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研 究 速 報

Nano-Structures Fabrication Technique for Biological Applications

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ABSTRACT

We succeeded to realize nano-structures using standard {100} silicon wafers and anisotropic etching; this technique makes use of the low etching rate in TMAH of {111} planes or silicon, in comparison to {100} planes, to adjusting the nanometric dimensions of the structures. No sub-micron lithography is required in this technique: it is compatible with normal UV photolithography. Neither complicated process steps are required: a succession of oxidation, oxide patterning and anisotropic etching only are needed. Nano-holes and nano-wires were made using this technique and possible applications of the structures are described for two BIO-MEMS projects.

INTRODUCTION

In the BIO-MEMS field, tiny structures are of great interest because of their dimensions close to the one of biological materials. Actually, cells dimensions are from few microns, to several ten microns. Proteins dimensions are until few ten microns. This is the reason why nano-structures maybe essential to manipulate cells without damaging them or to be able to analyse closer proteins.

The realization of nano-structures is not directly possible with normal UV photolithography (the usual minimum realizable patterns size is few microns). Sub-micronic lithography is possible using direct writing on the wafer with an electron beam. However, this is a very expensive technique which is time consuming. An other possibility is to perform a quite complicated process using SOI wafer[1]. But this technique, even if it gives very precise dimensions, requires expensive equipments and is time consuming too.

In this article, a cheap, simple and robust technique is proposed to realize nano-structure. The advantages of this technique are as follows:

— standard {100} silicon wafers can be used (no SOI needed);

— standard UV photolithography can be used (no EB lithography needed):

— the structures are realizable using oxidation and anisotropic etching with KOH and TMAH only.

The key of the technique is to take advantage of the slow silicon etching rate of the {111} planes in TMAH, compared to {100}, to control the dimensions of the nano-structures very precisely. The ratio between {111} and {100} etching rates is reported 1/30[2].

The principle of the fabrication technique will be first described, then two examples of fabricated nano-structures (nano-holes and nano-wires) will be shown; finally examples of application in the BIO-MEMS field will be explained.

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PRINCIPLE OF THE FABRICATION TECHNIQUE

The principle of the fabrication technique is described on the scheme Figure 1. The fundamental idea is to fabricate interconnected shapes made by anisotropic etching, in a way that their interconnections form a nano-structure.

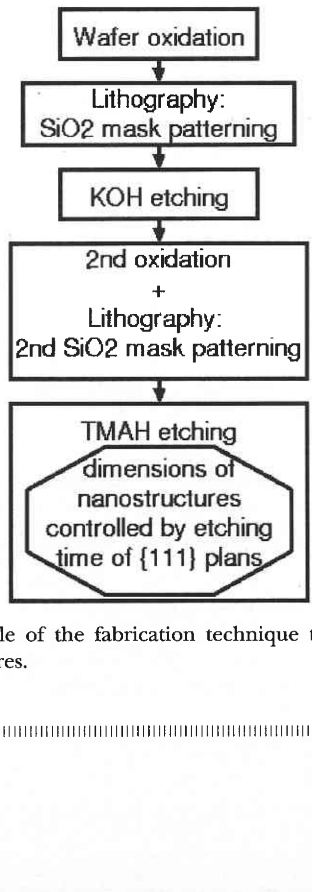

Figure 1: Principle of the fabrication technique to realize nano-structures.

All masks used for the anisotropic etchings are in SiO2, realized by thermal oxidation. SiO2 is used as a mask for two reasons: first, thermal oxidation is cheap and easy to be made; second, this mask is almost not etched in TMAH (the ratio of SiO2 etching rate and Si etching rate in TMAH 15% at 80 C is about 10^{-4}) and only slowly etched in KOH (the same ratio in KOH 33% at 80C is about 5.10^{-3}).

The first anisotropic etching with KOH allows to obtain patterns with precise dimensions defined by the SiO2 mask: there is almost no etching of {111} planes with KOH [12]. The structure obtained in this way has the same dimensions in width and length as the SiO2 mask.

The second anisotropic etching with TMAH allows to fabricate the nano-structures, adjusting their dimensions by controlling the etching time of the {111} planes. Actually, TMAH slowly etches the {111} plan of silicon, contrary to KOH which doesn't etch this plan. This etching rate of {111} plans in TMAH rate 15% was measured approximatly, about 0.03μm/min at 80 C:. Taking advantage of this slow etching, it is possible to control precisely by time this under-etching of {111} plan and then the nano-structures dimensions.

No extremely precise alignment and lithography is required for this process as the nanometric dimensions are dependent on the etching time in TMAH only. In the two process presented in the following part, and generally speaking for this technique, normal UV lithography can be used for the masks, with patterns even larger than several microns or ten microns in dimensions.

During the process, an important step is to protect from TMAH etching the structures made during the first KOH etching: that is why a second oxidation is essential. If there is not such a protection, the second V grooves interconnecting the first ones will create a hole which size will increase very rapidly, following the {100} plan etching rate, and not the {111} plan etching rate. On Figure 2, a scheme explains this phenomena.

Two types of nano-structures were realized using this technique: nano-holes and nano-wires.

EXAMPLES OF FABRICATION OF NANO-STRUCTURES: NANO-HOLES AND NANO-WIRES

These two structures are not the only one which can be made with this technique. It is possible to imagine many other one, just interconnecting in the right way shapes made by anisotropic etching. The nanometric dimensions of the structures is then adjusted just by controlling the etching time of {111} plans in TMAH.

With anisotropic etching, it is possible to realize mainly two kinds of shapes: V grooves shapes and rhombus grooves shapes. The V groove shape is obtained by direct anisotropic etching of a {100} silicon wafer through a rectangular mask. The rhombus groove shape needs a supplementary vertical etching of silicon, through the mask, before performing the anisotropic etching.

If standard V grooves are used, it is possible to realize holes with nanometer size, by crossing perpendicularly a front-side V groove with a backside V groove (Figure 3). At the intersection of both grooves a tiny hole appears which dimensions depend only on the etching time of the {111} planes in TMAH. A longer time in TMAH will enlarge the hole, but with a rate slow enough to be easily controllable.

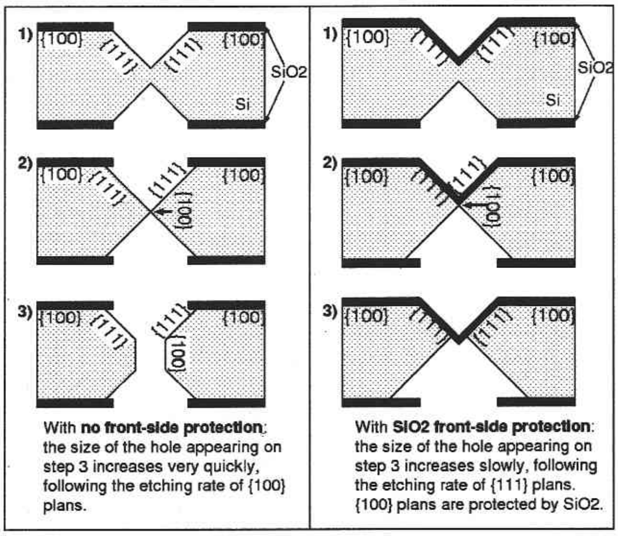

Figure 2: Formation of holes, with TMAH etching, at the interconnection between two V grooves, when there is or not SiO2 protection of one of the V grooves.

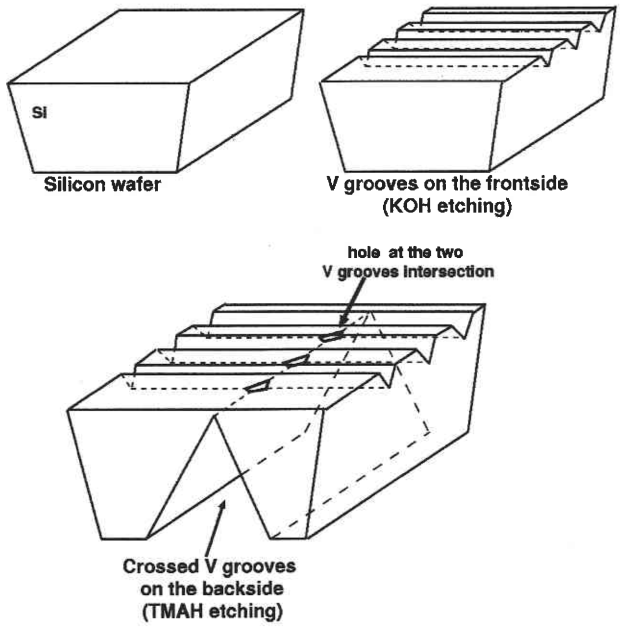

Figure 3: 3 D scheme of system made with standard V grooves. Tiny holes are obtained at the intersection between the front-side V grooves and the backside V grooves.

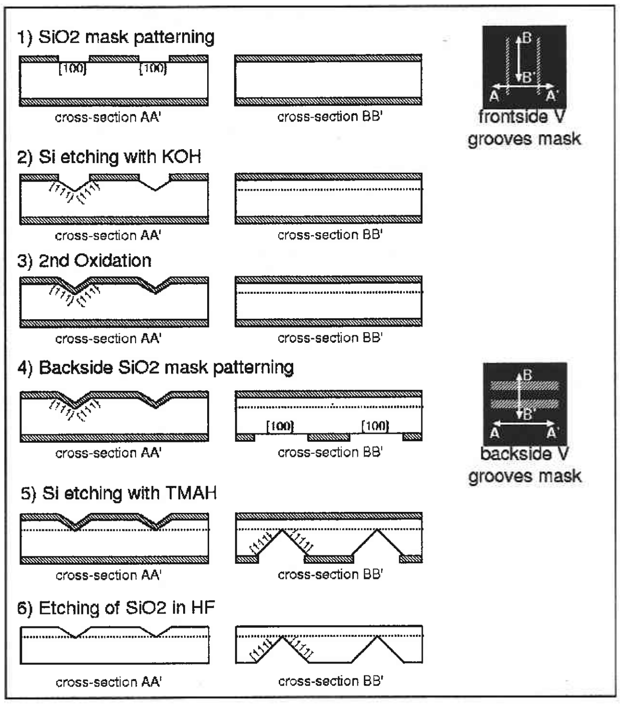

Figure 4: Detailed process chart of the fabrication of nano-holes.

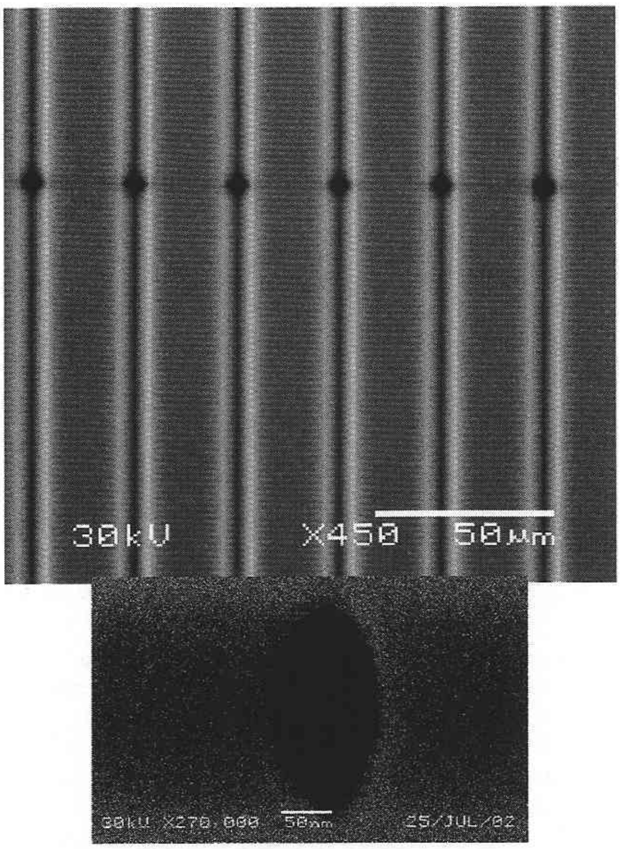

Figure 5: SEM pictures of holes obtained at the bottom of the front-side V grooves, according to the scheme Figure 3. The smallest ones obtained are 90 nm × 180 nm.

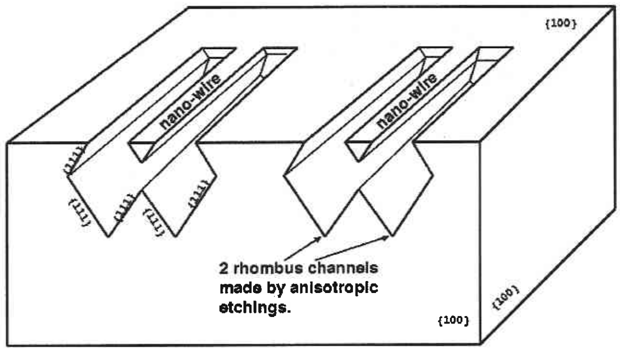

Figure 6: 3 D cross-section scheme of a system made with rhombus channels. They are obtained by adding a step of vertical etching of the substrate before each step of anisotropic etching. Nano-wires are made by interconnecting two rhombus grooves laterally.

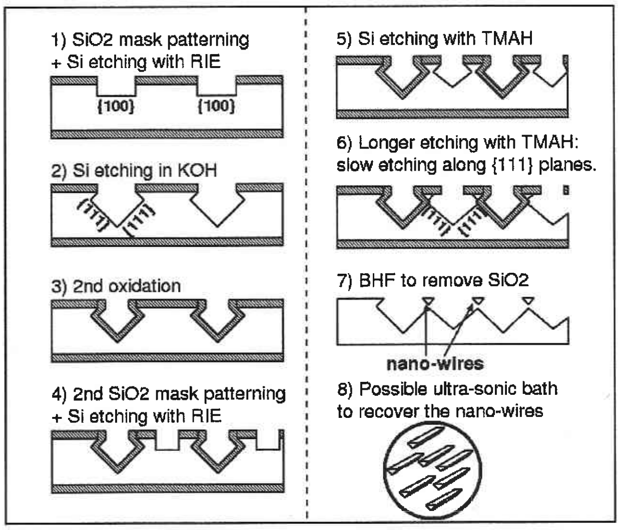

Figure 7: Process chart of the nano-wires fabrication. System seen in cross-section.

Figure 4 shows the detailed process of the structure presented on Figure 3.

One Figure 5 are shown SEM pictures of holes obtained with this technique, with two different dimensions: 4µm width holes and one of the smallest holes obtained: 90nm × 180nm.

If a vertical etching of silicon is performed before each anisotropical etching, the pattern shape is no more a V groove but a rhombus[3]. Taking advantage of this shape, nano-wires can be obtained by connecting two rhombus grooves together, in the lateral direction (Figure 6 and 7). Here the two anisotropic etching are both made on the front-side of the wafer. Again, the slow etching of {111} in TMAH allows precise control of the dimensions by etching time. Nano-wires can be obtained with dimensions as small as 100 nm in width and 15µm in length (Figure 8).

Both structures are really suitable for BIO-MEMS applications,

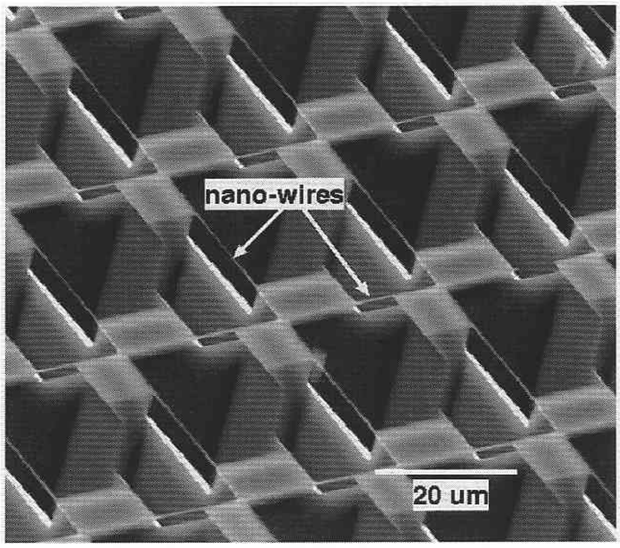

Figure 8: SEM picture of the realized nano-wires: 100 nanometersin width and from 5 to 15 micrometers in length, according to the mask pattern dimension.

because of their tiny dimensions.

EXAMPLES OF APPLICATIONS

One application with yeast cells and an other with motor protein are presented here.

The system with holes can be used to catch and to place cells precisely on a chip, by performing aspiration through the holes[4]. The aspiration creates a gentle water flow through each hole and cells in the water are dragged towards them. Figure 9 shows yeast cells (6μm in diameter) placed successfully as an array on holes. The system used for this experiment had rectangular holes, 2μm in width and several microns in length, because of the patterns design. The design can be improved in order to obtain square holes. Moreover, if smaller cells have to be placed in array, smaller holes can be easily realized just reducing the etching time in TMAH. In what concerns the nano-wires, one possible application is to recover these wires in solution (by performing ultra-sonic bath) in order to attach them on motor-molecules. Although a motor-moecule is too small to observe directly, the motion of attached objects could indicate the motion of the molecule.

CONCLUSIONS

In this article a cheap, simple and robust technique to make nano-structures with normal {100} silicon wafers and anisotropic etching was presented. It takes advantage of the slow and controllable, but not neglectible, etching of {111} plans in TMAH to adjust the nanometric dimensions of the structures.

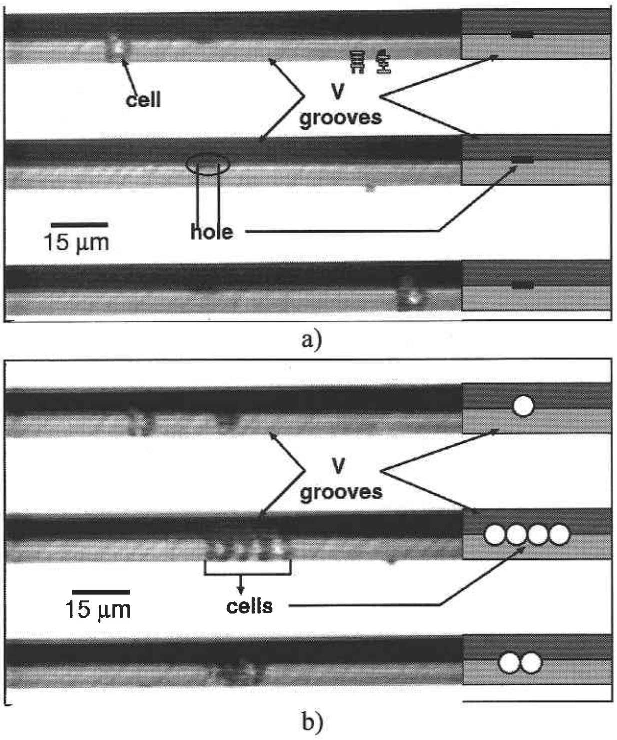

Figure 9: Photos of the holes system: a) before aspiration and b) after aspiration of yeast cells.

Two examples of realized nano-structures were described: nano-holes and nano-wires. These structures are really suitable for BIO-MEMS applications because of their tiny dimensions, close to the one of the biological materials.

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