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M. Maywald
Bergische Universität Wuppertal

R. J. Pylkki
TopoMetrix, Santa Clara

L. J. Balk
Bergische Universität Wuppertal

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IMAGING OF LOCAL THERMAL AND ELECTRICAL CONDUCTIVITY WITH SCANNING FORCE MICROSCOPY

M. Maywald, R.J. Pylkki[1], and L.J. Balk*

Bergische Universität Wuppertal, Lehrstuhl für Elektronik, D-42097 Wuppertal, Germany
[1]TopoMetrix, 5403 Betsy Ross Dr., Santa Clara, CA 95054-1162, U.S.A.

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Abstract

The evaluation of thermal and electrical properties has been demonstrated using a scanning force microscope under ambient air condition. For the first time, the investigation of thermal conductivity on a chemical vapor deposited (CVD) grown diamond surface has been performed with a lateral resolution below 200 nm. Depending on the growing parameters, structures consisting of lines of different thicknesses and spacings are visible. The determination of the electrical structure has been carried out with a resolution of 2 nm using the contact current mode (CCM) showing similar structures. The thermal images exhibit a high thermal conductivity mostly on the diamond crystals whereas the electrical conductivity reached its highest values between them.

Key words: Scanning force microscopy, thermal conductivity, electrical conductivity, chemical vapor deposition (CVD), diamond film.

*Address for correspondence:
Ludwig J. Balk
Sonderforschungsbereich 254,
Bergische Universität Gesamthochschule Wuppertal
Lehrstuhl fur Elektronik
Fuhlrottstraße 10,
D-42097 Wuppertal
Germany
Telephone number: (+49-202) 439 2972
FAX number: (+49-202) 439 3040

Introduction

Since the scanning force microscope (SFM) has been invented [1] many different applications have been developed to image specimen features with a high spatial resolution. These include magnetic [3], electrostatic [12], frictional [8], and thermal [7, 10, 13] properties. Up to now investigations of the thermal and electrical structure on a chemical vapor deposited (CVD) diamond film have not been performed with a resolution below 1 μm [5, 6] so that the data are always averaged over a given area. To overcome this problem and to improve the understanding of the growth parameters as well as the material properties on a nanometer scale, techniques with a high spatial resolution have to be applied. The aim of this paper is to demonstrate the possibilities of thermal and electrical conductivity measurements by means of a scanning force microscope on a diamond surface. Thermal properties were investigated by a scanning thermal microscope (SThM) [2] and the electrical conductivity was studied by contact current mode (CCM) [9]. Both applications were performed with a modified commercially available SFM (TMX 2000, TopoMetrix, Santa Clara, CA, U.S.A.) under ambient air condition. The investigated specimens were CVD diamond films of two different average thicknesses and crystal sizes. The specimens were undoped, polycrystalline and the diamond films were grown on a silicon substrate on a (111) surface. They have previously been investigated by cathodoluminescence [4]. The substrate of the thick diamond films has been removed by etching.

Experimental Procedures

Scanning thermal microscope (SThM)

To determine the relative thermal conductivity of a specimen we used a self heating design [2]. Figure 1 shows a schematic of the SThM probe. The probe consists mainly of a bent 75 μm silver Wollaston wire which has a 5 μm platinum core. The wire is fixed to a holder, and an aluminum foil is cemented onto the wire acting as a mirror. After the wire is properly adjusted, the end of the loop is etched, uncovering a small length of platinum. This exposed filament is the thermal element of the probe. Resistive heating occurs in the filament since it is the only portion of the probe with a significant resistance. The probe tip is part of a Wheatstone bridge to enable measurements at a constant probe sample temperature difference (e.g., $\Delta T = 40$ K). High thermal conductivity specimens will cause the thermal feedback loop in order to increase the applied voltage to keep the probe temperature constant. Therefore, the monitored signal is the applied voltage which is correlated directly to thermal conductivity [2], if it is assumed that the heat transfer through air is constant. The SFM is equipped with a photo diode, using the optical lever method, and operated in the constant force mode. Typically, spring constants of the Wollaston wire cantilevers are of about 5 N/m. Since the frequency response of the thermal signal is about 100 kHz [2], the limitation for scan speed depends only on the roughness of the surface. Here the scan speed was 3 lines/sec with a resolution of 200 x 200 pixels. Thermal and topographic images were obtained simultaneously.

Contact current mode (CCM)

The CCM [9] evaluates the current flow between two electrodes by measuring the voltage drop over a serial resistor R (see Fig. 2). Therefore, a voltage is applied between these electrodes to provide a current. The voltage in principle can be AC or DC in nature which allows the application of different amplification techniques (e.g., lock-in). In this case, a DC voltage (1 V) was selected. As can be seen in Figure 2, the electrodes are formed by the tip and the silver paint. To perform CCM measurements, the SFM must be equipped with a conducting tip. A standard silicon nitride cantilever was coated with a gold film of about 70 nm. The measured voltage drop across R (Fig. 2) was amplified and sent via an analogue to digital converter (ADC) to a computer, where the current was recalculated and displayed. The measuring programs were modified in such a manner that a simultaneous measurement of CCM and topography is possible. The scanned images have a resolution of 200 x 200 pixels and a scan rate of 3 lines/sec similar to the parameters of the thermal measurements.

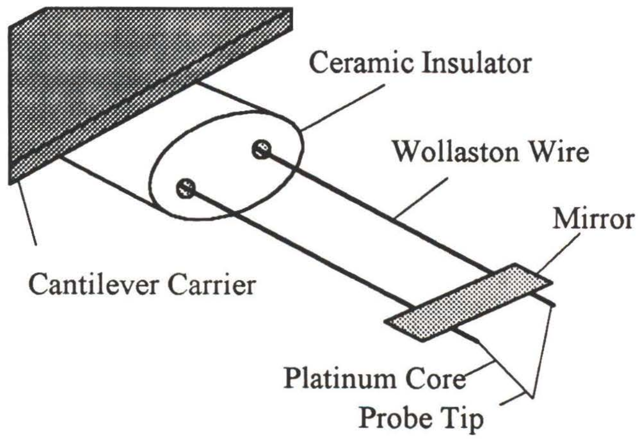


Figure 1. Schematic diagram of scanning thermal microscope probe.

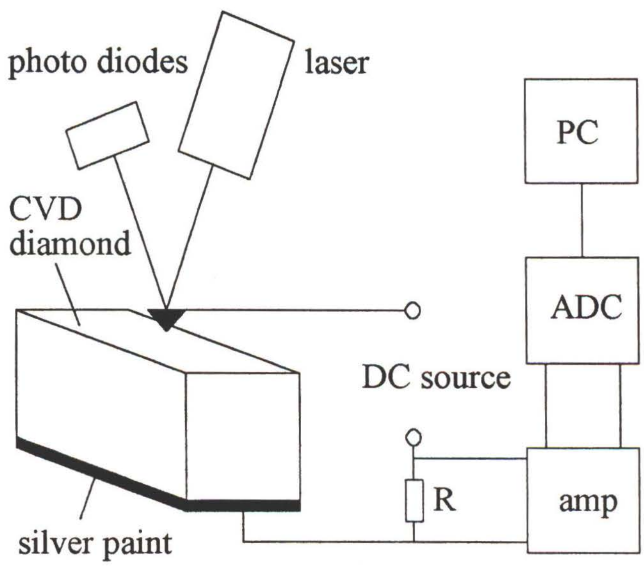


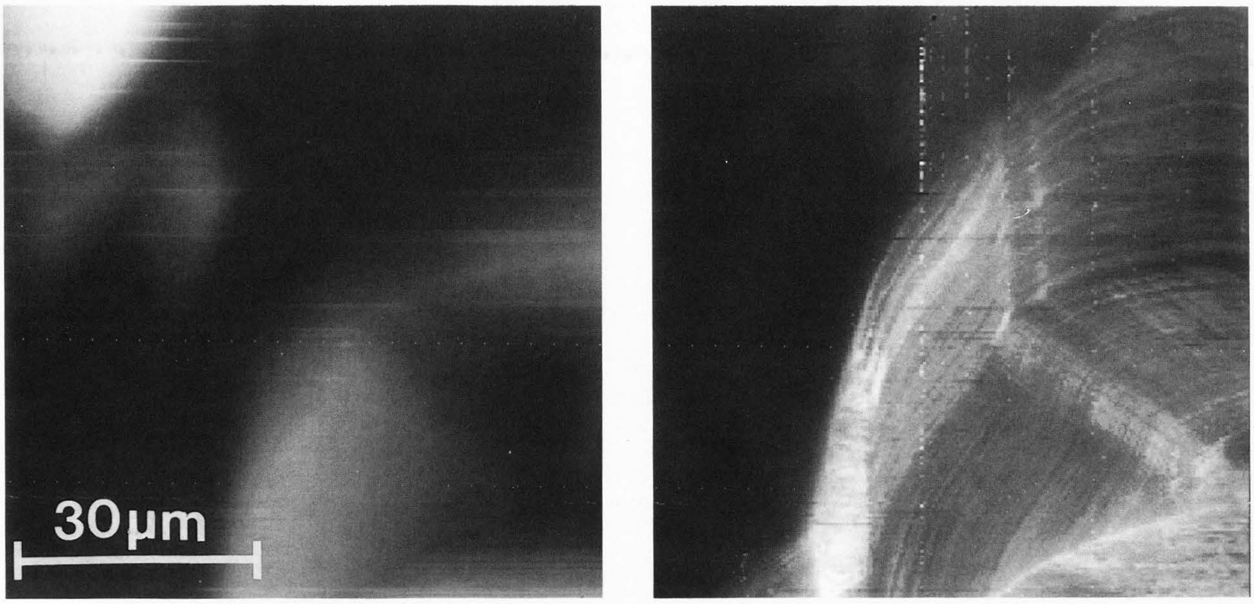
Figure 2. Schematic setup for contact current mode (CCM).

Results

Figure 3 shows simultaneously recorded topography and thermal images of the top side of a back etched thick diamond film at a scan range of 75 μm. The images were obtained on a CVD film of 100 μm thickness and of 20 μm average crystal size.

The bright areas in the topography image illustrate high topographical levels and hence diamond crystals. The overall height difference in this image is about 5 μm. A comparison with the corresponding thermal image demonstrates that the areas between diamond crystals show a lower thermal conductivity whereas the crystals itself are areas of relatively high thermal conductivity. The thermal conductivity corresponding to the very bright values in the image is twice as high as the thermal conductivity corresponding to the very dark values.

Furthermore, a laminar thermal structure is visible that includes lines of different thicknesses, orientations,



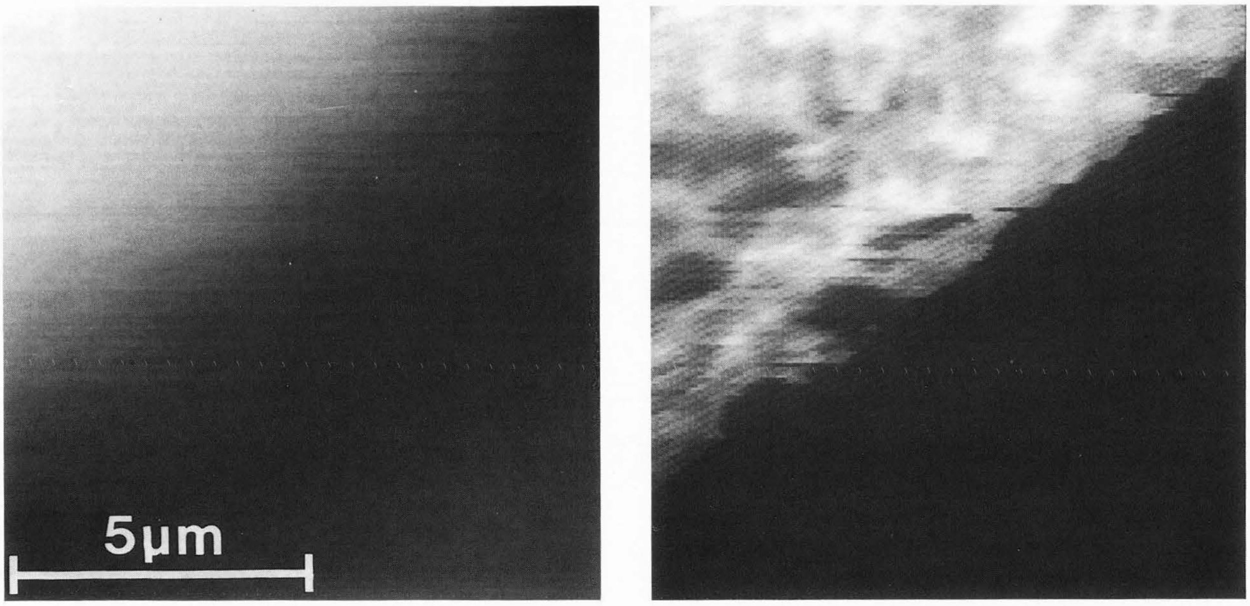


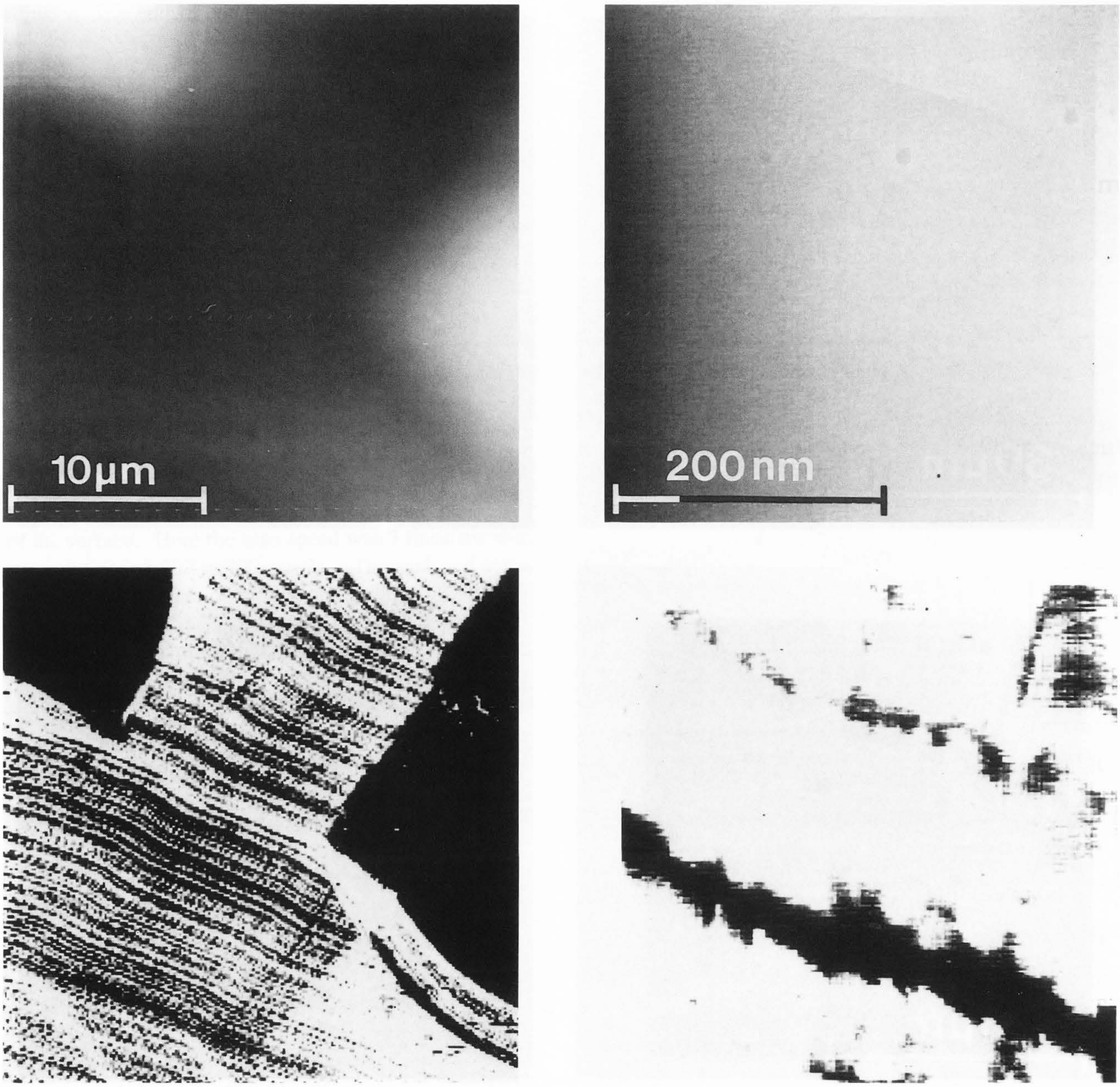
Figure 3 (top). (**a, left**) Topography image and (**b, right**) corresponding thermal image of a thick diamond film.

Figure 4 (bottom). (**a, left**) Topography image and (**b, right**) corresponding thermal image of a diamond/Si interface.

and spacings. The average spacing of the lines is in the range of 1 μm. A further comparison of both images illustrates that the lateral resolution of the thermal image is superior to the detected topography.

Figure 4 demonstrates thermal measurements on a thin (3 μm) CVD diamond film at the vicinity of a Si/diamond interface at a scan range of 10 μm. The topography image (Fig. 4a) illustrates a falling topography from the top left to the bottom right corner without any significant features. The corresponding thermal image (Fig. 4b) obviously shows a sharply distinguished interface and features of different thermal conductivity within the diamond film.

The lateral resolution was determined to be < 200 nm at the interface. The lateral resolution of the thermal image is therefore again much better than that of the topography image. Structures observed in Figure 4b are totally absent in this image.



Figures 5 (at top) and 6 (at bottom). (5a and 6a, at left) Topography images and **(5b and 6b, at right)** corresponding thermal images of a thick diamond film at two magnifications.

Figures 5a and 5b are topography and CCM images obtained on a thick diamond film (compare these to Figs. 3a and 3b) at a scan range of 25 μm. The topography image (Fig. 5a) indicates two diamond crystals, one in the top left corner and the other on the right side of the image. The same areas appear dark in the corresponding CCM image (Fig. 5b) which indicates a low current signal. The area between the crystals demonstrates of an electrical conductivity signal which contains inhomogeneously structured features.

The visible lines, similar to the thermal structure, contain different thicknesses, spacings, orientations, and electrical conductivities. The maximum value of the contact current was 4 nA. The absence of comparable structures in the topography image may be due to limited height resolution. A measurement at a higher magnification performed in the shallow areas (Fig. 6) shows slight topography variation with no visible structure at a scan range of 400 nm. The lateral resolution at the line boundary was determined by a linescan to be 2 nm and

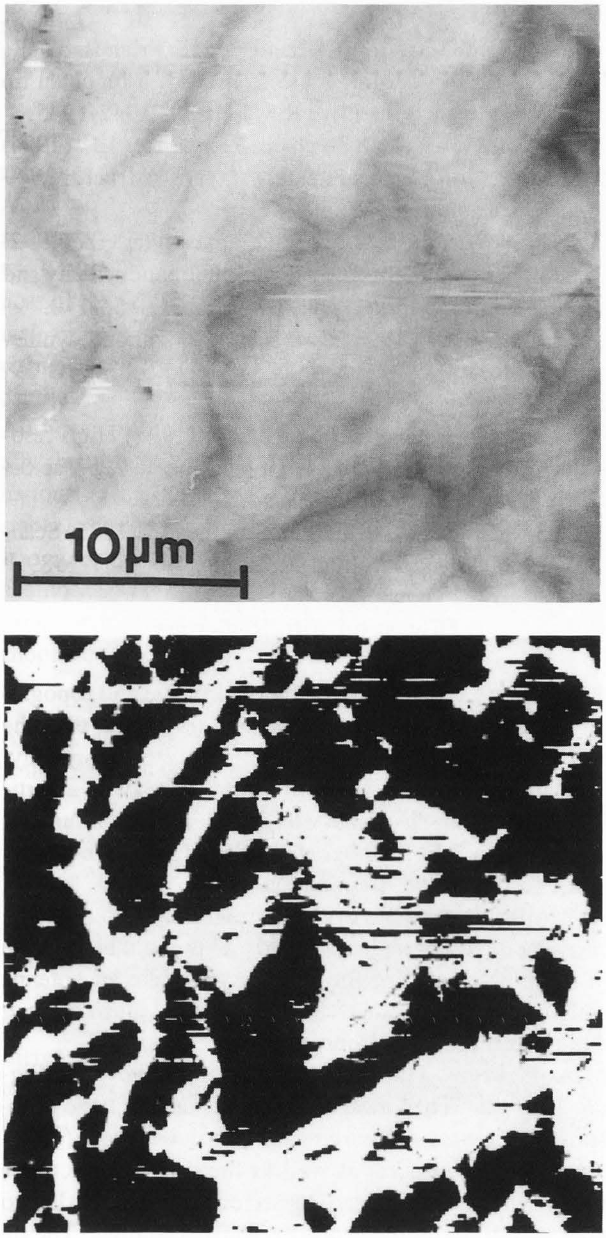


Figure 7. (a) Topography image and (b) corresponding thermal image of a thin diamond film.

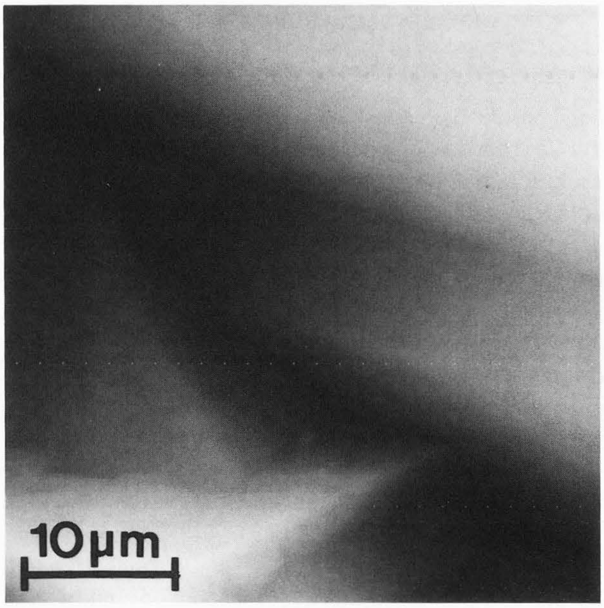


Figure 8. Topography image of a thick diamond film performed with an enhanced tip.

the line spacing to be approximately 150 nm.

Measurements performed on the thin diamond film are illustrated in Figure 7 at a scan range of 25 μm. The CCM image does not contain any structure similar to those observed on the thick diamond film although there is a correlation between Figures 7a and 7b. The areas of high electrical conductivity refer mostly to the areas of low topography although there are some high conducting areas on the crystals itself.

Discussion

Since the lateral resolution is limited by the tip geometry it should be improved by an improved tip design. The origin of the laminar structure occurring on the thick diamond film is still unclear, though similar features appear in topography when scanning with an enhanced tip (Fig. 8). The layered structure visible in some of the images is probably due to the growth parameters of the diamond film. Another indication for this is the measurement on the thin diamond film, because no kind of layered structure comparable to Figure 3b could be observed. The superior lateral resolution of the thermal image compared to the corresponding topographic image may be due to a low force sensitivity of the cantilever itself and due to cohesive forces of contamination layers on the surface.

The CCM measurements detected a high electrical conductivity mostly on the areas between the crystals where graphite or diamond-like materials are supposed to occur [11]. The nature of the electrical layers may also be due to the growth parameters mentioned above. The lateral resolution of the current images is about 2 nm. The inferior lateral resolution in topography may be caused by the gold coating of the cantilever which increases its spring constant and therefore decreases the force sensitivity. Furthermore, cohesive forces should occur in this case, too.

Conclusions

For the first time the thermal structure of CVD diamond has been demonstrated with a lateral resolution below 200 nm and the electrical structure with a lateral resolution of 2 nm which make both techniques suitable tools for a high resolution investigation of thermal and electrical conductivity. The thermal images show a relatively good conductivity on the diamond crystals as expected whereas the contact current signal reaches its highest values between the diamond crystals indicating a non-diamond material. The origin of the laminar structure occurring on some of the specimen investigated is still unclear and needs to be investigated in detail.

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Discussion with Reviewers

T. Thundat: Related to the thermal image and topography images in Figure 3: the authors claim that the thermal image shows more structures than topography. The laminar structures seen in Figure 3b may be an artifact from the tip shape. We have observed such laminar structures in AFM topographs of CVD diamond (only when shown in the illumination mode).

A. Majumdar: Some of the thermal images seem to be carrying the topographical signal. This could be because in the present set up, the probe works like an antenna and picks up any voltage changes from the piezo scanner due to features in the topography.

M.A. George: The cantilever design as shown in Figure 1 for the SThM measurements appears to have problems in that both the wire of the cantilever arms (designated Wollaston wire) as well as the probe can act as a spring which would lead to serious anomalies due to drag and strain on the probe tip while scanning. The tip of a standard AFM cantilever is fixed and rigid while the cantilever arms translate the motion due to the surface morphology of the sample.

M.P. D'Evelyn: Will not the applied voltage be sensitive to the thermal contact between probe and sample even more than to the thermal conductivity?

Authors: To affirm that the observed structures do not correspond to tip artifacts, we scanned the images at different scan angles. Therefore, the detected signal has no correspondence to tip artifacts. Changes due to topographical or contact (tip-specimen) variations could not be excluded in principle although scanning at different scan rates and with different applied loads led to the same results. To look at the effect of the piezo voltage the tip was scanned in a previous experiment in air with a modulated z-piezo. No variations in the thermal feedback loop have been observed.

T. Thundat: The authors should address how contamination effects affect thermal images.
A. Majumdar: It appears from their description of the scanning thermal microscopy technique that the tip is used as an resistive temperature device (RTD). Is it possible to show by any measurement that the total thermal signal obtained corresponds to just the resistance changes in the tip and not from any other portion of the cantilever?
Authors: In the case of diamond, contaminations generally should decrease the thermal conductivity. Contaminations will affect the images only if they are locally stable and cannot be identified throughout several scans. It is not likely to be seen that variations of the cantilever should affect a thermal image during several scans in the same way. Therefore, the detected signal changes should correspond to variations in the tip only.

A. Majumdar: The scanning of the sample was done using a scan rate of 3 Hz. I feel that the scan rate should be decided based on the frequency response of the cantilever.
A. Majumdar: How do the authors eliminate the contributions (noise) due to the electrostatic interaction between sample and the tip?
Authors: When scanning with a conductive tip over a surface in contact mode with a very slow scan speed, no charged carriers will be influenced onto the surface and therefore no electrostatic interactions should occur. As a proof of this statement, no leakage currents and no topographical variations have been observed corresponding to a change of the applied temperature.

A. Majumdar: Can the authors give any estimate of the typical tip diameter?
A. Majumdar: The authors claim that the lateral resolution is about 200 nm. I feel that the probe diameter will decide the lateral resolution of the technique. If the probe diameter itself is about 5-10 μm, how can one expect a lateral resolution of 200 nm?
M.A. George: In addition to an increased force constant, as mentioned by the authors, resolution is limited by the dimension and morphology of the probe tip. A standard silicon nitride cantilever coated with 70 nm of gold would decrease spatial resolution due to an increase in tip diameter.
M.A. George: What are the dimensions and the geometry of the probe tips? How was the lateral resolution determined for the different images: SThM, CCM and SPM?

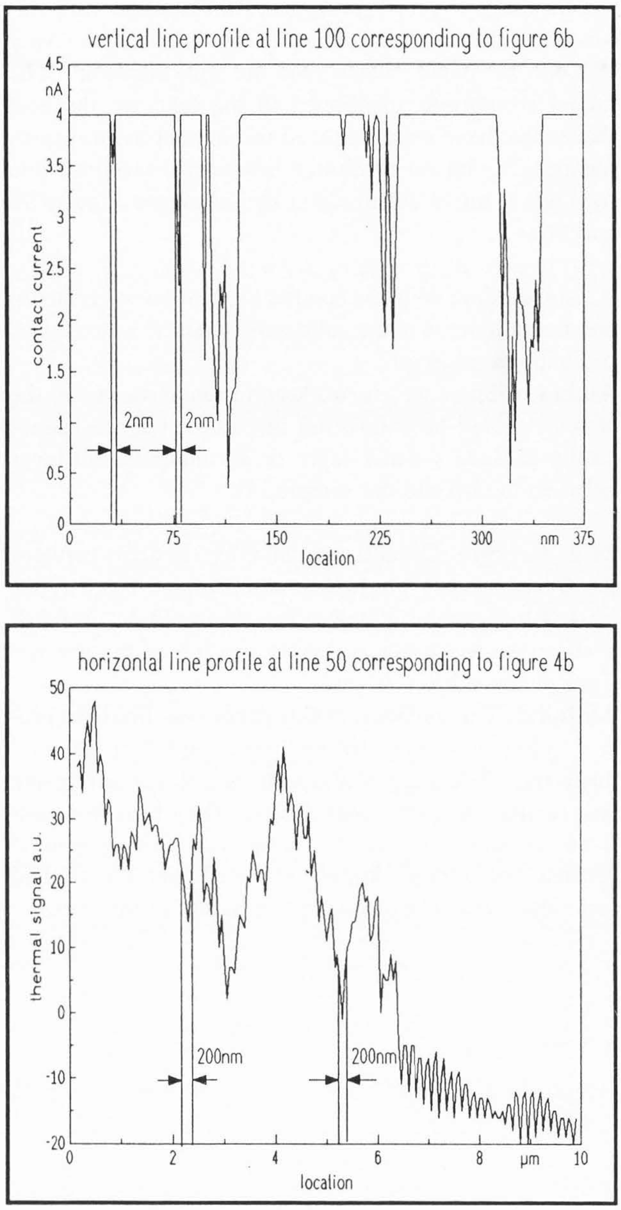

Figure 9. **(a, top)** Vertical line profile of the CCM signal corresponding to Figure 6b, and **(b, bottom)** horizontal line profile of the thermal signal corresponding to Figure 4b.

Authors: For the CCM measurements, the tip diameter was estimated by point contact measurements which led to a diameter of 1.8 nm.

An increase of tip diameter due to coating will not occur generally because each layer of coating atoms will follow the shape of the tip and therefore form a new tip of approximately the same size. The bent Wollaston platinum core has a diameter of 5 μm. But because of the bending, the topography, and the applied temperature, the effective heat emitting tip diameter can vary and become much smaller than the wire diameter. The lateral resolutions mentioned in the text are the best values that have been achieved throughout the measurements. The lateral resolution has been determined due to single features occurring in linescans (see Figures 9a and 9b).

A. Majumdar: What is conducting heat between the tip and the sample: is it air, solid-solid contact, water layer, or continuation layer?

Authors: Since we are working in repulsive mode, the contact should be solid-solid in nature, but we cannot totally exclude a water layer or a contamination layer between the tip and the sample.

M.A. George: The authors claim that the areas between the crystals consist of graphite and diamond-like carbon. This is too general a statement. It would be useful to perform a spectroscopic characterization of the specific films examined in this study.

Authors: The intention of this paper was firstly to present techniques for evaluating thermal and electrical conductivity. The suggested spectroscopic measurements will carried out in the near future. They have not been done yet, and therefore, the statements are very general because the authors know very well that the applied techniques are only of qualitative nature in this case.

M.A. George: Aside from the maximum measured contact current of 4 nA, what are some typical values for the as measured current as a function of surface features.

Authors: We have not observed any topographical features comparable to the electrical structures (see Fig. 6a). Therefore, we are not able to make any statements about the correlation of contact current signal to topographical features.

T. Thundat: Does the thermal conductivity have the same meaning at nanometer scale?

Authors: Up to now we have achieved a lateral resolution of about 200 nm. The obtained images show features that you probably would expect from other techniques but with a better lateral resolution. The question of what effects could be observed when reaching the scale of a few nanometers and if this still could be called thermal conductivity we cannot answer. Hopefully, future experiments could help us to answer this question.