Nanoscale phase transformation in Ge$_2$Sb$_2$Te$_5$ using encapsulated scanning probes and retraction force microscopy

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Encapsulated conducting probes that can sustain high currents are used to study the nanoscale properties of thin-film stacks comprising of a phase-change chalcogenide, Ge$_2$Sb$_2$Te$_5$. Scaling studies on this promising candidate for random-access memory devices had thus far required extensive lithography and nanoscale growth. This seriously hampers rapid materials characterization. This article describes the use of two key techniques, an encapsulated conductive probe and its use in retraction mode, whereby the attractive force between tip and sample is used to maintain electrical contact. The effective transformation of nanoscale dots of amorphous Ge$_2$Sb$_2$Te$_5$ into the crystalline state is achieved and the electrical conductivity of the transformed structures is probed. The use of retraction force microscopy in a robust manner is demonstrated by reading the conductivity of the crystalline dots. Both these techniques could enable rapid electrical characterization of nanoscale materials, without extensive nanopatterning, thus reducing material development cycles. © 2009 American Institute of Physics. [DOI: 10.1063/1.3204449]

I. INTRODUCTION

The atomic force microscope (AFM) (Ref. 1) is a powerful tool for the investigation of materials and devices with nanoscale resolution. However, its application in the conducting mode for commercially viable, nanoscale technologies has been severely hampered by unreliable nanoscale tip apexes,$^2$ Besides normal wear-associated unreliability, an additional problem incurred with such tips is their limited ability to sustain high currents. This problem is usually circumvented by the use of a larger tip apex, thus compromising on the achievable resolution. Addressing these issues successfully is crucial for electrical characterization at the nanoscale, an urgent need in the further development of nanoscale applications of these materials. One such example is the rapidly developing field of phase-change memory, where macroscale materials characterization has reached a reasonable level of maturity,$^3$ whereas nanoscale electrical characterization has often required patterning or other elaborate techniques, which, while being very useful, are time consuming and must be repeated for every material system.$^6$ In this article, we demonstrate the use of an encapsulated conductive probe (a probe having a conductive core and dielectric encapsulation) to locally change the phase of a stack of amorphous phase-change material. This creates nanoscale crystalline dots similar to those reported in Refs. 8 and 9. Two key concepts are demonstrated: (1) the effective use of encapsulated conductive probes for nanoscale electrical characterization of materials and (2) conduction-mode imaging with such tips in the retraction mode, where the enhanced attractive force between tip and sample is used to maintain reliable electrical contact.

II. DEMONSTRATION OF NANOSCALE PHASE TRANSFORMATIONS

Ge$_2$Sb$_2$Te$_5$ (GST) is considered to be the most promising material for use in phase-change-memory devices. The very high difference in conductivity between the crystalline and the amorphous states in this material can be exploited to store and retrieve data. In addition, both the high speed and the low power consumption incurred in the phase transformation of these materials have been shown to be attractive for future memory and data storage devices. Scaling these devices down to nanoscale dimensions has been a subject of recent interest because of the associated improvement in power consumption,$^10$ but methods to study scaling are cumbersome, especially because it requires extensive lithographic and/or growth techniques to study every new material composition at the length scales of interest. Probe-based approaches provide a convenient alternative to these techniques. Moreover, the influence of the GST thickness, different material compositions, electrode materials and other variables on switching behavior can be investigated rapidly using probes. One such instance is a phase-change cell, which in Fig. 1(a), is a contact-minimized (or “mushroom”) phase-change cell that is represented schematically. To minimize the writing power, the volume of GST that is crystallized must be kept small. This is done in such a cell by minimizing the contact area.$^4$ The probe-based characterization technique closely resembles such a cell, but without
incuring the requirements for patterning, and thus provides a convenient alternative for rapid material characterization of these thin films.

The details of fabricating encapsulated tips with platinum silicide apexes were reported previously, and such a tip is shown in Fig. 1(b). The spring constants for the cantilevers used in this article are ~1 N/m. In this article, the use of encapsulated tips to create crystalline dots in an amorphous GST background is described. The large difference in the electrical resistance between the crystalline and the amorphous phase of the GST is then used to image (or read) the crystalline dots on the amorphous background. In our films, we observe no appreciable change in the topography due to phase transformation, even though crystalline GST is known to be denser. This may be because of the combination of the small thickness of the GST film capped with a stiff carbon layer. By writing and then reading back the crystalline dots, we can characterize the performance of the encapsulated probes as well as extract important information regarding material and other parameters for a phase-change cell.

The phase-change media we use is a stack, as shown in Fig. 1. It consists of a bottom electrode of 120-nm-thick doped carbon layer, the amorphous GST phase-change medium (20 nm) capped with a thin layer of nitrogen-doped carbon (6 nm). All layers are sputtered using a commercial installation of the SDS-131 in-line coater. Carbon is dc sputtered at 4 kW in Argon (1.4 Pa, target-substrate distance: 42 mm) with some nitrogen doping (0.08 Pa or 6% of the Ar pressure), which reduces the resistivity of the carbon. Dynamic base pressures of around $3 \times 10^{-4}$ Pa are achieved during the process in the sputtering chamber. The Ge$_2$Sb$_2$Te$_5$ is also sputtered in situ in an adjacent (vacuum connected) chamber at 0.3 kW (Ar pressure 0.4 Pa, target-substrate distance: 42 mm).

The experimental setup consists of a home-built AFM with both optical deflection-sensing and conduction-mode operation capability. It is designed such that the micro-cantilevers can be mounted almost parallel to the sample. The sample itself is mounted on a commercial nanopositioner, and controlled using a DSP/FPGA board. A pulse generator is used to generate the write signals, and the resulting current is monitored using an oscilloscope. Writing on the stacks is typically accomplished by means of a 4 V, triangular pulse with a 100 $\mu$s period. Although GST can be switched with much shorter pulses, the selection of 100 $\mu$s pulse duration is primarily intended at obtaining good I-V measurements during the write process. An oscilloscope measures the applied voltage and the resulting current simultaneously. This can be used to obtain the characteristic switching behavior of phase-change materials. To write multiple crystalline dots, such write signals are applied at predetermined intervals as the tip slides along the surface.

For the read operation, a digital-to-analog converter is employed to generate the necessary constant voltage signal and a logarithmic amplifier measures the resulting current. The latter aids in measuring the dynamic range of the current across decades, as would be the case when the tip slides over alternating amorphous and crystalline phases of the GST. During the read process we are interested in knowing only the resistance map of the surface. It is undesirable to pass significantly large currents through the phase-change material during this process. This could inadvertently change the state of the phase-change material. To avoid such a scenario we typically use a series resistor to limit the current while reading and for the experiments presented in this article, a resistance value of 100 $\Omega$ is chosen. The voltage for reading must be below the threshold switching voltage for the GST stack, and accordingly we used a value of 0.5 V. Resistance measurements are obtained at a sampling time of 500 $\mu$s, while the scanner is being moved at 0.01 mm/s for a sampling distance of 5 nm.

A single phase transformation from the amorphous to the crystalline phase is termed a write operation, whereas the conductive map that shows the location of this crystalline dot is termed a read operation (consistent with the terms used in Refs. 8 and 9). We read by keeping the cantilever at ground potential; all write events are performed by keeping the sample at ground potential. For the experiments we chose a tip whose conductive core projects out of the encapsulation by 30–50 nm. The intention is to compare the performance

![Schematic cross section of the phase-change stack used in the experiments.](image-url)

**FIG. 1.** (Color online) (a) Schematic of contact-minimized (or “mushroom”) phase-change cell. (b) Scanning electron micrograph of a tip with a SiO$_2$ encapsulation. (c) Schematic cross section of the phase-change stack used in the experiments.
of an encapsulated probe with that of one without encapsulation (as is the case before the conducting core is worn down to such an extent that it is flush with the encapsulation). Initially, read and write experiments were performed on this tip with the projecting conductive core. By repeated write and read operations the projecting conducting core was gradually worn down until it was flush with the oxide. At this point, tip wear slows down to very low values\(^1\) and can be neglected for the experiments reported in this article. Now the conducting core has a much smaller diameter compared with the total diameter of the tip area that comes into contact with the sample. The evolution of the tip geometry is monitored by periodically measuring the force of adhesion. Approach curves are obtained at regular intervals, and the force of adhesion is calculated as the product of the spring constant \(k\) and the distance moved before snap off occurs (i.e., when the cantilever tip loses contact with the surface). Scanning electron micrographs of both the original tip and the worn-down tip are shown in Fig. 3.

A single crystalline dot that forms as a consequence of Joule heating can be represented by a phase transformation current-voltage (\(I-V\)) characteristic. Two such phase transformations are shown in Fig. 2. The lower part of the curve in Fig. 2 represents the high-resistance, amorphous state. The amorphous region exhibits a nonlinear \(I-V\) characteristic due to trap-hopping as described in Ref. 15. At a certain voltage (the threshold voltage), the system changes to a lower-resistance regime. A large amount of power (due to the higher current) will heat the material to a high temperature, eventually resulting in changing the state of the material to the crystalline state. The \(I-V\) characteristic for the configuration shown in Fig. 2 is not very sensitive to the presence of the \(\text{SiO}_2\) encapsulation, because \(\text{SiO}_2\) is a poor thermal conductor (thermal conductivity of 1.28 W/mK), and hence will not dissipate heat rapidly. The \(I-V\) characteristics are also reproducible in different areas of the material stack and is evidenced in Fig. 2, where the two phase transformation curves at different areas on the sample lie almost on top of each other.

After performing a series of phase transformations while scanning the surface (write operation), we read out the crystalline dots using a conduction image of the area. While scanning, the topographical variations of the surface are also recorded using the cantilever deflection signal. Thus, a topographical image of the surface is obtained simultaneously with the conduction map. The topography and current images corresponding to the crystalline dots written and read back with the original tip (with the conductive core projecting out) and with the worn-down tip are shown in Fig. 3. By comparing the topography images, it is evident that there is a significant difference in the image resolution. With the original tip, we can resolve the surface roughness thanks to the nanoscale fidelity of the tip and image feature sizes of roughly 25–35 nm before it had been worn down to the encapsulation. With the worn-down tip, however, it is impossible to resolve these features because of the much larger physical diameter of the tip. Owing to the tip convolution, we observe features with dimensions of approximately 150 nm, i.e., comparable to the diameter of the worn tip, as revealed by the scanning electron micrograph of the worn tip shown to the right in Fig. 3. This micrograph shows that the diameter of the inner core is \(\sim 48\) nm, whereas that of the total tip (including the encapsulation) is \(\sim 150\) nm.

The current images obtained during this read operation are plotted below the topographical images in Fig. 3. Dark areas correspond to the low conductance amorphous regions, and the bright dots to the crystalline regions. This conduction map indicates that the size of the crystalline dots is quite comparable for the sharp and the worn-down tip. There is only a marginal increase in the size of the crystalline dots when the tip is worn (image on the right). This is attributed to the increase in the dimension of the conductive core from the original tip to that of the worn-down tip due to its conical geometry. In any case, it is clear that the size of the crystalline dots is not linked to the total physical diameter of the tip (\(\sim 150\) nm); their size is on average \(~ 50\) nm and comparable to the measured diameter of the conducting core (48 nm). This confirms the confinement of the conduction to the electrical core of the tip and the limited role, if any, of meniscus-based conduction, which was expected to be minimal because of the hydrophobic nature of the carbon surface. Moreover, it provides evidence in favor of the hypothesis that much of the conduction occurs vertically through the layers and via the highly conducting bottom electrode, rather than laterally through the various layers in parallel, as implied in previous simulations of phase change using probes.\(^{16}\) Thus, the assumption that the phase change using probes is similar to that in a contact-minimized cell is valid.

The line scans reproduced in the bottom panels of Fig. 3 represent the resistance as the tip is scanned over a line of crystalline dots. The large difference in resistance between the amorphous region and the crystalline dots is evident in both line scans. However, even though the applied loading force of 50 nN is identical in both cases, we note that there is an increased signal-to-noise ratio while reading and writing with the worn tip. We attribute this improvement to two factors. (1) With the sharp tip, there is some medium derived noise that occurs as the tip probes the surface asperities, thus

![Phase Transformation IV 1](image-url)
coming into contact with parts of the surface via the side-walls of the tip; this noise is suppressed with the blunt tip on account of the dielectric encapsulation and the higher force of adhesion with the worn tip ensures a better tip-sample contact while sliding.

III. RETRACTION FORCE MICROSCOPY

Another interesting aspect of employing tips having large physical diameters is the ability to use these tips in the retractive regime, in which the tip probes the attractive regime of the tip-sample interaction potential. In regular operation, the actual force at the tip-sample interface can be assumed to be the sum of the normal applied force and the adhesion force. In the retractive regime, the actual force on the tip at the tip-sample interface is less than that exerted by the adhesive force alone by an amount equal to the force of retraction which is the product of $k$ and the distance by which the tip is retracted). Following from the work of Briscoe and Evans, the pressure-dependent shear stress is given by the second term on the right-hand side of the following expression:

$$
\tau = \tau_0 + \xi F_N^{1/2} \left( \pi a^2 \right) + \left( k_B T / V_0 \right) \ln \left( v / v_0 \right),
$$

(1)

where $\tau_0$, $\xi$, and $v_0$ are constants, and $a$ is the tip radius, which is a constant for a given configuration. The normal load $F_N$ is given by the applied and the adhesive forces: $F_N = F_{\text{Applied}} + F_{\text{Adhesion}}$. Gotsmann and Lantz also derive the relationship for the change in radius of a conical tip for a given sliding distance with velocity and pressure dependencies. Hence, it is expected that the lower loading force achieved by operating in retraction mode would result in lower tip wear because $F_{\text{Applied}}$ changes sign.

Regulation of the cantilever deflection in retraction mode is accomplished by first approaching the cantilever to the surface and then retracting it to the desired setpoint. In our experiments, the sample is moved toward the tip until the nominal applied force is 200 nN. This ensures that the conducting core is in contact with the surface. We then retract...
the sample to a predetermined point such that the applied force is negative (for instance, $-100$ nN) and then perform the scanning in closed loop to maintain this setpoint. The cantilever continues to be in contact with the surface because of the force of adhesion ($F_{\text{Adhesion}}$), albeit with a lower force corresponding to $F_{\text{Adhesion}} - F_{\text{Applied}}$, where $F_{\text{Applied}}$ is the restoring force of the cantilever, as the cantilever is retracted from the surface. The force of adhesion can be estimated from the standard approach curve as the product of the distance to snap off ($x$) and the spring constant $k$. This is shown schematically in the approach ramp of the cantilever to the surface in Fig. 4 (top). The cantilever approaches the surface, and once it is in contact follows the surface ramp (blue line). Thus, by scanning in the regime just prior to snap off, the effective force at the tip can be reduced to almost zero.

We performed the readout of the crystalline dots using this technique for a range of forces in retraction mode. The current is monitored while the cantilever slides on the surface at a predetermined deflection setpoint. The readout of a field of 20 crystalline dots (each $\sim 50$ nm in diameter) in an amorphous medium is shown in Fig. 4 (bottom) for different applied forces ($150$, $100$, and $50$ nN), at no applied force ($0$ nN) and in retraction mode ($-50$ and $-100$ nN). It is seen that the quality of the electrical readout is not significantly affected. This demonstrates that we are able to read previously written crystalline dots reliably in retraction mode.

While pressure reduction alone may not be a sufficient motivation, an additional, but very important aspect of using these tips robustly in the retraction mode is the ability to scan and image without having to apply a force in order to keep the cantilevers in contact. The ability of the cantilever tips to remain “stuck” to the surface while scanning, even for relatively large variations in long-range surface topography (such as wafer bow) could be useful, especially in future applications of large-scale, probe based technologies. Assuming that $F_{\text{adh}}$ in nanonewton is the adhesion force, the amount of variation in nanometer (such as due to wafer bow) that can be tolerated would be equal to $Z_0 + F_{\text{adh}}/k$, where $Z_0$ is the deflection setpoint in nanometer, while $k$ is the spring constant in N/m. Thus for the 1 N/m cantilevers used in this article, with a tip adhesion force $F_{\text{adh}}=200$ nN and scanning at a deflection setpoint $Z_0$ of 300 nm, this could translate to as much as 500 nm of tolerance.

So, in addition to the pressure reduction, there are two advantages to retraction force microscopy.

1. We can scan surfaces with large variations in long-range topography with no feedback control.
2. We have more tolerance against loss of tip contact while scanning topographically challenging surfaces.

One such relevant application is the nanometrology of wafers passing through a semiconductor fabrication line. The characterization of these wafers can be achieved while scanning at low forces, and any change in the force setpoint from the positive to the negative, due to wafer inhomogeneity, should not affect the electrical characterization, because of the inherent robustness of scanning these tips in the attractive regime. This lack of robustness has been an issue for applications that have tried to employ scanning with a negative setpoint. This is the first such demonstration of this technique in a robust manner in the contact mode and its combination with the encapsulated tips may prove to be a powerful tool for a variety of applications that require reliable, nanoscale, conduction-mode atomic force microscopy.

IV. CONCLUSION

In conclusion, the use of encapsulated conducting tips with platinum silicide conducting cores for conduction-mode AFM applications is demonstrated by creating nanoscale crystalline dots on an amorphous film of Ge$_2$Sb$_2$Te$_5$. In amorphous GST stacks, a phase change from the amorphous to the crystalline phase is accomplished. We use this to “write” crystalline dots in an amorphous background and experimentally verify that the size of these bits is confined to that of the conducting core. The concept of retraction force...
microscopy is introduced by exploiting the large contact area of the encapsulated tips. This is done without compromising on the achievable nanoscale electrical resolution. Both concepts demonstrate the simplicity and elegance with which thin films can be characterized rapidly. Thin-film characterization is important in a variety of fields, including data storage, thin-film photovoltaics, and nanometrology of semiconductor devices. Nanoscale electrical characterization is challenging, and these techniques could be the key to providing reliable and rapid electrical characterization. Combined with other forms of probe-based techniques, they could aid in the optimization not only of the material properties, but also in the design of various nanoscale device parts, thus drastically reducing device development cycles.

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