Structural Investigations of Phosphorous Doped Silicon Layers

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The structure and surface topography of low-pressure chemical vapor deposited silicon films have been studied using atomic force microscopy. The films were grown on the thermal oxide of a (100) silicon substrate at temperatures between 550 °C and 630 °C. For comparison two different ex-situ doping processes were used: doping from a $POCl_3$ source, and implantation. The measurements have been performed on undoped as-grown as well as on doped and annealed samples. Due to the much lower roughness of the layers amorphously deposited below 570 °C the roughness values of the doped samples are different by more than a factor of ten. Additionally, depending on the initial state of the as-grown layers the grain sizes and their shapes are very different for the doped layers although the annealing parameters are comparable.

1. Introduction

Thin films of conducting polycrystalline silicon prepared by low-pressure chemical vapor deposition (LPCVD) and subsequent doping are very important in the production of integrated circuits. Due to the wide range of their conductivity they are used in a large number of different applications. For instance, heavily doped films are used in the production of gate electrodes and interconnections, and films with high resistivity are used for resistors and floating gates.

Due to the progress in the production of integrated circuits the lateral dimensions of the components continuously decrease. As a consequence the requirement for a better understanding of the detailed properties of polycrystalline silicon layers and their dependence on the production conditions increases. During the last decades a lot of different characterization methods have been developed. All methods have disadvantages in common like a limited lateral resolution, an extensive preparation and a restriction to special surfaces. In contrary, scanning probe microscopy is a powerful tool for real-space surface imaging with high 3D resolution, and needs a minimum of preparation techniques for materials used in the semiconductor device production.

In this work we report on structure and surface roughness investigations of silicon films produced by LPCVD at different temperatures (550 °C – 630 °C) and subsequent doping. The measurements were performed with an atomic force microscope (AFM) operated in contact mode under high vacuum (HV) conditions. The samples have been taken from production-line testwafers, produced by high-volume equipment.

2. Experimental

The measurements have been performed using a UHV AFM with a 5 µm range tube-scanner driving the sample. Samples and tips can be exchanged in HV. Commercially available silicon nitride cantilevers characterized by a 36° interior angle and 20 nm radius of curvature have been used for the contact-mode-AFM measurements. Force calibration for individual cantilevers has been obtained by measuring the bending of the cantilevers as a function of the distance between dip and sample and taking into account the force constant of the cantilevers.

The investigated films have been deposited onto silicon dioxide thermally grown on (100) n-type silicon wafers. The deposition temperature for different samples was changed from 550 °C up to 630 °C in steps of 10 °C. The thickness of the as-deposited layers was between 250 and 270 nm. The ex-situ phosphorous doping was produced in two different ways: a) by a gaseous predeposition using a $POCl_3$ source at 900 °C, followed by a drive-in step at the same temperature and the deglazing in a buffered oxide etch (BOE), and b) by implantation of phosphorous and subsequent annealing of the films at 900 °C. Doping by implantation was applied to films grown at deposition temperatures below 600 °C only.

Prior to the scanning probe measurements an additional selective silicon dioxide etching has been applied to all samples by dipping them in 2% hydrofluoric acid (HF). This procedure is well known to produce surface passivation by H-termination of silicon dangling bonds. Immediately after etching, the samples have been mounted in the AFM chamber and were measured at high vacuum conditions at a residual gas pressure $< 2\times10^{-7}$ mbar. A comparison of HF-treated and untreated samples have shown that the HF passivation of the surface changes neither the topographic nor the grain structure of the samples [1].

The measurements have been performed with constant normal forces as low as 0.05 nN (normal forces below 1 nN only in vacuum) and ranging up to 10 nN. The roughness values discussed below have been evaluated from contact-mode AFM measurements. In order to exclude effects due to different tip qualities, all samples have been investigated using the same type of cantilevers and a low normal force of 0.1 nN. Additionally, for the comparison of the tip quality the first measurements of each tip have been done on a sample with narrow trenches. Previous measurements had shown that in high vacuum and for normal forces below 1 nN no degradation effects of the tips and the silicon surfaces are observable for more than 200 scans.

Areas have been chosen randomly on each sample. Samples taken from different areas of the wafer show no significant difference in structure and topography of the surface. At least 10 images of 1x1 μm^2 have been acquired on each sample. Roughness values have been calculated from each image using standard software, which fits a plane to the entire image, determines the deviation of each image point from this plane, and calculates the root-mean-square value R_{rms}. The roughness values for each layer have been obtained by averaging measurements of at least five areas.

3. Results

Previous measurements on as grown films [2] have shown that films grown at deposition temperatures below 570 °C are amorphous. Films grown above 600 °C are polycrystalline, and the hillocks were identified as the grains of the layer [3]. For deposition temperatures from 570 °C to 600 °C an amorphous matrix with an increasing number of embedded crystallites occurs. The roughness values of the amorphous films are below 1 nm and more than one order of magnitude smaller than those of the polycrystalline films (10 - 15 nm).

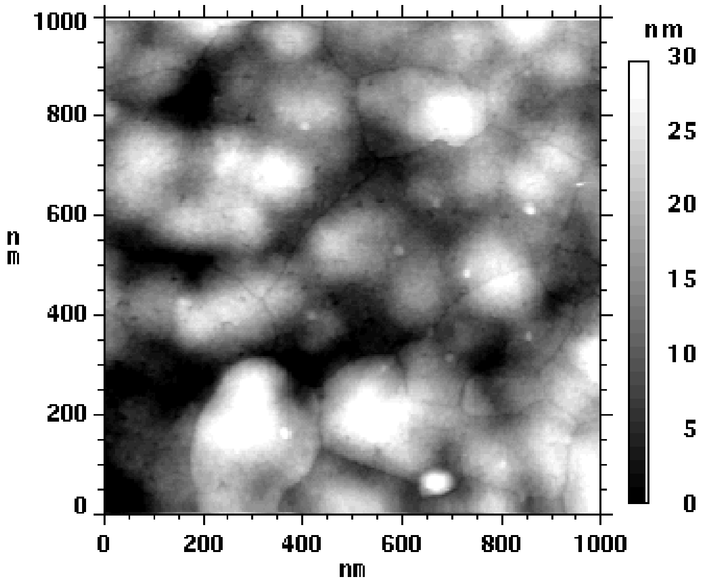

Fig. 1: Contact-mode-AFM topographic image of a doped polycrystalline silicon film grown at 620 °C.

A conventional topographic image of a doped silicon film grown at 620 °C is shown in Fig. 1. The image is dominated by a hillock structure with lateral features between 70 and 200 nm and is characteristic for all films grown at temperatures higher than 600 °C. A comparison of the topographies of doped and as-grown films show only slightly enlarged and flattened hillocks for the doped films with roughness values between 7 and 12 nm. In contrary to the as-grown films the hillocks of the doped samples can no longer be identified with the grains shown in Fig. 2. In this image the topographic data have been differentiated and convoluted to enhance short-scale corrugation. This way a well defined grain structure clearly becomes observable and resembles earlier TEM micrographs of similarly processed samples reported by Wada et al. [4]. Typical lateral dimensions of the grains are 350 nm, but ranging up to 500 nm.

Fig. 3 shows the topography of a doped silicon film grown at 560 °C. Due to the very low roughness values of the films grown below 570 °C the boundaries of the grains can be observed directly in the topography without differentiation. Fig. 3 is characteristic for all samples grown below 570 °C and both kinds of doping processes. The grains partially exhibit unusual elongated shapes with angles at the corners deviating strongly from 120°. Their lateral dimension ranges up to above 1000 nm. The roughness values for these films are between 1 and 2 nm.

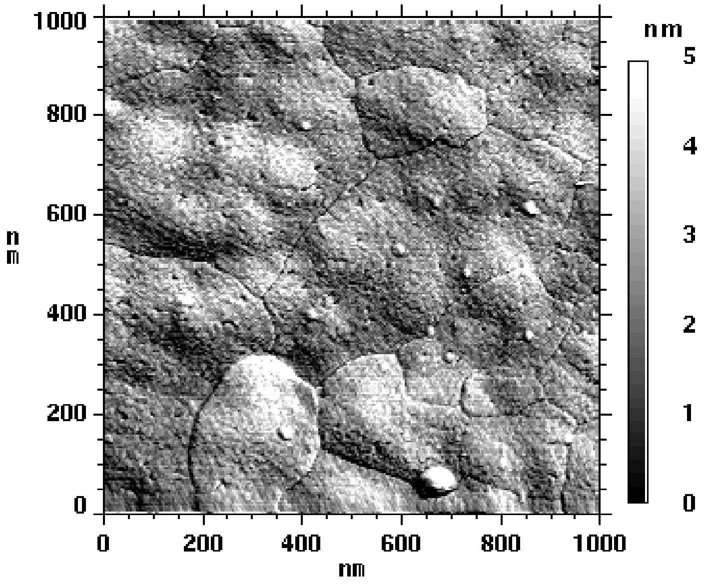

Fig. 2: Differentiated and convoluted image of Fig. 1, showing grain boundaries.

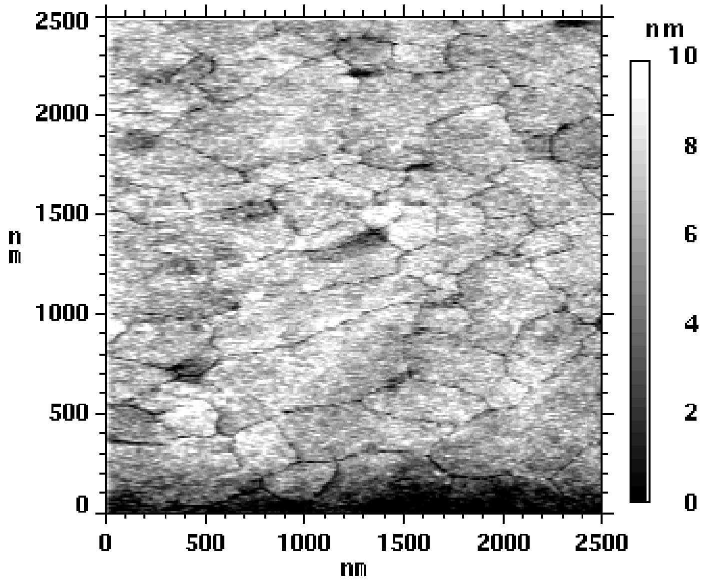

Fig. 3: Contact-mode-AFM topographic image of a doped polycrystalline silicon film grown at 560 °C.

4. Conclusion

The AFM techniques applied in this work have proven to be a powerful tool for the characterization of silicon layers taken out of the production line of the integrated circuit production. Particularly, for the first time it has been possible to distinguish between topographic hillocks and the grain structure of polycrystalline silicon films using only one characterization technique.

We have investigated undoped and doped films grown in a temperature range between 550 °C and 630 °C. The roughness values for the amorphous films grown below 570 °C are by more than a factor of ten smaller than those of the polycrystalline grown films above 600 °C. The doping and subsequent annealing at temperatures around 900 °C causes a complete recrystallization of the layers. Therefore the amorphous films become polycrystalline and the size of the grains of initially polycrystalline films increases. In contrary to this change in the structure of the films the topography of the films is only slightly modified. Consequently the roughness values do not change drastically due to the doping process.

In addition we have shown that the grain structures of initially amorphous and polycrystalline films are very different after the doping process although temperature and duration of the annealing processes are comparable. Doped films grown below 570 °C show partially unusual shapes of their grains with lateral dimensions in a wide range up to above 1000 nm. In contrast to this result doped films grown above 600 °C exhibit more regular shapes of their grains with lateral dimensions in a small range around 400 nm.

The reason for the different recrystallization behaviors is not completely understood. Due to the importance of homogeneous and flat polycrystalline silicon films for the production of integrated circuits with very large scale integration this behavior should be investigated in further experiments.

References

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