NT-MDT Spectrum Instruments

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Application Notes

Life Sciences

AUTOMATED SPM MEASUREMENTS

A. Alexeev and D. Wouters

Automatic measurements with SPM include automatic adjustment of scanning parameters, measurements of the programmed areas with SPM methods and automatic data analysis. The application areas of the automated SPM measurements are:

- Combinatorial Material Research (high throughput characterization of libraries of the samples with different chemical composition or of samples prepared at different conditions[1]).
- SPM measurements of macroscopic areas by moving of the scanning area over sample surface with the help of the positioning system. As a result areas with a size of several millimeters or centimeters can be measured by SPM (maximum scan range in commercial SPMs is limited by ~100 microns).
- SPM modifications (nanolithography) on the macroscopic areas.
- Quality control in industry (e.g. control of the CD/DVD disk surface).

The modified SPM Solver LS equipped with special software is an essential tool for automatic characterization and modification of surfaces with all basic SPM modes. Fig.1 shows the sample holder of the Solver LS for 4 standard 4-inch silicon wafers, each of them can consist of large amount of samples deposited, for example, by ink-jet printing.

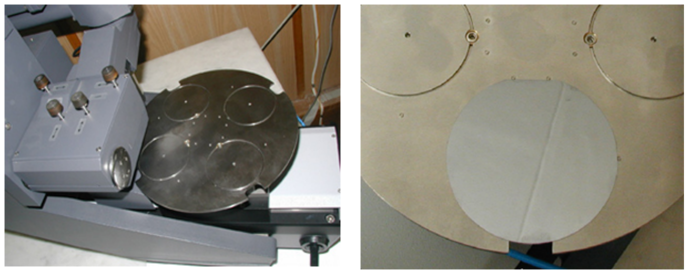

Fig. 1 Modified SOLVER LS for automated measurements (left). Positioning platform for four 4-inch silicon wafers (right).

Fig. 2 shows the menu of automated measurements for 25 points. The coordinates of each point are saved in the program. The software automatically captures an optical image of the current position (with resolution down to 1.5 microns), performs the SPM measurement, moves the sample to the next saved position etc. All saved data can be automatically processed by the software in order to obtain statistics or certain parameter for all the measured areas.

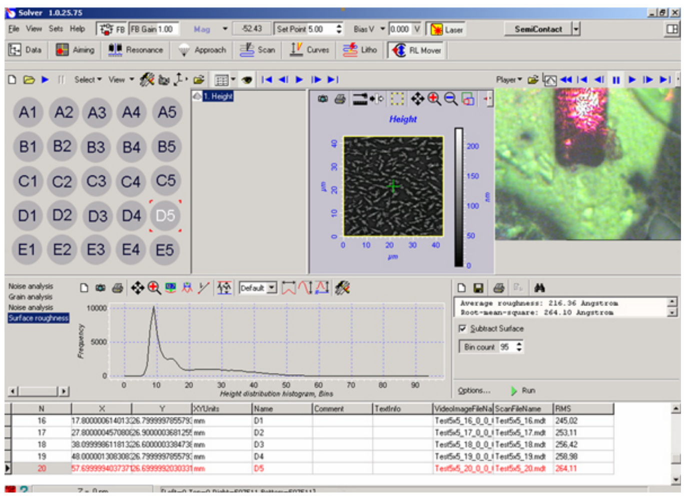

Fig. 2 Menu of the automated SPM.

Results that are described below were obtained in the group of Professor U.S. Schubert (Eindhoven University of Technology, The Netherlands, http://www.schubert-group.com) in collaboration with the Dutch Polymer Institute. The modified SOLVER LS for automatic measurements has been used for these experiments.

Automatic measurements of photo embossed polymer gratings

The selective irradiation through a mask of a sample containing a pre-polymer, monomers and a photo initiator causes the formation of periodically elevated relief structures (Fig.3). For applications in display technology one of the goal is to obtain the highest possible relief structures. The formation of the elevated structures is dependent on a large number of sample preparation conditions like the initial film thickness, composition, the period of the applied mask, intensity of the light and the temperature in the development stage.

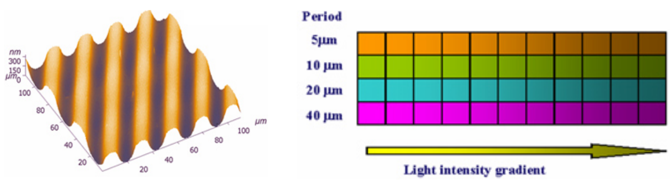

Fig. 3 AFM picture of the structure with 20 microns pitch (left); schematic presentation of the complete sample (right).

To investigate the optimal processing conditions, a combinatorial setup was chosen in which on a large substrate two parameters were varied simultaneously. The resulting sample consists of the four rows of polymer gratings with different pitches (5, 10, 20, and 40 microns). Each row consists of 11 areas that were prepared at different conditions (e.g. light intensity or a temperature gradient during development) (Fig.3, right). The total size of the sample is 25x102 mm. Automatic SPM analysis allows us to determine the proper conditions of the sample preparation, for instance, at which the maximum aspect ratio of the polymer grating is achieved. Fig.4 demonstrates the dependence of a grating height on light intensity (for the sample obtained by using intensity gradient mask). The sample consists of 44 areas: 4 pitches and 11 values of energy dose have been used for formation of polymer grating. Automatic measurements have been executed in tapping mode. The results of investigation are 4 dependences for each grating period that show changes of height with increasing energy dose. This information allows the determination of the optimal preparation conditions for the sample.

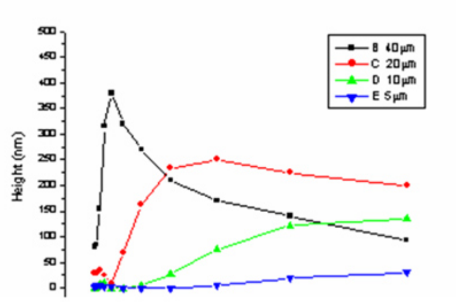

Fig. 4 Results of automatic measurements of the library of samples. Dependence of height of the polymer grating on light intensity (energy dose) for 4 pitches of grating: 5, 10, 20, and 40 microns.

Analysis of macroscopic areas

Measurements of the large areas with SPM are possible only by movement of the SPM head over sample surface by the positioning system. Analysis of thickness of the spin-coated polymer film over 3.5 cm distance has been performed with the help of automatic SPM. The spin-coated film deposited on silicon was scratched by knife (Fig.5) and thickness of film was measured in 19 positions along scratch (Fig.6). The coordinates of those positions have been saved in the software before scanning. The film thickness has been determined as distance between maximums on dependence of number of pixels on z-coordinates of pixels. The analysis of film thickness (Fig.6, right) shows that middle part of film is quite uniform; meanwhile 7 mm area near the edge of film has variable thickness indicating moving of material outside during spin-coating.

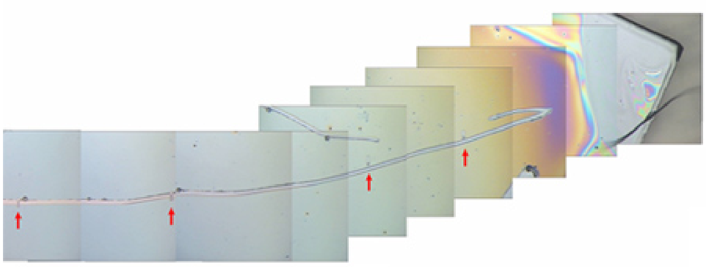

Fig. 5 Optical images of the scratch (scratch is indicated by red arrows).

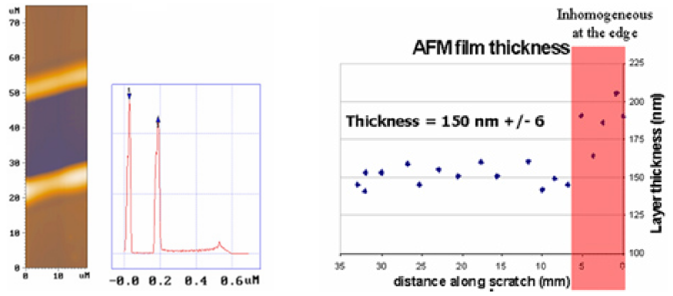

Fig. 6 SPM image of the scratch (left), determination of the film thickness as distance between peaks on statistics (center), final result: dependence of film thickness on distance along the scratch (right).

Automated nanolithography on macroarea

A monolayer of octadecyl trichlorsilane (OTS) deposited on a silicon wafer can be oxidized electrochemically by using conductive SPM tips[2]. In normal conditions a thin water layer is always present on the surface. Decomposition products thereof allow us to change the terminal -CH_3 groups of the OTS layer to –COOH by applying of voltage to the tip. The smallest modified area can be as small as the tip size (it depends also on humidity, applied voltage etc.). The result of oxidation is visible on lateral force image in contact mode. Translation of the lithographic pattern over large area by moving of the positioning stage forms macroscopic lithographic patterns with minimal detail lying in nanometer range. Fig.7 shows lateral force distribution for an oxidized OTS film. The unit of this pattern (Fig.7, left) has been translated over large an area by moving of positioning stage. Fig.7 right shows only part of the modified area that is larger than the scan size. In total 100 units (a 10 by 10) spanning 0.2 by 0.2 mm have been made in less then 2 hours.

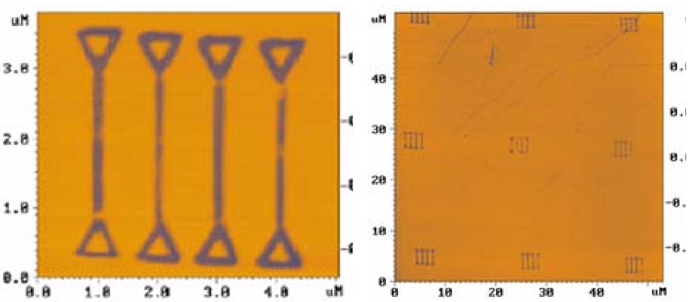

Fig. 7 Lateral force microscopy of oxidized areas: pattern unit (left), 9 units (right).

Acknowledgments

The authors would like to thank Dr. C. Sanchez, Dr. C.W.M. Bastiansen and Prof. Dr. D.J. Broer for samples used in this work, Dr. J. Loos for valuable discussions.

References

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CONTACT DETAILS

NT-MDT Spectrum Instruments
Proezd 4922, 4/3, Zelenograd, Moscow 124460, Russia
E-mail: info@ntmdt-si.com
Tel: +7-499-110-2050