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SiC/graphene-based test structures for the Kelvin probe microscopy instrumental function determination

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The paper proposes a method for determining the instrumental function for measuring the surface potential in the Kelvin probe microscopy mode. The method is based on the use of SiC samples with regions of single-layer and double-layer graphene as a test structure. The measurement of potential profiles along different directions on such a surface makes it possible to determine the instrumental function for measurements of the potential. Using the instrumental function, one can perform a deconvolution procedure and restore the exact surface potential.

Keywords: Scanning probe microscopy, Kelvin probe microscopy, deconvolution, graphene, silicon carbide.

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Scanning probe microscopy is used widely to probe various properties of the surface of nanostructured materials [1]. The study of surface potential by Kelvin probe microscopy is particularly important [2]. Owing to the long-range nature of the Coulomb interaction, Kelvin probe microscopy (KPM) offers a spatial resolution of 30–100 nm. The specific resolution value depends in a complex manner on the experimental conditions of surface scanning (probe–surface distance, shape and curvature radius of the probe tip, the presence of an adsorbed water layer on the surface, etc.) [3]. In view of this, the lateral dimensions of measured potential features are broadened in KPM surface scanning. In addition, measurements yield erroneously low values of the potential if scanned objects are smaller in size than the spatial resolution. It is relevant in this context to determine the instrumental function experimentally in KPM potential measurements and perform a deconvolution procedure with it [4]. This should help raise the accuracy of potential measurements and improve the resolution of KPM measurements. A special test sample is needed to determine the instrumental function. In the present study, we propose to use the surface of single-crystalline silicon carbide (SiC) with single-layer and double-layer graphene regions as such a sample [5].

The proposed test samples are SiC samples of polytypes 4*H* and 6*H* subjected to high-temperature annealing in a specific regime [6]. This annealing results in the formation of single-layer graphene (1LG) on the surface of the silicon face of SiC; in addition, islands of double-layer graphene (2LG) emerge in certain regions (Fig. 1). Since the work function of double-layer graphene differs approximately by 130 mV from the work function of single-layer graphene, potential „islands“ of roughly the same height form on the surface [7]. These islands have fairly sharp edges that may be used to determine the instrumental function. In order to do this, one needs to perform KPM scanning of a double-layer graphene island and extract profiles of sections of the potential step in different directions (Fig. 1). The measured profiles are convolutions of actual potential profiles $h(x)$ (steps with a sharp edge) and KPM instrumental function $A(x)$ in a given measurement

$$h_{exp}(x) = h(x) * A(x). \qquad (1)$$

Here and elsewhere, the operation of convolution of two functions is denoted by an asterisk.

Differentiating both parts of expression (1) with account for the fact that the actual potential profile is a fairly „narrow“ step, one obtains

$$h'_{exp}(x) = h'(x) * A(x) \approx \delta(x) * A(x) = A(x), \qquad (2)$$

where $\delta(x)$ is the Dirac delta function. Roughly speaking, the derivative of the measured step profile is the section of the instrumental function in a given direction.

Thus, to reconstruct the complete instrumental function, one needs just to obtain a set of potential profiles at different edges of a 2LG island (Fig. 1) and differentiate them. It should be noted that an instrumental function is not necessarily radially symmetric. The reasons for this are as follows: (1) the probe axis is normally tilted by 15–25° relative to the surface; (2) more often than not, the probe shape is not radially symmetric (e.g., pyramidal).

The obtained instrumental function may then be used to perform partial deconvolution of the surface potential in Gwyddion (modular open-source software for data analysis) [8].

A test sample for accurate measurements of the instrumental function should satisfy several requirements. Material layers with different potentials deposited onto the surface need to have sharp edges. It should be noted that 2LG islands on the surface of single-layer graphene on SiC meet this condition.

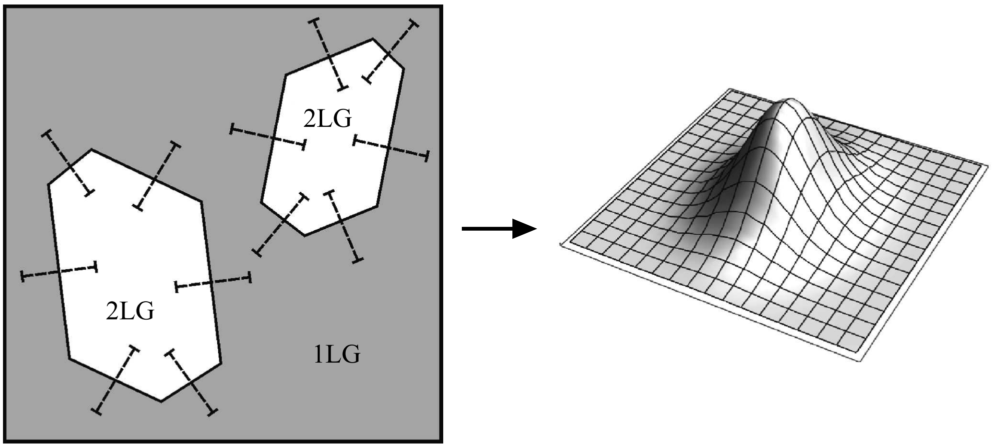

Figure 1. Left panel: schematic diagram of the test SiC sample coated with single-layer graphene (1LG) and featuring islands of double-layer graphene (2LG). Dashed segments denote the positions of section lines for the potential step between single-layer and double-layer graphene. Right panel: schematic image of the instrumental function in KPM potential measurements.

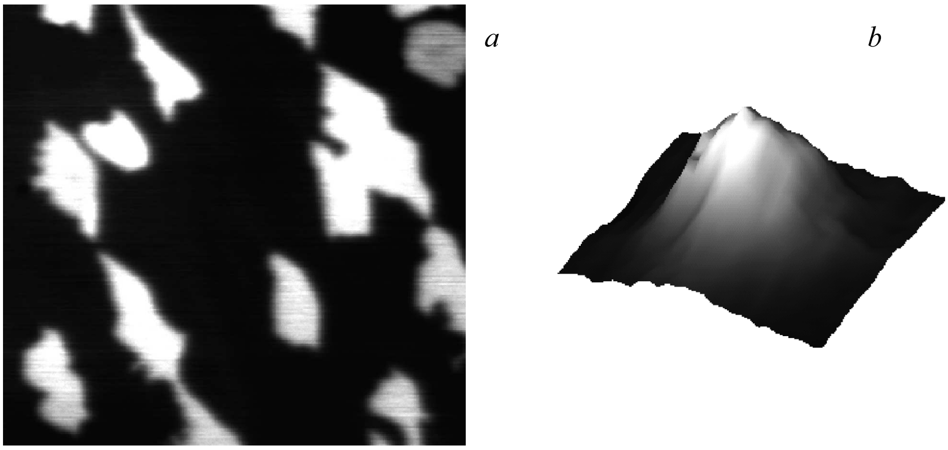

Figure 2. *a* — KPM image of the test sample surface (the field is $4 \times 4\,\mu$m in size) with islands of double-layer graphene (bright regions); *b* — instrumental function that defines the lateral resolution of KPM in measurements with a given probe under given scanning conditions.

On the one hand, 2LG islands should not be too large and fit in their entirety into the scanned region, thus allowing one to compile a set of sections in different directions. Ii ts fair to assume that islands with a characteristic lateral size $D = 500$ nm (or smaller) are the best suited for measurements. A KPM scan with 512×512 measured points and scanning pitch $\Delta = 2{-}4$ nm will then cover an entire island and provide the needed sections with fine discrete sampling.

On the other hand, 2LG islands need not be too small: their size should be multiple times greater than the characteristic lateral resolution of the KPM technique. As was already noted, the resolution of KPM depends on a number of experimental conditions (probe−surface distance, shape and curvature radius of the probe tip, the presence of an adsorbed water layer on the surface) and may vary from 30 to 100 nm. It needs to be mentioned here that a spatial resolution at the nanometer level may be achieved using certain advanced KPM techniques. In the present study, we lease these advanced techniques aside, since they require different types of test structures. Thus, the optimum size of test islands for the standard KPM method is 300-500 nm.

In addition, islands of double-layer graphene should cover a considerable fraction of the surface (so as to eliminate the need to search for an island). Specifically, 30–70% of the surface area need to be covered by double-layer graphene. It is also worth noting that 2LG islands on SiC often turn out to be highly elongated. Their „long“ axis is normally aligned with steps on the SiC surface [6]. Since islands of double-layer graphene on the test structure should not be stretched markedly in any direction, a specific technological growth regime is needed for such samples. Graphene was grown by SiC surface sublimation at a temperature of $1750 \pm 20^{\circ}$C in argon atmosphere under a pressure of 750 ± 20 Torr. Commercial 4*H*-SiC substrates with a

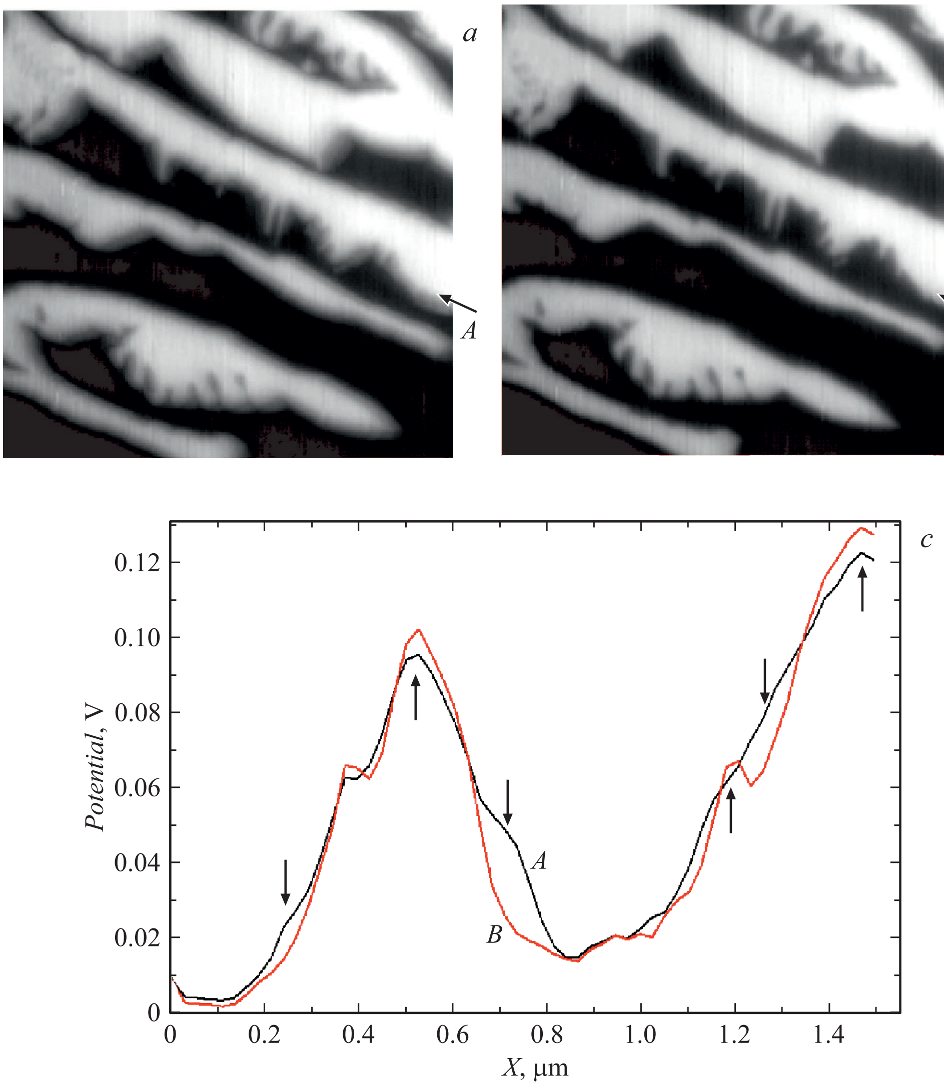

Figure 3. *a* — KPM image of the surface of the studied sample (the field is 4 × 4 μm in size); *b* — KPM image of the same surface after deconvolution; *c* — potential profiles (*A* and *B*) measured at sites indicated with arrows in the top panels. Arrows in the lower panel denote the regions where measured KPM profile *A* differ from profile *B* reconstructed by deconvolution.

(0001) ± 0.25° orientation of the working face were used. These substrates were cut into individual samples 5 × 5 mm in size.

Figure 2, *a* shows the KPM image of the proposed test sample with 2LG islands 300–1000 nm in size on the SiC/graphene surface. Since the distance between 2LG islands is comparable to their characteristic size, it is easy to find a target island at almost any point on the sample. Having extracted profiles in several different directions at the edges of islands, one may then construct an instrumental function (see the example in Fig. 2, *b*). The obtained instrumental function is bell-shaped, and its characteristic width is around $L = 70$ nm. This L value is the lateral resolution of the KPM technique for a given probe and given scanning conditions.

The instrumental function may be used to reconstruct the exact value of the measured KPM potential. The procedure of deconvolution of the measured KPM potential and the instrumental function is applied for this purpose. Figure 3, *a* shows the experimental KPM image, while Fig. 3, *b* presents the image after deconvolution. One may notice that the edges of islands are somewhat diffuse in the initial image, but become sharper after deconvolution. Figure 3, *c* presents the comparison of potential profiles for the initial KPM image (profile *A*) and the KPM image after deconvolution (profile *B*). The approximate site of measurement of these profiles is denoted with arrows in Figs. 3, *a* and *b*. It can be seen that the lateral dimensions of measured features decrease somewhat after deconvolution of the initial image and the instrumental function. These regions are denoted with arrows in Fig. 3, *c*. In addition, the values corresponding to peaks of measured potential features in the initial image are lower than the ones obtained after deconvolution. Specifically, the 2LG potential step height in the right part of the profile became close to 0.13 V, which is the actual potential value in this case, after deconvolution. All of these changes are attributable to the fact that the instrumental function reduces the measured potential values and broadens the measured features in experiments. This problem may be solved in part by measuring the instrumental function and performing a deconvolution procedure.

We note in conclusion that a new type of a test surface for calibration of Kelvin probe microscopy measurements and determination of the KPM instrumental function was proposed. The test surface is a specially annealed SiC sample that is coated with single-layer graphene and features a sufficiently dense surface array of islands of double-layer graphene 300-1000 nm in size. Having measured potential profiles in several different directions on this surface, one may determine the instrumental function for probe-type potential measurements. It was demonstrated that the procedure of deconvolution of the initial KPM image and the measured instrumental function provides an opportunity to improve the lateral resolution of the KPM technique slightly and determine the surface potential more accurately.

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Conflict of interest

The authors declare that they have no conflict of interest.

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