tip with a surface layer of corresponding thickness.

Completely different conditions are used to yield topographical information at light tapping with $r_{sp} \approx 1$, where significant tip indentation does not take place and the corresponding negative phase shift (see Fig. 2b) indicates that the overall tip–sample interactions are attractive. Many TMAFM studies using these conditions, including recently reported theoretical [22,23] and experimental [24,25] studies dealing with non-contact investigations, have been published. A more detailed analysis of height images recorded at $r_{sp} \approx 1$ is, however, beyond the scope of the present work, as the described experimental results are only sufficient to recommend light tapping with $r_{sp} \approx 1$ to extract the desired topographical information. Phase shift plots as well as indentation plots in dependence on r_{sp} in Figs. 2b and 3b, respectively, reveal nearly identical curves in matrix and lamellar areas for $0.95 < r_{sp} \leqslant 1$. This is a clear indication that at $r_{sp} \approx 1$ height images are not superimposed by the morphological structures.

5. Concluding remarks

Systematic tapping mode AFM (TMAFM) investigations of a blend consisting of 25 wt.% high density polyethylene and 75 wt.% ethylene/1-octene copolymer were carried out. For these studies height and phase images were recorded simultaneously using a free amplitude $A_0 \approx 40$ nm of the oscillating cantilever and set-point amplitude ratios, $r_{sp} = A_{sp}/A_0$, ranging from 0.95 to 0.1, where A_{sp} is the reduced set-point amplitude of the tapping cantilever. Force-probe mode measurements were also carried out at $A_0 \approx 40$ nm both in a matrix area of the blend and at a site of a crystalline lamellae bundle. Based on the resulting amplitude versus distance and phase shift versus distance curves a contrast interpretation of the height and phase images at different r_{sp} was carried out. It showed that only at light tapping, with r_{sp} close to 1, does the profile in the height image present a true surface topography, while a much harder tapping with $r_{sp} \approx 0.5$ is necessary to observe maximum phase shift contrast between stiff and soft regions of the blend. Thus, height and phase images with optimum topographical and morphological information, respectively, cannot simultaneously be monitored with conventional use of TMAFM.

Therefore, interleave scanning was used to simultaneously record a height image at light tapping, with r_{sp} close to 1 in the main scan, and a phase image with maximum contrast at correspondingly harder tapping in the interleaved scan. Additionally, local differences in indentation were made visible in amplitude images by a combination of the interleave scanning with negative lift mode.

Acknowledgements

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