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Strain-based scanning probe microscopies for functional materials, biological structures, and electrochemical systems

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Abstract

Strain and electromechanical coupling are ubiquitous in nature, and exist in many processes involved in information technology, energy conversion, and biological phenomena. Strain-based scanning probe microscopy (s-SPM) techniques, especially piezoresponse force microscopy (PFM) and electrochemical strain microscopy (ESM), have emerged as powerful tools to probe and manipulate materials, structures, and systems at the nanoscale. In this review, we will present the fundamentals of s-SPM and a variety of its operational modes, introduce its applications in scientifically or technologically important functional materials, electrochemical systems, and biological structures, and discuss some of its challenges and potential opportunities. By detecting dynamic strains associated with underlying microscopic processes excited by a scanning probe, high sensitivity and unprecedented spatial resolution can be obtained, though caution must be exercised to distinguish different microscopic mechanisms, and quantitative interpretation of the s-SPM data remains challenging. We expect that s-SPM will continue to provide great insight into functional materials and structures, and will play a valuable role in the emerging field of materiomics.
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1. Introduction

Driven by the rapid advances in nanostructured materials and systems over the last two decades, scanning probe microscopy (SPM) has emerged as a powerful tool to probe and manipulate materials, structures, and systems at the nanoscale. While SPM was originally developed in the 1980s to study the atomic forces between samples under investigation and a scanning probe tip, a variety of other imaging modes of SPM have since been proposed and implemented, expanding the capabilities of SPM to a wide range of functional properties. Of particular interest here are strain-based SPM (s-SPM) techniques that detect dynamic strain excited in a sample using a scanning probe, including piezoresponse force microscopy (PFM), conceived in 1990s [1,2], and recently developed electrochemical strain microscopy (ESM) [3,4] and piezomagnetic force microscopy (PmFM) [5,6]. These techniques have been applied to study functional materials such as piezoelectrics, ferroelectrics, and multiferroics, electrochemical devices including lithium ion batteries and solid oxide fuel cells, as well as biological materials and structures. They have not only provided unprecedented insights into the microscopic mechanisms of these materials at the nanoscale, but also enabled new discoveries as well as novel nanostructure fabrications and manipulations. Thus we feel it is quite appropriate and timely for us to survey the current state of art of s-SPM in this inaugural issue of Journal of Materiomics.

Strain is ubiquitous in nature, and exists in many processes involved in information technology, energy conversion, and

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biological phenomena. Many of the strains are electromechanical in nature, and the coupling between electric field and mechanical deformation underpins the functionality of materials and systems as diversified as ferroelectrics and multiferroics, electrochemical cells, and biological systems. In ferroelectrics, strain is directly coupled to polarization [7], and thus can be used to study complex phenomena involving polarization reversal, domain wall pinning, and multiferroic interaction. During electrochemical conversion and transport, a key attribute is electrochemical strain resulting from changes in either ionic concentration or valence state [8−12], which might not be desirable for device operation, yet provide valuable information on local electrochemistry that is otherwise difficult to detect. The very basis of biological functionalities is electromechanics [13−15], including nerve-controlled muscle contraction at macroscale, hearing at microscale, and voltage-controlled ion channels at nanoscale. It appears that strain and electromechanical coupling are directly relevant to many existing and emerging aspects of materials science and nanobiotechnology, and s-SPM techniques, by detecting dynamic strain associated with underlying microscopic processes, offer a set of power tools to probe such materials and systems with high sensitivity and resolution.

In this short review, we will present the fundamentals of s-SPM and a variety of its operational modes, introduce its applications in a number of scientifically or technologically important areas, and discuss some of its challenges and potential opportunities. This does not mean to be a comprehensive survey of the state of art. Instead, we plan to sample some of the progress in methodology development, present selected applications in functional materials, electrochemical systems, and biological structures, and highlight a few endeavors in resolving microscopic mechanisms and quantitative analysis associated with s-SPM.

2. Fundamentals of strain-based scanning probe microscopies

2.1. Basic operations

Scanning probe microscopy (SPM) was developed to detect tiny interactions between a scanning probe tip and the sample surface under investigation. As shown in Fig. 1(a), an SPM system includes four core components: (1) a cantilever that serves as a force sensor; (2) a laser photodiode that measures cantilever deflection; (3) a scanner that moves the sample relative to the cantilever in two-dimensions (2D); and (4) a data acquisition and control unit. When the cantilever is brought close to the sample surface by an actuator in the z direction, the interaction between the probe tip and the sample surface will attract or repel the cantilever, resulting in a deflection s that is measured by photodiode. If the spring constant k of the cantilever is known or calibrated, then the interaction force can be calculated as

$$F = ks. \tag{1}$$

Meanwhile the displacement Z of the entire cantilever along the z axis is independently controlled, and the tip-sample distance D can be determined as

$$D = Z + s \tag{2}$$

when an appropriate sign convention is adopted, allowing us to measure the tip-sample interaction force F as a function of their distance D. Interested readers can refer to monograph by Greg Haugstad for more in-depth discussions on SPM [16].

The imaging mechanism of s-SPM is based on the detection of dynamic strain or displacement of the sample. An AC voltage is applied to the sample through the conductive probe in contact with the sample, which often serves as the top electrode, with the bottom electrode grounded underneath the sample, as shown in Fig. 1(b). Since the probe tip is in contact with the sample surface, the cantilever is bent upward, and the set point, i.e. the force exerted by the cantilever to the sample surface, is usually fixed by feedback control. As such, the topography of the sample surface can be mapped simultaneously with the surface displacement. The applied electric field will trigger a localized surface vibration of the sample due to its electromechanical coupling, which in turn induces vibration of the cantilever that can be measured by the photodiode. Two types of surface displacement can be measured, one is the vertical one arising from normal strain, as shown in Fig. 2(a), and the corresponding mode is referred to as vertical s-SPM; the other is the lateral displacement arising from shear strain, as shown in Fig. 2(b), and the corresponding

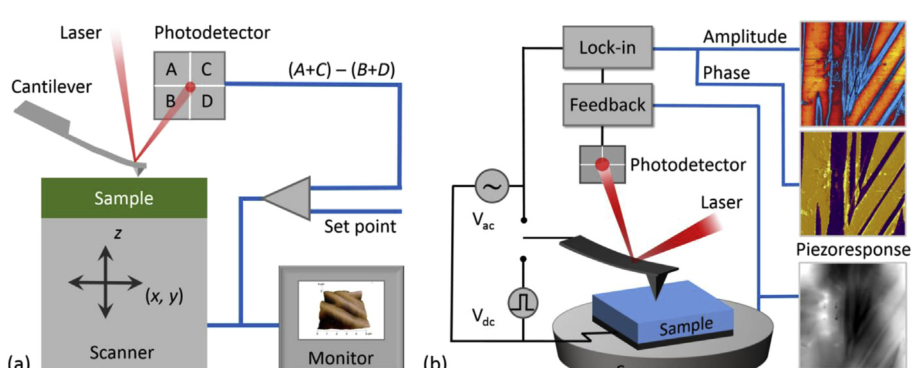

Fig. 1. Configurations of a typical SPM system (a) and s-SPM setup (b).

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