Towards Automation in the Characterization of Nano-Structured Materials and Devices

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ABSTRACT

The combination of a confocal Raman microscope and an atomic force microscope (AFM) in an automated microscope system is used to study large samples in terms of chemical composition and morphological conformation on the nanometer scale. Recent developments in CCD detectors and ultra-high throughput spectrometer enable the acquisition of Raman spectra in microseconds, thus enabling the acquisition of 2D spectral arrays consisting of ten-thousands spectra in less than 5 minutes. The evaluation of spectral features, such as peak intensity, peak position etc, lead to Raman images revealing either chemical or stress distributions within the analyzed materials. The automated sample positioner, allows the automated execution of predefined measurement sequences on any user defined selection of measurement points on the sample. By turning the microscope turret, the confocal Raman microscope is transformed into an AFM, allowing to perform high resolution topographical images on the same pre-selected positions of the sample.

Keywords: Confocal Raman Microscopy, AFM, automated system for large sample analysis

1 INTRODUCTION

The characterization of nanostructured materials implies knowledge about their chemical and structural properties, leading to a growing demand for characterization methods for heterogeneous materials on the nanometer scale. However, certain properties are difficult to study with conventional characterization techniques due to either limited resolution or the inability to chemically differentiate materials without inflicting damage or using invasive techniques such as staining. By combining various analytical techniques such as Raman spectroscopy, confocal microscopy and AFM in one instrument, the same sample area can be analyzed with all implemented methods, leading to a better understanding of nanostructured materials.

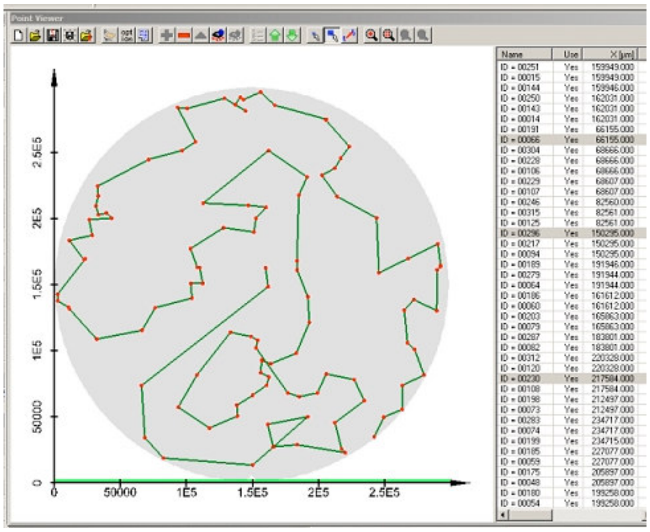
Raman spectroscopy, a chemical analysis technique, combined with confocal microscopy enables the unique Raman imaging of heterogeneous materials [1-8]. The power of Raman imaging stems from the high chemical information content of molecular vibrational spectra. In the Raman spectral imaging mode, a complete Raman spectrum is recorded at every image pixel, leading to a two-dimensional array consisting of ten-thousands of complete Raman spectra. From this array images are extracted by analyzing various spectral features (sum, peak position, peak width, etc). Differences in chemical composition, although completely invisible in optical images, will be apparent in the Raman image and can be analyzed with a resolution down to 200 nm [3,6,9]. The recently implemented high speed EMCCD camera and ultra-high throughput spectrometer (UHTS 300) allow the reduction in Raman data acquisition time by another factor of 10 [10, 11]. If higher resolution is required, by simply turning the microscope turret, the confocal Raman microscope can be transformed into an AFM. Using this imaging technique, structures below the diffraction limit can be visualized from the same sample area [9].

This article describes further instrumental improvements which allow the automated analysis of large samples. Beside the acquisition of large area scans [12], through special scripting functions it is possible to predefine measurement sequences on any user defined selection of measurement points on the sample, guaranteeing the most comprehensive surface analysis tool for systematic and routine research tasks.

2 INSTRUMENTATION

The new developed alpha500 RA microscope from WITec (www.witec.de) is a highly modular and flexible microscopy system. It combines a high throughput confocal Raman microscope for 3D chemical imaging and an AFM for high resolution morphological imaging in an automated system for large samples. An image of the instrument is shown in Fig. 1. The piezo scanner, with a scan range of 200x200 μm^2, is mounted on top of a motorized x-y-z stage. This stage is driven by stepper motors and allows an expansion of the scanning range in x-y direction to 150x100 mm^2 with a step size of 100 nm. Besides expanding the scanning range, this stage can be used to perform multi-area/multi-point measurements on any user-defined number of measurement points. An example of such a point raster is shown in Fig. 2. The table on the right side lists the coordinates of the points of interest, whereas the graph on the left side displays the points of interest and the travel path for automated point measurements. Through scripting functions several consecutive tasks can be executed automatically, without any online process control by an operator during the measurements.

Fig. 1: The alpha500 AR microscope, highlighting the motorized xy-stage (1) and the high accuracy piezo scanner (2)



Name	Use	X [µm]
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ID = 00144	Yes	159946.000
ID = 00250	Yes	162031.000
ID = 00143	Yes	162031.000
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ID = 00191	Yes	66155.000
ID = 00066	Yes	66155.000
ID = 00304	Yes	68666.000
ID = 00228	Yes	68666.000
ID = 00106	Yes	68666.000
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ID = 00246	Yes	82560.000
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ID = 00125	Yes	82561.000
ID = 00296	Yes	150295.000
ID = 00217	Yes	150295.000
ID = 00094	Yes	150295.000
ID = 00189	Yes	191946.000
ID = 00279	Yes	191944.000
ID = 00064	Yes	191944.000
ID = 00186	Yes	161612.000
ID = 00060	Yes	161612.000
ID = 00203	Yes	165863.000
ID = 00079	Yes	165863.000
ID = 00287	Yes	183801.000
ID = 00082	Yes	183801.000
ID = 00312	Yes	220328.000
ID = 00120	Yes	220328.000
ID = 00230	Yes	217584.000
ID = 00108	Yes	217584.000
ID = 00198	Yes	212497.000
ID = 00073	Yes	212497.000
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ID = 00175	Yes	205897.000
ID = 00048	Yes	205897.000
ID = 00180	Yes	199258.000
ID = 00054	Yes	199258.000

Fig. 2: Example of point raster for automated multi-point /multi-area measurements.

3 EXAMPLE MEASUREMENTS AND DISCUSSIONS

A series of AFM and Raman measurements were performed on a DRAM (dynamic random access memory) chip (Infineon Technologies), to prove the automation capabilities of the alpha500 RA. In a first step white light video images were recorded on three different positions of the sample (Fig. 3). For this procedure the following scripts were used:

a) auto-illumination for optimum white light illumination of the sample,
b) auto-focus performs an auto-focus based on the video images,
c) the snapshot acquires a video image in each preselected point.

The video images from Fig. 3 were recorded with an Olympus 100x (NA = 0.95) objective and reveal the different pattern selected from the DRAM chip.

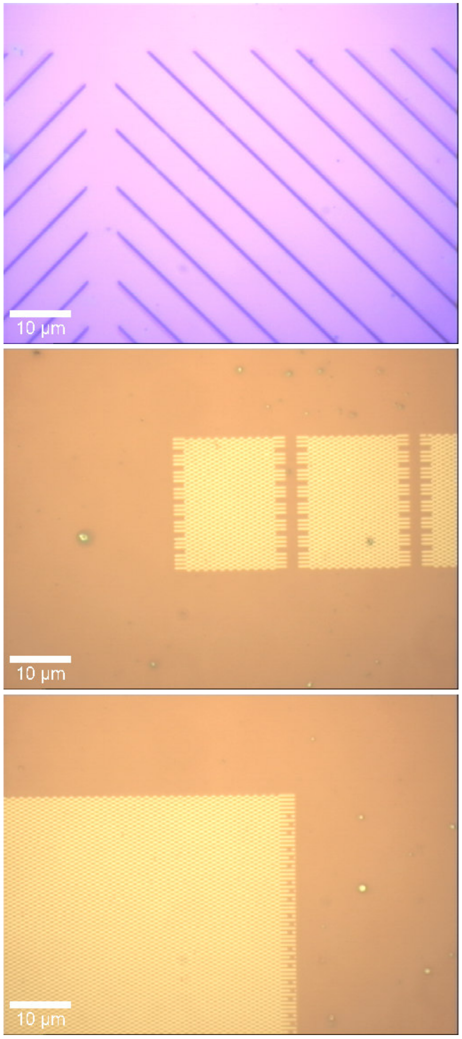

Fig. 3: Automated recorded video images from three different areas of the DRAM.

Parts of the three selected areas shown in Fig. 3 were then imaged in Raman spectral imaging. In this imaging mode, a complete Raman spectrum is recorded in every image pixel, leading to a 2D array of 150x150 Raman spectra. The integration time for each individual Raman spectrum was 0.01 s, thus the array of 22500 Raman spectra was recorded in less than 4 minutes. By evaluating the position of the first order Si Raman band at 520/cm, stress images of the selected areas can be obtained. As shown in a previous paper [13], indents in Si produce a stress field which expands around the indent as a function of load force. Fig. 4 shows the stress images obtained from the three pre-selected areas.

By simply turning the microscope turret, the confocal Raman microscope was transformed to an AFM. By using the same positioning raster as used before for the acquisition of the Raman images, and the additional auto-tip-approach scripting function, AFM images were recorded from the same sample areas. Fig. 5 shows the AFM images recorded from the three selected sample areas. The line structure in area one consists of parallel grooves which are 80 nm deep. The pits from areas two and three have a diameter of 300 nm and a depth of 300 nm.

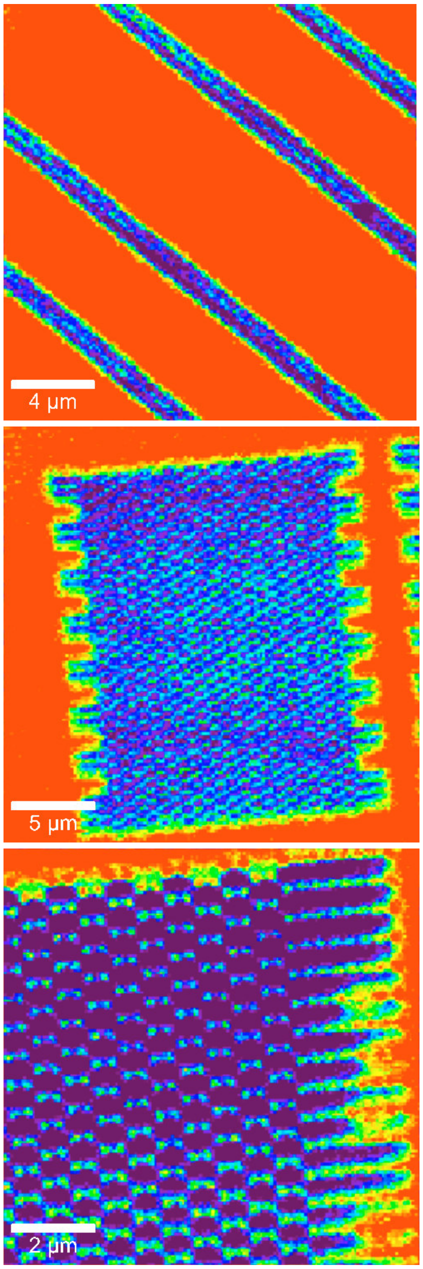

Fig. 4: Raman stress images recorded on three different areas of the DRAM.

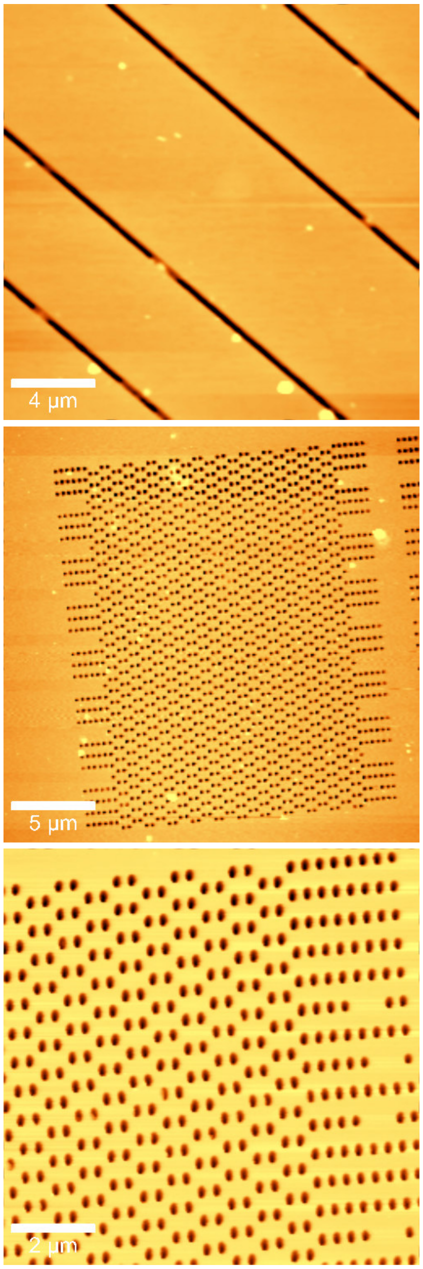

Fig. 5: AFM topography images measured from the three different areas of the DRAM.

4 CONCLUDING REMARKS

The capabilities of an automated confocal Raman AFM system are demonstrated. By combining two nondestructive sample analysis methods in one automated system, one and the same sample area can be characterized by the implemented measuring methods. Multi-point/multi-area measurements can be routinely performed on various areas of the sample.

The motorized sample stage can be used to perform large overview scans, whereas the additional piezo-scan-stage allows the acquisition of high resolution images from selected areas of the overview image [12].

The implemented scripting routines such as auto-focus, auto-AFM-tip-approach, etc. guarantee standardized routine measurements procedures without any online process control by an operator during measurement.

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