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A scanning tunnelling microscope study of groove structures in polycarbonate optical discs

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The groove structure in polycarbonate substrates, commonly used in the fabrication of optical discs, has been studied with the scanning tunnelling microscope. Comparative studies of the same structures were also performed using more conventional scanning electron microscopy and transmission electron microscopy techniques. These studies illustrate the ability to characterize the shape of man-made structures that are commonly recorded in these polymer-based materials. The scanning tunnelling microscope images show the superiority of this technique for detailed cross-sectional studies of the profiles of structures with typical dimensions ~ 500 nm in width by ~ 50 nm in depth.

1. Introduction

Due to its relatively low cost and high storage density, optical recording is quickly becoming an important part of the data storage industry. In recent years, compact discs with digitally encoded music have taken a major share of the music recording business from traditional analogue techniques. CD-ROM and write-once optical disc systems are being marketed by several companies, and allow for 200 to 500 megabytes of information on a single 13.0 cm disc. Even though this technology is relatively new, it is already the least expensive way to store and access large amounts of information. Future developments will include erasable optical storage media and drives which may compete directly with magnetic hard discs and tape.

A primary technical advantage of optical recording arises from the fact that one can write and read with a focused laser diode beam with a spot size of less than one micrometre. The beam can also provide information which enables a drive to stay in focus. Grooves or other features on the medium enable an optical disc drive to address and read a single track of data. All of this can be done with the optical head (laser, optics, detectors and objective lens) more than a millimetre away from the outside of the disc package. Thus, one can achieve up to 10^8 bits cm^{-2} while the read/write head is fairly far from the disc.

The critical parameters in optical recording are the size, shape and depth of the features in which the information is encoded. As already mentioned, grooves are often used so that a single track can be quickly found and so that the read/write beam can be kept on the data. There are several proposed methods for deriving a signal which can be used for tracking. One of the most commonly used systems is push-pull tracking in which a split detector is used to detect the read beam. The difference signal between the two halves of the detector crosses zero whenever the beam is centred either on the groove or on the land (the space between the grooves). At other beam positions, the diffraction from the groove causes unequal intensity on the two halves of the detector. Thus the difference signal from a split detector can be used as a tracking error signal. When fed back through a servo system, this signal is used to keep the beam on track.

The amplitude of the tracking error signal will depend critically on the depth and width of the groove. Although a depth of $\lambda/4$ (λ being the laser wavelength) gives the largest tracking error signal, usually a value of $\lambda/6$ to $\lambda/8$ is used in order to minimize interaction between the tracking and focus servo systems.

The symmetry of the groove geometry is also important. Any asymmetry will lead to an offset in the tracking position from the centre of the groove or land. In addition, the degree to which the land (or groove bottom) is flat could be important in determining the amplitudes of the data signal and noise level, as well as possibly affecting the best write and read powers for the medium. While useful information about the average structure of the grooves is often obtained from studies of the diffraction pattern of incident light, new techniques to better characterize these structures are clearly required as the need for high-density storage media increases.

A schematic illustration of the groove geometry

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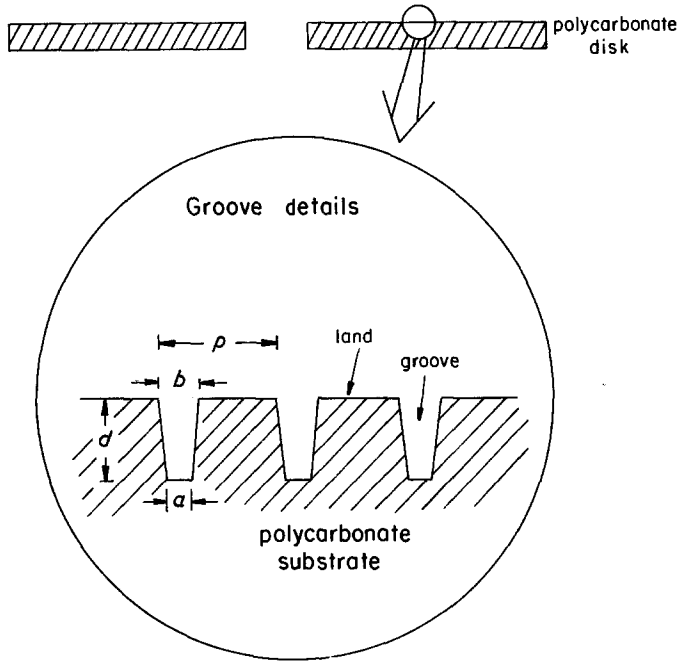

Figure 1 A cross-section of a polycarbonate disc with an enlarged schematic view of the groove profiles that are of interest in this study. The periodicity of the grooves is denoted by p. The depth of each groove is indicated by d, while the width of the bottom and top of the grooves are indicated by the parameters a and b, respectively. Note that the vertical scale is expanded relative to the horizontal scales so as to exaggerate the depth of the grooves.

placed on these discs is shown in Fig. 1. Discs are usually 1.2 mm thick, and the active layer is on the surface farthest from the laser. The groove structure is periodic, with a characteristic spacing given by the parameter p along the radial direction of the disc. As discussed above, each groove should be identical to all others and should be characterized by a high degree of symmetry. The bottom and edges of the grooves should be smooth and homogeneous to better reflect the light from the incident diode laser. The depth of the groove, d, is determined by the wavelength of the incident diode laser light as well as by the requirements of the drive for low interaction between focus and tracking signals.

Typical dimensions of the groove geometry are known. For instance, the periodicity p is often around 1.6 μm, while the depths d are about 1/8 to 1/4 of a wavelength. Since most optical recording systems use diode lasers at 830 or 780 nm, and since the substrates have an index of refraction of about 1.5 to 1.6, depths as small as 60 nm are frequently encountered. Although transmission electron microscopy (TEM) and scanning electron microscopy (SEM) have been used to examine features of this size, the new technique of scanning tunnelling microscopy (STM) offers unique opportunities to obtain high-resolution topographical images of the groove structure. In this paper we have investigated the topography of the groove structure in commercially available optical disc substrates made by injection-moulding of polycarbonate.

The purpose of this paper is to demonstrate the utility of STM in studying substrates and media used in optical recording. In addition, we will show that asymmetry, defects and local tilt, all potential problems for any optical disc substrate, can be measured quantitatively. Although we have not specifically addressed the uniformity of the substrates examined in this work, the measurements described here could easily be done in several spots on a substrate. This would provide a quantitative description of the uniformity of the disc.

2. Sample preparation and experimental details

The polycarbonate substrates chosen for this study are available commercially and were made by an injection-moulding process. The grooves are created during the moulding process by the stamper, a mould insert. The stamper is made from a master by a replication process using electrodeposition. There are typically two intermediates between the original master and the final stamper; the first is called the father and the second is called the mother. The glass master is made by writing on a photoresist layer with a focused argon-ion laser beam [1]. Features as small as 0.3 μm can be written on the master. The master is then developed, coated with silver, and electroplated with nickel to make the father. Usually, the father is replicated (via electroplating nickel) to make several mothers, which are in turn replicated into several sons. Thus a single master is used to make many stampers. The details of this process have been reviewed [1]. It is clear that defects and other surface features can be created at any step in the above process, and they will eventually show up on the polycarbonate substrate. Although in this study we have looked only at the final substrates, STM clearly will be equally useful in studying masters, fathers and stampers.

To adequately investigate the utility of STM techniques, we have chosen two different discs for this initial study. Each is characterized by different groove geometry and will be designated in the following text as Sample A and Sample B. In order to prepare samples for study, selected regions of the discs were cut into 1 cm $\times$ 1 cm pieces using a pair of heavy-duty shears. Although there was slight distortion near the sample edges, areas away from the edges were believed not to be affected by shearing. Most of the sample preparation was conducted in a clean-room environment.

The samples cut in this way were first coated with thin gold and platinum films using ion-beam sputtering (IBS). This step was required to produce a conducting layer on the insulating polycarbonate substrate. The

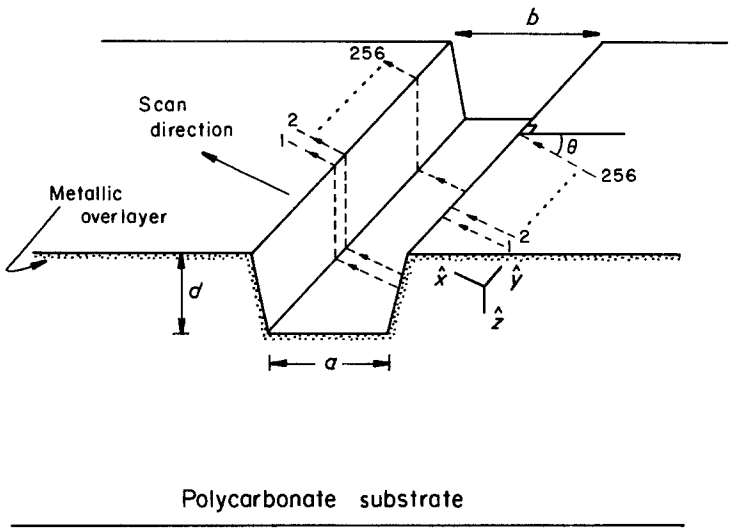

Figure 2 An illustration of the scanning geometry used in the scanning tunnelling microscopy studies. The tungsten tip carrying a tunnelling current of ~ 1 nA is rastered across the metallic coating sputtered on to the polycarbonate substrate. The rastering direction (x) occurs at an angle θ with respect to the direction normal to the groove direction. Typically, θ is estimated to be less than ~ 10°. The tip motion in the z direction required to maintain a constant tunnelling current provides the topographical information about the groove geometry.

sputtering was performed in a vacuum chamber using pure argon gas at a pressure of 10^{-5} torr while the polycarbonate substrates were held at room temperature. This technique is known to produce homogeneous thin films [2]. The sputtering was performed on a B370 EM-Microsputterer from Ion Tech Ltd (Teddington, Middlesex, U.K.). The thickness of the sputtered layers was measured using a quartz crystal thickness monitor.

The scanning tunnelling microscope used in this study was a standard "pocket-size" microscope operating in air in the constant-current mode [3]. In the studies reported here, an electrochemically etched tungsten tip was used to provide the tunnelling current. Further details regarding the principles of the STM are given elsewhere [4, 5]. The scan rates of the tip across the polycarbonate surface were ~ 0.1 Hz. A computer was available to digitize the voltages applied to the x, y and z piezoelectric transducers at 512 × 256 regular intervals. The voltages on the piezoelectric transducers were transformed into positions by calibrating their voltage response in a separate experiment against the known lattice spacing of graphite. In addition to direct recording of STM line scans on a chart recorder, the STM images could be displayed on a graphics monitor for further examination. Typically tunnelling voltages of 0.5 V and tunnelling currents of ~ 2 nA were used during the course of this study.

For the purposes of the STM study, knowledge of the orientation of the groove structure in the metal-coated polycarbonate substrates with respect to the scanning direction was required. In this study, the polycarbonate substrates were carefully positioned on the sample stage of the STM with the aid of an optical microscope. Considerable care was exercised to align the groove structures in a direction perpendicular to the x-scanning direction of the STM (Fig. 2). This allows scans to be made through the groove structure at an angle θ measured from the perpendicular to the groove as illustrated in Fig. 2. Because we are not able to set θ precisely to zero, the lateral groove dimensions determined by the STM may therefore appear longer by a factor of $1/\cos\theta$. Since Θ_{max} is estimated to be ~ 10°, the maximum error in the width measurement of a groove dimension is expected to be ~ ± 1.5%.

In order to provide corroborating evidence about the overall surface topography of the grooved discs, single-stage carbon replicas of the sample surfaces were prepared for TEM study. To prepare these replicas, the polycarbonate sample was first shadowed with Au–Pd at a shallow angle (typically 30° in this study). This shadowing was then followed by the deposition of a thin (~ 30 nm thick) carbon layer. The shadowing and subsequent carbon deposition were conducted in a vacuum evaporator at a pressure of 10^{-6} torr. The polycarbonate disc was then carefully dissolved using methylene chloride. The carbon/Au–Pd replicas produced in this way were placed on a standard 3 mm diameter copper grid for further TEM examination.

Some of the studies of the polycarbonate substrate required thin sectioning of the coated discs in a direction perpendicular to the grooves. This was done by carefully sectioning the discs in an LKB Ultramicrotome to a thickness of typically 100 nm. The thin sections were placed on a copper grid and carbon-coated before TEM studies were made.

3. Results

An SEM micrograph of an IBS gold-coated polycarbonate disc (Sample B) revealing the general groove and land morphologies is shown in Fig. 3. It is evident from this figure that the nominal width of the grooves in Sample B is ~ 0.5 μm. A nominal periodicity of the groove structure was determined to be ~ 1.5 μm. No grains of gold were resolved by the SEM even at magnifications as high as 50 000 ×.

A complementary TEM study was also performed to investigate the quality of the gold-overlayer. In order to accomplish this, ultrathin cross-sections of the IBS gold-coated disc were produced as described above and imaged in the TEM. The heavy line evident in Fig. 4 illustrates the groove profile due to the gold coating and provides good evidence that the coating layer continuously blankets the entire groove geometry. Furthermore, the thickness of the gold overlayer determined from these pictures is in reasonable agreement with the readings from the thin-film deposition thickness monitor. The uneven nature of the groove profile is an artefact that results from the microtome thin-sectioning techniques employed.

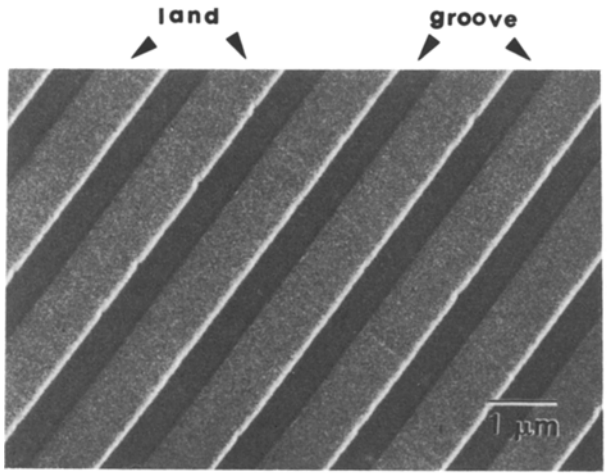

Figure 3 SEM micrograph of Sample B showing the overall surface morphology in a grooved polycarbonate optical disc.

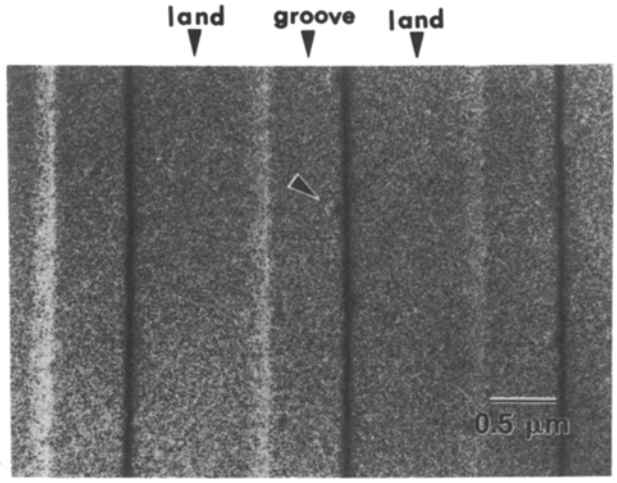

Figure 5 TEM micrograph of a carbon replica of an ion-beam sputtered gold-coated polycarbonate disc (Sample B). The grain size of some of the isolated gold particles that have agglomerated on the surface is evident from careful study of this picture. The large blister-like features (arrow) are believed to result from defects in the polycarbonate substrates.

TEM studies of carbon replicas made from the surface of the IBS gold-coated disc were made to further characterize the grain structure of the gold-coating. Fig. 5 shows a representative micrograph from Sample B that reveals some isolated gold particles which have sizes around 10 nm in diameter. Also evident from this figure are large "blisters" (arrow in Fig. 5) in the polycarbonate disc with typical dimensions of 100 nm across. These features are not associated with the sputtering process but are rather defects in the substrate. As such, they have important implications in producing potential reading errors in the marked discs.

The quality of typical STM images obtained for Samples A and B are shown respectively in Figs 6 and 7. These figures show a sequence of line-scans recorded by the STM in which the grooves in the disc are clearly evident. The apparent tilt evident in each image occurs because the tunnelling microtip is never at right-angles to the sample surface under study. The large apparent magnitude of the tilt is misleading and is caused by the large difference between the z and x axis scales in these figures. The actual tilt can be readily calculated using the appropriate length scales and is found to be $\sim 5°$.

An important consideration regarding the quality of these images is the possibility of producing a tunnelling current from different microtips during the course of an STM scan. The possibility of tunnelling from more than one microtip increases with the overall length of the scan and the roughness of the surface. In spite of these concerns, the high quality of the images obtained in this study provides good evidence that tunnelling from only one microtip is important. If tunnelling from two different microtips was occurring as the tip descended or ascended along the wall of the groove, several adjacent line-scans would show a sharp vertical discontinuity. This discontinuity would be related to the vertical offset distance between the two microtips located on the STM tunnelling tip. The STM images no doubt contain a distortion due to the finite resolution of the tunnelling tip. However, the dimensions of the groove are so much greater than the lateral resolution (~ 0.2 nm) of the STM, that the degree of distortion is insignificant.

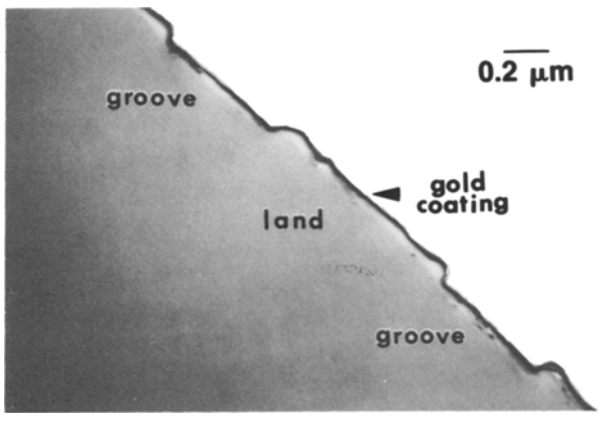

Figure 4 TEM micrograph of an ultrathin cross-section obtained from an ion-beam sputtered gold-coated disc (Sample B) illustrating the continuous coating of the gold overlayer (dark band).

A number of previously unobserved features are evident from these STM scans. First, it is apparent that a sizeable groove asymmetry is present, especially in Sample B. Second, different degrees of surface roughness are observed between the land and groove bottom when compared to the edges of the grooves. Third, blister-like imperfections, similar to those observed in Fig. 5, are also apparent from the STM images. In addition, the detailed topography of the grooves can readily be assessed from the STM images. This capability is an inherent feature of the STM technique and is illustrated for Sample A by plotting a single STM line-scan in Fig. 8. The details of the groove structure can clearly be resolved and precise measurements of the topography can be readily made.

Because of the importance of groove geometry for optical disc recording, further SEM and TEM studies were conducted to find corroborating evidence for the groove asymmetry revealed from the STM images. Fig. 9 is a TEM replica micrograph showing typical groove structures in Sample A. TEM replicas of Sample B, shadowed from opposite directions (but always

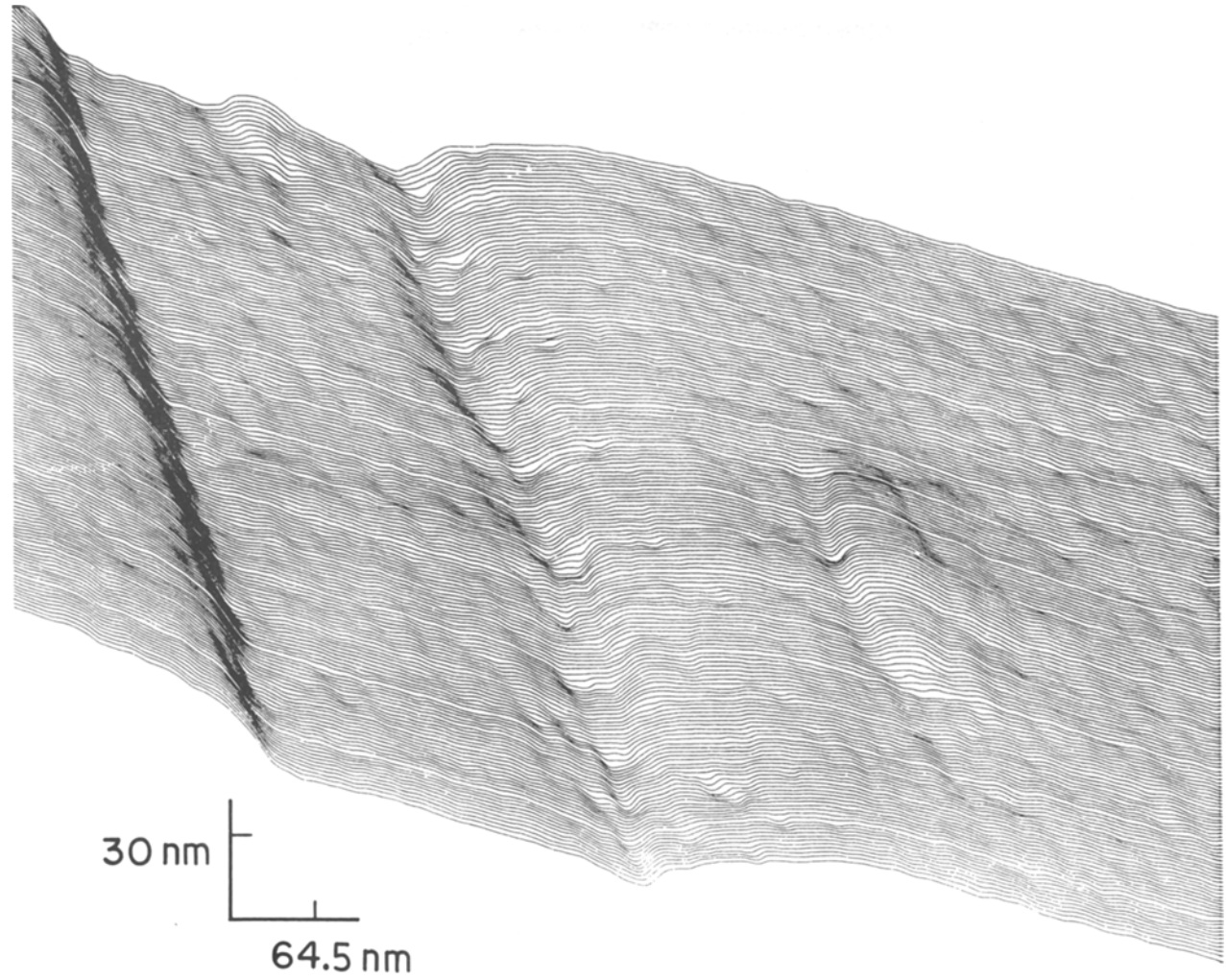

Figure 6 STM picture of the groove structure of Sample A. The total scan is 1000 nm in the x direction by 449 nm in the y direction. Note that the z-axis scale is roughly 2.5 times the x-axis scale and hence produces a distorted real-space image of the groove geometry. This enlargement of the z-axis scale, a characteristic feature of all STM images, allows the detailed topographic information on the groove structure evident in this figure.

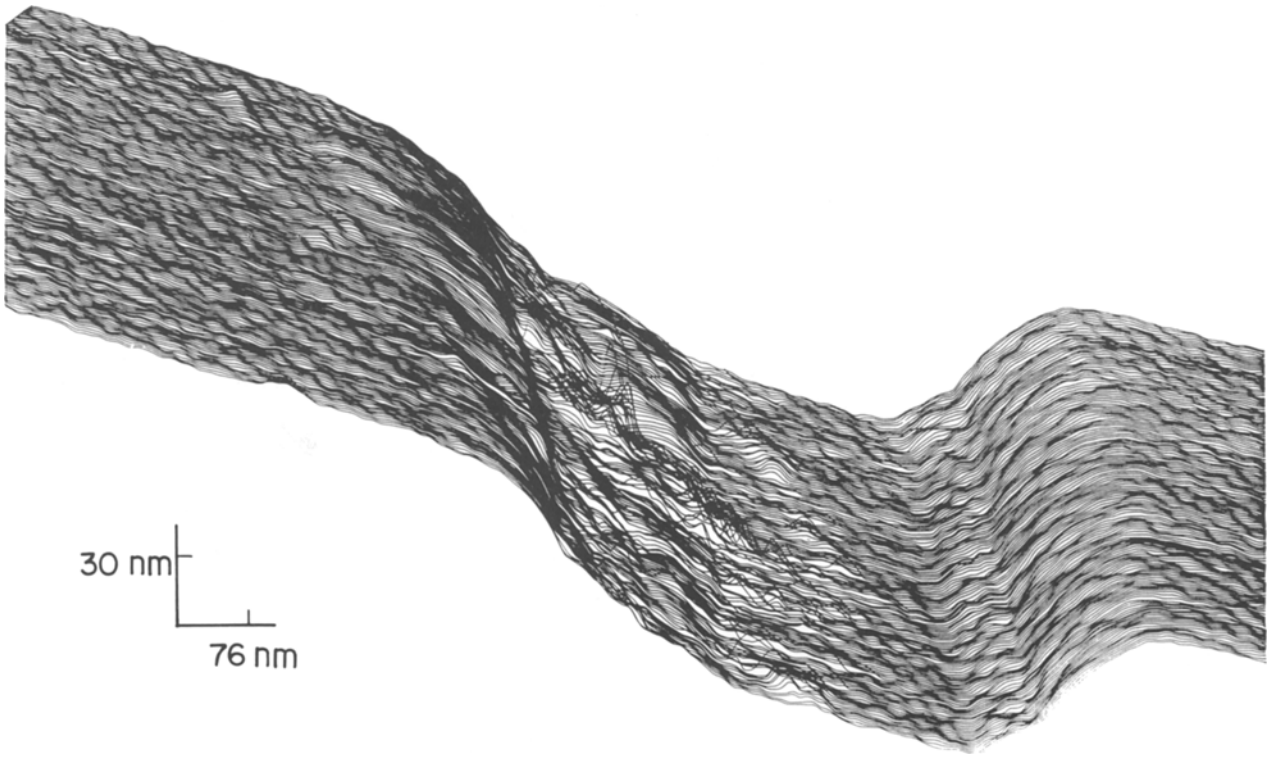

Figure 7 STM picture of the groove structure of Sample B. The total scan is 1475 nm in the x direction by 395 nm in the y direction. Note that the z-axis scale is roughly 2.5 times the x-axis scale as in Fig. 6.

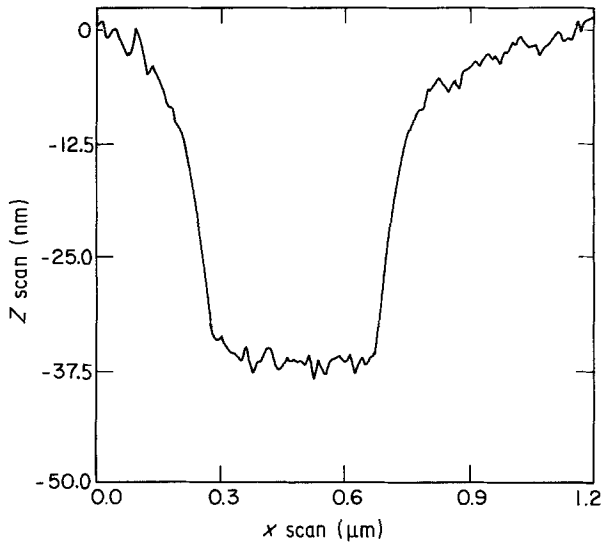

Figure 8 A plot of line-scan No. 2 obtained during the STM imaging of Sample A. The solid line connects the position of the tip along the z direction at 512 equally spaced points along the x direction. The details of the groove structure are clearly shown in this figure.

normal to the grooves), are also shown in Fig. 10. The depth of the groove can be estimated from the white shadow width, while the slope of the groove edge can be estimated from the dark shadow width. The accuracy of the depth determined by this method depends on the accuracy of the measured shadowing angle as well as on the TEM resolution.

Comparing Figs 9 and 10b, it is evident that Sample B has wider and deeper grooves than Sample A. In addition, significant groove asymmetry was also observed in Sample B, in agreement with the STM results. This asymmetry in groove profile is evident from Fig. 10, where the inner edge (arrow in Fig. 10a) was found to have a much steeper slope than the outer edge (arrow in Fig. 10b). Further TEM studies of ultrathin cross-sections provide a means of direct imaging of the groove profile. An example of this technique for Sample B is shown in Fig. 11 and the essential details revealed in the STM study are reproduced.

Table I compares the dimensions of the grooves from Sample B using three techniques – STM, TEM/replica, and TEM/cross-section. The agreement between the techniques is clearly excellent. STM, however, has advantages for this type of investigation which include

(a) the ability to provide complete topographical information from a single sample; and

(b) straightforward sample preparation (comparable to SEM).

TABLE I A comparison of the important optical disc groove dimensions (Fig. 1) determined from three different microscopy techniques

Technique	Sample A		Sample B	
	a (nm)	d (nm)	a (nm)	d (nm)
STM	380	38	540	80
TEM (cross-section)	–	–	570	76
TEM (replica)	370*	45*	550	78

*A sample similar to Sample A but from a different batch.

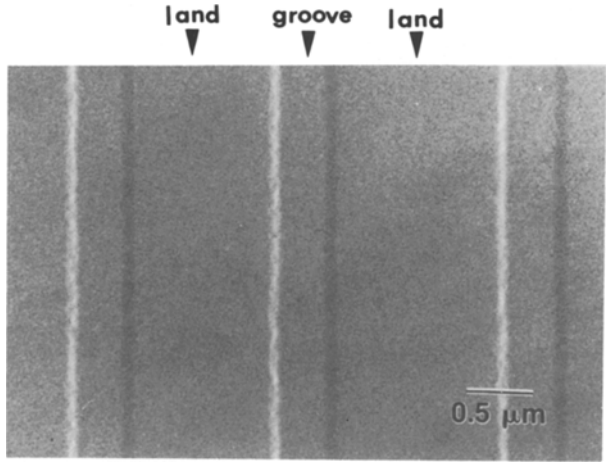

Figure 9 TEM micrograph of replica from Sample A. The shadowing direction is normal to the grooves and radially outward.

As mentioned briefly above, a careful examination of Figs 6 and 7 indicates variable surface roughness at different places on the disc. The land and bottom of the grooves appear generally smoother in the STM scans than the edges of the grooves. In order to determine whether this is an artefact of the deposition techniques employed, new samples were prepared using different metal overlayers with different thicknesses. Figs 12 and 13 show high-resolution STM scans of this surface roughness on Sample B for ~4 nm platinum

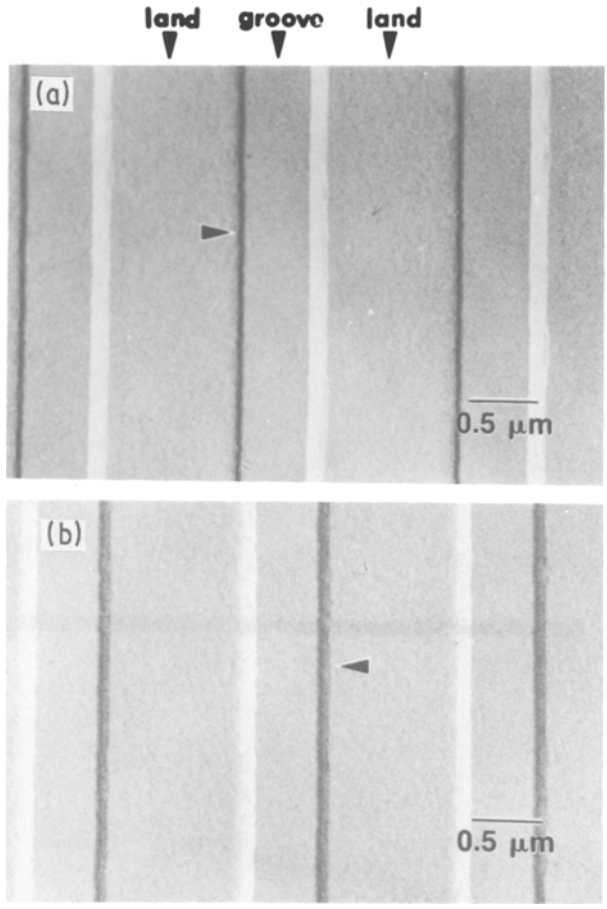

Figure 10 TEM micrographs of replicas from Sample B shadowed from opposite directions, normal to the grooves, illustrate the asymmetry of the groove structure. In (a) the shadowing direction is radially inward while for (b) the shadowing direction is radially outward.

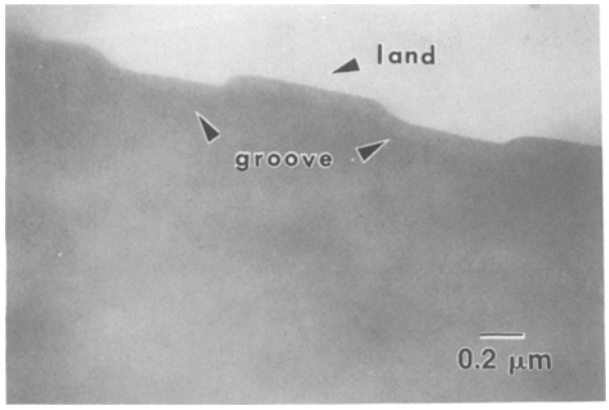

Figure 11 TEM micrograph of an ultrathin cross-section of a sample similar to Sample B illustrates the groove profile. The groove asymmetry in this sample is also evident from this picture.

coating and ~10 nm gold coating. The platinum-coated sample has features of characteristic dimension of ~2 nm while the gold-coated sample has mound-like structures with dimensions of ~10 nm. For the case of platinum, this structure is similar in detail to the recent STM studies of platinum films on mica substrates reported by Vazquez *et al.* [6]. It is evident from the present study that the platinum-coated sample produces a finer texture than the gold, in agreement with prior expectations based on established coating techniques developed for SEM and TEM work.

In addition to this fine structure that can be associated with the coating material, STM scans did reveal an oriented structure which is not correlated with the coating. This structure can be seen in Fig. 14, which shows a high-resolution STM scan on Sample B with ~10 nm gold coating. The features observed in this scan are elongated structures, some tens of nanometres in length. This preferential elongation is not associated with a thermal drift of the piezoelectric transducers because it is most prominent near the groove edges (see Fig. 7). This structure is probably associated with an alignment of the gold coating along a structure inherent in the polycarbonate substrate that presumably occurs during the injection-moulding of the substrate.

In addition to those features that are evident from the STM images presented above, a number of other important results have emerged from this initial study of optical discs using the STM.

Clear evidence for asymmetry, roughness and variability in the grooves has been found. STM allows features down to about 5 nm to be observed and characterized. However, care must be taken in the coating technique, and the metal coating does produce a microstructure which will obscure features at much smaller sizes. However, clear differences in microstructure in different regions can be observed and are probably related to the process used in manufacturing the discs.

The quantitative numbers obtained from STM topographs have three primary sources of uncertainty. The first is the absolute calibration of the piezoelectric transducers. Second, the metal coating might slightly alter the relative topology. This should be a very small effect when thin (10 nm) coatings are used as they were

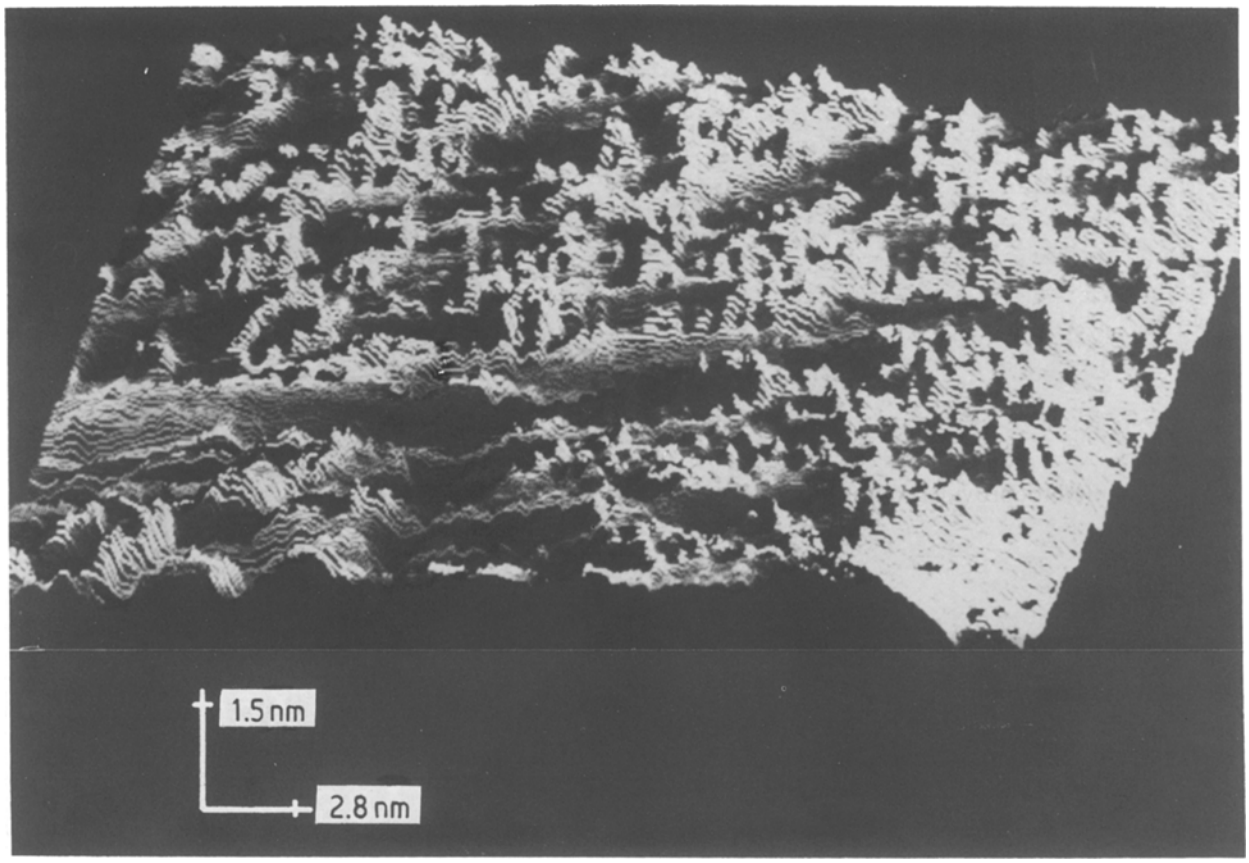

Figure 12 STM image of Sample B with 4 nm layer of platinum. The image presented is 50 nm × 50 nm and illustrates the surface morphology of the platinum coating on the polycarbonate substrate.

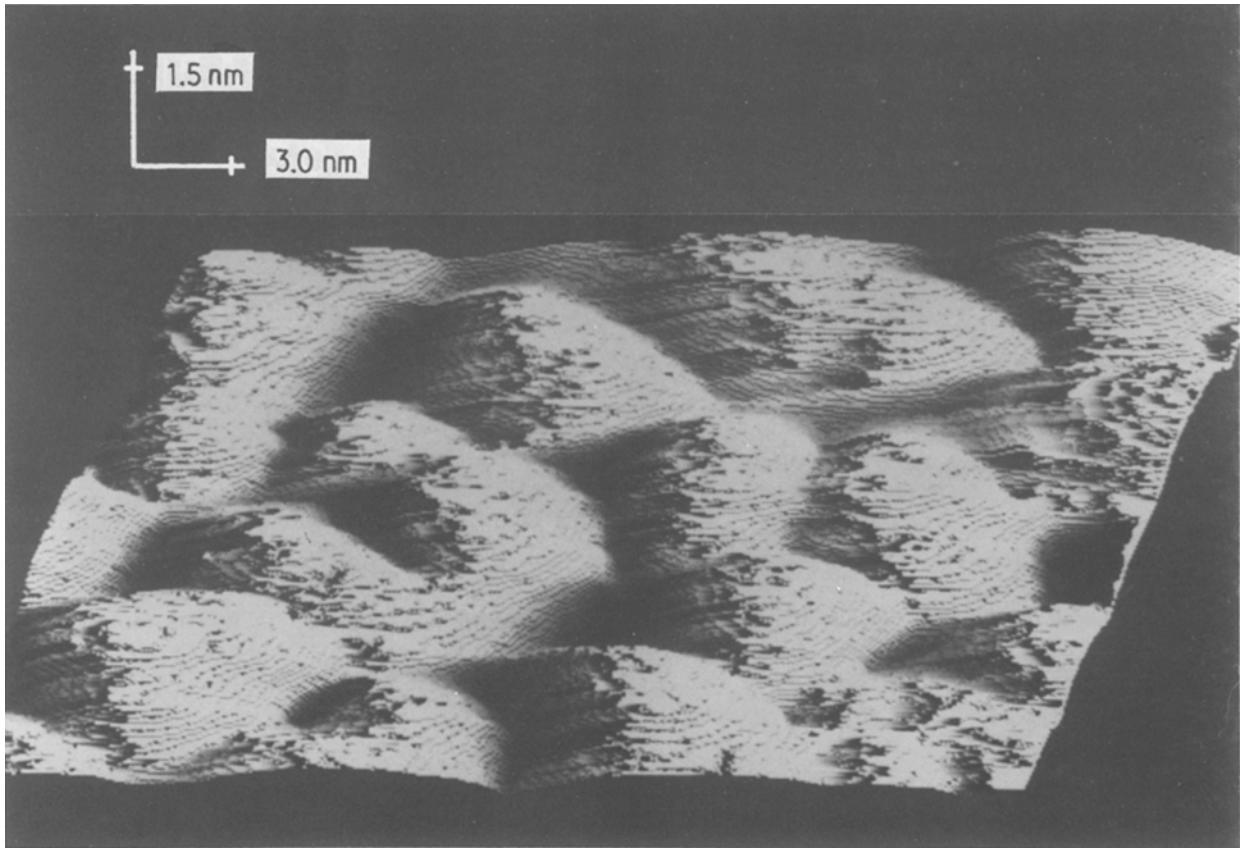

Figure 13 STM image of Sample B with ~ 10 nm layer of gold. The image presented is 53 nm × 53 nm and illustrates the surface morphology of the gold coating on the polycarbonate substrate. Mound-like structures are evident with typical dimensions of ~ 10 nm in diameter.

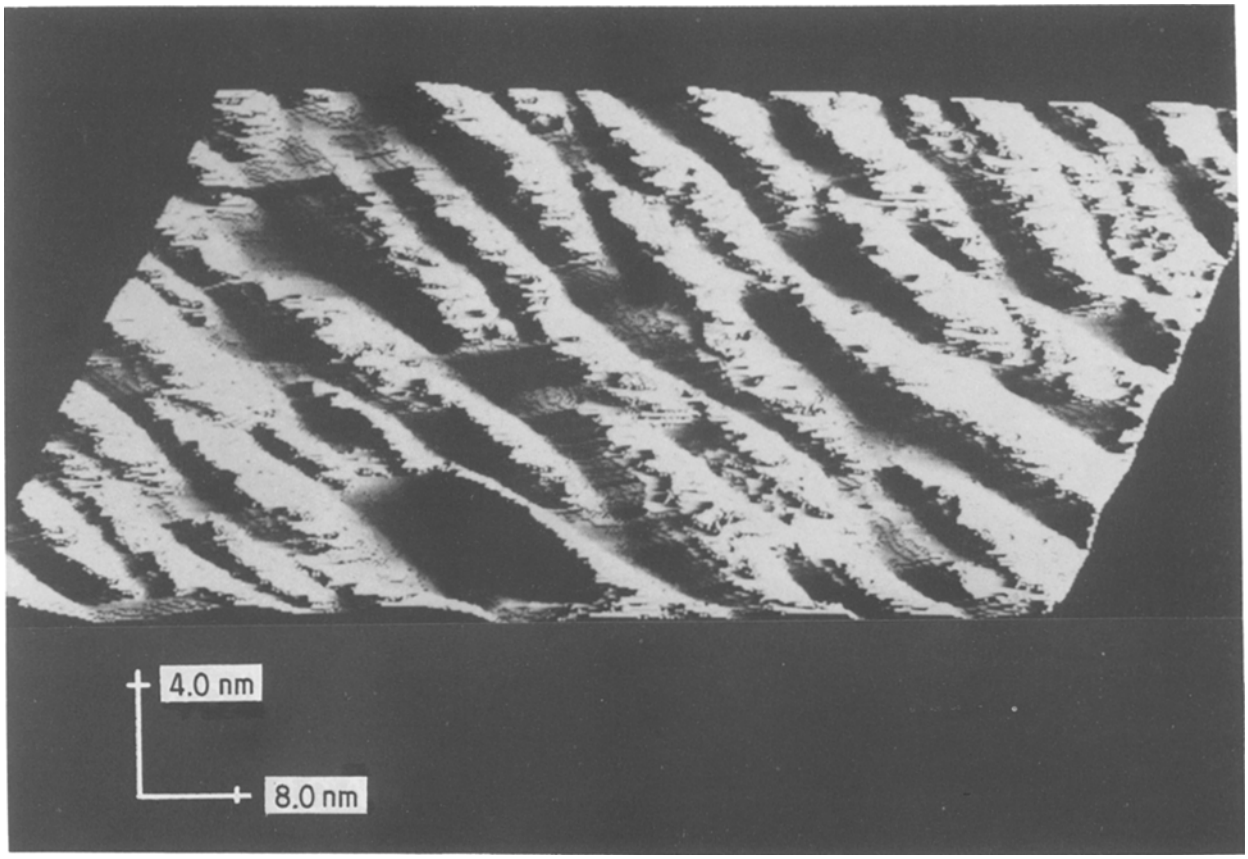

Figure 14 STM image of Sample B with ~ 10 nm layer of gold. The image presented is 140 nm × 140 nm and illustrates the long-range, chain-like alignment observed on the polycarbonate substrate.

in this study. Third, the exact scan orientation relative to a groove is not known. Optical alignment has been used to minimize this effect, keeping it to under 5%.

4. Conclusions

The shape of grooves on two different optical disc substrates that have been ion-beam sputtered with gold and platinum metal overlayers of different thicknesses has been measured. The coatings were made using ion-beam sputtering in order to minimize any microstructure effects of the coating. This study shows that metal coatings with a thickness of ~4–10 nm are sufficient to allow reliable STM images of polymer substrates. Although this is certainly sufficient for studying such gross features as the grooves in optical discs (sizes > 10 nm) we have also observed features on the surface of these discs on a much smaller size scale. The samples were coated using different metals and different thicknesses in order to help determine the degree to which the observed fine structure is due to the coating or is intrinsic to the polymer surface. Evidence was found that the fine structure is intrinsic to the surface and is probably caused by the details of the moulding process. This idea is supported by the differences in fine structure on the land and in the bottom of the grooves.

Thus we have demonstrated the utility of STM in characterizing polymeric optical disc substrates. This work also demonstrated that STM can easily be used to examine the surface structure of polymers on length scales greater than 5 to 10 nm. We can conclude that STM is a powerful tool both for studies of fabricated polymeric parts and for fundamental studies of microstructural effects in polymer alloys and composites.

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References

1. G. BOUUWHUIS, J. BRAAT, A. HUIJSER, J. PASMAN, G. VAN ROSMALEN and K. SHOUHAMER IMMINK, "Principals of Optical Disc Systems" (Adam Hilger, Bristol and Boston, 1985) pp. 189–209.
2. C. S. CLAY and G. W. PEACE, *J. Microsc.* **123** (1981) 25.
3. CH. GERBER, G. BINNIG, H. FUCHS, O. MARTI and H. ROHRER, *Rev. Sci. Instrum.* **57** (1986) 221.
4. P. K. HANSMA and J. TERSOFF, *J. Appl. Phys.* **61** (1987) R1.
5. Proceedings of the First International Conference on Scanning Tunnelling Microscopy, edited by N. Garcia, *Surf. Sci.* **181** (1987).
6. L. VAZQUEZ, J. M. GOMEZ RODRIGUEZ, J. GOMEZ HERRERO, A. M. BARO, N. GARCIA, J. C. CANULLO and A. J. ARVIA, *ibid.* **181** (1987) 98.

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