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Mapping buried nanostructures using subsurface ultrasonic resonance force microscopy

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ABSTRACT

Nondestructive subsurface nanoimaging of buried nanostructures is considered to be extremely challenging and is essential for the reliable manufacturing of nanotechnology products such as three-dimensional (3D) transistors, 3D NAND memory, and future quantum electronics. In scanning probe microscopy (SPM), a microcantilever with a sharp tip can measure the properties of a surface with nanometer resolution. SPM combined with ultrasound excitation, known as ultrasound SPM, has shown the capability to image buried nanoscale features. In this paper, the development of a modified type of ultrasound SPM called subsurface ultrasonic resonance force microscopy (SSURFM) is reported. The capability and versatility of this method is demonstrated by the subsurface imaging of various samples including rigid structures buried under a soft matrix (aluminum under a polymer), rigid structures buried under multiple layers (aluminum under a polymer and titanium layer), and rigid structures under a rigid matrix (aluminum under silicon oxide). Furthermore, tuning and optimization of the image contrast are reported. The experimental results provide possible new industrial metrology and inspection solutions for nanostructures buried below the surface.

1. Introduction

The nondestructive imaging of nanoscale features embedded inside a surrounding matrix is becoming more and more important for the manufacturing of high-performance semiconductor devices such as three-dimensional (3D) transistors, 3D NAND memory, and future quantum electronics. For example, such a capability is needed for defect inspection [1], characterizing nanolayers in extreme ultraviolet (EUV) lithography masks [2], and semiconductor wafer metrology [3]. The same functionality is required for biomedical research and diagnostic systems for identifying the internal components of cells or tissues [4]. Today, such features are most commonly imaged optically [3], but the industry is reaching physical limits regarding the resolution [5], material contrast [3], and opacity of the intervening layers [6]. Academic research has suggested an interesting candidate technology to enable the imaging of these embedded nanostructures with a very high resolving power and independent of the optical properties of the materials by combining scanning probe microscopy (SPM) [7] with ultrasound excitation [8,9]. Multiple research groups have experimentally shown the possibility of the imaging of samples such as small particles embedded in a polymer matrix [10,11] or voids in materials [12–14]. With these methods, the contrast for the buried features is usually ascribed to the mechanical properties of the stack of materials under the SPM tip [15–18]. Here, we report the development of an optimized subsurface SPM technique aimed at industrially relevant cases where the materials are typically closer in mechanical properties and therefore more difficult to distinguish compared to the typical reports in the literature cited above. This technique is based on a combination of the two contrast mechanisms currently described in literature, which are therefore shortly reviewed below.

The use of SPM with ultrasound for the visualization of subsurface structures was first reported by Kolosov and Yamanaka [19,20], who called their method ultrasonic force microscopy (UFM). In UFM, the sample is acoustically excited at ultrasonic frequencies of a few megahertz — typically at least an order of magnitude above the contact resonance frequency of the SPM cantilever [19]. By introducing amplitude modulation at a few kilohertz and monitoring the cantilever's response at this frequency, the subsurface features can be imaged as follows. At the ultrasound frequency far above the contact resonance, the mechanical impedance of the cantilever is increased, or, in other words, the cantilever moves with an amplitude that is very small

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compared to the excitation amplitude [19]. As the sample surface oscillates, but the SPM cantilever including the tip cannot follow this motion, the sample has to indent itself deeply upon the tip with each oscillation cycle. The nonlinear nature of the tip–sample interaction then causes the cantilever to experience an effective time-averaged force, which depends on the amplitude and force–indentation relationship [19,20]. The force–indentation relationship in turn depends on the mechanical properties of all the material that is affected by the induced stress field in the sample, which can include structures buried below the sample surface. Therefore, monitoring the time-averaged force on the cantilever as a function of the ultrasound amplitude allows for the visualization of subsurface features [21,22].

Later, other methods combining SPM with ultrasound based on the resonance properties of an SPM cantilever in contact with a surface (so-called contact resonance SPM [CR-SPM]) were developed for subsurface imaging [17,23]. According to the Euler–Bernoulli model of a cantilever beam, the equation of motion of the beam has boundary conditions at the tip end which are formed by the mechanical properties of the contact between the tip and the sample. Most importantly, the resonance frequency of the beam is sensitive to the material properties around the contact region. The resonance is sufficiently sensitive to these material properties that it is possible to obtain images of subsurface features via monitoring of the resonance frequency (or the amplitude when the beam is excited at a fixed frequency close to the resonance) [11,14]. Different methods based on this concept have been invented, where the differences involve excitation of either the tip [24–26] or sample [25,27] or measurement of either the amplitude [24,28] or resonance frequency [13,17,29] of the cantilever. All of these methods excite the cantilever and measure its response at the same frequency, which is chosen at or close to one of the cantilever's contact resonances.

From the literature discussed above, there are two different approaches for measuring mechanical properties with SPM with a sufficient sensitivity to use such measurements to visualize buried structures. On the one hand, UFM exploits the increased mechanical impedance at very high frequencies to introduce large indentations, the effects of which are measured at very low frequencies. On the other hand, CR-SPM exploits the sensitivity of the shift in the contact resonance frequency to the material properties in an extended volume around the tip–sample contact. In this paper, we present the results of a subsurface probe microscopy technique that combines and benefits from the physics behind both UFM and CR-SPM subsurface techniques and that we call subsurface ultrasonic resonance force microscopy (SSURFM). With SSURFM, we combine the large mechanical impedance of the cantilever at very high ultrasound frequencies (as in UFM) with the sensitivity of the cantilever's resonance to the mechanical properties (as in CR-SPM). We show the applicability and sensitivity of SSURFM by reporting experimental results for a variety of material combinations that are relevant over a wide range of industrial applications: rigid/rigid (metal in glass), multilayers (metal in a polymer beneath a metal top layer), and rigid/soft (metal in a polymer), which we have not seen imaged elsewhere in the literature.

This paper is organized as follows. Section 2 describes the SSURFM method. In Section 3, experimental measurements are presented. Sections 4 and 5 present the discussion and conclusions, respectively.

2. Description of the method

2.1. SSURFM method

SSURFM uses a high-frequency ultrasound wave at a frequency that is typically a few tens of megahertz—far above the contact resonance, as in UFM [19]. However, in contrast to UFM, this wave has a sinusoidal amplitude modulation applied at a frequency equal to or close to the cantilever's contact resonance frequency. The cantilever's response is measured at this modulation frequency. Thus, from UFM, we utilize the idea regarding the use of a high-frequency ultrasound carrier wave to make use of the high mechanical impedance of the cantilever and generate large indentations. However, in contrast to UFM, the cantilever's response is measured at a frequency coinciding with one of the resonance frequencies of the cantilever in contact with the surface to enhance the UFM response with the Q factor of this resonance and to additionally obtain contrast from the shift in the contact resonance, which is the basis for the CR-SPM methods [17]. The SSURFM method can be applied from either the tip or sample side in an experimental setup, as for CR-SPM [17,24] and UFM [20,30], although in our experimental setup, ultrasound is normally generated using a piezoelectric transducer located below the sample. Compared to other methods proposed for subsurface imaging, such as heterodyne force microscopy (HFM) [31] or resonant difference-frequency atomic force ultrasonic microscopy (RDF-AFUM) [32], which need to excite both the tip and sample, this is an advantage for several applications where the sample size is large. In these cases, excitation of the sample at a very high frequency is not practical; thus, the ability to perform subsurface imaging via tip excitation is a clear technical advantage.

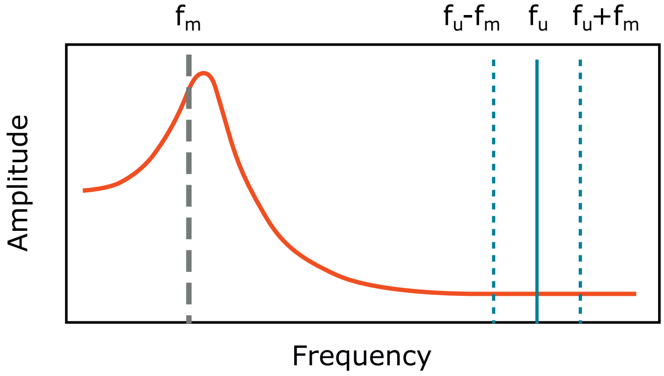

Fig. 1. Schematic of the frequency components of the ultrasound excitation signal (at f_u, $f_u - f_m$, and $f_u + f_m$) and measurement signal (at f_m). The solid curve is the cantilever response function with the first contact resonance.

The ultrasound excitation signal can be written as $A(t) = A_0[0.5\sin(2\pi(f_u - f_m)t) + \sin(2\pi f_u t) + 0.5\sin(2\pi(f_u + f_m)t)]$, where A_0 is the excitation amplitude, f_u is the high ultrasound frequency far above the cantilever's resonance, and f_m the modulation frequency. f_u should be chosen at or near a resonance frequency of the ultrasound transducer for maximum efficiency, whereas f_m should be chosen at or close to the cantilever's contact resonance. The frequency spectrum of this amplitude-modulated signal is shown in Fig. 1. Although there is no signal component in the ultrasound excitation signal at the cantilever's contact resonance, the nonlinear tip–sample interaction acts as a frequency mixer [33], creating new signal components in the cantilever motion. These components have frequencies that are a linear combination of the excitation frequencies, notably containing a component with the modulation frequency f_m.

2.2. Experimental setup

An ultrasound wave is generated using an ultrasound transducer with a suitably high resonance frequency f_u. We have used transducers mounted below the sample, which consist of a piezoelectric material embedded in a damping material to minimize