

# Atomic Force Microscope As a Tool for Nanometer Scale Surface Patterning

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## ABSTRACT

Progress in scanning probe microscopy (SPM) has transformed scanning tunneling microscopes (STMs) and atomic force microscopes (AFMs) from measuring devices into technological tools. Local surface modification by scanning probe allows us to treat materials with resolution from the angstrom to micron level [1-3]. The demonstration of single electron transistor creation by LAO [4] opens the way to the development of industrial nanolithography processing.

However, an insufficient knowledge in detailed understanding of the LAO mechanism limits the integration of this process into bulk production. The first experiments in STM tip oxidation of the hydrogen passivated Si surface was demonstrated in [5]. It was shown that electrical and structural properties of the positive-biased surface are changed irreversibly in air at room temperature under the tip effect. The common explanation of these changes in surface topography is formation of the oxide under the tip. The tendencies obtained in various works of the oxide pattern shape and its growth kinetics on the conditions of tip-induced treatment allows us to propose an electrochemical mechanism of LAO. Significant effect of the humidity on oxidation velocity is observed in [6]. This fact confirms the necessity of adsorbed electrolyte layer presence to produce oxide that is in good agreement with the electrochemical model. Moreover, there are other technological parameters that affect the oxidation kinetics. For example, the conductivity of an oxidized material affects the oxidation rate [7].

**Keywords:** nanolithography, AFM, anodic, oxidation.

## 1 EXPERIMENT

Nanopatterning was performed on thin titanium layers ( $2 \pm 1$  and  $8 \pm 1$  nm) evaporated by a cathode arc deposition technique [8] on thermally-oxidized silicon substrate. Such evaporation method allows to deposit of ultrathin continuous amorphous films with a surface roughness about 0.1 nm. LAO can be done on Si or GaAs wafers as well. Low value roughness is dramatically important because the surface morphology is changed within few nanometers during oxidation.

A commercial scanning probe laboratory Ntegra developed by NT-MDT Co. has used for LAO process. As probes for nanooxidation and further topography measurements silicon cantilevers with hard and high-conductive coatings based on  $W_2C$  have used [9]. Experimental scheme of local anodic oxidation process is shown (see Fig. 1). Several force constant lever types have been chosen, generally in the range of 10–20 N/m. The stiff silicon cantilevers had resonant frequencies in the range of 100–200 kHz respectively. Experiments in LAO were carried out in 10-50% of free cantilever oscillation amplitude. Free cantilever amplitude range about 60-500 Å. This mode allows one to increase the lifetime of the conductive cantilevers and thus to raise the nanolithography process reproductivity. Using environmental control in SPM chamber we were able to vary a relative humidity during LAO within range 10-90%. By the software it was possible to apply bias voltage on tip or on sample within  $\pm 10V$  with accuracy 0,001V and voltage impulse duration 0,01-1000ms.

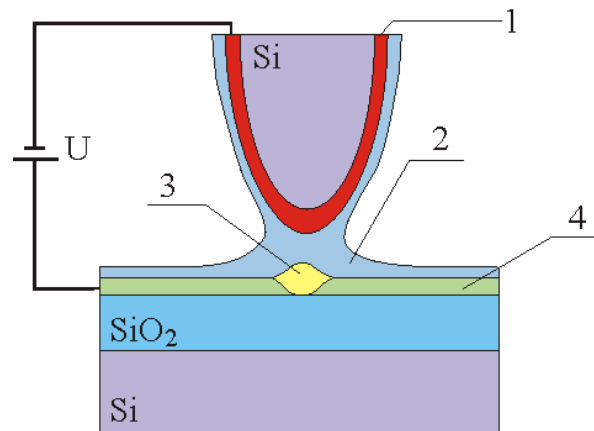


Figure 1: General scheme of LAO process. -1 Conductive coating, -2 Adsorption bridge, -3 Anodic oxide, -4 Metal film.

## 2 RESULTS AND DISCUSSION

The main idea of this article is to show possibilities in development of local anodic oxidation for surface patterning on nanoscale with reproducible and linear patterns even on relatively large areas. Physical-chemical model of local anodic oxidation of thin titanium films were described in our previous article [10] and here we would like to focus on its applications.

It is well known that conventional piezotube scanners are characterized by creep, drift and nonlinearity effects. These disturbance causes to unparallel oxide lines formed by LAO (see Fig. 2).

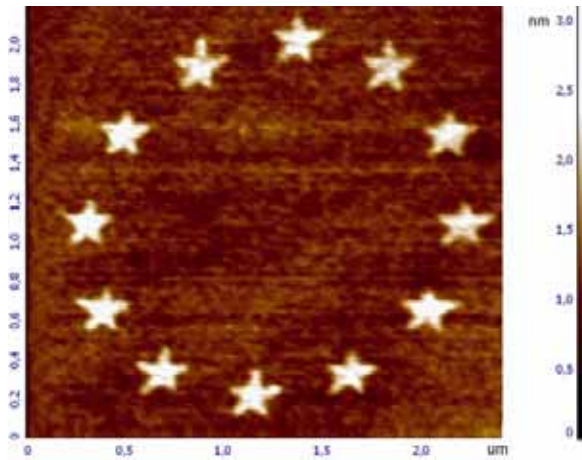


Figure 2: AFM image of Ti film after LAO process. Patterning and measurements were done with open-loop scanner. The small distortion of a ring stars is clearly visible.

In our experimental set up we have used scanning probe microscope with integrated capacitive sensors for closed-loop control scanner movement. Capacitance sensors are characterized by low noises for instance about 10ppm. Another important value of using capacitance sensors integrated in a piezotube scanner is improved linearity, thus scanners equipped with such sensors residual nonlinearity 0,1% in all three axis can be achieved. That high linearity enables for SPM to compete in metrological field for example to create by LAO method nanometer scale test gratings for AFM or SNOM calibration.

We made an attempt to make a patterning on large areas by using linearised scanner. As a sample we used intrinsic type of GaAs wafer in order to extend patterning time. On a figure 3 is demonstrated AFM image of GaAs surface after LAO patterning during 10minutes. Oxidizing started from left bottom corner of a square and then carried out continuously till tip returned back to the same position. On figure 4 shown cross sections over x and y directions. The distance between the left and right lines comparing with top and bottom lines within one pixel on AFM image.

quality is 512x512 data points over 25x25 micrometer area, so scan step is 48 nm.

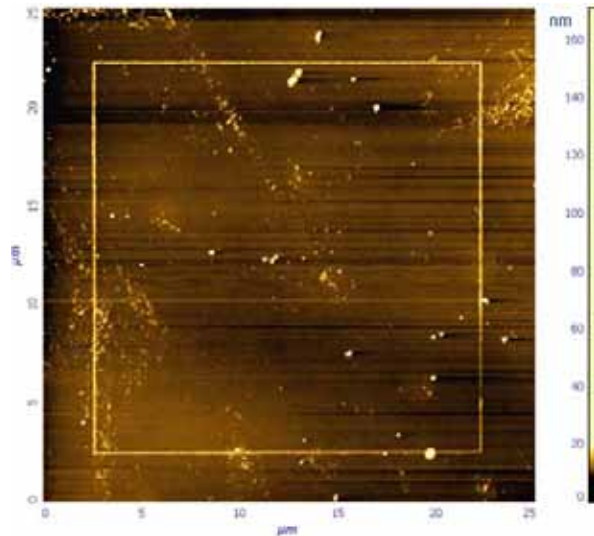


Figure 3: AFM image of GaAs surface after LAO process. In order to make this pattern probe started from left bottom corner made a square path and returned back within 10minutes.

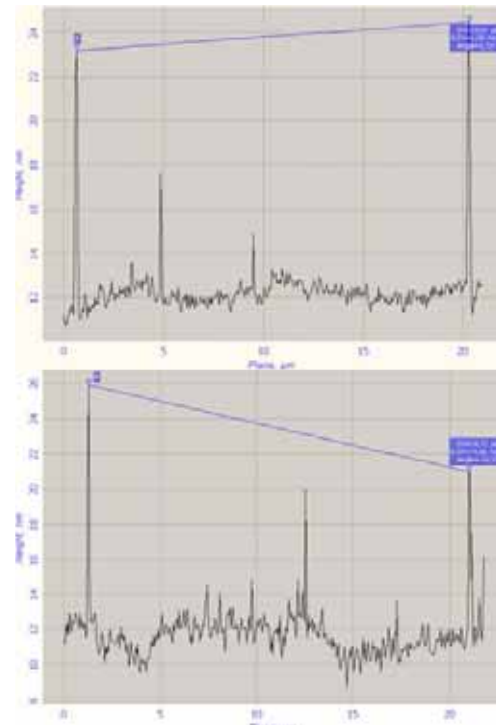


Figure 4: Cross-sections from figure 3, top image along X axis, bottom image along Y axis. The difference in distance between top-bottom and right-left lines is 50 nm, that determined AFM pixels resolution.

In order to check how good a tip returns back to starting point we have made a zoom in left bottom corner of square patters with 512x512 data point over 5x5 micrometers. On a figure 5 a close look on lines intersection is shown. The overlapping is 54nm and 29nm over 20micrometers lines in X and Y directions respectively.

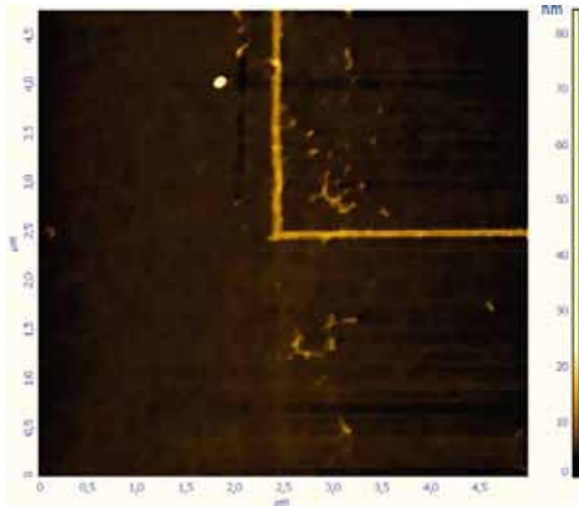


Figure 5: Zoom in to left bottom corner of AFM image shown in figure 3. Line width on half of height is 50nm.

Another interesting application of LAO process can be 2D nanopatterning. Looking for applications of the LAO process we are offering the method for ultrathin dielectric films formation. If we move the tip with step less than diameter of the oxide spot and apply variable voltage or impulse duration in each point according to some low, then within scan area we can modify the surface. For incarnation of the described idea the SPM software has been modified by such way that it is possible to load a mask as bitmap grayscale format. A loading mask may contain any information which we would like to transfer on surface by LAO, it can be drawing of nano-integrated circuit or just a portrait. In first step software is estimating a histogram of point's distribution over a grayscale image and associates the brightness in to percentage within 0-100% range. Then during LAO in every point of the raster we apply constant voltage but vary the voltage impulse duration according to certain brightness level of the particular point of the loaded mask. After performing such scanning with simultaneous LAO process it is necessary to check what's happened with the surface, for that we scan the same area in resonant mode AFM with same tip. On the figure 6 is shown AFM image of the modified surface of titanium film. Such image creation takes about 5-10min.

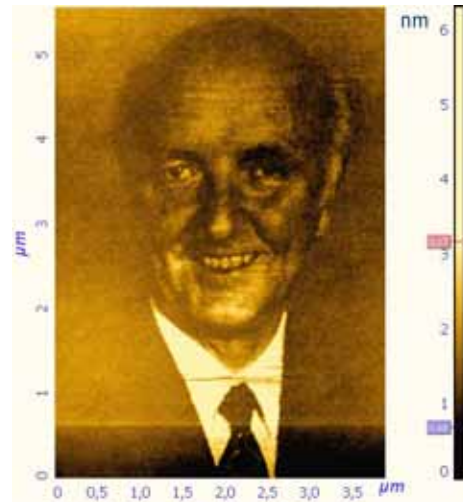


Figure 6: AFM image of Ti film after raster mode LAO.

### 3 CONCLUSIONS

In this work the possibility of LAO process accuracy improving is demonstrated. The main factor that can improve patterning process reproducibility are reduction of piezoscanner creep effect by integrating closed loop operation sensors for scanning control. Another important thing is a thermal drift that can also dramatically reduced buy making closed temperature stabilized chamber around a scanner, tip and sample.

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