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Etching Microwave Silicon [EMSi]-Microwave Enhanced Fast Deep Anisotropic Etching of Silicon for Micro-Electromechanical Systems [MEMS]

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A new method of fast wet anisotropic silicon micromachining is described. The new process provides significantly more rapid etching which is applicable in deep silicon micromachining for micro-electromechanical systems (MEMS). The process characteristics in a KOH:water mixture with a wide range of concentrations (3M–10M) and temperatures (60°C–90°C) are presented. The etching of silicon in an NH_4OH:water solution and spectacular etching in deionised water have been achieved. MEMS structures fabricated by the new process in a new machine are shown.

1. Introduction

Selective, deep, wet anisotropic etching of monocrystalline silicon is a technique commonly used for the fabrication of three-dimensional micromechanical structures. The structures' spatial form depends on etchant properties, the crystallographic orientation of the silicon wafer and the orientation of the mask pattern in relation to the planar crystallographic directions of the wafer.[1–3] These structures are the main parts of integrated silicon microsystem MEMS.

The silicon microsystems market attains turnovers of billions of dollars yearly.[4] Typical products are micromechanical pressure and acceleration sensors, ink-jet heads, optical fiber couplers, silicon pumps and chemical analysers.

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The etching of monocrystalline silicon is carried out in alkaline solutions with organic and inorganic bases. KOH:water solutions with the addition of isopropyl alcohol (IPA), ethylenediamine with pyrocatechol or pyrazine (EDP) and tetramethyl ammonium hydroxide (TMAH) are the most widely employed. Water solutions of NH_4OH, LiOH, CsOH are used occasionally.

The etching process is thermally activated. Typically the temperature range used is 80°C–90°C. The etching rates and the quality of the etched planes depends on the temperature and the concentration of the etchant. The process is accelerated imperceptibly in boiling etchants but the anisotropy and selectivity in relation to the mask are worse. Below 60°C etching rates decrease dramatically. These effects are the most apparent for KOH:water solutions.

The common disadvantage of wet silicon etching, regardless of the etchant used, is the low etching rate. The etching rate $V_{(100)}$ does not exceed 1.2 μm/min at 80°C for the (100) plane which is the most important plane from the point of view of fabrication (Fig. 1).

The (100) plane etching rate for KOH:water solution in the limited temperature range <100°C does not exceed 4.1 μm/min and anisotropy ($V_{(100)}$:$V_{(111)}$) is poor.[1] For EDP solutions at 117°C, the etching rate is similar but the anisotropy is poor ($V_{(100)}$:$V_{(110)}$:$V_{(111)}$=2:2.6:0.85 μm/min).[1,5]

The low etching rate of the (100) plane means that the process is very time consuming. For example, the etching time of 20-μm-thick silicon membranes on a 3" wafer (380 μm thick) at 80°C is 360 min and at 65°C is over 1000 min (not considering the poor quality of the etched planes).

(a)

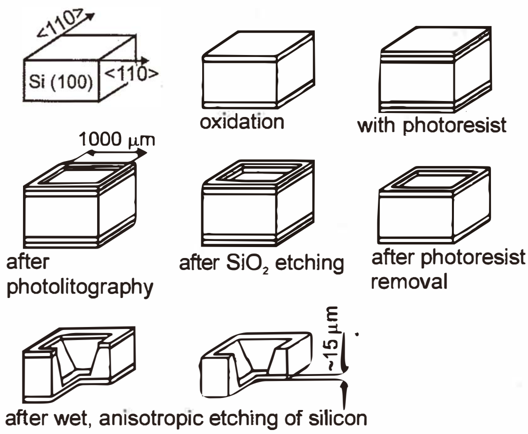


Fig. 1. MEMS structures obtained by wet, anisotropic etching - some examples:(a) fabrication of thin silicon membrane for pressure sensor.

(b)

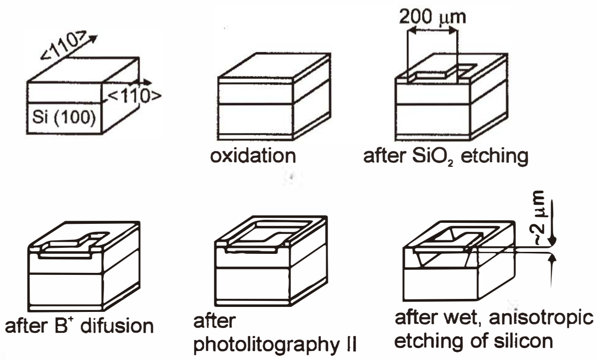


Fig. 1. (continued) MEMS structures obtained by wet, anisotropic etching - some examples: (b) fabrication of one-side hang beam using stop-diffusion, p^+ region is not etched.

2. Microwave Enhanced Fast Silicon Etching - EMSi

Fast silicon etching, Etching Microwave Silicon (EMSi), was developed in Poland in 1998.[6] The silicon wafers are placed inside a dielectric reaction chamber which is full of the etchant. The reaction chamber is placed inside a microwave resonator. According to this method, microwaves (300 MHz–10 GHz) irradiate the etchant. The microwave irradiation causes the excitation and heating of the etchant.

The etching process has three steps. At the beginning (step 1) full-power microwaves heat and excite the etchant rapidly, then (step 2) the microwave is reduced and the temperature is allowed to stabilise to the desired level. Finally, (step 3) microwave power is switched off and the etchant is allowed to cool to the ambient temperature.

The schematic and typical process characteristics for EMSi etching (temperature, microwaves power and pressure vs time) are shown in Fig. 2.

EMSi etching has been tested in laboratory-scale experiments for KOH:water solution.

Two types of etching set-up have been tested. The first setup is tightly, hermetically closed for processes being tested under elevated pressure and the second is equipped with an open chamber for etching below the boiling point of an etchant under atmospheric pressure. The etchant temperature was measured by use of a thin-film temperature-dependent thermistor and/or by an infrared sensor.

The increase of etching rate with order of magnitude is the most characteristic feature of the EMSi process.

The greatest etching rate of the (100) plane $V_{(100)}$ obtained in our test reached $V_{(100)} = 100\ \mu m/min$ for 3M KOH solution at 104°C and 7 MPa in a closed reactor. The anisotropy $V_{(100)}:V_{(111)}$ is low (~10). Significant damage to the mask was observed and therefore the etchant selectivity in relation to the mask (Si_3N_4) was poor. The process is too rapid and is uncontrollable.

(a)

(b)

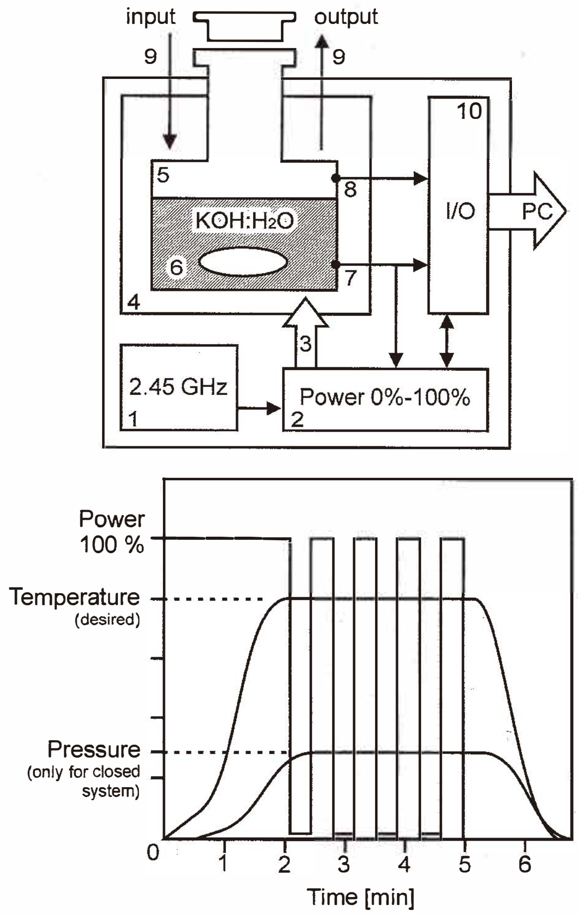


Fig. 2. EMSi etching: (a) scheme of the setup: 1-microwave generator, 2-power supply unit, 3-connector, 4-single or multimode resonator, 5-reaction chamber, 6-silicon substrate, 7-temperature sensor, 8-pressure sensor, 9-cooling water, 10-I/O. (b) process characteristics: pressure, microwave power and temperature vs time.

However, a repeatable and controllable EMSi process has been obtained for a system operating under atmospheric pressure. The basic characteristics of the EMSi process for the open system (under atmospheric pressure) are shown in Fig. 3.

Increase of the etching rate from 10 to approximately 50 times has been observed. Process selectivity in relation to the Si_3N_4 mask is about 1:10000. Point mask damage or overetching is not observed. The etching does not depend on the process temperature. The etching anisotropy decreases with the increase of solution molarity - this is inverse to the anisotropy of conventional etching. $V_{(100)}:V_{(111)}$ is about 30 for 3M–5M KOH:water

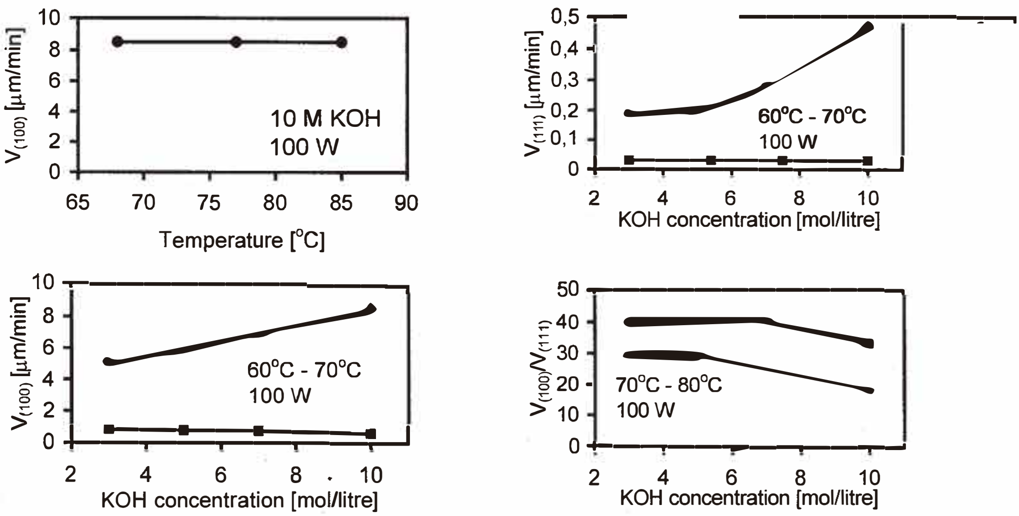


Fig. 3. Atmospheric pressure (open system) etching characteristics: (100) plane etching rate ($V_{(100)}$), (111) plane etching rate ($V_{(111)}$) and anisotropy $V_{(100)}$:$V_{(111)}$ vs temperature and molarity of KOH mixture for open system (atmospheric pressure), maximal microwave power level 100 W. EMSi process: round points, the standard process: square points.[1]

solutions, and it decreases to 20 for the 10M KOH:water solution. The most advantageous property of the EMSi process is its high etch rate below 70°C and the excellent quality of the etched planes, particularly the (100) plane, obtained at low temperatures (60°C–70°C) (Figs. 4, 5 and 6). This is impossible in conventional etching where the very low etching rate at low temperature – below 70°C – extends the etching time to 1000 min; therefore, no interest has been shown by researchers with regard to such low process temperatures.

In our studies, the preferential etching of single-crystal silicon in deionised water has been observed. We used a tightly closed reactor fulfilled with pure deionised 8 MΩcm water.

The DI water was heated to approximately 180°C by microwaves. The pressure inside the chamber increased to 4 MPa.

Typical anisotropic etching was observed. Well-defined (111) planes underetched around the Si_3N_4/SiO_2 mask are visible in the Scanning Electron Microscope (SEM) images of the etched cavities in Fig. 7. The DI water anisotropic etching of monocrystalline silicon obtained here indirectly confirms Palik's and Seidel's studies on the mechanism of silicon preferential etching.[1,5] These studies assume that hydroxide groups are necessary in the etching process and the role of cations is considered to be negligible.[10]

The equipment which has been used at this stage of research is insufficient for use in more detailed investigations of the nature of the EMSi process.

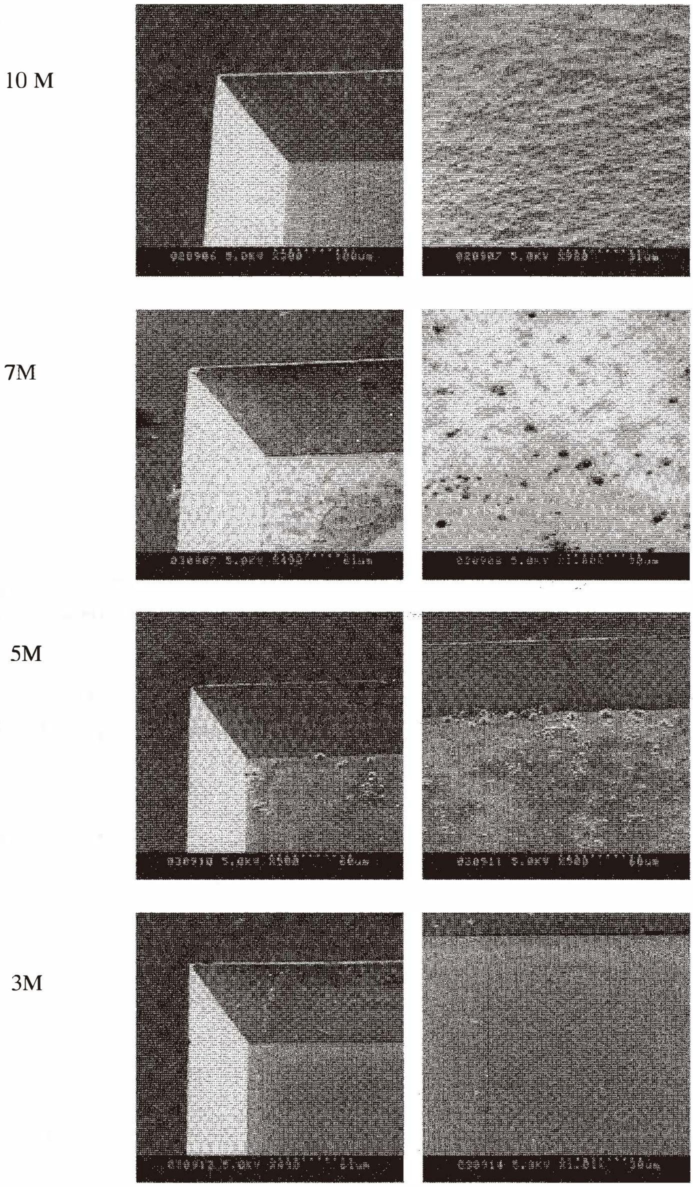


Fig. 4. SEM images of EMSi process: deep cavities etched in (100) silicon, molarity of KOH changed from 10M to 3M, temperature 70°C.

(a) (b)

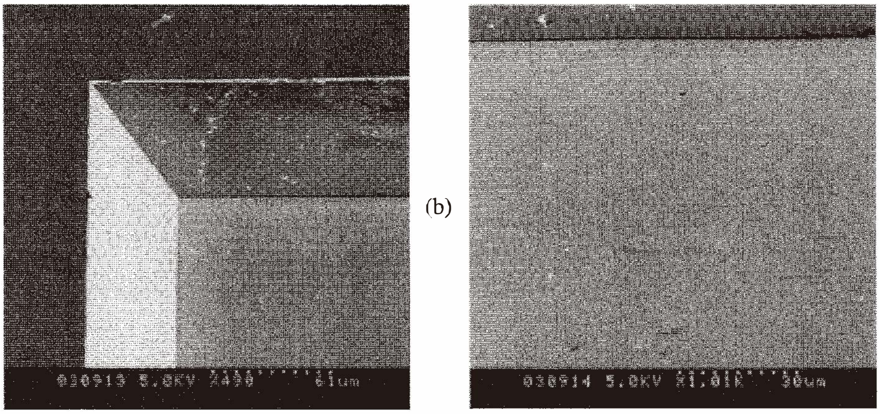


Fig. 5. EMSi process: membranes etched in (100) silicon, 3M KOH:water, 60°C, $V_{(100)}$ = 5.56 μm/min: a) SEM × 200, b) SEM × 1000; note the perfectly smooth (100) surface (no hillocks).

The "activation" of DI water by the electromagnetic field (microwave) excitation seems to be the only source of hydroxide group generation. This is a very interesting point which will be explored in our future studies.

The EMSi process in the open chamber has been tested with an NH_4OH:water mixture.

The obtained etched surfaces are unexpectedly very rough although the etching rate of the (100) plane is 10 times (approximately) higher than that reported in the literature for the appropriate temperature level.[11](Fig. 8)

The industrial applicability of the EMSi process has not yet been determined. However, the advantageous properties of the EMSi in relation to the disadvantages of the conventional method will probably lead to the wide adoption of the EMSi method (Table 1).

The new etching process has been tested in the laboratory-scale fabrication of micromachined structures.

We used 3" (100)-oriented, n-type double-sided polished silicon wafers covered with a Si_3N_4/SiO_2 mask in which 1000 × 1000 μm^2 square windows were photolithographically formed. The open-chamber EMSi process was carried out in approximately 40 min at 70°C in 5M KOH. Thin, uniformly etched, smooth flat membranes have been fabricated (Fig. 9).

The 20-minute-long EMSi process of bossed membrane etching in 5M KOH at 80°C was used to form the structure of the micromachined capacitive pressure sensor. The EMSi process was then followed by standard thermal anisotropic etching in 7M KOH with isopropyl alcohol (IPA) at 80°C for 15 min. This process finally formed the geometry of the boss and defined the thickness of the membrane of the pressure sensor structure.

The micromachined structure was covered with a SiO_2 magnetron sputtered layer on the front side, and a TiWAu ohmic contact was formed on the back of the chip.

Finally, the chip was anodically bonded to a glass substrate with a NiCr electrode. The cross-sectional view, the chip and the substrate before being assembled in a package and characteristics of the sensor are presented in Fig. 10.

10 M

7M

5M

3M

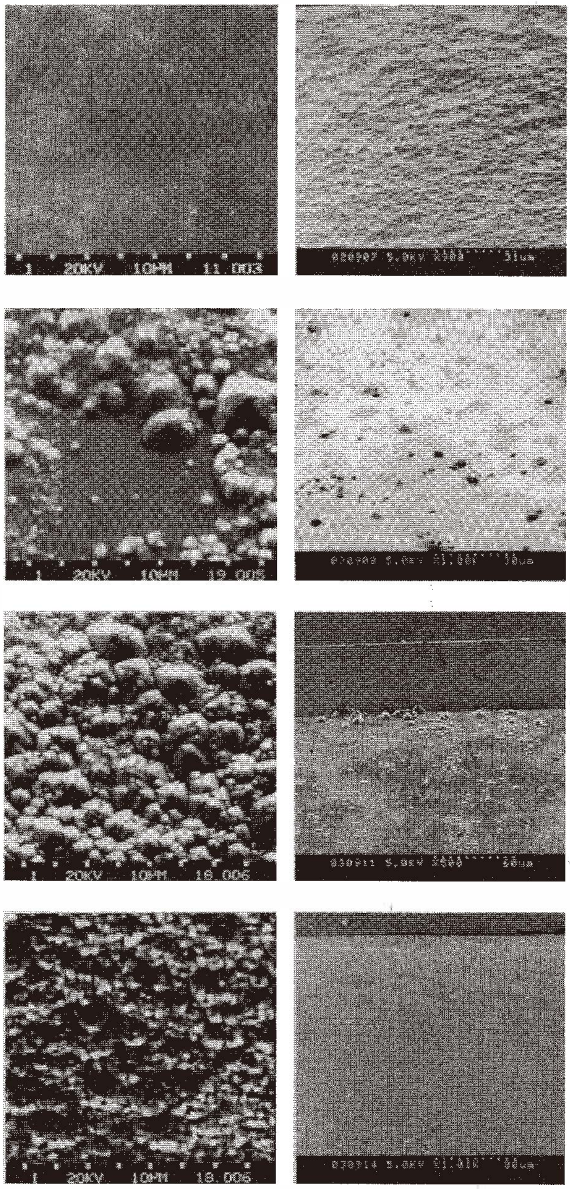
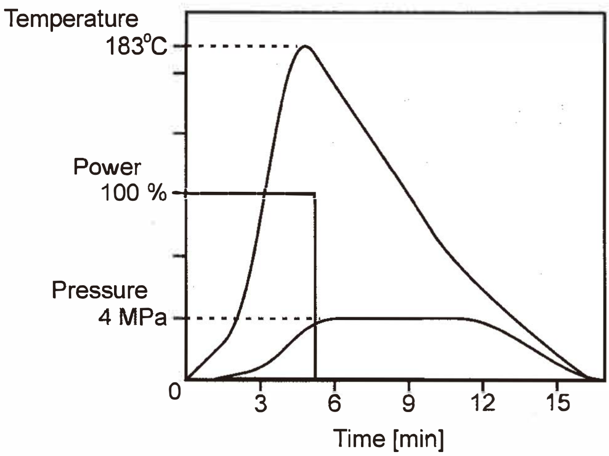


Fig. 6. A comparison of methods: SEM images of bottom, (100)-oriented planes of deep cavity anisotropically etched in (100) wafer: images on left hand side, thermally activated process, images on right hand side, EMSi process, molarity of KOH changed from 10M to 3M, temperature <70°C.

(a)



(b)

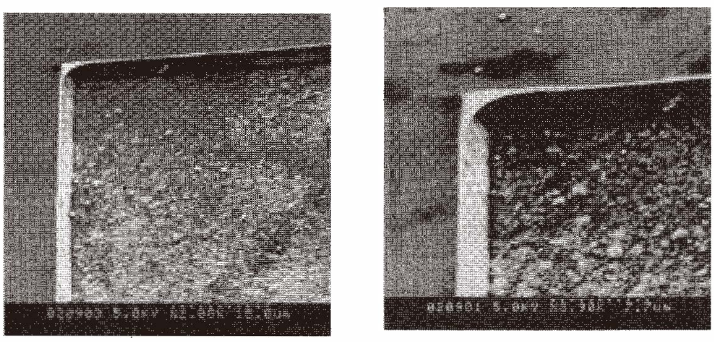


Fig. 7. EMSi process: etching of silicon in deionised water: (a) process parameters vs time, (b) etched pattern, SEM images magnification: left × 2000, right × 3900.

The EMSi process has been recently used in the fabrication of mold-type (transfer mold technique) field-emission arrays of SiC tips described by Górecka-Drzazga and others.(12)

The standard fabrication procedure of SiC mold arrays consists of two thermally activated anisotropic etching processes of a silicon substrate in KOH.

The first etching process forms an array of microminiature inversed pyramide-like cavities formed by four (111) planes. Typically cavities are less than 1 μm high, and are a few microns apart from each other. Cavities are filled with a SiC emissive layer, the substrate is bonded to the glass substrate and the silicon is etched away in a lengthy anisotropic etching process in 7M KOH:water at 80°C (Fig. 11). The average process time is 360 min (6 h).

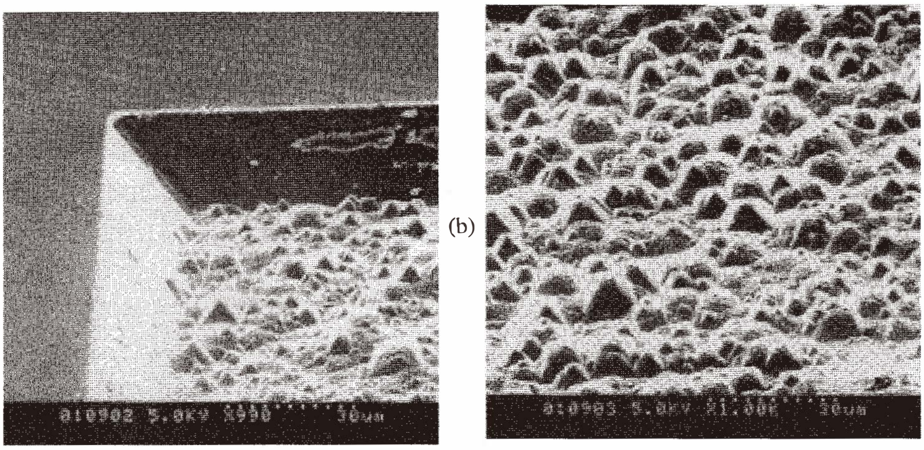
(a) (b)


Fig. 8. SEM images of (100)-oriented etched plane, 6M NH_4OH, T = 111°C, $V_{(100)}$ = 2 μm/min; note the large number of hillocks, SEM image magnification: left × 990, right × 1000.

Table 1
Comparison of etching methods in KOH.

Standard procedures	Microwave enhanced
low etching rates	high etching rates
$V_{(100)} \approx 1$ μm/min at 80°C	$V_{(100)} \leq 10$ μm/min at 80°C
etching rates depend on etchant temperature	etching rates depend on microwave power "pumped" into solutions
hillocks formed in low concentration KOH and/or low temperature	no hillocks
good anisotropy (10 M KOH~40–50) higher for stronger solutions	sufficient anisotropy 30–20 higher for weaker solutions
batch process	single wafer process, batch process possible
long (hours)	short (< 60 min)
etching rates at 60°C: negligible	etching rates at 60°C: high (~5 μm/min)
etching rates in 3 M KOH: negligible	etching rates in 3 M KOH: high (~5 μm/min)
SiO_2, Si_3N_4 mask	Si_3N_4 mask only; selectivity 1:10000
60°C process impossible	60°C process good

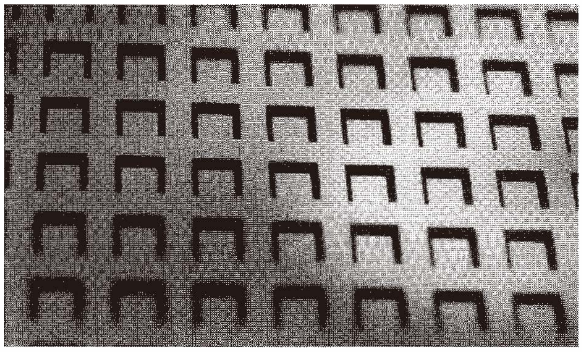
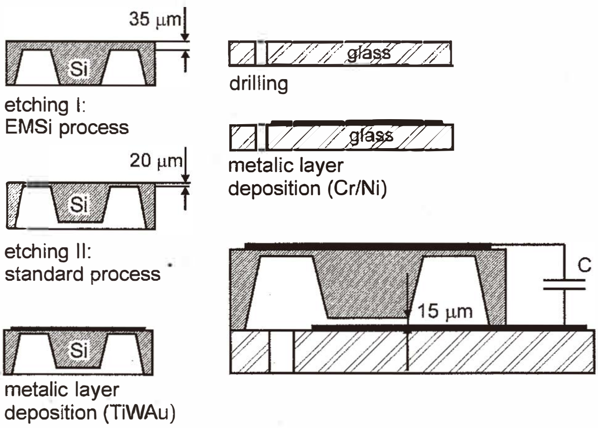


Fig. 9. MEMS structures etched by EMSi process; 20-μm-thick, 1000 × 1000 μm^2 membranes of piezoresistive pressure sensors etched in 380-μm-thick (100) substrate.

(a)



(b)

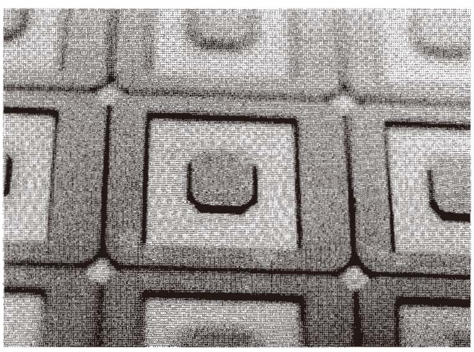


Fig. 10. Capacitive pressure sensors micromachined by EMSi process structure: (a) cross-section and fabrication steps and (b) the sensor chip after EMSi etching.

(c)

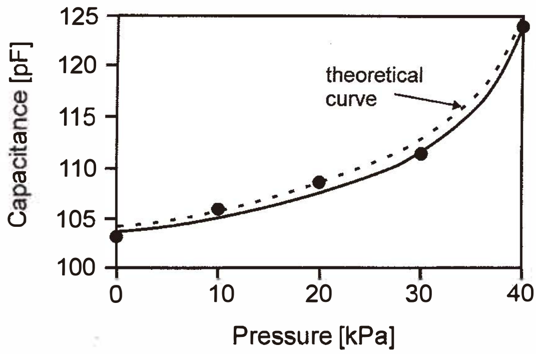


Fig. 10. (continued) Capacitive pressure sensors with micromachined by EMSi process structure: (c) output characteristic.

In our new procedure, final etching of the silicon substrate consists of fast EMSi process (approximately 40 min in 5M KOH at 80°C) followed by the short (20 min) standard anisotropic process in thermally activated KOH solution.

The MOLD array fabrication process has been shortened by almost 10 times – very reproducible and high-quality SiC tips have been obtained (Fig. 12).(13)

3. Conclusions

The EMSi process is a new method for fast, deep, anisotropic etching of silicon for MEMS. The EMSi process reduces the time required for strenuous silicon micromachining by conventional wet, anisotropic etching. The considerable increase of the etching rate does not significantly degrade the etching parameters (anisotropy, selectivity) in comparison with the conventional method. The quality of the etched planes is higher.

The mechanism and properties of the EMSi process are not yet clarified.

It is known that the etching rate depends on the microwave power level "pumped" into the etching solution and does not depend on the temperature. It is known that the pressure plays an essential role in increasing the etching rate.

In this work we have presented MEMS devices which have been produced by the EMSi etching process for the first time. This confirms the technical applicability of the EMSi process.

At the present stage of the investigation of the EMSi process, many questions remain unanswered:

• is this process competitive in comparison to standard etching procedures?

• what are the anisotropy, selectivity, underetching and stop-diffusion characteristics of the new method?

•what is the nature of the EMSi process and chemistry? Are an electrochemical etch stop or other etchants possible?

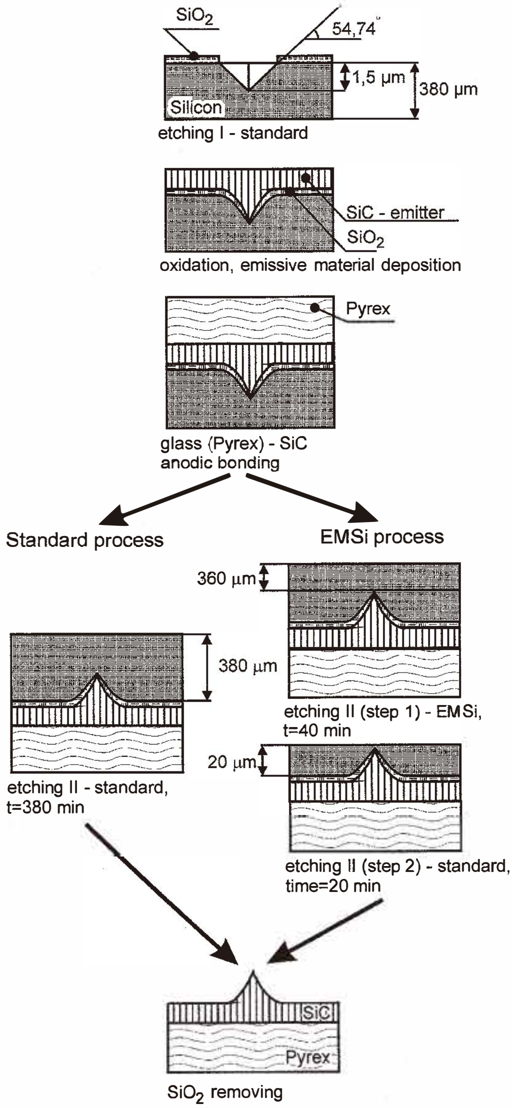


Fig. 11. A mold-type array fabrication procedure, standard process, thermally excited etching procedures (left branch) and the new EMSi process (right branch).

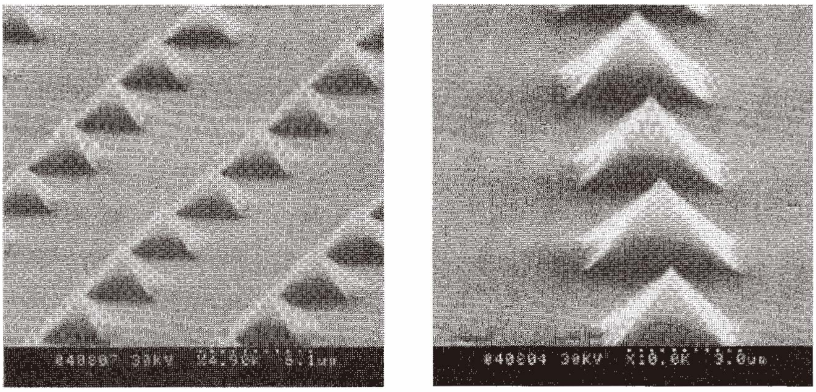


Fig. 12. The SiC mold-type array fabricated using the EMSi process: SEM image magnification: left × 4900, right × 10000.

To understand the mechanism and to make the EMSi process and machine suitable for industrial application, much remains must be done.

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