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Development of the photostimulated Kelvin Probe Force Microscopy setup. The tool for nanoscale diagnostic of optoelectrical properties of the surface.

Abstract. *In this paper we describe the development of the photostimulated Kelvin Probe Force Microscopy (ps-KPFM) measurement technique allowing to perform mapping of the electrical response of the material to optical excitation. The general information concerning the principles of the measurement setup and the data acquisition procedures as well as the examples of collected data is shown. In particular, the solution allowing to perform a number of measurements according to specific protocol is here sescribed. The developed system is capable of delivering the information about the electrical properties changes due to the specific light wawelength illumination of LED's in range from 390 nm to 940 nm as well as for the xenon lamp.*

Streszczenie. *W niniejszej pracy zaprezentowano konstrukcję systemu pomiarowego fotostymulowanej mikroskopii sił z sondą Kelvina (ps-KPFM), umożliwiającą mapowanie odpowiedzi elektrycznej powierzchni na pobudzenie optyczne. Przedstawiono kluczowe informacje dotyczące konstrukcji, procedur pomiarowych, jak również zaprezentowano przykłady wyników pomiarowych. Do szczególnych cech opisanego układu należy możliwość wykonywania pomiarów z wykorzystaniem wcześniej opracowanych protokołów. Zbudowany układ pomiarowy umożliwia uzyskanie informacji o optoelektrycznych właściwościach powierzchni dla pobudzenia źródłami światła LED w przedziale od 390 nm do 940 nm oraz lampą ksenonową. (**Opracowanie systemu pomiarowego fotostymulowanej mikroskopii sił z sondą Kelvina. Narzędzie do diagnostyki optoelektrycznych właściwości powierzchni w nanoskali**).*

Keywords: atomic force microscopy, Kelvin Probe Force Microscopy, optical stimulation, optoelectrical properties.
Słowa kluczowe: mikroskopia sił atomowych, mikroskopia sił z sondą Kelvina, pobudzenie optyczne, właściwości optoelektryczne.

Introduction

The development of scanning probe microscopy techniques provided insight into submicron properties of the materials and nanostructures [1, 2]. The variety of measured properties such as: mechanical, thermal, electrical, magnetic and optical allows to investigate various phenomena occurring at nanoscale and too subtle to be observed without such a tools [1, 3]. A continuous progress in the nanomaterials development created the need for more sophisticated diagnostic techniques, as complex properties has to be investigated. The electrical response to the light illumination is one of such a phenomena. Such a information is essential while new photosensitive device or materials are developed. In particular, the nanomaterials for optoelectronic applications are the subject of such a tests [4]. Therefore the combination of already known methods such as C-AFM (conductive atomic force microscopy) and KPFM (Kelvin Force Probe Microscopy) [5-7] with optical excitation solutions were introduced [8, 9]. It should be underlined, that there are some home-made setups already used in the investigation of the materials, however due to simple design, their measurement abilities are limited. The most frequent drawback is the ability to acquire only set of single measurements in one point instead of mapping. Also, the changes of the illumination intensity and wavelength can be performed manually, therefore it is difficult to run a series of measurements on various samples exactly according to specific protocol. As the solution providing more advanced functionalities was desired due to research on the polymer-based photovoltaic materials, we have developed and tested advanced system providing the photostimulated Kelvin Probe Force Microscopy measurements. In this paper the principles of the setup will be presented as well as the test results. It should be emphasized, that the integration of various components of such a system is essential, and it was achieved due to the flexible hardware and software of the commercial AFM as well as home-made modules and control codes. We show that developed system allows to perform the measurements of the electrical response of the surface due to the optical excitations using various wavelengths. Moreover, the user can edit the measurement protocol in order to perform the measurements in repetitive fashion.

Technique principles

The Kelvin Probe Force Microscopy technique allows to perform mapping of the contact potential value by the balancing of the bias voltage of the scanning tip and the voltage of the surface. As the tip-sample setup can be considered as the capacitor, the interactions between them are related to the presence of the electrostatic force, thus by measuring the response of the probe vibrating perpendicularly to the surface, one can minimize the forces and the voltage unbalance. Equations 1-3 define the three component forces acting between tip and sample:

$$F_{DC}(z) = \frac{1}{2}\frac{dC(z)}{dZ}\left[\left(\Delta V_{DC} - V_{surf}\right)^2 + \frac{1}{2}V_{AC}^2\right] \tag{1}$$

$$F_{1\omega} = \frac{dC}{dZ}\left[\left(\Delta V_{DC} - V_{surf}\right)^2 V_{AC}\sin(\omega t)\right] \tag{2}$$

$$F_{2\omega} = \frac{1}{4}\frac{dC(z)}{dZ}V_{AC}\cos(2\omega t) \tag{3}$$

where: C – capacitance of the tip-sample setup, Z – tip-sample distance, V_{DC} – DC voltage applied between tip and sample, V_{AC} – AC voltage with the pulsation ω applied to the tip, V_{surf} – the surface potential related to the work function of the surface and the tip. The most popular KPFM measurement solution, is so called two pass technique, basing on a lift mode method, where $F_{1\omega}$ is used to perform the voltage compensation.

In this approach, the first pass of the probe allows to acquire the profile of the surface using intermittent contact, in the second pass (return), the probe detects the electrostatic forces at specific height over the surface (fig. 1). In terms of utilization additional light sources causing certain response of the surface in ps-KPFM, the two pass method is desired, as it allows to avoid the unwanted influence of the illuminating light on the laser-based probe deflection detection system while the profile of the surface is acquired. It should be underlined, that conductive probes has to used in this mode (for instance covered with PtIr, Au or conductive diamond).

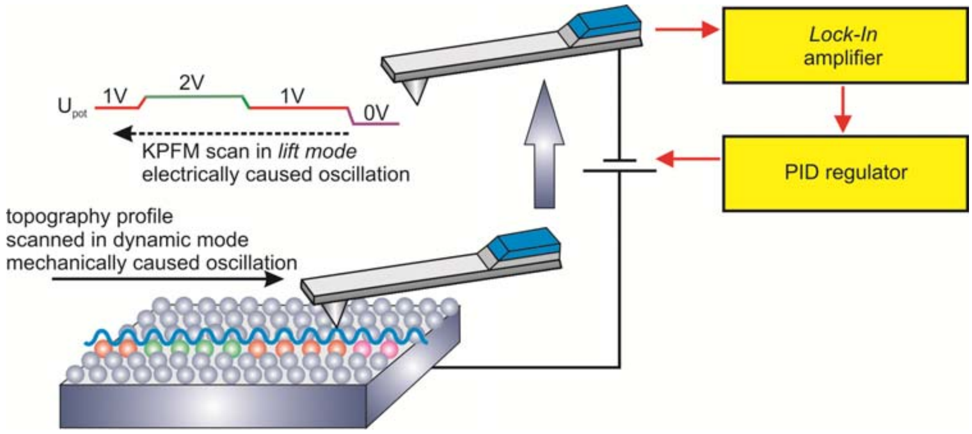


Fig. 1. The principles of the measurement procedure in KPFM technique using two pass mode

The setup development

The setup was developed basing on the commercial AFM system Innova from Bruker (former Veeco). Due to the specific hardware and software features provided by the producer, this setup allows to perform the integration with auxiliary units in order to obtain desired functionality. Such advantage was used formerly in the implementation of several different experimental upgrades and modifications [11-16]. In case of described mode, following features were used: pixel synchronization with handshaking, auxiliary analogue signal acquisition and internal signals connections mapping edition.

As a main part of the auxiliary unit, PXI data acquisition and processing system from National Instruments programmed using LabView 2013 environment was utilized. In order to provide desired computational performance, in particular the response time to external signals, Real Time regime was enabled [12, 15, 16]. Additionally, home-made electronic units, including the LED's drivers providing linear current control as well as the kHz frequency switching were designed and assembled. The simplified diagram of the setup is presented in figure 2.

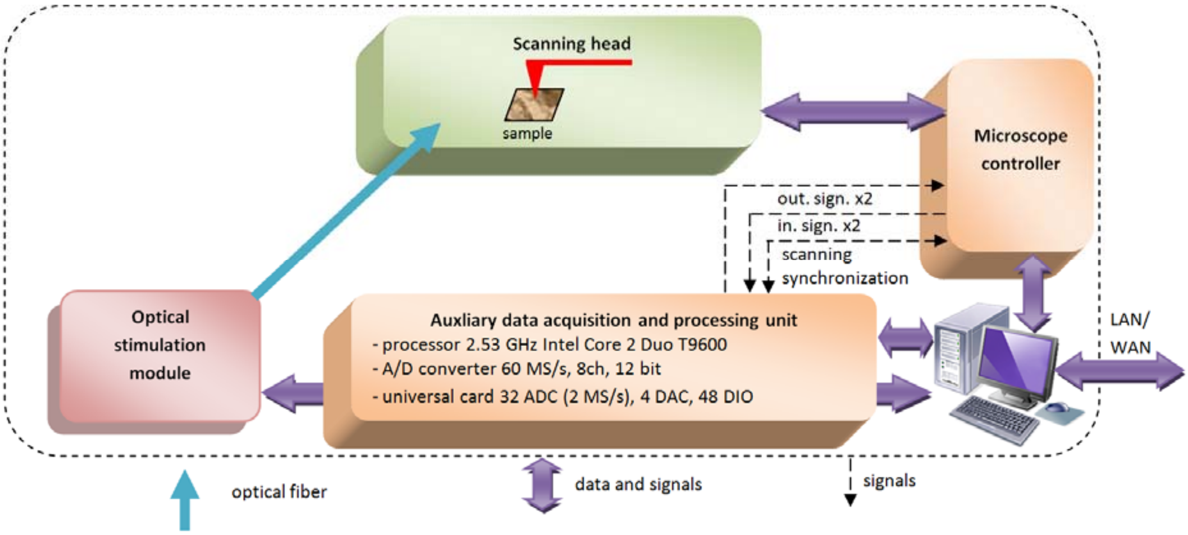


Fig. 2. The simplified diagram of developed measurement setup

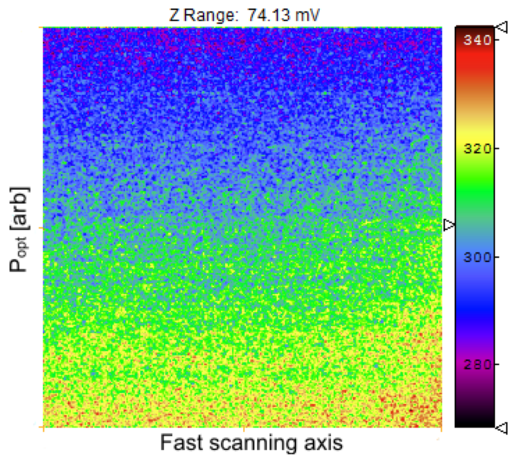
In order to investigate the response of the material in possibly wide range of the visible light, a number of the LED's was used emitting the light at following wavelengths: 390 nm, 460 nm, 525 nm, 624 nm and 940 nm. Additionally, the xenon lamp was used to provide the light spectra similar to the natural sunlight. This approach allows to test the materials for photovoltaic systems in typical conditions. In order to avoid the heating of the scanning head, the optical fiber was used to guide the light to the sample. The user can determine used light source and the light intensity. As in general one expects the series of the measurements for various wavelengths and light intensities, the measurement protocol can be prepared by the user and launched along with the scanning process of small, homogenous surface. The examples of the measurement results are shown in following section.

In order to avoid the illuminating light influence on the probe's deflection laser detection system, the system enables the LED's only when second scan (lift mode) is performed. Therefore the surface profile can be imaged precisely and with minimal risk of artifacts presence.

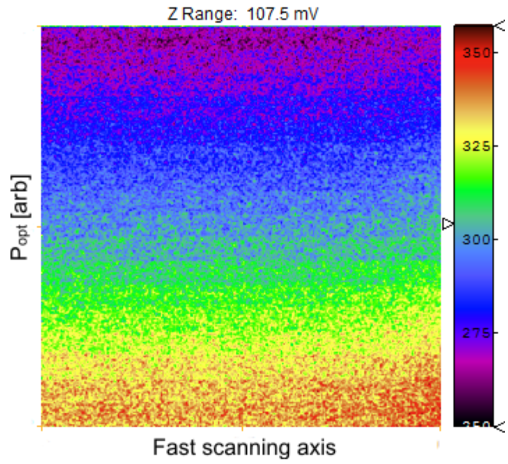
It should be underlined, that due to required repeatability of the measurement conditions, the calibrated photodiode is used to verify if the illuminating light intensity is at certain level. The small footprint of the photodiode allows to place it next to the sample and to verify the light intensity periodically if necessary.

Preliminary measurement results

The test measurements were performed using polycrystalline silicon solar panel. Figure 3 shows to surface potential images acquired at that same spot for two wavelengths: 940 nm and 624 nm. The illuminating power was increased gradually every 30 scanned lines using that same protocol. The acquisition of such amount of data (approx. 15 000 samples per single setting) allows to obtain average value and reduce the impact of the noise. The difference of the response of the surface is clearly visible.



a)



b)

Fig. 3. The surface potential maps acquired for the polycrystalline silicon solar panel using 940 nm (a) and 624 nm (b) wavelengths. The illuminating power was increased linearly every 30 scanning lines

Once the data is acquired as the image, further processing and analysis allows to read the surface potential changes.

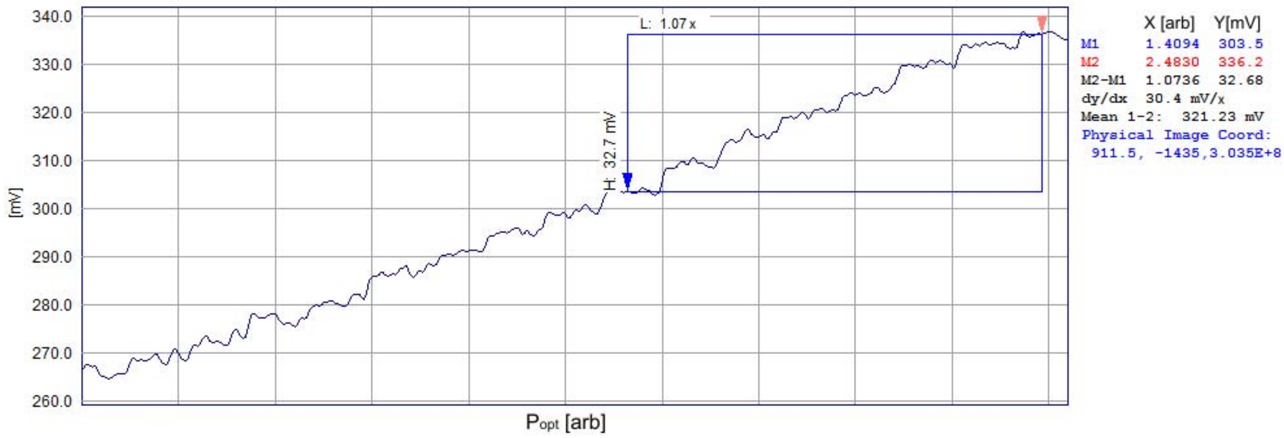


Fig. 4. The profile of the surface potential image acquired for 624 nm on polycrystalline silicon solar panel

It should be underlined, that although the outcome of presented measurements is a 2D map, it does not represent the image of the surface, but the response of the surface to the optical excitation at certain spot. Developed software allows however to perform batch acquisition of a series of surface potential maps along with topography images, where the parameters of the image-to-image optical excitation parameters changes are defined by the user. Figure 4 shows the crossection of the image shown in figure 3 acquired for 624 nm. As the measurement protocol is determined (wavelength, power), one can correlated the outcome with the illumination parameters.

Figure 5 shows the example of the outcome of series of the measurements, which provided the information about the dynamics of the electrical response of the surface to the illuminating light. Moreover, one can also compare the sensitivity to specific wavelengths. It should be underlined, that the system was successfully used in the investigation of the advanced composite material for photovoltaic [17]. Further investigations will be performed in near future.

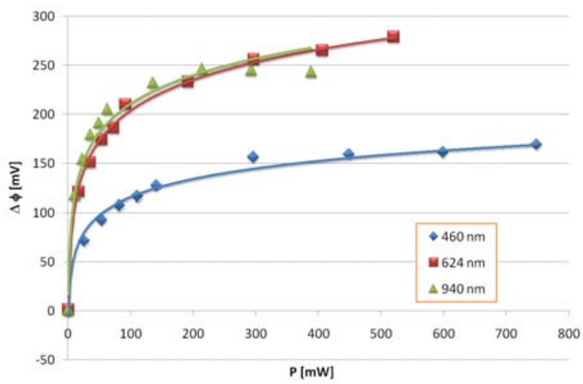


Fig. 5. The graph showing the changes of the electrical properties of the surface due to the light illumination

Summary and outlook

In this work the principles, development description as well as the examples of the test results of the photostimulated Kelvin Probe Force Microscopy are presented. In order to perform repeatable measurements, the solution allows to perform the procedure according to previously defined protocol. Such an approach enables comparison of various measurements data. Such an advantage was possible due to the full integration of the commercial AFM, advanced data acquisition and processing unit as well as the home-made modules.

It should be underlined, that due to the principles of laser-based, optical detection of the AFM probe, one has to take in the account the presence of the 650 nm illumination of the surface, and the artifacts caused by the response of the surface to this illumination. This issue may be however overcome by utilizing the probes with electrical control of the tip-sample interaction force. In such a case the system can work in complete darkness. One can however, in case of the typical detection system, perform the standard measurements for 650 nm wavelength and determine the power of the laser light delivered to the surface. Obtained data would need only the extrapolation towards 0 mW power.

After successful tests, further exploitation of the system is planned in order to perform the investigation of the materials for the organic photovoltaic systems.

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