Compositional Mapping of Materials with Single-Pass Kelvin Force Microscopy

Abstract
This note describes the applications of single-pass Kelvin force microscopy (KFM) to a broad range of samples. Particularly, it will be shown that KFM substantially complements phase imaging in compositional mapping of complex multicomponent materials.

Description of Single-Pass Kelvin Force Microscopy Measurements
Currently, the expansion of AFM applications is governed by a multifrequency detection of mechanical and electric tip-sample force interactions. In studies of local electric properties, these advances are related to single-pass Kelvin force microscopy (KFM) that can be implemented in different frequencies set-ups. For the past couple of years we have explored various single-pass KFM implementations, which are schematically shown in Fig. 1. This diagram illustrates that a combined detection of the mechanical and electric tip-sample interactions can be realized with amplitude modulation (AM) and frequency modulation modes (FM). Most of the experiments so far were conducted with the AM detection of the tip-sample forces that are used for topography profiling at the resonant frequency of the probe. Simultaneously with the topography measurements the electrostatic force interactions between the conducting probe and the sample are stimulated with AC voltage applied to the probe at much lower frequency. These electrostatic forces are measured with the AM or FM detection and their nullification with DC voltage in the KFM servo operation provide the quantitative surface potential data. Recently we have started using AFM measurements with FM servo for topography profiling and two related KFM modes: FM-AM and FM-FM are currently under scrutiny.
Single-pass KFM studies have been known for a while [1–2] and these measurements are usually performed with the probe scanning a sample in the non-contact mode. We have applied the single-pass KFM mode in the intermittent contact mode at set-point amplitudes in the 0.9–0.5A0 range (A0 — the oscillating amplitude of the non-interacting probe). On one hand, at these conditions a short contact of the conducting probe with a sample (even when it is a semiconductor or metal) does not perturb these specimens. On another hand, having the probe in the immediate vicinity of the sample is favorable for the sensitive KFM measurements with high spatial resolution. This is the definite advantage of the single-pass KFM compared to the Lift mode detection [3], in which the probe is removed away from the sample.

The main features and peculiarities of the AM-AM and AM-FM modes were explored on the samples of fluoroalkanes FnHm [FnHm = CF3(CF2)n(CH2)mCH3] whose self-assemblies exhibit strong surface potential due to the vertical orientation of the chains carrying the molecular dipole at the \-CH2-CF2- bond [4]. A set of F12H20 self-assembled structures (most are toroids) on Si substrate was examined in KFM multi-frequency experiments in which, in addition to the surface potential images at 5kHz, the \(\frac{dC}{dZ}\) were recorded at 10kHz Fig. 2. These measurements were performed at averaged probe-sample distances of 1nm, 7nm, 15nm and 30nm (top to bottom columns in Fig. 2). The images indicate that surface potential of these structures does not depend on the separation and its value (around -0.7 V) is consistent with predominantly vertical alignment of the molecular chains. The same self-assemblies in the \(\frac{dC}{dZ}\) maps exhibit the most pronounced contrast at 7nm separation. Actually, the \(\frac{dC}{dZ}\) contrast, which generally is associated with dielectric constant, uniquely reveals a partitioning of the toroids into seven sectors. The surface potential images in Fig. 2 were obtained in AM-FM mode. In comparing the AM-FM and AM-AM modes applied for detection of the electrostatic forces we found out that the FM detection of the electrostatic forces has definite advantages in agreement with the results obtained earlier in UHV [5]. It requires lower AC voltages (usually less than 1V) and in most cases provides higher and more correct surface potential data. Actually, our first measurements in the FM-AM and FM-FM modes are showing the similar trend. This example also shows that in the multifrequency AFM measurements there is the increasing number of variables for the experiment optimization. Therefore a researcher should be aware about making the correct choice of imaging mode, finding the optimal imaging parameters, choosing the most appropriated probe and a sample preparation.

The sensitivity and resolution of KFM are rather complicated issues. The sensitivity is essential in mapping the small potential variations in the 1–10mV range. Besides electronics the probe dimensions are one of the important factors influencing the signal-to-noise ratio in KFM experiments. The images obtained with the larger probe (tip apex around 60 nm) show substantially lower noise level in the sub-10 mV range compared to the sharp probe with the 20-nm apex. Therefore when nanometer...
spatial resolution is not essential the larger probes can be chosen for sensitive potential measurements. The latter can also benefit from slow scanning rates. As regarding the spatial resolution a strict criterion is the width of the surface potential transient region at the step between the substrate and adsorbate having different potential values. The analysis of the surface potential profiles at the steps of the fluoroalkane self-assembles shows that true spatial resolution is around 20 nm comparable with the size of the commercial conducting probes. For KFM compositional imaging of heterogeneous materials the spatial resolution of visualization of the heterogeneities is also important and we resolved the 2 nm features in such surface potential images [2].

As KFM applications broaden, this technique was applied to a wide range of the samples in different environments. The KFM studies in the intermittent contact mode, which were carried out in high humidity [4] revealed that screening of the sample surface potential was less substantial compared to the results of KFM experiments conducted in the non-contact mode. The imaging of the fluoroalkanes in high humidity demonstrates this capability, Fig. 3. In these images a water adsorbate is seen between two domains of the fluoroalkanes, which exhibit strongly negative patterns in the surface potential images. At RH > 95% the value of the surface potential of F14H20 is around -0.6 V that is only slightly smaller than at ambient conditions (-0.75 V).

Below we will overview the applications of single-pass KFM to different materials: metals and semiconductors, organics and polymers.

Metals and Semiconductors
Surface potential of metals is directly related to their work function that varies in the 4 V–5.5 V range for most common metals [6]. KFM can be employed for compositional images of metal alloys and even polycrystalline metal samples because the surface potential is different on various crystalline facets. The examples of KFM images of incomplete alloys of Bi with Sn are shown in Fig. 4. In these soldering materials KFM reveals domain morphology of this compound. The domains of Bi and Sn are distinguished in surface potential images.
images due to the difference of their work functions. Actually, the surface potential change of 0.2 V, which was found in freshly prepared samples, corresponds well to the difference of these metals’ work functions [6]. The surface potential images and the cross-sections in Figs. 4 demonstrate that the contrast disappears with time due to the oxidation of metals (particularly Sn) in air. The flat surface of the Bi/Sn sample was prepared by hot pressing between two smooth Si plates, which were removed after the sample was chilled to room temperature. Most likely, this procedure induced some internal stresses that have been released at room temperature. The related change of the sample surface is seen from the topography images in Figs. 4.

The use of KFM for compositional imaging of metals and semiconductors is further demonstrated by imaging of the test structures made by FIB deposition of Pt and SiO$_2$ lines on surfaces of Si wafer and graphite [7]. The images of these cross structures are shown in Figs. 5–6. The contrast of the lines differentiates them in the surface potential images. The quantitative data are seen from the cross-section profiles placed under the images. The potential of the Pt strips on both substrates are close to that of the conducting probe. The potential of SiO$_2$ strips, which are 20nm in height, differs from that of Si substrate that has only 2–3nm of naturally grown oxide film. Although the Pt and SiO$_2$ lines on graphite are partially “lost” amidst the numerous surface steps, the surface potential pattern shows only the material-related contrast, Figs. 6. The comparison of the lines’ dimensions in the topography images with their surface potential blueprints indicates that the effective probe is substantially larger in the electric measurements. This means that a larger part of the probe is involved in the electrostatic tip-sample interactions compared to the tip-sample intermittent mechanical contact. At the current stage of KFM developments and applications the use of these and similar standards is useful for understanding the basics of this method. Such fundamental studies can be further assisted by computer

Figure 5. Topography and surface potential images of FIB-deposited lines of Pt and SiO$_2$ on Si substrate. Scan size 40 µm. The contrast covers the height and potential changes in the 0–60 nm and 0–1 V ranges. The cross-sections profiles taken along the directions pointed with the white dotted lines are placed underneath the images.

Figure 6. Topography and surface potential images of FIB-deposited lines of Pt and SiO$_2$ on graphite. Scan size 40 µm. The contrast covers the height and potential changes in the 0–100 nm and 0–3 V ranges. The cross-sections profiles taken along the directions pointed with the white dotted lines are placed underneath the images.
simulation of the electrostatic force interactions, the surface potential and $dC/dZ$ of metals and semiconductors.

The more sophisticated standards, which have surface layers with different doping type and level, are employed for verification of different AFM-based electric techniques capability of quantitative tracking these industrially important properties. KFM images of the IMEC standard (n-type Si staircase T9 [8]) on the surface with the doped layers running perpendicular are shown in Fig. 7 together with the resistivity and doping profiles determined by different methods.

The face with the layers having doping levels increasing from $1 \times 10^{14}$ to $1 \times 10^{18}$ cm$^{-3}$ does not show noticeable surface corrugations. The layers do manifest themselves in the surface potential image as the bands whose contrast increases with the increase of the doping level. The potential varies 250 mV on the locations covering 4 orders of magnitude of dopant density. This data set is in good agreement with the earlier experimental results obtained on Si wafers with different doping density [9]. The difference of 0.240 eV between Fermi levels of the n-Si samples with $1.1 \times 10^{14}$ and $7 \times 10^{19}$ cm$^{-3}$ doping densities was estimated from the surface potential data. The well-pronounced steps of the potential profile (Fig. 7) correlates well to the width of the differently doped layers. However, it is rather premature to claim that surface potential provides the direct quantitative measure of the doping density. In many cases the potential differences detected within the single image are relatively reliable. Less reproducible are absolute potential values recorded in different images. Surface contamination, varying oxide coverage and environmental effects are among the possible reasons of the poor reproducibility.

Low-Dimensional Semiconductor Structures

Low-dimensional semiconductor and metal systems offer variety of unique structural, optical and electric properties. Some of these highly ordered materials can be prepared in mild conditions by self-assembly. The formation of CdTe nanowires is one of the related examples. It has been shown that crystalline nanowires are built up of CdTe nanoparticles in water solution upon controlled removal of organic stabilizers [10]. The strong dipole-dipole interactions were suggested as the driving force of self-organization of the nanoparticles into the pearl necklace aggregates that re-crystallized into the nanowires. The formation of the CdTe nanowires from suspension of the nanoparticles during its drying on mica substrate was monitored with KFM [11]. The topography and surface potential images, which were recorded at the end of this process, are shown in Figs. 8. These images show individual CdTe nanowires, which are 20–30 nm in width and few nm in height, as well as their arrays whose growth was likely nucleated from thicker linear structures. The surface potential image is dominated by bright contrast (approx. 250 mV) on the locations covering 4 orders of magnitude of dopant density. This data set is in good agreement with the earlier experimental results obtained on Si wafers with different doping density [9]. The difference of 0.240 eV between Fermi levels of the n-Si samples with $1.1 \times 10^{14}$ and $7 \times 10^{19}$ cm$^{-3}$ doping densities was estimated from the surface potential data. The well-pronounced steps of the potential profile (Fig. 7) correlates well to the width of the differently doped layers. However, it is rather premature to claim that surface potential provides the direct quantitative measure of the doping density. In many cases the potential differences detected within the single image are relatively reliable. Less reproducible are absolute potential values recorded in different images. Surface contamination, varying oxide coverage and environmental effects are among the possible reasons of the poor reproducibility.

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Figure 7. Topography and surface potential images of T9 standard sample with layers having different doping level. Scan size 25 µm × 30 µm. The contrast in the topography image covers the height corrugations in the 0–20 nm range. The resistivity (dark blue) and doping (magenta) profiles are placed underneath the topography image. The cross-section profile along the direction marked with a white dashed line in the surface potential image is shown below it.

Figure 8. Topography and surface potential of CdTe nanowires formed from a suspension of CdTe nanoparticles. Scan size 2 µm. The contrast covers the height and potential variations in the 0–22 nm and 0–1 V ranges.
0.5 V) of the nanowires. Additionally, to nanowires, there are also the small nanoparticles (20–30 nm in diameter), which are brighter than the nanowires in the topography images but darker in the surface potential image. These features can be assigned to the traces of the organic stabilizers of the CdTe nanoparticles.

The KFM results suggest that self-assembly of CdTe nanoparticles into crystalline nanowires is accompanied by structural transitions leading to the formation or realignment of molecular dipoles. Dipole moments in CdTe nanoparticles with cubic lattice are not expected unless they appear at structural defects that induce asymmetry of molecular structure. Therefore, the formation of the nanowires entails the radical changes of local symmetry of molecular groups.

**Organic Electronic Materials**

Current developments of organic materials for electronics strongly depend on understanding structure-property relationships in molecular films applied in field effect transistors, light emitting diodes, and organic solar cells. Getting such knowledge becomes non-trivial for objects consisting of a single sheet of molecules where electron transport has a two-dimensional character and the molecular scale imperfections need to be examined. AFM and, particularly, electric modes can be very helpful in the characterization of such structures and related devices. This has been proved in numerous studies of semiconducting molecular layers of oligo-thiophene [9] and pentacene [10]. Single-pass KFM can further improve the characterization of these materials.

KFM images of a pentacene adsorbate, which was evaporated on Si substrate in vacuum, are shown in Figs. 9. This is the single layer film with several 2nd layer dendritic structures. The 1st layer is composed of tightly packed vertically standing molecules and it has the domain substructure. The domains boundaries are best seen as bright features in the surface potential image. This contrast is observed only when the stimulating AC voltage is above 3 V that indicates on the voltage-induced dipoles at the domains’ edges. The surface potential of the dendritic structures, whose height also indicated on the vertical orientation of the molecules, is more positive (~80 mV) than that of the 1st layer, and it does not depend on a magnitude of the stimulated voltage. Actually, the smaller potential change of 50 mV was observed between the 1st and 2nd pentacene layers on Si using the Lift mode [11]. The nature of the surface potential contrast of pentacene layers is not yet established. The possible contribution of the interfacial dipoles is considered as a factor leading to the observed surface potential [12].

Self-assembled monolayer transistors in which oligo-thiophene molecules form a single layer on dielectric Si gate have been recently tested [13]. In this case the devices can be obtained by spin-casting of the thiophene solution of appropriate concentration or by immersing the substrate in it. KFM studies of these devices reveal the grain boundaries in the single layer and also a presence of extra material in form of multilayers. Both structural features can be considered as imperfections which influence semiconductor behavior of the molecular film. The multilayer morphology of one of the adsorbates of the 7-thiophene with a hydrocarbon tail is evident from KFM images in Figs. 10. The topography image demonstrated the multilayer domains formed on the
single molecular layer that completely covers the Si substrate. The surface potential of these top layers decreases with height and the difference is reaching 0.5 V.

**Heterogeneous Polymers**

Compositional mapping of heterogeneous polymers is usually achieved in phase imaging performed at elevated tip-sample forces [14]. The phase contrast is assigned to differences of local mechanical properties and variations of energy dissipated in the tip-sample interactions. Surface potential images can play a similar role in identifying the surface locations with different electric properties. Fluorinated polymers are good candidates for compositional analysis because they usually possess molecular dipoles moments that lead to surface potential contrast [15]. A range of polymer materials that might be sensed by KFM can be expanded by including acrylic polymers. Macroscopic Kelvin probe studies of thin films of poly(methyl methacrylate) (PMMA) indicated that their surface potential depend on stereoregularity and molecular conformations of this polymer [16]. Therefore KFM might distinguish PMMA domains in blends and block copolymers with other polymers. This is indeed the case as seen from the images of PS/PMMA blends (PS-polystyrene) with different composition and symmetric block copolymers. The KFM images of a 20-nm thick film of 3PS/7PMMA film (weight ratio of the components is 3:7), which was deposited on Si substrate by spin-casting, are shown in Figs. 11. The scanning was performed in the upwards direction and the stimulated AC voltage changed from 0.5 V to 6 V in the middle of the scan. This change caused a drop of the potential noise and the related drastic improvement of the surface potential image. The binary contrast pattern with the averaged difference of ~60 mV was detected in the top part of this image where the dark patches are a minor component. These patches correspond to the surface dimples seen in the topography image. The increase of the electrostatic force interactions was favorable for the potential’s noise reduction in air. When the KFM study of this sample was performed in humid atmosphere (RH>95%) the surface potential noise was smaller even at low stimulating AC voltages, Figs. 12. The surface potential exhibits the binary patterns similar to one observed at high AC voltages in air. Additionally, the multiple bright droplets, which are located inside the dimples, have
appeared in the topography image. They might present the condensed water droplets which form a relatively large contact angle that indicates on hydrophobicity of the underlying locations. Therefore the dimples with the droplets can be tentatively assigned to polystyrene domains, which are expected to be hydrophobic.

As we mentioned before, the spatial resolution of compositional imaging with single-pass KFM is in the nanometer range. The additional evidence comes with KFM imaging of PS-b-PMMA block copolymer. The images of the 50-nm film of this block copolymer on Si are presented in Figs. 13. These images show the phase separation pattern with 30-nm wide blocks. The phase image recorded simultaneously with the images did not show the contrast variations. The phase contrast becomes noticeable when the imaging is performed at larger tip-samples forces and with stiffer probes than the ones (Olympus and Mikromasch Pt-coated probes with 4–5N/m stiffness) we are regularly using for KFM studies.

Studies in humid environment can be helpful for compositional imaging of the materials with hydrophilic components. This result was obtained in imaging of two industrial polymer materials: perfluorinated membrane (Nafion™) and conducting blend of poly(3,4-ethylenedioxythiophene) and poly(styrenesulfonate) [PEDOT:PSS]. In Nafion polymer, a hydrophobic polytetrafluoroethylene backbone coexists with hydrophilic -SO₃H⁺ acid groups connected to the backbone via -O-CF-CF₂-CF₂-O-CF₂-CF₂- side chains. Due to its outstanding chemical stability and proton conductivity this polymer is used as the proton exchange membrane in electrolyte fuel cells. The cell functioning substantially depends on ion conductivity in the polymer membrane that is defined by its microphase separation morphology. In other words, a nanoscale distribution of hydrophilic and hydrophobic regions is the most essential for optimal membrane performance.

The Nafion membranes are intensively examined using microscopic (TEM, AFM) and diffraction (small angle X-ray scattering, SAXS, and neutron scattering) methods in attempts to clarify the film nanostructure. The recent model based on the SAXS data suggests that cylindrical hydrophilic domains arranged in parallel water channels with a mean size of about 2.4 nm [17]. High-resolution visualization of Nafion film morphology with AFM revealed the ionic domains of ~4 nm in size [19] is consistent with TEM data. Most of AFM experiments on Nafion samples were conducted in air and the measurements under water did not show a well resolved nanostructure.

As it was shown above in the studies of PS/PMMA blends, the 5500 scanning probe microscope (Agilent Technologies) is the most suitable for AFM and KFM measurements at high humidity. The images obtained during studies of Nafion films at different
humidity are presented in Figs. 14–15. In the imaging of Nafion films at elevated humidity one should take into account that the membrane is swelled with water and becomes much softer than at normal and low humidity. The gentle imaging of swelled films at RH=60% and at RH>95% was achieved with the regular Si probe which were driven in oscillatory amplitude modulation mode at small amplitude around 1 nm. The topography images recorded at these operational conditions exhibit the same pattern independent on humidity. The phase images are quite different and numerous dark spots of 2–3 nm in size appeared in the image recorded at higher humidity. These regions can be assigned to softer locations such as water channels described in the model introduced in [17]. This nanoscale phase separated morphology is similar to that recorded in the ambient-conditions AFM images [19]. Remarkably, KFM images with different magnification, which were recorded at RH>95%, show that the surface potential pattern is characterized by numerous 3–4 nm spots with lower potential, Figs. 14. Most likely, these spots are observed on the water-enriched locations and their slightly larger dimensions can be related to the larger size of the conducting probes that are employed for KFM. The assignment of lower potential regions to the swelled locations is supported by the data presented below.

Intrinsically conducting PEDOT:PSS blends are often used as a component in organic solar cells and light emitting diodes. Therefore morphology and electric properties of PEDOT:PSS films are among the factors influencing the cell and diode performance. PEDOT is a hydrophobic and conducting component, whereas hydrophilic PSS is an insulator. The correlation between the blend composition, film morphology and conductivity is examined by variety of the techniques with emphasis on local current studies with conducting AFM methods [20]. Particularly, it was shown that topography depressions correlate with low conductivity locations and the number of such locations correlates with amount
of PSS component. TEM studies of morphology PEDOT:PSS films reveal the phase separation in this material [21]. According to the suggested model PEDOT:PSS films are composed of spherical grains with diameter in the 50–80 nm range. Based on EDX analysis it has been assumed that these grains consist of a 5–10 nm-thick PSS-rich shell and a PEDOT-rich core.

PEDOT:PSS blends are made commercially and they are distributed as water suspensions. Conductivity of these blends depends on their composition and highly conducting PEDOT:PSS (1:2.6) material was used for the preparation of films on Si substrate by spin-casting. These films were examined with KFM in the broad range of humidity. The images of the same location of the 50-nm PEDOT:PSS film, which were obtained at extremely dry and wet conditions, are shown in Figs. 16. A comparison of the topography images the change of humidity led to smoothing of surface morphology and the numerous deep dimples became less pronounced. The change of the surface potential images is much more drastic and a homogeneous pattern seen at dry conditions converted into a binary map with the 200 mV potential difference between the components. These images were obtained at low forces therefore the phase images don’t exhibit any noticeable contrast. The darker spots in the surface potential map are located at the dimples. These regions can be assigned to PSS-rich material because the conductive AFM measurements (not shown here) revealed lower conductivity compared to their surroundings that are most likely enriched in PEDOT. The lower surface potential of hydrophilic inclusions can be tentatively attributed to apparent dipole moment developed in the locations swelled (or filled) with water. In any case, KFM imaging of PEDOT:PSS film at high humidity provides a compositional map of this material and thus improves the understanding of the structure-property relationship.

**Conclusions**

Single-pass KFM mode with FM detection of the electrostatic forces improves sensitivity and spatial resolution of surface potential imaging when applied in the intermittent contact mode. The extension of KFM applications to a wide range of materials shows its exceptional value for compositional imaging. In this function KFM complements phase imaging by advancing to earlier not accessible areas such as metal alloys. Due to the stiffness range of commercial probes phase imaging is typically useful for studies of heterogeneous soft materials with high image contrast being achieved in operation at elevated forces. The use of phase imaging to hard materials is quite limited. The KFM studies in different environments are also bringing new and valuable information about morphology of heterogeneous polymers. The area of environmental AFM will further benefit from local electric and mechanical measurements with multiple frequency detection.

**Acknowledgments**

The materials that were used in studies described in this note were kindly provided by Prof. M. Moeller (DWI, Aachen, Germany), Prof. N. Kotov (University of Michigan), Dr. R. Berger (MPIP, Mainz, Germany) and Dr. V. Kalihari (University of Minnesota). The part of the experiments described above was conducted during S. Magonov’s research visits to MPIP in Mainz and DWI in Aachen.
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Printed in USA, March 25, 2010
5990-5480EN