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Writing nanometer-scale pits in sputtered carbon films using the scanning tunneling microscope

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A reproducible method of the formation of pits in a sputtered carbon surface with a platinum-iridium tunneling tip is presented for possible use in lithography or data storage applications. Thin carbon films are sputtered on top of chrome and gold metallic underlayers on a silicon substrate. Overall surface roughness of the carbon films is under 3 Å. Holes are produced in the carbon film by applying short voltage pulses (4–8 V in height, 250 ns–100 μs in length) across the tunneling gap. An array of holes written in the carbon demonstrate reproducibility and the feasibility of using this multilayered structure in a data storage system. © *1999 American Institute of Physics.* [S0003-6951(99)00923-7]

The ability of the scanning tunneling microscope (STM) for surface modification has been investigated by many groups motivated by a desire to realize atomically controlled surfaces and to fabricate atom size devices.[1,2] Important potential applications of these surface modifications are high-resolution lithography and high density data storage. A number of techniques employing the STM for surface modification have been demonstrated. Most controllable and repeatable surface modification techniques typically involve using higher voltages or currents across the tunneling junction or mechanical indentation of the surface with the tip. Voltage pulses across the gap have been used to produce 400 nm pits in highly oriented pyrolytic graphite (HOPG),[3] nanometer sized pits and hillocks in Ag(111),[4] and 2 nm diameter pits in gold.[5] Applying a voltage pulse to MoS_2 surface in a H_2 atmosphere caused surface marks to be produced by a gas-solid reaction that enhanced the evaporation of top-layer sulfur atoms.[6] The STM has also been utilized to produce nanometer scale oxide patterns on titanium by tip induced anodization.[7] In this letter, we present a technique for the production of pits in a sputtered amorphous carbon film. These written pits vary in diameter from 3.5 to 70.0 nm depending on the different parameters used during writing.

The media samples were deposited on a Lesker radio-frequency (rf)-magnetron sputtering system. The substrates were 2 in. (111) silicon wafers. These Si wafers were then sputtered etched to remove any native oxide layer. The first layer deposited was gold at 75 W dc and 5 mTorr for 10 s. Next, a chrome layer was deposited at 100 W dc and 5 mTorr for 5 s. The carbon target was then presputtered to remove any absorbed oxygen or nitrogen. Finally, the carbon layer was deposited at 40 W rf and 5 mTorr for 2 h. The deposition rates on our system for each of these materials are 67.8, 28.8, and 0.9 nm/min for gold, chrome, and carbon, respectively.

Previous attempts by other groups to make marks in a substrate have concentrated primarily on crystalline materials. However, because the sputtered carbon film is amorphous, the surface roughness of the film becomes important in order to be able to distinguish the written marks from the background surface undulations. The as-deposited film's surface topography was scanned by a Digital Instruments Dimension 3000 STM using mechanically cut Pt/Ir tips. Scans of square areas 100 nm on a side were made at various points on the surface. Surface roughness values, as determined by the STM, were, at worst case, below 0.3 nm root-mean-square (rms) roughness with a maximum out of plane asperity height of 3 nm on the 100 nm×100 nm scanned areas. Typical values were 0.198 nm rms roughness and asperity height of 2.2 nm. A larger area scan is shown in Fig. 1 demonstrating the smoothness of the deposited films.

A typical pit is created by applying a voltage pulse to the Pt/Ir tips while positioned in tunneling mode over the carbon film. The voltage pulse is applied by placing a HP 112A Pulse Generator in series with the normal bias voltage between the tip and the substrate by use of an external Digital Instruments Signal Access Module. The smallest hole that can be reliably reproduced at this time is on the order of 3.5 nm in diameter as shown in Fig. 2. This hole was created by a 4 V 250 ns pulse. A reduction in either the pulse height or length seems to produce no mark in the substrate. It is un-

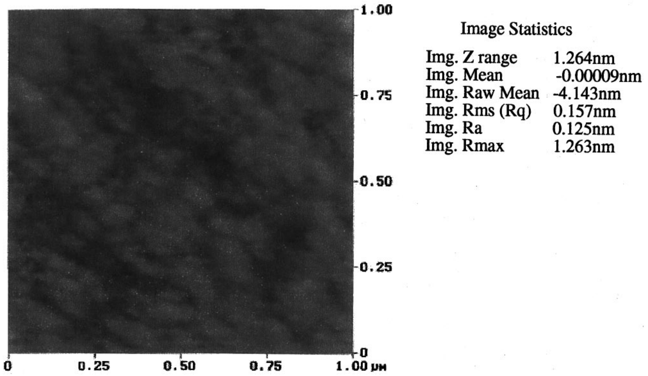

Image Statistics

Img. Z range	1.264nm
Img. Mean	-0.00009nm
Img. Raw Mean	-4.143nm
Img. Rms (Rq)	0.157nm
Img. Ra	0.125nm
Img. Rmax	1.263nm

FIG. 1. Surface roughness scan of the top sputtered carbon layer. Over an area of 1 μm^2, the rms roughness is 0.157 nm and the maximum asperity height is 1.264 nm.

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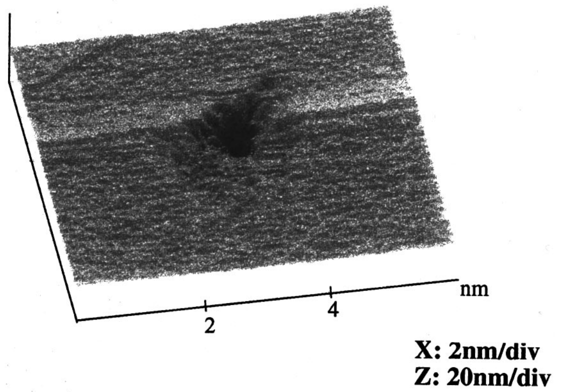

FIG. 2. A pit written in the sputtered film. The diameter on the pit is approximately 3.5 nm with a depth of roughly 4 nm. The bit was written by a 4 V, 250 ns voltage pulse.

clear at this point whether no mark is made due to the inability to distinguish small marks from the background roughness or due to the presence of a ''threshold'' voltage as is suggested by others if the writing mechanism is attributed to sublimation of surface atoms induced by tunneling electrons (SITE).[8] No marks can be reliably produced if a tungsten tip is substituted for the Pt/Ir tips up to pulse heights of 8.5 V, which also seems to agree with Kondo *et al.* results. Joule heating does not seem to be involved with the writing process in this material system. Using the method of Flores *et al.*,[9] the temperature increase at the substrate surface is estimated to be less than 70 K, which is probably not enough to cause surface modification.

The major parameters affecting the bit size were determined to be the voltage pulse height and length. The tunneling current setpoint, which is related to the height of the tip above the carbon surface when the pulse is applied, had some effect on the hole diameter but not as large as the pulse characteristics. A graph of these results is shown in Fig. 3. The thicknesses of the underlying metal layers and of the carbon layers did not have a measurable effect on the hole size.

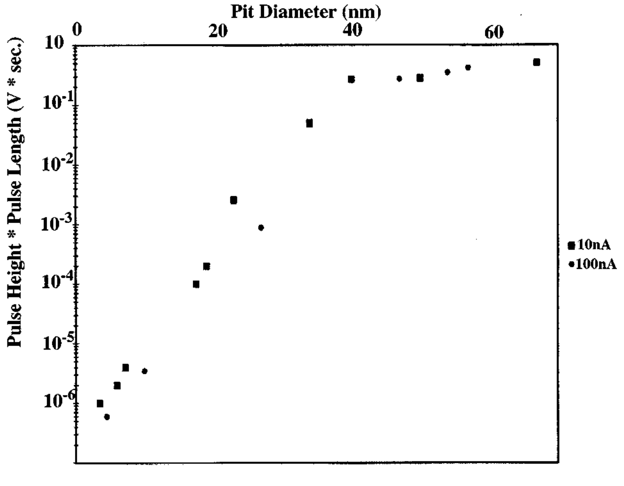

FIG. 3. Graph of pit diameter vs the product of the voltage pulse height and pulse time. Increasing the setpoint current is seen to reduce the pit diameter slightly. The thicknesses of the deposited films did not show any affect on the pit diameter.

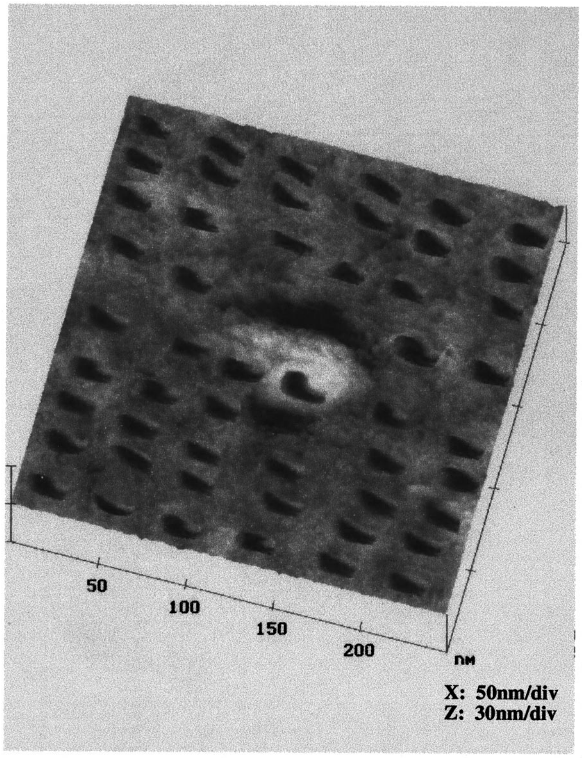

FIG. 4. A 6×9 array of pits written in the deposited film. The average pit diameter is on the order of 25–40 nm.

An array of pits was written in the carbon surface to investigate the feasibility of using this material structure in a data-storage system. For the pit array shown in Fig. 4, the tip was pulsed while performing a line scan above the top carbon layer which could account for the elongated pit structure in the direction of motion. Average pit size in this figure is 25 nm×25 nm. Two pits seem to have run together in the middle of the array, creating the larger bump which is present.

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