Metrological characterization of nm-range dynamic etalons using a heterodyne interferometer

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Abstract. Test structures used for calibration of scanning probe microscopes have certain limitations. They are short-lived, their work surface becomes coated with microparticles over time, and it gradually wears out by contact with the measuring probe, resulting in the etalon geometry deformation. Dynamic etalons allow calibrating SPMs in ranges from pm to nm. In this article we present the results of dynamic etalon metrological characteristics research using an SPM equipped with tree-coordinate heterodyne laser interferometer. Obtained data indicates stability of piezoelectric modulus and absence of piezoelectric hysteresis phenomena in the etalon samples used.

1. Introduction

Reaching the subnanometer resolution and accuracy of the dimension measurements during topography characterization of micro- and nanostructures is provided by using scanning probe (tunneling, atomic force) and scanning electron microscopies [1]. The main problem regarding linear dimensions measurement in the nanometer range is associated with intricate and often ambiguous relationships between the measured object and its image in the scanning microscope [2]. The recorded image does not definitely agree with the real profile of the measured surface topography element of the object. At present, various topography etalons of the geometric dimensions (height, period) remain the only possible means of calibration of scanning probe microscopes (SPMs). They represent an array of periodic structures of specific geometrical shapes mounted on the surface of a silicon single crystal square plate, its surface oriented parallel to the crystallographic plane (100) [3]. The transfer of the length unit from etalon to SPM is carried out by comparing the results of etalon surface profile measurements using the device being calibrated, with the etalon technical data as measured by the metrological instrument (device equipped with a laser interferometer) [4]. However, such etalons are short-lived. Their work surface becomes coated with microparticles over time, and it gradually wears out by contact with the measuring probe, causing changes in the etalon’s geometric dimensions. Besides, the measurement results of surface profile always depend on the shape of the probe, since the image obtained using a scanning probe microscope (SPM) represents convolution of the profile surface of the probe and the surface of the topography etalon.

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Instead of the static ones, the so-called dynamic action etalons may be used that are free from these drawbacks. Dynamic etalons of length unit allow to simplify the calibration procedure of SPMs and greatly enhance the accuracy of the results. The principle of dynamic action is based on the controlled movement of the surface of material with inverse piezoelectric effect, under the influence of a regulated voltage. This etalon allows us to replace a set of static measures, producing the specified required displacements in the range from tens of microns to tens of picometers. They are durable, and their operation does not require any special shape of the probe tip. Due to high resonance frequencies (over 10 kHz), dynamic etalons can measure the SPM response time and the dynamic parameters of its nanopositioning system. However, just as any static etalon, the dynamic one requires calibration using a metrological SPM. This paper presents the results of metrological characteristics study of dynamic action etalons carried out by means of a heterodyne laser interferometer.

2. Dynamic etalon of geometric dimensions for scanning probe microscopes calibration

At present, two types of dynamic (tunable) etalons have been well-studied. They differ in the active piezoelectric material: piezo-ceramic etalons [5] and single crystal piezomaterial ones (typically, lithium niobate) [6]. Both these materials change their dimensions when the control voltage is applied. The advantage of ceramics is that the piezoelectric modulus (factor of voltage-displacement conversion) is tenfold that of the single crystal, i.e. for the same magnitude of displacement of the etalon’s surface, the applied voltage for ceramics would be less. Disadvantages of the ceramic etalon are the instability of the piezoelectric modulus and the dependence of deformation on the electric field frequency. Piezo-ceramics always has polarizing noise due to fluctuations in the electrical domain dipoles. Ambient temperature, heating of ceramics during operation, and mechanical stresses also affect the voltage-displacement conversion. Etalons based on a single crystal have more stable piezoelectric modulus, while the voltage-limiting restrictions can be eliminated through design modification, in particular, by using the multilayer structures that significantly extend the range of attainable displacements. In the present study we investigated the characteristics of the etalons made of single-crystal piezo-ceramics.

Figure 1 presents a schematic design of dynamic etalons of vertical and horizontal displacement. Type of displacement depends on the relative orientation of the crystallographic axes and the external electric field. A single crystal is mounted between two platforms, one of which serves as a carrier, the second being the reference.

![Figure 1. Schematic design of vertical and horizontal displacement dynamic etalons](image)

When calibrating the SPM etalon of vertical type, it is located on a microscope table, and SPM probe is brought to its reference surface. Control voltage is applied to the etalon with a certain magnitude, and the etalon’s surface is displaced by a known amount, which is measured using SPM servosystem. Etalon of horizontal displacement has an array of reference marks (typically a series of parallel submicron grooves) on its surface. During operation, the etalon surface moves and SPM scanning system records the movement of the reference marks with respect to their initial position. Values obtained from SPM are compared to the corresponding calibration data of etalons. In this way SPM measurement channels are calibrated using dynamic etalons.

3. Heterodyne laser interferometer NanoScan-3Di
The research of characteristics of the single-crystal piezoelectric dynamic etalon was conducted in the measuring SPM NanoScan-3Di [7]. This device ensures the accuracy of the data through the use of hard piezo-resonant probes and accommodation of a three-channel heterodyne interferometer that controls movement of the piezoelectric table. The radiation source in the interferometer is a frequency-stabilized He-Ne laser with a power of 1mW, and a relative instability of wavelength $10^{-9}$ for 8 hours. Dimensions of the optical interferometer unit are $260 \times 260 \times 60$ mm, which allows embedding it inside the commercially available SPMs, including SPM NanoScan-3D [8].

Special design features including placing the triple-prisms on the moving axes of table in three orthogonal planes allowed minimizing Abbe error that is typical for measuring interferometers [2]. Reducing the influence of temperature fluctuations is achieved by removal of the laser radiation source outside the optical scheme [9]. Interferometer allows measurements in the range of $100 \times 100 \times 10$ mm along the axes X, Y, Z, respectively, with a resolution of 0.01 nm.

4. The results of dynamic etalon characterisation

This work deals with a study of the metrological characteristics of dynamic etalons of vertical and horizontal displacement. Dynamic etalon was placed on the surface of a nanopositioner PI P-733.3 XY(Z) [10] of metrological probe microscope NanoScan-3Di. Scanning probe of SPM was brought to the surface of dynamic etalon, and the automatic control system was activated to maintain the contact of the probe tip with the etalon surface. After that, the pulsed control voltage of rectangular or trapezoidal shape with adjustable magnitude and period was applied to the etalon. Several series of measurements were performed for the applied control voltage in the range 1–1000 V. The etalon surface was measured using the interferometer. The precautions of acoustic and thermal insulation of measuring SPM greatly reduced phase noise in the interferometer that was determined by the temperature drift of the structure and by the fluctuations of the refractive index of air in the measuring arms. Measurements of dynamic etalons were carried out in a special enclosure box after continuous thermostatting for 3–6 hours. In this manner, monotonic drift of the SPM piezoelectric table positioning was eliminated, and phase noise associated with the convective air flow inside the box was less than 2 nm.

For example, figure 2 demonstrates the time-resolved movement of a dynamic etalon at a voltage of 100 V when the displacement range was 74.56–75.30 nm.

![Figure 2. Time-resolved movement of a dynamic etalon at a voltage of 100 V](image)

Figure 3 demonstrates the displacement of the etalon’s surface when a voltage of 10 V was applied. Here, the displacement range was 0.95–1.23 nm.
Data obtained by the interferometer was processed using an algorithm involving gradient filtering based on the convolution with the second derivative of the Gaussian function, and the extraction of areas corresponding to the maximum and minimum coordinates of the etalon’s surface position from the original data set. Figure 4 shows the resulting data set obtained after applying the processing algorithm.

The histogram distribution of maxima and minima of the etalon’s positions was then built on the basis of acquired data. The displacement of the etalon’s surface is defined as the algebraic difference between the values of the histogram peaks corresponding to the maximum and minimum position of the etalon (see figure 5).
Using the above mentioned algorithm, we obtained the displacements of etalons for different control voltages applied. For voltage 100 V the displacement was 73.91 ± 0.41 nm (see figure 3), and for 10 V it was 0.97 ± 0.13 nm (see figure 4). The displacement plots of two etalons’ surfaces as functions of applied voltage in the range 100–1000 V are shown in figure 6. These measurements were performed both for increasing (black curves) and decreasing (red curves) voltage.

Analysis of the curves shows that the dependence of displacement on the applied voltage for samples no. 1 and no. 2 can be approximated with a function of \( y = kx + b \). Factor \( k \) stands for the sensitivity of the etalons to the control voltage and is expressed in pm/V. Sensitivity of etalon no. 1 was 196.2 ± 8.5 pm/V, and of the etalon no. 2 — 70.2 ± 1.3 pm/V. Sample no. 1 examined with the increasing voltage had \( k_i = 196.4 ± 8.5 \) pm/V, and with the decreasing voltage — \( k_d = 195.8 ± 2.1 \) pm/V. Meanwhile, for sample no. 2 these values were \( k_i = 70.8 ± 1.3 \) pm/V and \( k_d = 69.5 ± 0.4 \) pm/V, respectively. The relative discrepancy of the factor \( k \) may be defined as \( K = (k_i - k_d)/k_d \) and used to estimate the nonlinearity of piezoelectric material. For sample no. 1 it is 0.4%, and 1.8% — for the sample no. 2. These values do not exceed the random error in determining the slope coefficient. Hence the results show that the piezoelectric material of the samples does not exhibit hysteresis effects.
5. Conclusion
The metrological SPM NanoScan-3Di equipped with three-axis heterodyne interferometer was used to study the characteristics of two single-crystal dynamic etalons of vertical displacement. The use of a heterodyne laser interferometer allowed us to calibrate etalons in the nanometer range with an accuracy of 0.1 nm. The dynamic range of the test etalon samples was 200 nm and 80 nm. Dependencies of the etalon’s surface displacement on the control voltage were obtained for increasing and decreasing voltage magnitude. The obtained trends were approximated with functions $y = kx + b$, resulting in sensitivity of the etalons $196.2 \pm 8.4 \text{ pm/V}$ and $70.2 \pm 1.3 \text{ pm/V}$, and non-linearity of the etalons 0.4% and 1.8%, respectively, indicating the stability of piezoelectric modulus and the absence of piezoelectric hysteresis phenomena.

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