Reference gage calibration methods of probe nanometry systems

S S Antsyferov, D A Karabanov, K E Rusanov and K N Fazilova

MIREA - Russian Technological University, 78 Vernadsky Ave., Moscow, 119454, Russia

1 E-mail: fazilova@mirea.ru

Abstract. Reference gage calibration methods of probe nanometry systems are considered in this work. Existing methods for calibrating reference standards provide high measurement accuracy, but at the same time are distinguished by a high complexity of practical implementation, in particular, the interferometric method, or low accuracy, but with simple practical implementation. Therefore, the urgent task is to develop a calibration methodology that provides a sufficiently high measurement accuracy with a relatively simple practical implementation. The paper proposes a methodology based on the combined use of two methods: a comparative assessment of the step heights of the calibrated gages using one of the probe nanometry systems and the precision measurement of the step height of one of the calibrated gages by computer three-dimensional reconstruction of images in a scanning electron microscope. The components of the measurement uncertainty arising when determining the height of the steps of the calibration standards are investigated. The calculated value of the expanded uncertainty showed that the achievable measurement accuracy is comparable to the accuracy of the interferometric method with a simpler practical implementation. Practical testing of the proposed methodology has shown that the use of calibration techniques developed on its basis provides high accuracy and reproducibility of the results obtained.

1. Introduction

Probe nanometry (SPN) systems are widely used to observe and measure the parameters of nanostructured objects. Probe nanometry systems perform the functions of not only observation, but also measurement of the parameters of nanostructured objects. Due to this, they need appropriate metrological support and, first of all, methods and technical means of calibration of reference gages [1-8]. It should be noted that the existing methods for calibrating reference standards are often difficult to use, laborious, time-consuming and do not provide the required accuracy in some cases.

Currently, the main methods for calibrating reference calibration standards are the direct measurement method using the AFM of the Solver PRO type and the interferometric method based on the combination of the AFM with a laser nanodisplacement meter (figure 1).
The main advantages and disadvantages of these methods are shown in table 1.

Table 1. Advantages and disadvantages of existing calibration methods for reference standards.

| Advantages                        | Direct method | Interferometric method                                      |
|-----------------------------------|---------------|-------------------------------------------------------------|
| • ease of use.                    |               | • high accuracy (the step height of the measure is about 500 nm is determined with an error of ± 2 nm). |
| • low accuracy (the step height of the measure is about 500 nm is determined with an error of ± 20 nm). |               | • the complexity of conjugation of 3 measuring instruments (AFM and 2 interferometers, which control the movements along the X and Z scanning axes); |
|                                   |               | • failure of the given operation (combination) by the manufacturer; |
|                                   |               | • significant dimensions of the devices used; |
|                                   |               | • the complexity of meeting the requirements of parallelism of the displacements of the measuring mirrors of the interferometers to the scanning axes of the AFM due to the effect of deformation of the geometric shape of the piezoscanner; |
|                                   |               | • long duration of calibration operations.                  |

The purpose of the work is to develop a methodology for calibrating reference gages that provides high measurement accuracy with a relatively simple practical implementation.

2. Methodology for the calibration of reference gages
The proposed methodology (figure 2) is based on the methods of preliminary comparative assessment of the step heights of the calibrated gages and precision measurement of the step height of one of the calibrated gages.
The comparative assessment method provides the following procedure:

- Measurement of the step height, calibrated gages and step height of the reference gage using a stylus profilometer.
- Determination of the ratios of the step heights of the k-th calibrated gages with the step height of the comparison gage

$$\gamma_k = \frac{h_k}{h}$$  \hspace{1cm} (1)

The precision measurement method is based on the operation of three-dimensional reconstruction of images in SEM. At the same time, in order to achieve high accuracy in determining the height of the step, a 2 nm thick gold film, which has an island character, is applied to its lower and upper planes by magnetron sputtering in vacuum (figure 3).
This method involves performing the following operations:

- Spraying of a reference measure of gold particles on the surface in order to create reference points on the surface of the gage.
- Formation of images of the reference gage using SEM with the slope of the measure ±α.
- Measurement of the distance between the reference points l of the obtained images (figure 4)

\[
l = s \sum_{i=1}^{N} (n_1 - n_2)_i \frac{1}{N}
\]

where S is the pixel size;

\(n_1-n_2\) is the difference in the values of coordinates along the Y-axis of some point \(P_i\) (figure 4);

\(N\) is the number of measurements.

- Determination of the height of the reference gage

\[
H_{ref} = \frac{l}{2 \sin \alpha}
\]

As a result, the height of the calibrated gages is determined according to the following relationship:

\[
h_k = \gamma_k \times H_{ref}
\]
Figure 4. Scheme for determining the step height of the reference gage.

The uncertainty of measurement arising in determining the height of the steps includes the components indicated in table 2.

Table 2. Uncertainty components of measuring the height of reference gages steps.

| Component                                                      | Value   | Assessment type | Number of degrees of freedom (n-1) |
|---------------------------------------------------------------|---------|-----------------|-----------------------------------|
| Relative standard measurement uncertainty due to inaccuracy in determining the increase in SEM during its calibration, $\mu_M$ | 0.001   | A / B           | -                                 |
| Relative standard measurement uncertainty due to inaccuracy in determining the angular position of the goniometric stage, $\mu_\alpha$ | 0.001   | A / B           | -                                 |
| Relative standard measurement uncertainty for comparison on a stylus profilometer, $\mu_K$ | 0.0011  | A               | 29                                |
| Relative standard parallax measurement uncertainty, $\mu_p$   | 0.001   | A / B           | -                                 |
| Relative standard deviation of the arithmetic mean of the height of the calibrated standard, $\mu_{em}$ | 0.0004  | A               | 29                                |
| Relative standard deviation of the arithmetic mean height of a reference gage determined by a precision method, $\mu_{mp}$ | 0.0026  | A               | 29                                |

As a result, the expanded measurement uncertainty when determining the height of the steps of the calibrated gages (the nominal value of the heights is 475 nm) with the coverage factor $k=2$ ($P=0.95$) is calculated by the formula:

$$\mu = 2H_{ref} \sqrt{\mu_M^2 + \mu_\alpha^2 + \mu_K^2 + \mu_p^2 + \mu_{em}^2 + \mu_{mp}^2} = 3.2 \mu_m$$  

(5)
Thus, a methodology for calibrating reference gages has been developed, based on the comparison of signals obtained by the probe nanometry system and on the precision estimation of the step height of the reference calibration gage by means of computer three-dimensional reconstruction of images in SEM. The obtained value of the expanded measurement uncertainty arising in the calibration of reference gages shows that the accuracy of the proposed methodology for calibration of gages is comparable to the accuracy of the interferometric method.

3. Conclusion
The proposed methodology provides higher quality indicators in comparison with the known methods: a significant reduction in measurement time, simpler practical use, sufficient measurement accuracy, lower cost.

Practical approbation of the methodology has shown that the use of the methods developed on its basis provides high accuracy and reproducibility of the results obtained, the possibility of calibrating a wide class of SPM and high-performance indicators of the calibration processes.

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