Features of combining scanning probe microscope with optical and scanning electron microscopes

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Abstract. The designs of combining a scanning probe microscope (SPM) with an optical microscope (OM) or scanning electron microscope (SEM) have been presented. To reduce the size of the SPM units the self-sensing probes and a piezo-inertial displacement system are proposed to use. The peculiarities of working of such SPM units are discussed. The examples of visualization of different nature objects in combined systems SPM-OM and SPM-SEM have been presented.

1. Introduction
There are two main experimental approaches to visualizing the structure or mapping the properties of objects with nanometre spatial resolution. In one case, a focused beam of particles or radiation interacts with the sample under study. The beam source is far from the sample and the result of beam-sample interaction is detected. Otherwise, a sharp solid-state probe interacts with the sample under study. In this case, a sharp solid-state probe is located near the surface on a distance \( \lambda < \lambda \), where \( \lambda \) - the probe-sample interaction decay length, and the probe-sample interaction is detected. Since the interaction of focused beams and sharp probes with the sample surface has a different nature, the combination of different experimental “beam” and “probe” approaches in one device [1-5] provides more complete information about the object under research.

2. Combined system of coarse approach and scanning
It is obvious that the compact design of the scanning probe microscope (SPM) assembly is optimal for combining it with an optical microscope (OM) or scanning electron microscope (SEM). To reduce the size of the SPM assembly, the commonly used mechanical design based on a stepper motor with complicated mechanical components has been excluded from the coarse approach system of the probe with the sample. Instead of a system on the base of a stepper motor, a simpler system on the base of a 1D piezo-inertial driver has been used.

In the Figure 1a, the principal scheme of 1D a piezo-inertial motor is presented. A stack multilayer piezo actuator (2) is attached to the fixed base (1) on one side. A titanium guide rod (4) is attached to the opposite side of the stack multilayer piezo actuator. The titanium carriage (3) is pressed against the surface of the titanium guide rod (4). Asymmetric electric pulses (Fig. 1b) with a sharp and smooth front are applied to the piezo actuator (2). Under the action of a sharp front, the piezo actuator (2) sharply stretches or contracts depending on the sign of the electric voltage. In this case, the inertial force acting on the carriage (3) is greater than the friction force acting between the surface of the guide rod (4) and the surface of the carriage (3). As a result, the carriage (3) slides along the surface of the guide rod (4). Under the action of a smooth front, the friction force is greater than the inertial one and...
slippage does not occur. The up or down moving direction of a carriage can be reversed by changing a sign of control voltage. Work reproducibility of a piezo motor with such design strongly depends on the quality of polishing of the titanium surfaces of friction pair. We applied mechanical polishing of titanium surfaces to the “orange peel” state.

The magnitude of the single step of the carriage movement depends on the force of the carriage pressing against the guide rod and the amplitude of the control pulse. It is clear that the application of a smoothly varying voltage to the piezo actuator provides a smooth movement of the carriage together with guide rod. In our case, the range of smooth movement (1D scanning) is ~7 μm, with a range of step by step movement of ~10 mm. On the carriage of the piezo motor, a probe sensor or a piezo tube scanner with a sample holder or a probe sensor were mounted. Also, it is clear that by combining 1D piezo motors it is possible to create 2D and 3D piezo drivers. Since the piezo motor can operate both as a linear stepper motor and as a smooth scanner, it is possible to use them both for coarse approach of the tip to the sample and for stabilization of the interaction between tip and sample in the constant force mode, when the SPM feedback loop is closed. Therefore, breaking and closing the feedback loop between the consecutive steps of the piezo motor, you can measure the step size by measuring the corresponding displacement of the scanner (Fig. 1c). When the guide rod is vertically positioned, the step up is smaller than the step down due to the action of gravity (Fig. 1c). As can be seen from Figure 1d, the average step of the piezo motor is ~ 100 nm and the minimum and maximum steps are ~ 50 nm and 150 nm, respectively.

3. Probe sensor
To reduce the size of the SPM unit, the quite bulky optical circuit usually used to measure the deflection of the cantilever has been excluded also from the SPM units design intended for SPM-OM and SPM-SEM combinations. Instead of usual Si cantilever, two types of self-sensing probe sensors have been used. One probe is a tuning-fork sensor fabricated from two piezo tubes with replaceable W tip [6] and another is a Si piezoresistive cantilever sensor [7].

The advantage of such kind of probe sensors compared with a standard Si cantilevers with optical registration of cantilever deviation is absence of any alignment after the probe’s substitution. Tuning-fork sensor on the base of piezo tubes and replaceable W tip has a Q factor of about 30, while Q factor of Si piezoresistive cantilever is about 200. Therefore, tuning-fork sensor has a smaller sensitivity compared with Si piezoresistive cantilever.
Nevertheless, the sensitivity of tuning-fork sensor on the base of piezo tubes is enough for many experimental cases. For example, the tuning-fork sensor with W probe is preferable to a Si cantilever when operating in dynamic force nanolithography mode. From the practical point of view, a sensor on the base of piezo tubes with replaceable W tip is very convenient, because the users can fabricate the W tip themselves by electrochemical etching of tungsten wire with (100-150) μm diameter.

It should be note that at small tilt angle of cantilever (~10 degree) relative to the sample surface, which usually installed for a standard cantilever and at a standard value of a reference signal on input of feedback loop of SPM, the amplitude of cantilever oscillations begins decreasing earlier than a needle starts tapping on the sample surface. We attribute this fact to the action on the cantilever of an air flow reflected from the sample surface. Indeed, the area of the piezo cantilever is larger than that of a conventional cantilever, since the Wheatstone bridge is located on its surface. It is experimentally found that if a reflected air flow entering the cantilever surface is decreased by increasing a tilt angle between sample surface and cantilever plane (up to 40 degrees), this effect disappears. This effect also disappears in a vacuum. Therefore, when working with a piezo resistive cantilever in tapping mode, it is necessary to select a reference signal for the feedback loop different from the signal corresponding to the case of a conventional cantilever. In this case, the operating point on the probe-sample approach curve should correspond to a larger value of the decreasing of the cantilever’s oscillations amplitude.

4. SPM-OM and SPM-SEM schemes

Figure 1e shows a scheme of SPM unit combined with the OM lens. Figure 1f shows a scheme of the SPM unit located on the goniometer of the SEM under the pole piece of the SEM objective electron lens. The gap between sample surface and the pole piece of the objective lens is 7 mm. In the case of a combination of SPM-OM (Fig. 1e), the probe moves in the (X, Y) plane under the OM light beam using screws. It is sufficient to use only 1D piezo-inertial mover to capture the interaction between the probe and the sample. To choose the place on the sample, a 2D OM stage is used. The piezo tube is used for scanning by the probe at X, Y directions. The stack multilayer piezo actuator is used both for fine scanning by the probe at Z direction and for stepwise coarse approach of the probe to the sample. In the case of a SPM-SEM combination (Fig. 1f), a 2D piezo-inertial mover is used to move the probe under the electron beam. To select a region on the sample surface, the SEM goniometer is used. In this case, the piezo tube is used to scan the sample at the X, Y directions, and the stack multilayer piezo actuator is used both for fine scanning the sample in the Z direction and for coarse approach of the sample to the probe using the 1D piezo-inertial mover.

**Figure 2.** Probe sensor displacement along the Z direction during scanning: 1 – piezo scanner (X, Y) in the form of piezo tube with length L, 2 – probe sensor with length l, 3 – compensating piezo actuator in the form of bimorph membrane, α – small angle of piezo tube displacement during scanning, x_{12} – the displacement of the end of the piezo tube along the X direction, h – the displacement of the end of the probe sensor along the Z direction.
It should be noted that for scheme shown in Figure 1e, a gap between the tip and sample surface is increased during scanning. Indeed, in frame of rough geometrical model, it can be assumed that the free end of the piezo tube (1) moves around a circle of a large radius and probe sensor (2) rotates at a small angle $\alpha \sim x_{12}/L_p$ during scanning, as it is shown in Figure 2, where $x_{12}$ is the probe displacement during scanning from point 1 to point 2 in X direction, $L_p$ is the distance from the attachment point of the probe to the centre of the circle being approximately equal to the length $L$ of the piezo tube (1). As a result, the gap between the tip and sample surface is increased by the amount $h \sim x_{12} l/L$, where $l$ is the probe sensor length. For example, at $L \sim 30$ mm, $l \sim 10$ mm, and $x_{12} \sim 20$ µm, the gap increasing $h$ will be $\sim 7$ µm. This deviation is compensated by SPM feedback loop and looks like a slope on the SPM image. Of course, this slope can be deleted by mathematical treatment; however, the SPM working diapason along Z axis will be decreased. To compensate the working diapason decreasing, the extra piezo actuator in the form of bimorph membrane (3) is attached to the free end of the piezo tube. As a result, the working diapason on Z direction is $\sim 20$ µm. The SPM lens shown in Figure 1e, supplemented by a compensating piezo-bimorph (not shown in Fig. 1e), provides a spatial resolution of $\sim 1$ nm with a scanning range of 20x20 µm$^2$. The circuit shown in Figure 1f is devoid of the disadvantage described above, since on this scheme along the XY coordinates not the probe, but the sample moves. In this scheme, the scanning diapason is 20x20x7 µm$^3$ with the same spatial resolution.

5. Examples of SPM-OM and SPM-SEM images

Figures 3 and 4 present the results of SPM-OM and SPM-SEM application. Combining methods, firstly, makes it possible to move the SPM probe to the desired point under the control of OM or SEM and to carry out accurate measurements along the Z coordinate, which is rather difficult to do without

![Figure 3](image-url)

*Figure 3.* (a), (b), (c) SPM-OM images of polymer 2D-hologram; (d), (e), (f) semiconductor CCD-matrix; (g), (h), (k) diffraction grid on the Si surface. (a), (d), (g) OM images; (b), (e), (h) SPM images; (c), (f), (k) cross sections of SPM images.
Figure 4. SPM-SEM images of steel surface and a result of a dynamic force nanolithography performed under an electron beam control; (a) SEM image of the steel surface, (b) SPM image received simultaneously with SEM image, (c) SEM image of microstructure created on the CD disk by dynamic force nanolithography.

using SPM. Secondly, the combination of different visualization methods in one experiment allows more accurate deciphering the micro- and nanostructure of the object. For example, on the optical image of the polymer hologram surface (Fig. 3a), periodically located large bands with a period of ~50 μm and a fine structure consisting of round objects with a typical size of ~1 μm are clearly visible. At the same time, on the SPM image both these large bands and a fine striped structure with a period of ~1 μm are observed (Fig. 3b,c), but the round objects with a typical size of ~1 μm are absent. Comparing optical and SPM images, it can be assumed that the fine striped structure on the surface of a polymer is not resolved by OM. It can be also assumed that the round objects on the optical image (Fig. 1a) are water micro drops condensed on the polymer surface and reflecting light. These micro drops of water are not visible on the SPM image, because the SPM probe penetrates through them and gives an image of the real surface of the polymer. It is interesting to compare the OM and SPM images of the diffraction grating (Fig. 3g,h).

In an optical image (Fig. 3g), the surface of the diffraction grid looks like a plane divided by narrow channels into square micro cells. From this image, the lattice period can be measured correctly. Although the correct data on the micro relief of the surface can be extracted only from the SPM image (Fig. 3h) and its cross section (Fig. 3k). This is due to the fact that in a vertical incidence, light is equally reflected from the surface of the protruding and recessed cells. SPM with nanometre accuracy allows measuring the height of the steps equal to 400 nm. Finally, SPM-OM-SEM combination fundamentally expands the possibilities of the experiment, for example, it becomes possible to measure local mechanical, electrical, and magnetic characteristics on the sample surface using a variety of SPM techniques.

6. Conclusion
Using piezo-inertial linear motors and self-sensing probes allows creating compact SPM modules for combining it with optical and electron microscopes. Due to the exclusion from the design stepper motors and an optical scheme for measuring the cantilever deflection, the SPM modules are easily placed on the lens of an optical microscope or goniometer of a scanning electron microscope. Since the scanning probe, scanning electron, and optical microscopes have a fundamentally different nature of contrast, their combination significantly expands the volume of information received in one experiment and increases the accuracy of nanodiagnostics of materials.

Acknowledgements
The work was carried out with the support of the Ministry of Education and Science of the Russian Federation, State Project No. 075-00780-19-00 (Subject No. 0074-2019-0007).
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