Solid State Communications 246 (2016) 17–22

Contents lists available at ScienceDirect

Solid State Communications

journal homepage: www.elsevier.com/locate/ssc

Fast-track communication

The role of substrates and environment in piezoresponse force microscopy: A case study with regular glass slides

Shilpa Sanwlani*, Mohammad Balal, Shubhra Jyotsna, Goutam Sheet

Department of Physical Sciences, Indian Institute of Science Education and Research Mohali, Punjab 140306, India

ARTICLE INFO

Article history:
Received 29 April 2016
Accepted 21 July 2016
Available online 28 July 2016

Keywords:
Piezoresponse force microscopy (PFM)
Ferroelectrics
Hysteresis
Piezoelectrics

ABSTRACT

Piezoresponse force microscopy (PFM) is a powerful tool for probing nanometer-scale ferroelectric and piezoelectric properties. Hysteretic switching of the phase and amplitude of the PFM response are believed to be the hallmark of ferroelectric and piezoelectric behavior respectively. However, the application of PFM is limited by the fact that similar hysteretic effects may also arise from mechanisms not related to ferroelectricity or piezoelectricity. In this paper we report our studies on regular glass slides that show ferroelectric-like signal without being ferroelectric and frequently used as a substrate in PFM experiments. We demonstrate how the substrates and other environmental factors like relative humidity and experimental conditions may influence the PFM results on novel materials.

Piezoresponse force microscopy (PFM) is one of the most powerful techniques used for imaging and identifying domains in ferroelectric and piezoelectric materials [1–6]. This is done by bringing a sharp electrically conductive tip in contact with the surface of the material and applying an external bias to the conductive tip. An electromechanical interaction between the cantilever tip and the sample determines the sample response to an applied sweeping dc electric field. As the electric field sweeps, an ac signal rides on the sweeping field which is used as an excitation signal in order to track the sample characteristics [7,8]. A phase switch of the response and hysteresis in phase vs. dc voltage plot is known as an evidence of local polarization switching indicating ferroelectricity, whereas, hysteresis in amplitude with field indicates piezoelectric behavior [8].

It is seen that in some of the materials, which do not have ferroelectric ordering may show strikingly similar hysteretic switching behavior under a PFM [9]. The origin of hysteresis in such materials is poorly understood. Such observations are usually attributed to the local electrostatic charge accumulation or local electrochemical effects. The role of the electrostatic effects are usually mitigated by employing the switching spectroscopy PFM (SS-PFM), protocol pioneered by Jesse et al., in which the dc-bias is applied in a sequence of electric pulses and the measurements are done in the “off”-states of those pulses [10–12]. Since the electrostatic charges are expected to relax much faster than the ferroelectric polarization, this technique removes the electrostatic components from the measured data to a large extent. While the electrochemical effects cannot be entirely removed, whether electrochemistry contributes to the observed switching or not can be investigated by performing topographic imaging after performing PFM spectroscopy. In case of electro-chemical reactions taking place under the tip additional nano-structures are known to grow as an end product of the said electrochemical reaction that can be imaged by regular non-contact topographic imaging [13–18]. In fact, recently the tip-induced electrochemistry has been exploited in a new characterization technique namely Electrochemical Strain Microscopy (ESM) [1] which has been applied to enormous number of non-piezoelectric but electrochemically active materials such as in biological molecules [19,20], corrosion and in medical science [1,21–24]. It was observed that these substances exhibit piezoelectric and ferroelectric-like behavior on interaction with the electrically conductive tip. In PFM spectroscopy on such materials spectral features such as hysteretic phase switching along with the hysteresis in amplitude (butterfly loops) can be observed as well. In some cases it was also shown that such ferro-electric-like signal might be associated with hysteretic switching behavior of the substrate or the base material on which the samples are mounted [9].

One of the popular based materials on which PFM samples are usually mounted is the regular glass slides. The primary chemical component of such glass slides is amorphous silica. However, most of them are known to contain significant amount of rare-earth impurities like sodium [25,26]. Sodium containing glass slides are known to be electrochemically active [1]. However, from structural point of view glass slides are not expected to be ferroelectric or

* Corresponding author.
E-mail address: ships2105@gmail.com (S. Sanwlani).

http://dx.doi.org/10.1016/j.ssc.2016.07.023
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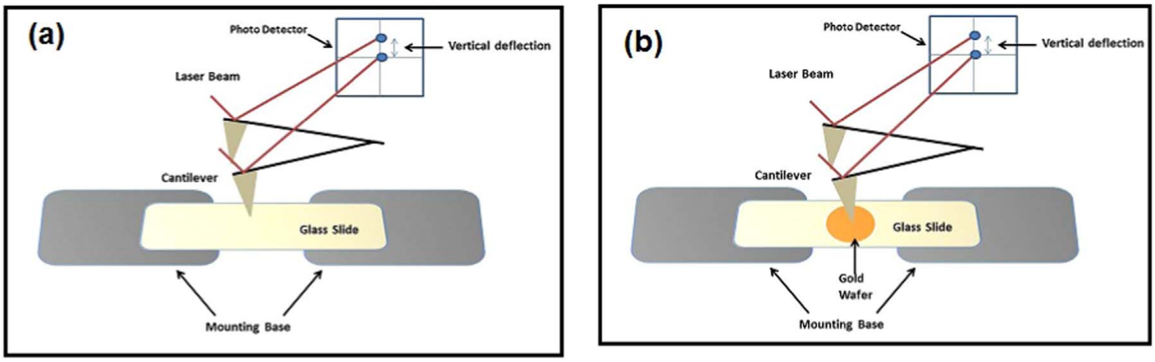

Fig. 1. (Color online) (a) Schematic diagram of piezoresponse force microscopy (PFM) on a regular glass slide. (b) Schematic diagram of PFM on a gold (Au) wafer mounted on the glass slide.

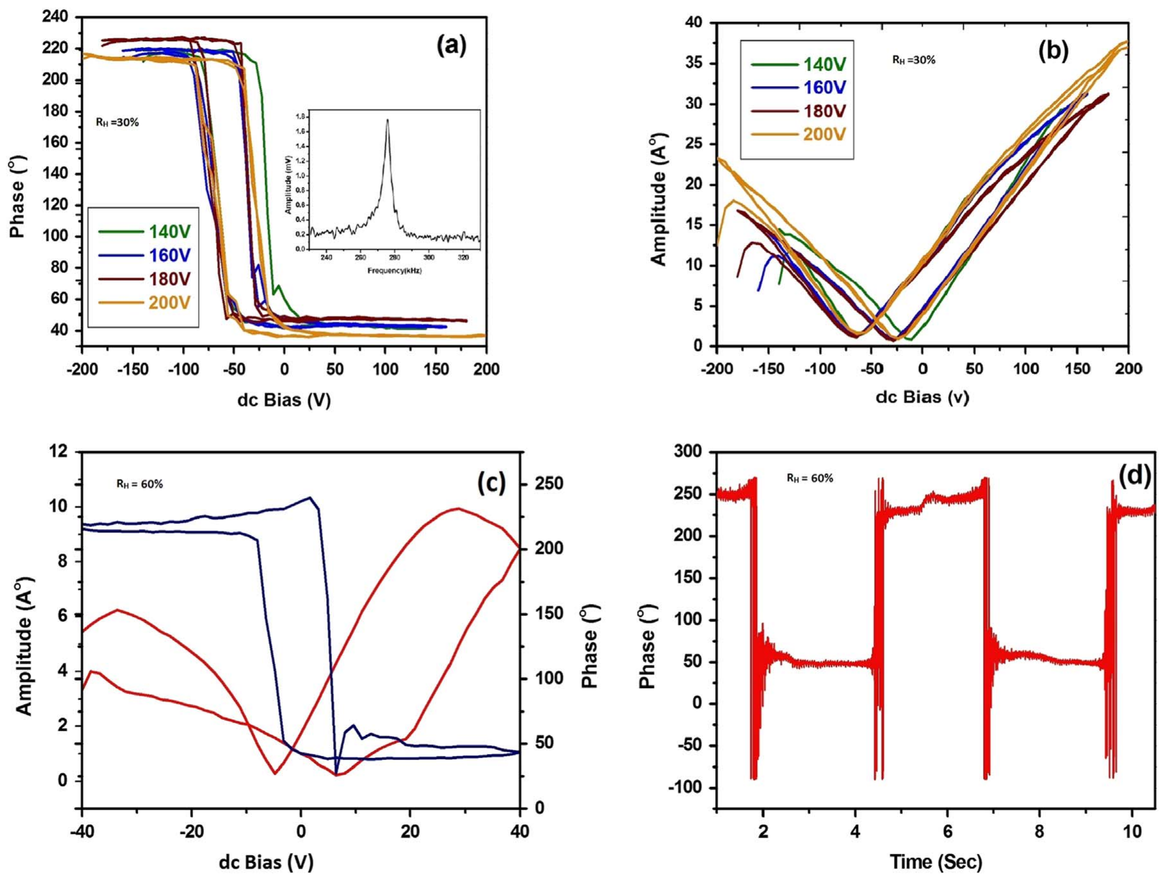

Fig. 2. (a) PFM phase hysteresis on glass slide up to 200 V with inset showing that in-contact resonance frequency of the cantilever tip varied between 270 kHz and 280 kHz. (b) Butterfly loops on glass slide up to 200 V. (c) PFM hysteresis and butterfly loops observed in glass slide at 160 V at relative humidity 60%. (d) Typical phase-switching in time domain at relative humidity 60%.

piezo-electric. This makes the slides a natural choice as substrates for PFM measurements [27]. Recently it has been shown that hysteretic effects in sodium-glass slides might originate from electrochemical reactions taking place on the surface of the glass slide [1]. In Ref. [1] it was shown that during the PFM spectroscopic measurements on glass slides using conductive tips, nanometer size topographic structures grew that could be imaged in the non-contact AFM mode after the spectroscopic measurements [28]. This observation is a proof of the electro-chemical processes in a small region on the sample surface underneath the tip [29,30]. However, on other glass slides with different components, signature of electrochemical reaction is not found by topographic imaging after the spectroscopic experiments. Therefore, the hysteresis effects observed on low sodium glass slides cannot be simply attributed to electrochemically induced switching. Other experimental and environmental factors need to be considered. In addition, our observation has been that every material that is mounted on a glass slide shows hysteretic effects under a piezoresponse force microscope. Therefore, the role of substrates in PFM experiments should be investigated.

In this paper we report our measurements on a wide variety of samples that do not show hysteresis in PFM response normally, but do show hysteretic phase and amplitude switching when mounted on glass slides. We also performed spectroscopic measurements on a diverse variety of samples including paper which is known to be a good dielectric and a small piece of thin