STM Characterization of a LPCVD-Polycristalline Silicon Sample

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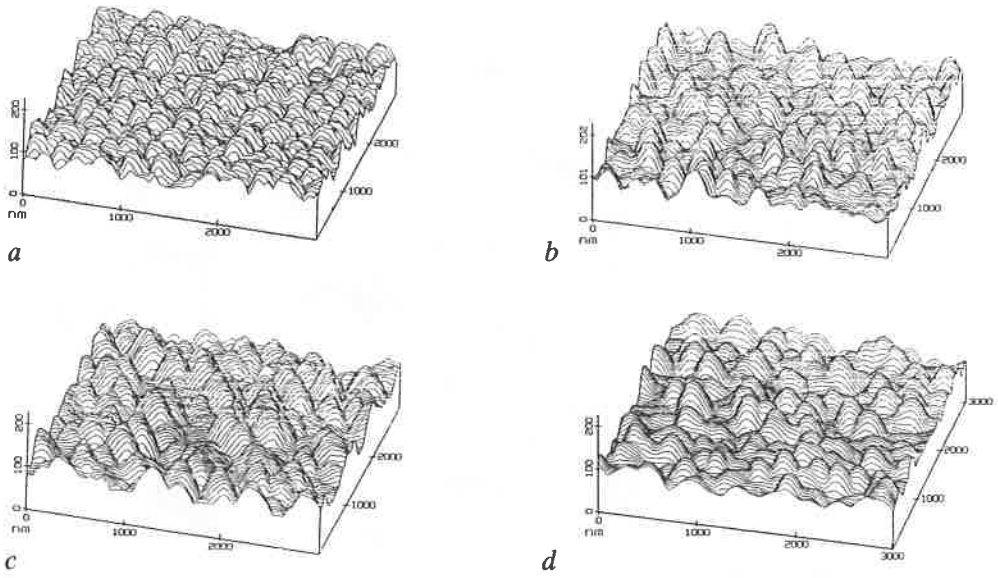
Introduction

Scanning tunneling microscopy (STM) and now atomic force microscopy (AFM) are very powerful techniques for surface characterization.

In this paper, results concerning STM study of polycrystalline silicon surfaces are presented.

All AFM and STM measurements were made in air using a Digital Instruments Nanoscope II. Pt-Ir tips and Si_3N_4 cantilevers were used for STM and AFM observations, respectively.

A 4600 Å-LPCVD polycrystalline silicon layer was deposited on a 235 Å silicon oxide layer which was itself deposited on a (001)-oriented monocrystalline substrate. The polycrystalline silicon was phosphorous implanted at a dose of $9.10^{15}cm^{-2}$.



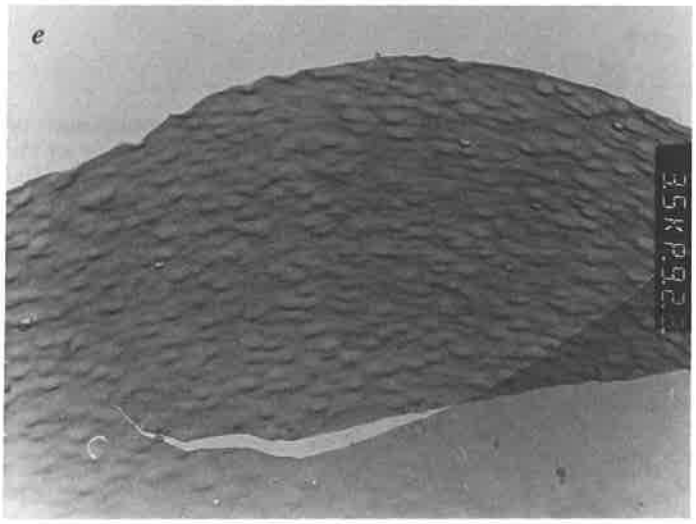


FIGURE 1. - *Comparison between STM, AFM and TEM images of the grains of polycrystalline silicon :*
(a) STM image of polycrystalline silicon grains after etching with HF diluted solution.
(b) STM image of polycrystalline silicon grains after the treatment with buffered oxide etch.
(c) STM image of polycrystalline silicon grains coated with Pt-C layer.
(d) AFM characterisation of polycrystalline silicon grains without any preparation.
(e) TEM carbon replica of the surface.

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Results and discussion

Surface study

In order to determine the surface roughness and the shape of polycrystalline silicon grains, the sample was observed by STM. Then, STM results were compared to AFM and TEM results obtained on similar samples.

The surface was studied after two different chemical treatments. One sample was subjected to a diluted fluorhydric acid solution (1 HF : 100 H_2O), another to a buffered oxide etch (1 HF : 7 NH_4F). *Figures 1a* and *1b* show the surfaces after these treatments. The grains presented a rounded aspect. Their mean sizes were valued at 260 nm in the first case, at 240 nm in the second case.

STM investigation was also done after a metallic deposition. A Pt-C 5 nm-thick layer was evaporated on the surface before STM observation, in order to allow a good electron flow. Results are shown on *figure 1c*. Mean grain size was 240 nm.

Then, the sample was observed by AFM without any preparation *(figure 1d)*. A carbon replica of the surface was made to determine the size of the grains by TEM *(figure 1e)*. The mean dimension of the grains by these two techniques was 250 nm and 220 nm, respectively.

Table I summarizes all previous results.

TABLE I. - *Estimation of polycrystalline grains size.*

	Mean dimension (nm)	**Roughness** (nm)
STM results		
Deox. HF/H_2O	260	12.0
Deox. HF/NH_4F	240	11.4
With Pt-C layer	240	11.1
AFM results	250	10.6
TEM results	220	

Comparing these different characterization techniques, it was possible to establish that STM allows to characterize the deoxidized surface of highly-doped polycristalline silicon.

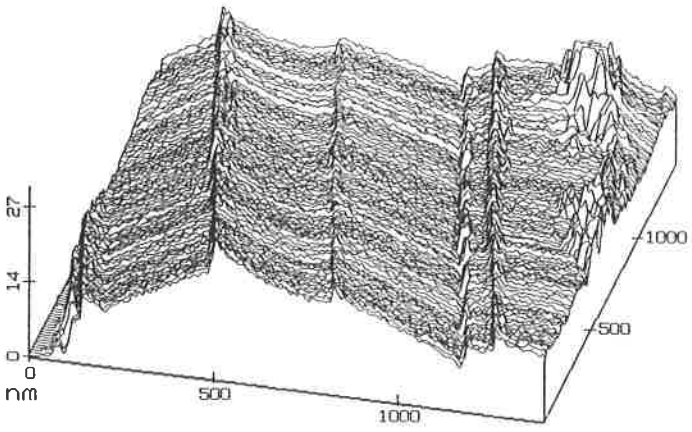


FIGURE 2. - *The cross-section immediatly observed after cleavage and metallization.*

Cross-section study

The sample was studied by STM immediatly after cleavage and metallization. The high roughness of the cross-section coupled with the fact that the region of interest was near the edge of the sample rendered STM observation very difficult (see *figure 2*).

We concluded that a more sophisticated preparation technique was required : the sample was cleaved, the two pieces were glued face to face together, and polished *(figure 3)*. The oxide layer between the polycristalline silicon and the substrate was revealed by a buffered oxide etch (1 HF : 7 NH_4F). Finally, the sample was metallized to allow STM observation *(figure 4)*.

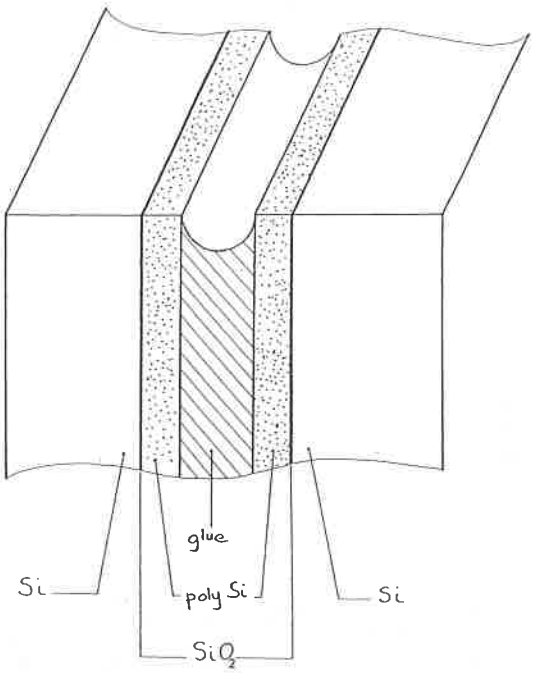


FIGURE 3. - *The two pieces were glued face to face together and polished.*

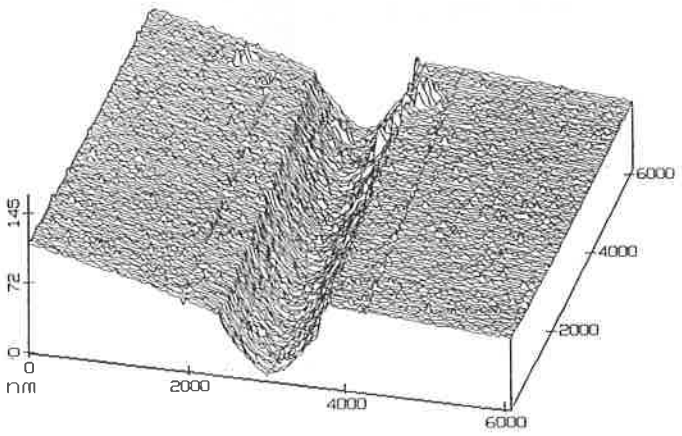
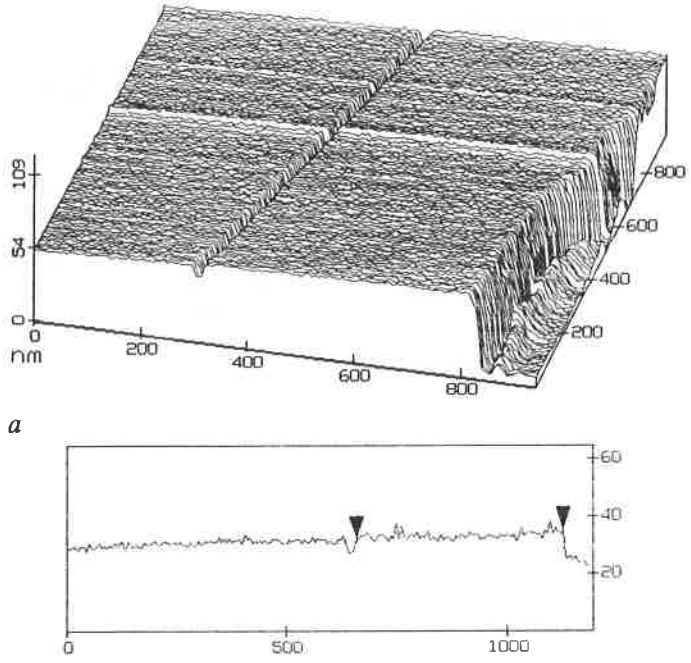


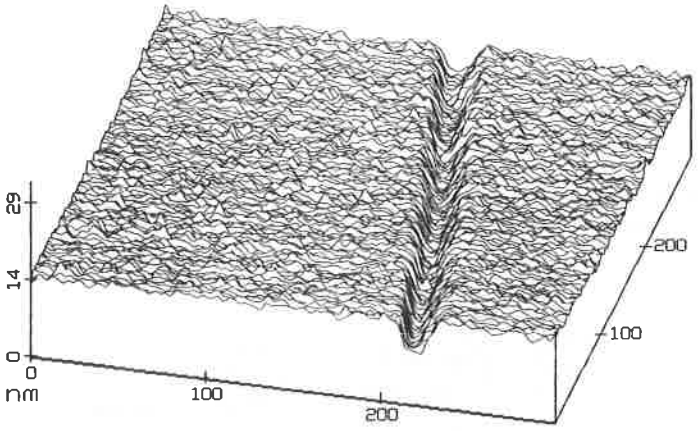
FIGURE 4. - *General STM image of the sample ; it is possible to distinguish the two fine oxide trenches.*

It was thus possible to estimate the oxide layer thickness *(figure 5)*. However, when using Pt-C coated samples, we were not able to find any difference between monocristalline and polycristalline silicon. We were only able to tell them apart when observing the samples with AFM since no metallization is required *(figure 6)*.

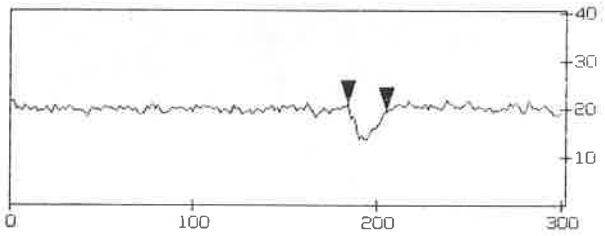


a

Horizontal distance (nm)	*470.32*	*Spectral period (nm)*
Vertical distance (nm)	*3.19*	*DC*
Angle (deg.)	*0.39*	

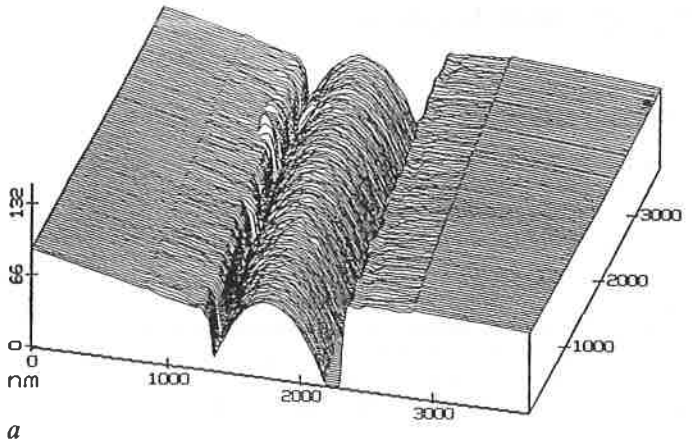


b

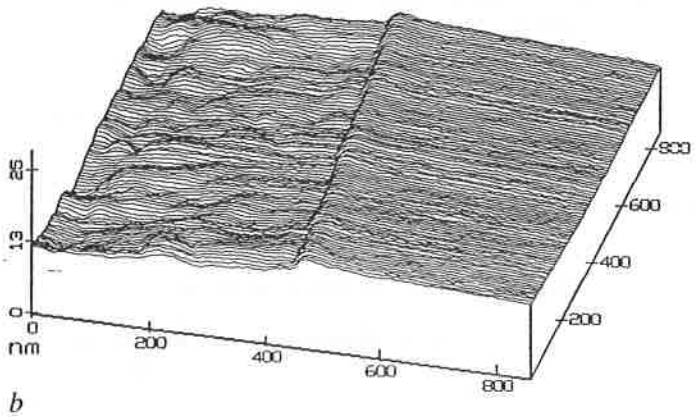


Horizontal distance (nm)	*21.28*	*Spectral period (nm)*
Vertical distance (nm)	*1.43*	*DC*
Angle (deg.)	*3.83*	

FIGURE 5. - *More detailed images.*
(a) Estimation of the thickness of the LPCVD polycristalline silicon layer.
(b) Estimation of the thickness of the oxide layer.



a



b

FIGURE 6. - *AFM characterization of the cross-section.*
(a) General AFM image.
(b) More detailed image : the roughness of polycrystalline and monocrystalline silicon was different.

Scanning tunneling microscopy allows imaging of the surface of highly-doped polycrystalline silicon in air, after oxide elimination. The results obtained are consistent with TEM results. STM characterization requires a very simple preparation of the surface while STM cross-section study requires a more sophisticated preparation. After polishing, differences between constituting elements of the sample are very small, thus limiting the resolution of the method.

Acknowledgements

We wish to thank B. Faure for her contributions to TEM analysis.