Crystalline Surfaces for Laser Metrology

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Abstract:

The number of methodological recommendations has been pronounced to describe correctly low-dimensional structures up to atomic resolution and to fabricate crystalline surfaces for applicability in nanoscale metrology. Technology of an extremely flat mirror for optical and laser metrology has been demonstrated on the base of crystalline surface. Stepped crystalline surface with controlled steps distribution is a precise calibrator at nanoscale measurements which is suitable for high-resolution techniques. The precision of measurements performed by atomic-force microscopy (AFM) and high-resolution electron microscopy (HREM) for solving problems of metrology and diagnostics of solid nanostructures is discussed.

Introduction

Characterization of surface morphology, atomic structure and geometrical sizes of low-dimensional systems is extremely important for industry and clear understanding of physical nature of nanomaterials for science. Because of the quantum character of interaction between the measurement tool and the measurement object at the nanoscale, it is difficult to distinguish perturbations induced by the measurement process from the measurements proper [1]. Therefore, the characteristics and properties of nanostructures created with the use of nanotechnologies impose specific requirements to measurement tools and metrological support. Such measurement tools have to possess new functional capabilities, extended ranges of measurements, and elevated accuracy, which imposes stringent requirements on traceability. Currently, much attention is focused on improving the metrological certainty of nanotechnologies, in particular, metrologies of linear measurements in the nanoscale range and the development of short-length standards to provide unified measurements of nanoobject sizes. Thus, it is necessary to create primary standards by using high-resolution lithographic technologies and also by using elements of the natural surface relief (for instance, monatomic steps [2]) whose size is traceable to fundamental natural constants (crystallographic parameters of crystals, for instance, of silicon [3]). In the latter case, a problem of certification of natural constants arises, which has not yet been solved.

The priority in basic research in the field of nanotechnologies belongs to methods of scanning tunneling microscopy (STM) and atomic-force microscopy (AFM), which are characterized by a high resolution in the normal-to-surface direction and allow visualization not only of the atomic, but also of the electronic and magnetic structure of the surface. Precise measurements in the bulk of nanostructures and analysis of their atomic structure are performed by methods of transmission high-resolution electron microscopy (HREM). Other in situ electron microscopy methods based on diffraction contrast allow obtaining unique information about the processes on an atomically clean surface in ultra-high vacuum in the temperature range of 20-1300°C, despite their resolution of ~1 nm. An example of such a method is ultra-high vacuum reflection electron microscopy (UHV REM) [4,5]. Direct visualization of self-organization processes proceeding on the crystal surface and in the bulk of the crystal under various actions offers new possibilities for forming nano-objects with a controlled structure and properties.

The objective of the present work is to demonstrate the possibility of using the effect of self-organization of monatomic steps on an atomically clean Si(111) surface to fabricate a precision test object for AFM calibration. A digital analysis of HREM images is used to measure the height of the monatomic step on the Si surface.

Self-organization of monatomic steps on an atomically clean Si(111) surface

The crystal surface declination related to the accuracy of cutting the ingot into the wafers determines the density of steps, which can reach $\sim 10^{10}$ cm$\sim^{2}$ in the best cases. Heating in UHV REM is accompanied by the transition of silicon atoms from the steps to an adsorbed state on terraces and their subsequent evaporation; as a result, the steps move and an atomically clean surface is formed. One of the first attempts to use such a surface for AFM calibration was made back in 1996 [6]. Because of the small distance between the steps and their zigzagging shapes, however, the AFM-measured step height was 0.35 ± 0.03 nm. To obtain a surface with a low density of monatomic steps, one can use the effect of their self-organization, which depends on temperature and direction of electric current passing during heating [4]. The essence of this effect is the formation of bunches (clusters) of steps on the surface, which are separated by almost singular areas with a low density of monatomic steps. Knowing the laws of self-organization and having a possibility of in situ observations in UHV REM, one can control the surface relief at the atomic level and, as will be demonstrated below, create test objects for calibration of *z*-measurements in AFM without using lithographic technologies.

When a required distribution of step bunches and monatomic steps on the Si(111) surface was obtained, the sample was taken out from the UHV chamber and was studied in air by means of AFM [Solver P-47H and Ntegra (NT-MDT)] with the use of ordinary cantilevers. When the sample is brought into the air, the atomically clean surface is covered with a natural oxide layer whose thickness increases from ~0.7 to ~5.0 nm during several years. The oxide layer is assumed to reproduce well the monatomic step height [7], though this height has never been quantified by direct structural methods.

HREM application for measuring the height of the monatomic step

To measure the monatomic step height by the HREM method and ensure good accuracy of its replication by the oxide layer, we used cross sections of a step-bunched surface prepared by a standard technology of gluing of two frontal surfaces and subsequent etching of the thin glued samples with a focused ion beam. The HREM measurements were performed by a JEM-4000EX electron microscope with an acceleration voltage of 400 keV, which has a point-to-point resolution of 0.16 nm and a line-to-line resolution of 0.10 nm. For the oxide step to be visualized, the sample surface was additionally covered by a Ti layer 9 nm thick prior to its cross-sectional cutting. The study shows that visualization of the monatomic step on the Si surface (Si-step) and oxide strongly depends on the thickness of the region transmitted by electrons. The former is well seen in an extremely thin sample (~10 nm), whereas the latter can only be observed in a thick sample (~30 nm) owing to the difference in electron absorption in the oxide and titanium films. Moreover, in the latter case, there appears a contrast jump on the Si-step because of the extreme smoothness of the Si-oxide interface and the presence of weak strains; therefore, the step height can be measured. It is seen from Fig. 1 that the Si-step height corresponds to the distance between the (111) planes in the bulk of the Si sample with accuracy that can be reached in a routine analysis of HREM images.

A digital analysis of the HREM image taken in a thick crystal allowed us not only to improve the accuracy of Si-step height measurement, but also to measure the oxide step height [7]. The power spectrum (Fast Fourier Transform) of the HREM image of the Si crystal shows that the lattice parameters of Si can be measured within ±0.001 nm, i.e., the heights of both steps on the Si surface and in the oxide layer can be measured with the same accuracy. On the intensity profiles taken on the left and on the right of the step, normal to the surface, the oxide layer is visualized in the form of a high double peak with a subsequent cave corresponding to the oxide-Si interface location. Therefore, the shift of these peaks on the superimposed profiles shows the height of the monatomic step, which is equal to 0.314 ±

0.001 nm. Note that the small peaks induced by the crystalline structure of Si on the superimposed profiles coincide with high accuracy. The measured results testify that the height of the monatomic steps on the Si surface and in the oxide layer coincide within the measurement accuracy.

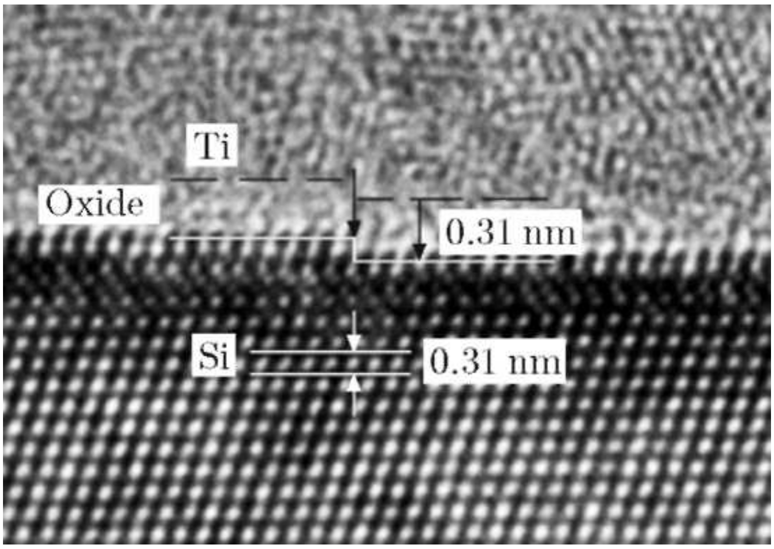

Fig. 2: HREM image of the monatomic step on the Si(111) surface in a thin sample.

Ensuring traceability of AFM measurements at the nanoscale

Reduction of the characteristic sizes of the calibration measure to several nanometers or fractions of a nanometer considerably complicates creation of a periodic relief by lithographic methods with sufficient accuracy and reproducibility, especially in the *z* direction. Typically atomic steps formed on the surface of crystalline graphite or mica due to spalling or stratification is usually used for *z* measurements in the nanometer and subnanometer ranges. Spalling, however, does not allow regular reproduction of step arrays with a given distribution and large atomically flat terraces. As was shown above, the use of the self-organization effect on an atomically clean Si(111) surface during heating in UHV REM allows creating surface areas with a given density of monatomic steps and the distance between them (tens of microns) whose height is 0.314±0.001 nm, including situations after the natural oxide film is formed.

Figure 2a shows a three-dimensional AFM image of the step-bunched surface, which was obtained in the phase shift (phase contrast) regime. The enlarged topographic AFM image of monatomic steps on a smooth terrace in the frame indicated by the dashed line and a corresponding spectrum of heights are shown in Figs. 2b and 2c. The size of the scanning area in AFM was approximately 5 *x* 5 µm, and the average error of monatomic step height measurement was 0.001 nm (~0.3%).

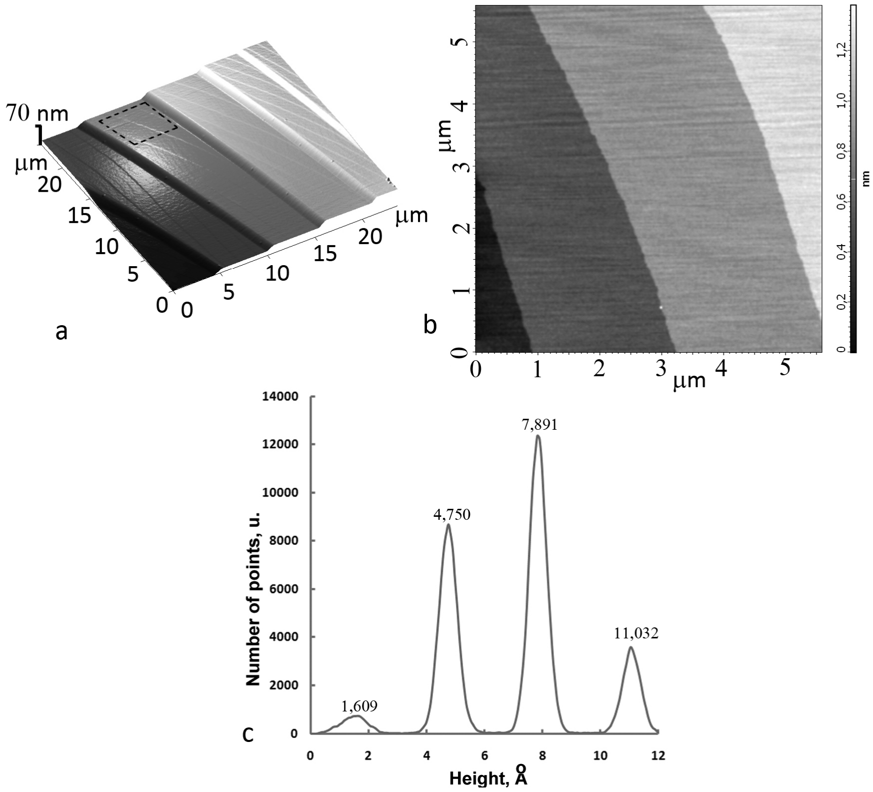

Fig. 2: AFM diagnostics of the Si(111) surface: (a) phase contrast of bunches (large steps) and monatomic steps (thin lines); (b) AFM image of monatomic steps separated by atomically flat terraces 2 μm in wide; (c) statistical distribution of points over the height.

Using self-organization effects, one can create step bunches with a countable number of closely located monatomic steps, which, as a whole, can be used for AFM calibration. The height of such a step bunch is multiple to the number of monatomic steps in it; if the height of an individual step is known, the height of such a step bunch can be measured by the AFM method with high accuracy. As an example, we determined the height of a bunch containing 28 steps with an estimated theoretical height of 28x0.314=8.792 nm. The AFM measurement yielded 8.82 nm, i.e., the measurement error was 0.34%. At the moment, small lots of test objects on the basis of the step-bunched Si(111) surface are fabricated for AFM calibration in the range from 0.3 to 100 nm.

For on-line control the instrumentation facility, we have developed another test-object the nanoscale. This test object is also based on step-bunched Si(111) surface but additionally contains negative islands of 0.08 nm of deep at flat singular areas between monatomic steps [8]. Such negative islands can be prepared in ultrahigh vacuum reflection electron microscopy by rapid cooling (quenching) of specimens from high temperature and then visualized by AFM. This nano-relief is controlled by vacancy generation in the regions where the (7×7) superstructure forms and accompanied by additional disordered layer formation at surface areas not undergoing structural reconstruction. Visualization of triangular islands at the quenched Si surface characterizes the ability of routine AFM techniques to measure nanoobjects 0.1 nm of height**.**

Conclusion

Self-assembly effects occurring at atomically clean surface during high temperature anneals provide the formation of ordered arrays of monatomic steps assembled by step bunches or almost singular surface areas with widely spaced monatomic steps suitable for the calibration of optic and laser interferometer and atomic force microscopes. The monatomic step height at the silicon (111) surface was attested by the high resolution transmission electron microscopy followed by software analysis and found to be equal to interplanar spacing (0.314 nm) in the volume of silicon crystal with ±0.001 nm of accuracy. Monatomic step is replicated with the same accuracy by native oxide film covering the stepped silicon surface thus making available precise AFM calibration at the nanoscale at ambient conditions. Over the last few years, the test object developed on the basis of monatomic steps is used for calibrating scanning probe microscopes The traceability of such surface measurements is discussed.

[1] Gotszalk T.P et al., Optica Applicata, 2007 XXXVII, No. 4, 397
[2] Fedina L.I. et al., Optoelectronics, Instrument. and Data Processing, 46 (2010) 301
[3] Martin J et al Metrologia 35 (1998) 811.
[4] Latyshev A.V. et al. Surf. Sci. 213 (1989) 157.
[5] Yagi K. Surf. Sci. Rep. 17 (1993) 305.
[6] Suzuki M. et al J. Vac. Scie. Technol. A14(3) (1996) 1228.
[7]. L.I. Fedina et al,. Meas. Sci. Technol. 21 (2010), 054004, pp. 1-6.
[8] D.A. Nasimov et al, Phys. Low-Dimens. Struct. 3/4 (2003) 157.