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A New and Simple Procedure for Electric Potential Mapping by Phase Contrast in AFM

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Electrostatic phenomena have been intensively studied after the 17th and 18th centuries with contribution from many scientists like Coulomb, Faraday, Volta, Ampère, Maxwell, DuFay and others. Based on electrostatic principles, many important technologies have been developed, such as electrostatic painting [1], electrospinning [2] and photocopying [3]. However, even considering this long history and the great number of resources available and widely used in the study of electric phenomena in insulators, there are still many open questions, for example, how can charge carriers in insulators be detected, identified and quantified?

Electrostatic charging of insulators is poorly known and it often goes out of control because the charge carriers are unknown, in almost every case, resulting in many practical problems, including serious recent industrial accidents. Different authors have put forward new proposals but a persistent problem is the difficulty to produce repeatable and predictable electric potential patterns [4,5]. The study of electrostatic patterns on dielectrics and other solids has been greatly improved by the introduction of electric mode techniques in Scanning Probe Microscopy [6]. The scanning electric potential microscopy (SEPM) technique allows the detection of electrostatic patterns adjacent to an insulator surface with 10-20 nm spatial resolution [7]. However, the instrument setup is time-consuming and the experiment total time is often too long.

In this work, we propose a new and practical method for determining the electric gradient over the surface of a sample, based on phase contrast in atomic force microscopy (AFM) periodic contact mode. Phase contrast can be produced by topographic variations, as well as by changes in hardness, viscosity and other physico-chemical surface properties. The separation of the contributions of each different property was done by proper choice of the probe (material, elastic constant, resonance frequency, etc) and of the acquisition parameters (interaction force, probe amplitude, phase detection angle, etc).

Scanning Phase Contrast Electric Potential (SPCEPM) - Experiments were performed in a modified Discoverer TMX 2010 (TopoMetrix) commercial instrument, using a biased Pt-coated Si probe that oscillates in its resonant frequency. Initially, the phase angle (θ) between photodetector output (r) and the electric signal that drives the probe is made equal to 0° and the probe is moved closer to the sample surface, until the oscillation amplitude is one-half of the free oscillation amplitude. During scanning, the topographic image is acquired by monitoring the voltage applied by the feedback system to cancel the amplitude displacement in the oscillation. Simultaneously, the phase signal is recorded and used to make the phase contrast image. The signal r is delayed ($\Delta\theta < 0$) when the electrostatic forces between the probe and the scanned region are attractive and is advanced ($\Delta\theta > 0$) when the electrostatic force is repulsive.

Scanning Electric Potential Microscopy (SEPM) - Experiments were performed in a Discoverer TMX 2010 (TopoMetrix) instrument with the aim of comparing the images obtained by this technique with the phase contrast electric mode results. SEPM uses the standard non-contact AFM setup, but the sample is scanned with Pt-coated Si tips with a 20 nm nominal radius. An AC signal is fed 10 kHz below the frequency of the normal AFM oscillator, which matches the natural frequency of mechanical oscillation of the cantilever-tip system (40-70 kHz). The mechanical oscillation of the tip is tracked by the photodetector and analyzed by two feedback loops. The first loop is used in the conventional way to control the distance between tip and sample surface, while the sample is scanned at constant oscillation amplitude. The second loop is used to minimize the electric field between the tip and the sample, as follows: the second lock-in amplifier measures the tip vibration at the AC frequency oscillation during scanning and adds a DC bias to the tip to cancel the phase displacement in the AC oscillation. The SEPM image is built using the DC voltage fed to the tip.

Standard sample preparation and polarization – Silica thin films were formed by silicon wafer oxidation and partially covered with sets of interdigitated parallel gold electrodes, using microlithography techniques. AFM, SEPM and phase contrast electric images were acquired maintaining one set of electrodes grounded and the other biased at -5V or +5V.

Figure 1 shows the topography and the electric images obtained by phase contrast from the same area of an insulator sample covered with an array of biased parallel gold electrodes. The scanning probe was biased with -13V (Figures 1b-c) and +13V (Figure 1d). The electrodes were alternately biased with -5V and 0V (Figure 1b) and +5V and 0V (Figures 1c-d).

Positive electrodes appear darker when the tip is negative biased and *vice-versa*. Bright and dark regions show the advance and the delay in the signal, resulting from repulsive and attractive electrostatic forces, respectively. The voltage in the border of the electrodes has the same signal of the tip bias, independently of the electrode voltage (Figures 1b-d). These borders were examined by AFM and the micrograph acquired at higher magnification (Figure 1e) showed scattered gold particles at the border. These particles probably have poor electric contact with the electrode strip and thus they are polarized by the biased tip during scanning in phase contrast electric mode

Under these conditions, the electrostatic effect on phase contrast formation predominates over viscoelastic and other contributions due to sample uniformity. The proposed method makes possible to map the domains with excess of electrical charges, even in the presence of large topographic variations (≈ 200 nm).

The SPCEPM results were compared with SEPM experiments. Figure 2 shows AFM and SEPM images from another region of this same standard sample. The SEPM images (Figures 2b-c) show that the set of positively biased electrodes acquires a positive potential relative to the other set. In SEPM, the measured voltage is absolute, resulting from the bias applied to the electrodes, electrostatic charges, relative humidity, work function and other factors that can change the surface potential.

In the SEPM images (Figure 2) the electric potentials measured over the silica surface are always more positive than the electrodes mean voltage, while in the electric contrast images (Figure 1), the silica voltage is negative when the probe is biased at +13V and positive when the probe is at –13V.

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This can be explained because the tip DC bias in SEPM cancels the electric field between the tip and the sample, while the permanent tip DC bias in phase contrast electric mode can polarize the silica. When the biased tip touches the silica surface, it transfer charge by either injection or abstraction [6]. Direct tip-surface contact overrules the silica polarization induced by the strip electrodes, since the tip is biased at a large potential and the distance to be travelled by charge carriers is very small.

The new SPCEPM is a very practical and fast method, when compared to SEPM. This technique can be easily used in any conventional AFM instrument with a module for periodic contact and software/hardware for phase detection. Other great advantage of this method is the possibility of performing experiments by applying different bias to the probe and acquiring electric map simultaneously. SPCEPM is currently a qualitative mapping technique, but further work will be done to convert it into a quantitative technique as SEPM. Both methods can produce essential information to elucidate the nature of charge carriers in insulators. [8]

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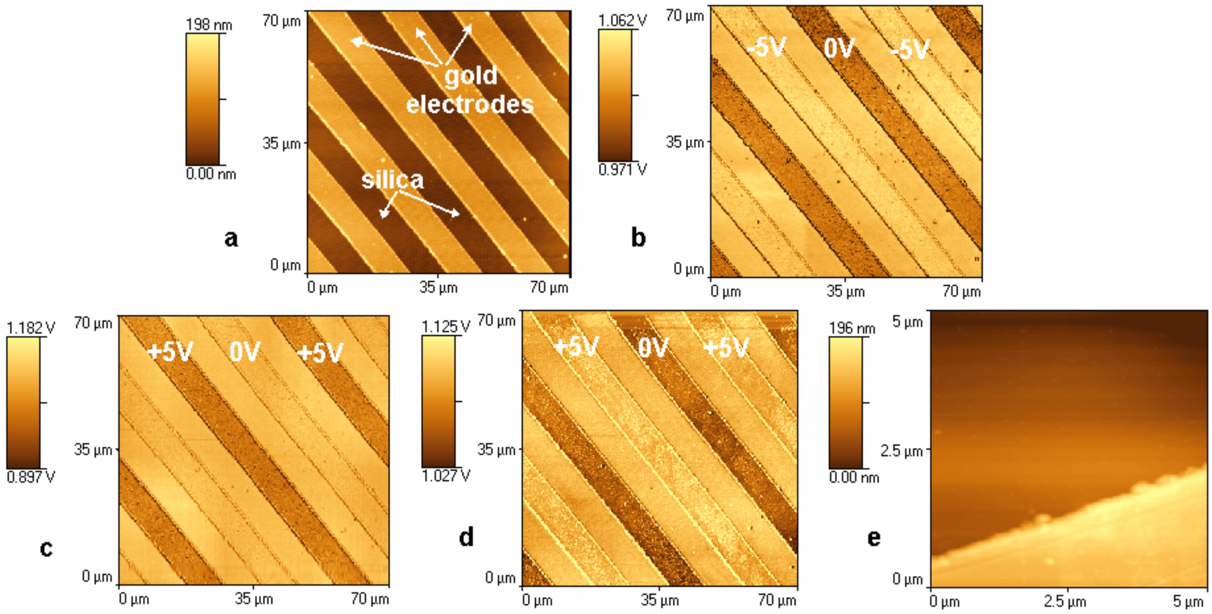

Fig. 1. (a) Topography and (b-d) electric contrast images from the same area of a silica surface with biased parallel gold electrodes. The probe was biased with -13V (b-c) and +13 V (d). The electrodes were alternately biased with -5V and 0V (b) and +5V and 0V (c-d). The voltages of the scales are the lock-in phase output. (e) Topography of the electrode border at higher magnification.

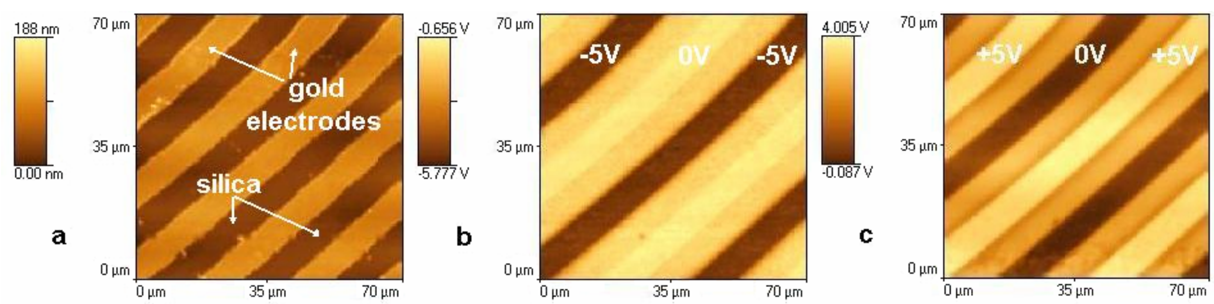

Fig. 2. (a) Topography and (b-c) SEPM images from the same area of a silica surface with biased parallel gold electrodes. (b) Electrodes alternately biased at -5V and 0V and (c) +5V and 0V.