

## Porous silicon bulk micromachining for thermally isolated membrane formation

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### Abstract

A novel low thermal budget technique is proposed for the preparation of thermally isolated silicon membranes. The selective formation of porous silicon in a p-type silicon wafer results in an undercut profile below the implanted n-type silicon regions. The sacrificial porous layer is subsequently removed in a dilute KOH solution. A non-stoichiometric LPCVD nitride layer combination forms the suspension of the single-crystalline silicon membranes. This technique eliminates the need for epitaxial substrates and backside alignment, and proves to be very efficient in the realization of a high-temperature micro-hotplate operating with minimum power consumption for the purpose of integrated gas sensors.

**Keywords:** Bulk micromachining; Membranes; Porous silicon

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### 1. Introduction

Recent advances in microsystem technology are directed towards the integration of arrays of sensing elements with the evaluation logic for an enhanced sensitivity and selectivity for various gas compounds. In the sensor arrays both semi-conducting (adsorptive) and catalytic (combustion type) sensor elements operated at different elevated temperatures can provide the required flexibility and sensitivity with the appropriate processing of the output characteristics.

Combustive and semiconductive oxide gas sensors operate at elevated temperatures between 300 and 600°C. In discrete devices, e.g., pellistors, this temperature can be sustained at the cost of high power consumption (0.2–0.6 W) of the sensor element. Such a high dissipation is not even allowable in standard transducers operating with 4 mA at 12 V. The single sensing element for integration must have a power dissipation of less than 10 mW. Consequently, the efficient thermal isolation of the heated sensing area is of crucial importance. Membrane size is the main limitation for convection and radiative losses, while the conductive losses depend mainly on the cross section of the membrane and distance between the heater and silicon support.

Suspended thin membranes of mostly silicon nitride or oxinitride [1] are usually formed by preferential alkaline wet chemical etching, often by means of double-sided alignment. The formation of suspended silicon membranes by these techniques is only possible with a highly doped etch-stop diffusion and the application of a toxic EDP etchant. In many applications, however, a moderately doped silicon membrane is required for reasons of electronic construction.

The present approach is aimed at the development of a novel technique addressing all the above concerns primarily in a micro-hotplate application.

(a) Suspension of the low-mass silicon membrane is possible by a reduced-stress non-stoichiometric silicon nitride bridge supported at a few suspension points along the perimeter of the membrane.

(b) The silicon membrane is formed by a novel, low-cost bulk-micromachining technique using laterally selective porous silicon formation by anodization. This fully self-aligned technique requires no double-sided alignment.

(c) The heating element is formed from a polysilicon resistor in place of metal. The occupied area for equal performance can thereby be considerably reduced due to the difference in specific resistivity by up to five orders of magnitude. In this scheme the suspended silicon membrane serves as a heat-distribution plate.

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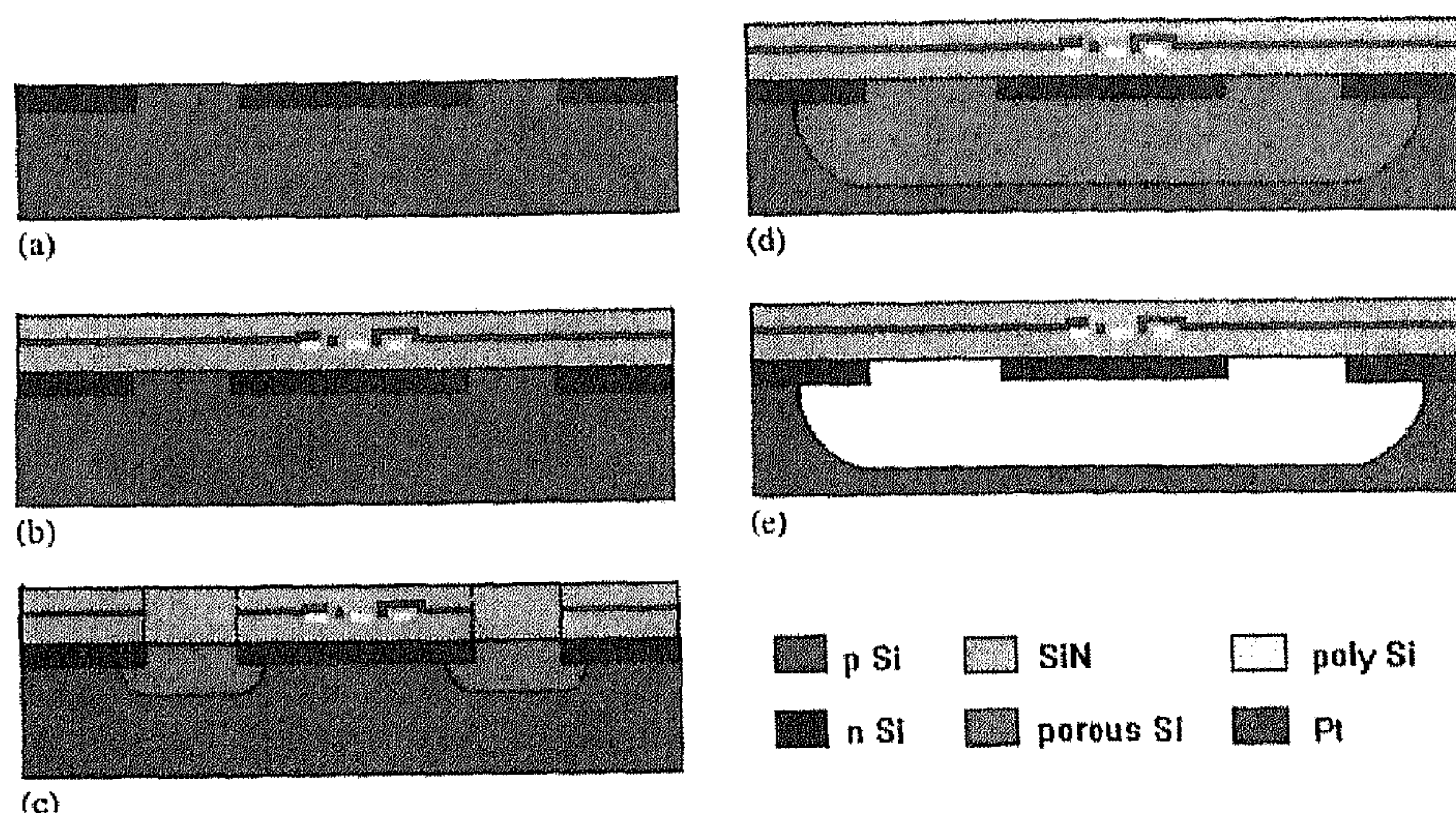


Fig. 1. Process sequence of bulk-micromachined suspended silicon membrane micro-hotplates.

## 2. Experimental

The process sequence is shown in Fig. 1. The p-type 4–6  $\Omega \text{ cm}$   $\langle 100 \rangle$  Si wafer was selectively doped with phosphorus to a surface concentration of  $1.2 \times 10^{17} \text{ cm}^{-3}$  using an oxide mask to obtain about 5  $\mu\text{m}$  deep n-type regions. After oxide removal non-stoichiometric silicon nitride of 0.8  $\mu\text{m}$  thickness was deposited by LPCVD at 800°C and 0.5 mbar from  $\text{SiH}_2\text{Cl}_2:\text{NH}_3 = 3.8:1$ , resulting in a refractive index of  $n = 2.16$  at HeNe, which corresponds to a composition of roughly Si:N = 0.93:1 (Fig. 1(a)).

The heating element was formed from a doped polysilicon resistor meander on the membrane. Electrical connection was provided through contact holes in the second 0.2  $\mu\text{m}$  non-stoichiometric nitride layer. The Pt thermometer resistor and the wiring on the hotplate were formed simultaneously, followed by the deposition of a third encapsulating non-stoichiometric nitride layer of 0.8  $\mu\text{m}$  thickness. After the appropriate patterning and nitride removal the selective anodization using an Al backside contact could start through the nitride window (Fig. 1(b)).

The porous Si layer was formed by anodization in the dark at room temperature using a complex current profile. The electrolyte consisted of hydrofluoric acid (HF) 50 wt.% and absolute ethanol in 7:3 volume ratio. A porous silicon layer thickness of 72  $\mu\text{m}$  was obtained, sufficient to completely undercut the 100  $\mu\text{m} \times 100 \mu\text{m}$  silicon membrane (Fig. 1(c)).

The removal of the porous phase, i.e., the formation of the air gap, was done by a selective etch using 2 wt.% KOH solution at room temperature (Fig. 1(d)).

## 3. Results and discussion

The top view of a typical micro-hotplate is shown in the optical micrograph in Fig. 2. The membrane size is 100  $\mu\text{m} \times 100 \mu\text{m}$  and the width and length of the suspension are

20  $\mu\text{m}$  and 110  $\mu\text{m}$ , respectively. The SEM cross section of the same structure is shown in Fig. 3(a) and (b) at different view angles. The 5  $\mu\text{m}$  thick Si membrane hanging on the nitride suspension has a smooth bottom and well-defined dimensions. Note the characteristic profile of the cavity underneath the membrane. The excellent mechanical stability is evident even after cleavage.

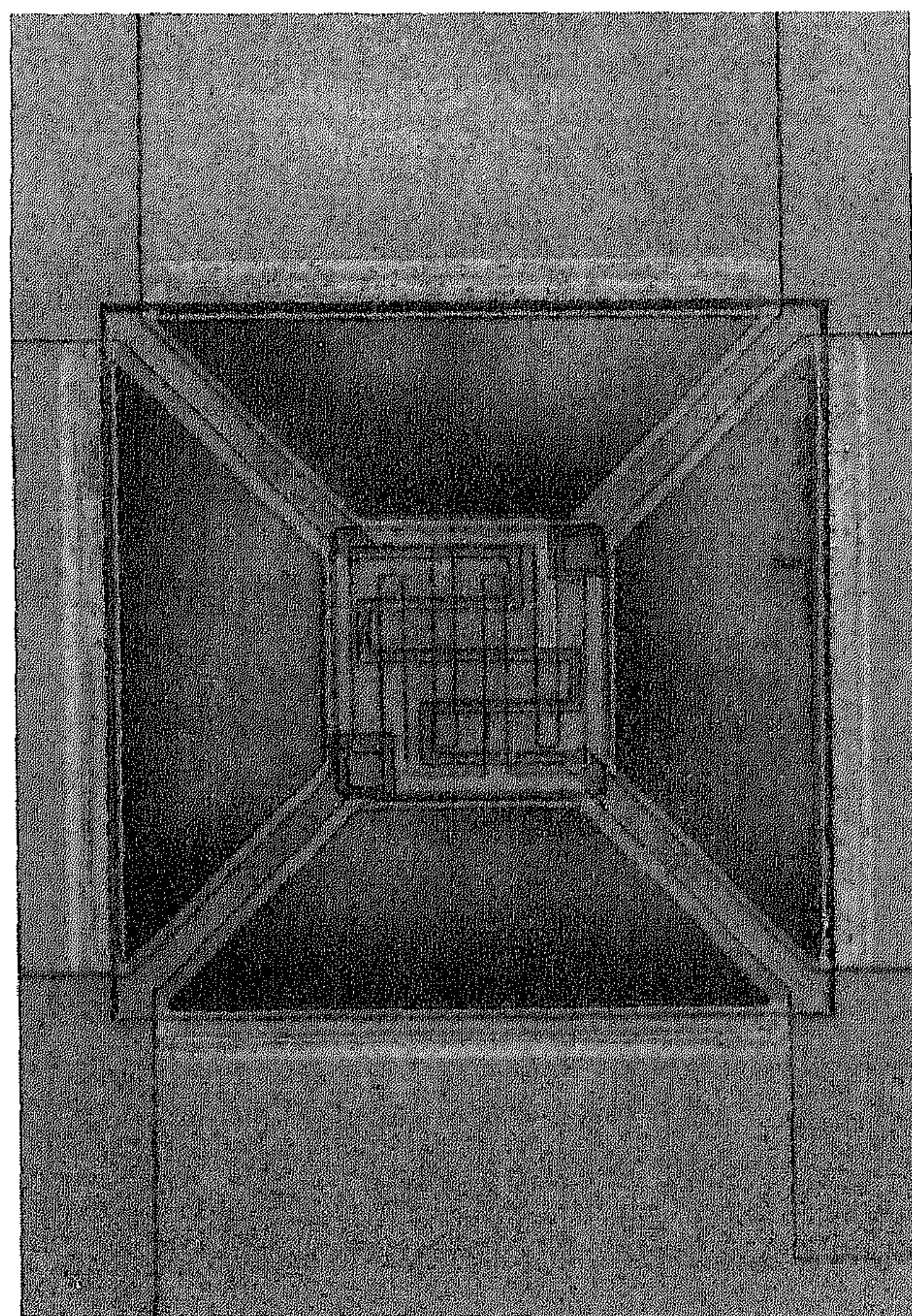


Fig. 2. Optical micrograph of a 100  $\mu\text{m} \times 100 \mu\text{m}$  nitride suspended micro-hotplate with Pt thermometer resistor and polysilicon heating element.



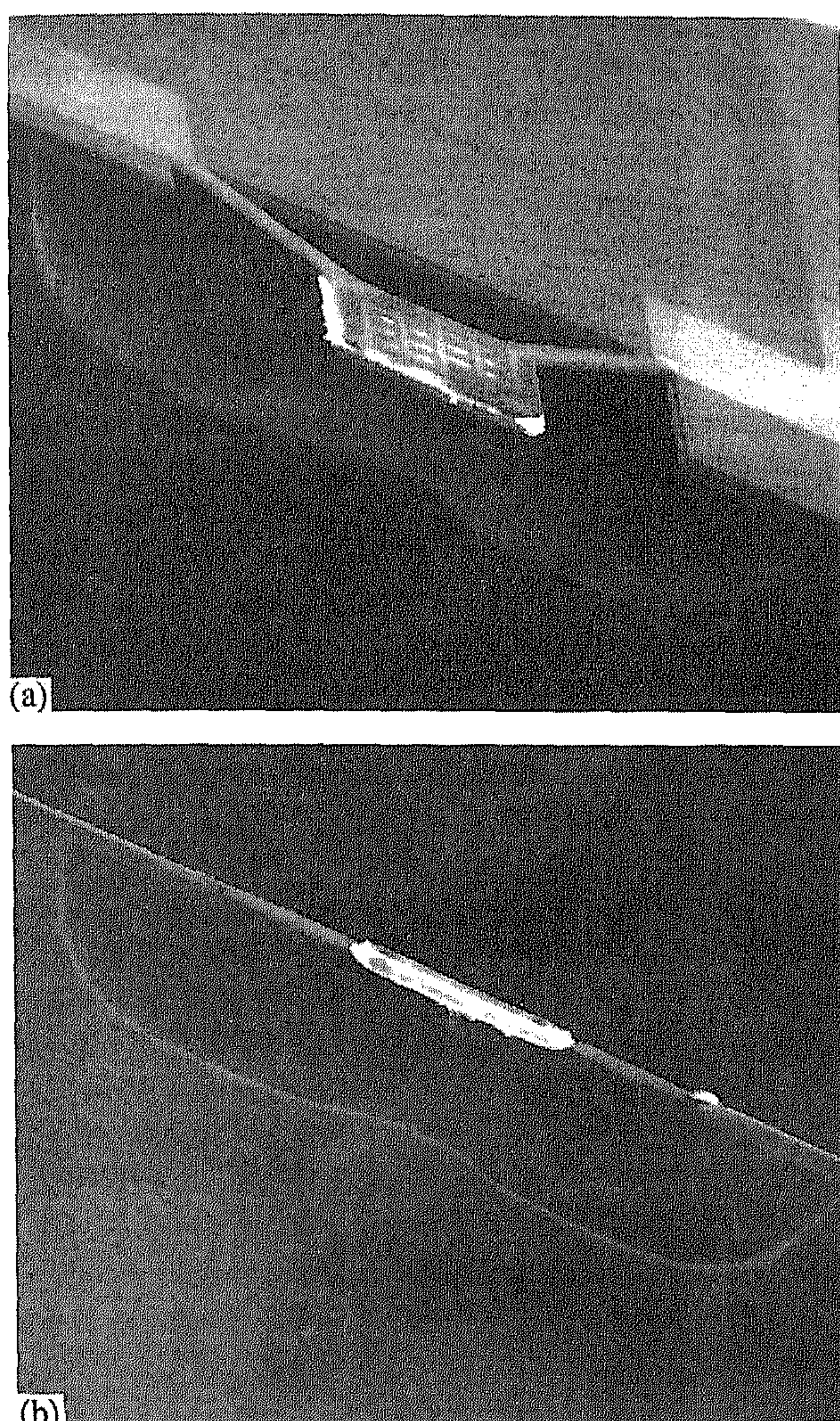


Fig. 3. SEM cross sections of a cleaved micro-hotplate structure viewed at a tilt angle of  $30^\circ$  (a) and perpendicularly (b). The  $5\text{ }\mu\text{m}$  thick suspended silicon membrane can clearly be distinguished. The size of the micro-hotplate is  $100\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$ .

Porous silicon was first used in oxidized form for SOI-like CMOS integration [2], and recently applied to the fabrication of micromechanical membranes as a sacrificial layer by using epitaxial etch-stops [3]. Formation of porous silicon in eous HF solutions requires the presence of holes at the interface. This implicitly offers the possibility of lateral selectivity, since the non-illuminated moderately doped n-type surface is not affected, while p-type surfaces are attacked in the electrolyte leading to the formation of pores. According to Fig. 4 an exceptional selectivity between n- and p-regions is obtained. The non-stoichiometric nitride protective layer has a limited but sufficiently low etch rate in the anodization solution according to Fig. 5.

Due to the isotropic nature of the porous layer formation in the cavity and the dimensional requirements regarding the undercut, a relatively thick porous layer has to be formed. This, however, is very critical from the point of view of mechanical stability of the developing porous layer. For a given electrolyte composition the porous layer characteristics

are controlled by current density and time. In order to maintain the integrity and mechanical stability during formation of the sponge-like porous phase, the porosity has to be changed in depth [4]. The computer-controlled anodization method developed in our laboratory offers an excellent control over porosity and thickness by adjusting the injected current. Fig. 6 is a plot of the applied current-density profile during anodization.

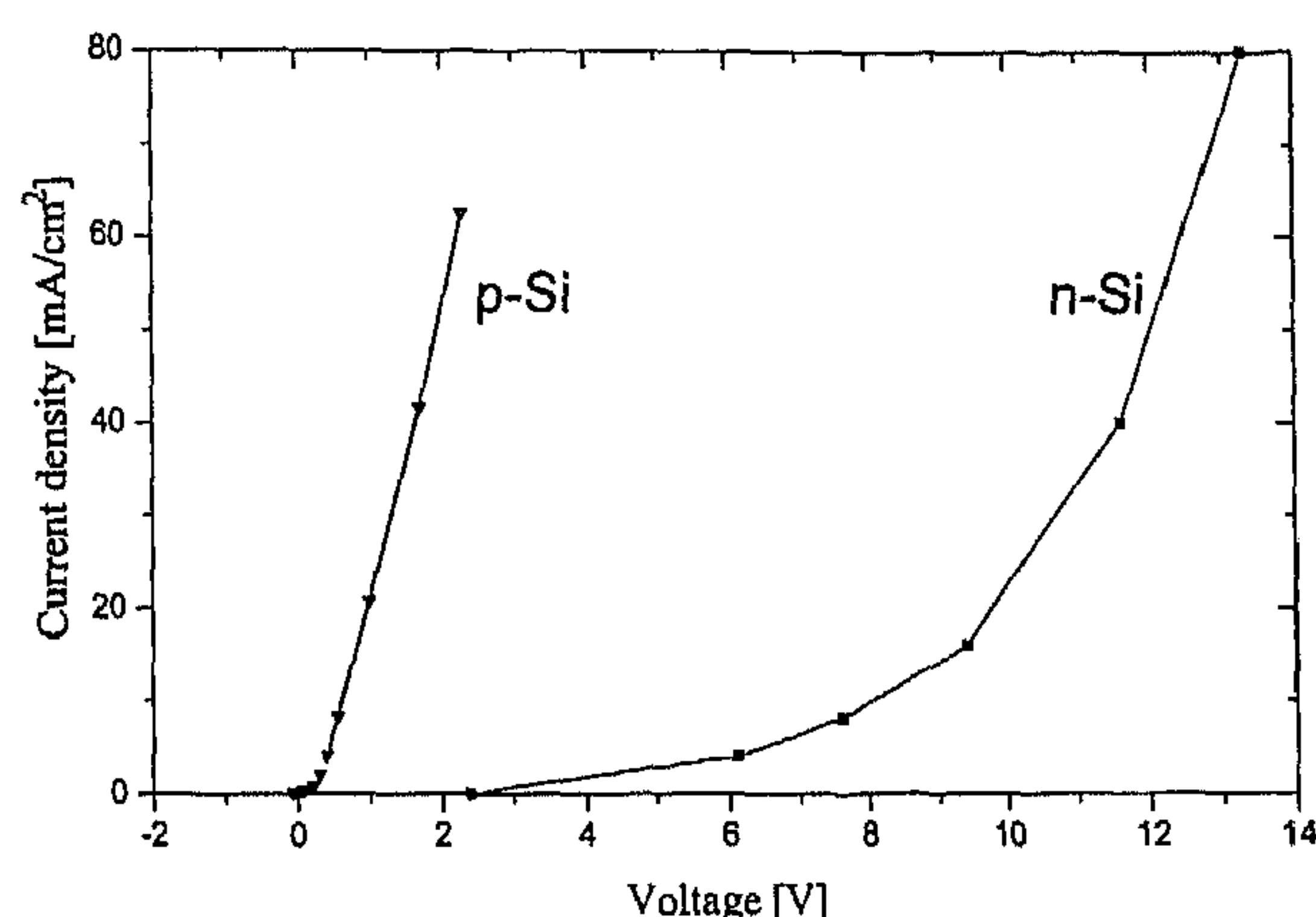


Fig. 4. Anodization current-voltage plot on p-type and n-type silicon in the applied electrolyte, reflecting the excellent obtainable lateral selectivity.

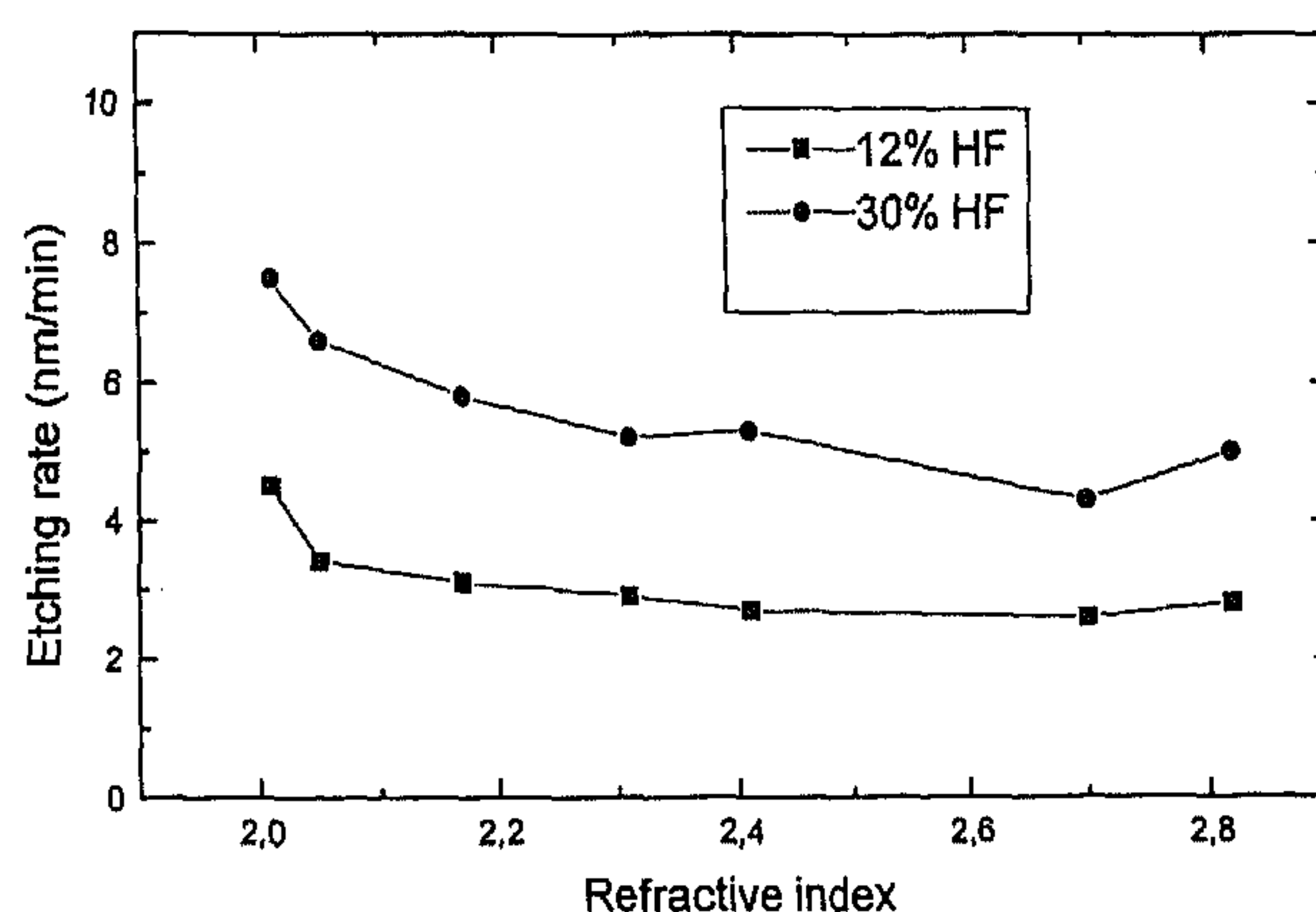


Fig. 5. Etching rate of silicon-rich nitride layers vs. refractive index in different HF solutions.

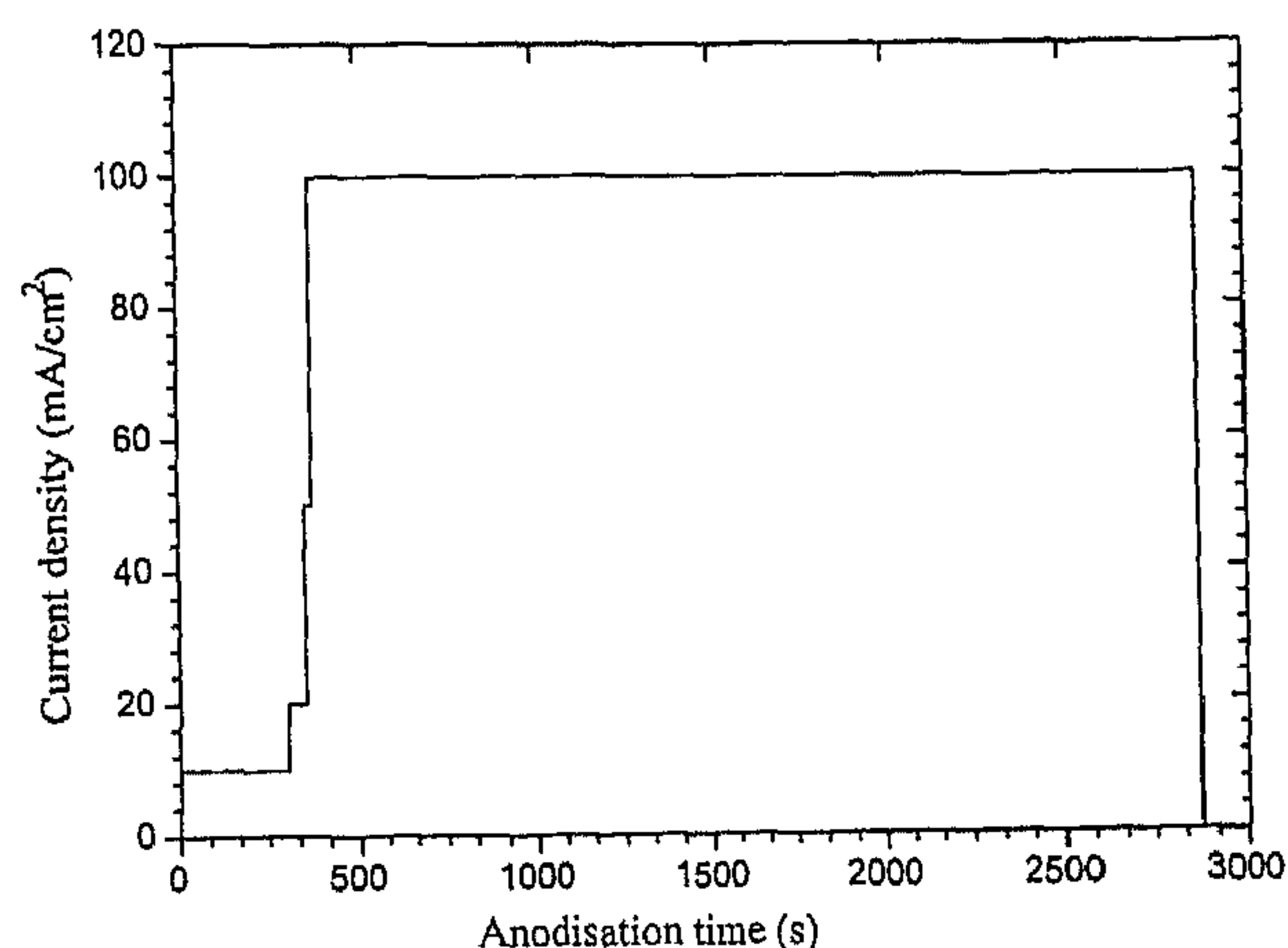


Fig. 6. Anodization current program for the preparation of mechanically stable  $72\text{ }\mu\text{m}$  thick porous silicon layer on  $4\text{--}6\text{ }\Omega\text{ cm}$  p-type (100) Si.



The result is a quasi-isotropic porous layer formed underneath the unattacked n-type silicon. Continuing the etching leads to the touching of the fronts from both sides of the 'masking' layer, which is in our case the n-type island, i.e., the membrane to be formed and/or the silicon nitride suspension. The characteristic etching front is clearly visible on the SEM pictures in Fig. 3. The only limitation in the silicon membrane formation by this micromachining technique is the minimum depth of the cavity being at least half of the lateral dimension of the freestanding membrane. However, constraints of wafer thickness and mechanical stability have to be considered too. Note that the mass and dimensions of the suspended single-crystal membrane can be excellently controlled by the light n-doping profile. The smooth bottom of the membrane and the moderate doping level facilitate most kinds of pre- and postprocessing.

The micro-hotplate shown in Fig. 2 has been heated by constant-current bias. The temperature was measured by a calibrated resistor of  $300\ \Omega$  composed of a  $300\ \text{\AA}$  Ti adhesive layer and  $1000\ \text{\AA}$  Pt. The resulting temperature versus heating power plots for some membranes with 10 and  $20\ \mu\text{m}$  wide suspension beams are shown in Fig. 7. The preferred uniform temperature distribution across the low thermal mass membrane is provided by the silicon plate of high heat diffusivity. The temperature of  $\approx 200^\circ\text{C}$  achieved with an input power of  $15\ \text{mW}$  is already very promising. Unfortunately, the non-optimized Pt metallization and silicon-rich nitride encapsulation developed rather high dissipation losses in the heating resistor wiring to the bond pad. Due to the long final nitride CVD step at high temperature ( $800^\circ\text{C}$ ,  $\approx 300\ \text{min}$ ) a silicidation of the Pt wiring occurred accompanied by the drastic deterioration of the metal characteristics (specific resistivity and temperature coefficient). Regarding the mechanical and thermal stability and reliability of operation, the micro-hotplate endured between room temperature and a  $15\ \text{mW}$  load several hundred repeated cycles without any degradation of performance. However, there is room for an

improved design. The present deficiencies and shortcomings are therefore expected to be overcome by improved geometry and process optimization.

#### 4. Conclusions

We described a novel bulk-micromachining process with low thermal budget by the use of a simple selective sacrificial porous silicon technique. The one-sided structuring process, being fully self-aligned, is compatible with microsystem integration. It is capable of fulfilling the stringent low power consumption requirements in micro-hotplates for gas-sensing elements, but it can also be applied to the fabrication of a variety of micromechanical structures. The described micro-hotplate preparation technique is intended to form a basis for the fabrication of integrated gas-sensing arrays. The different adsorptive and combustive sensing layers in the classical manner can be selectively deposited on the top of the micro-hotplates preceding cavity formation, but in that case they must be able to withstand subsequent processing. More appealing is the approach of selective even double-side coating after completion of the suspended structure (see, e.g., Ref. [5]). Moreover, the uniform-temperature silicon membrane offers by porous etching the unique possibility of specific surface enhancement on the bottom, which is essential in micropellistor-type applications.

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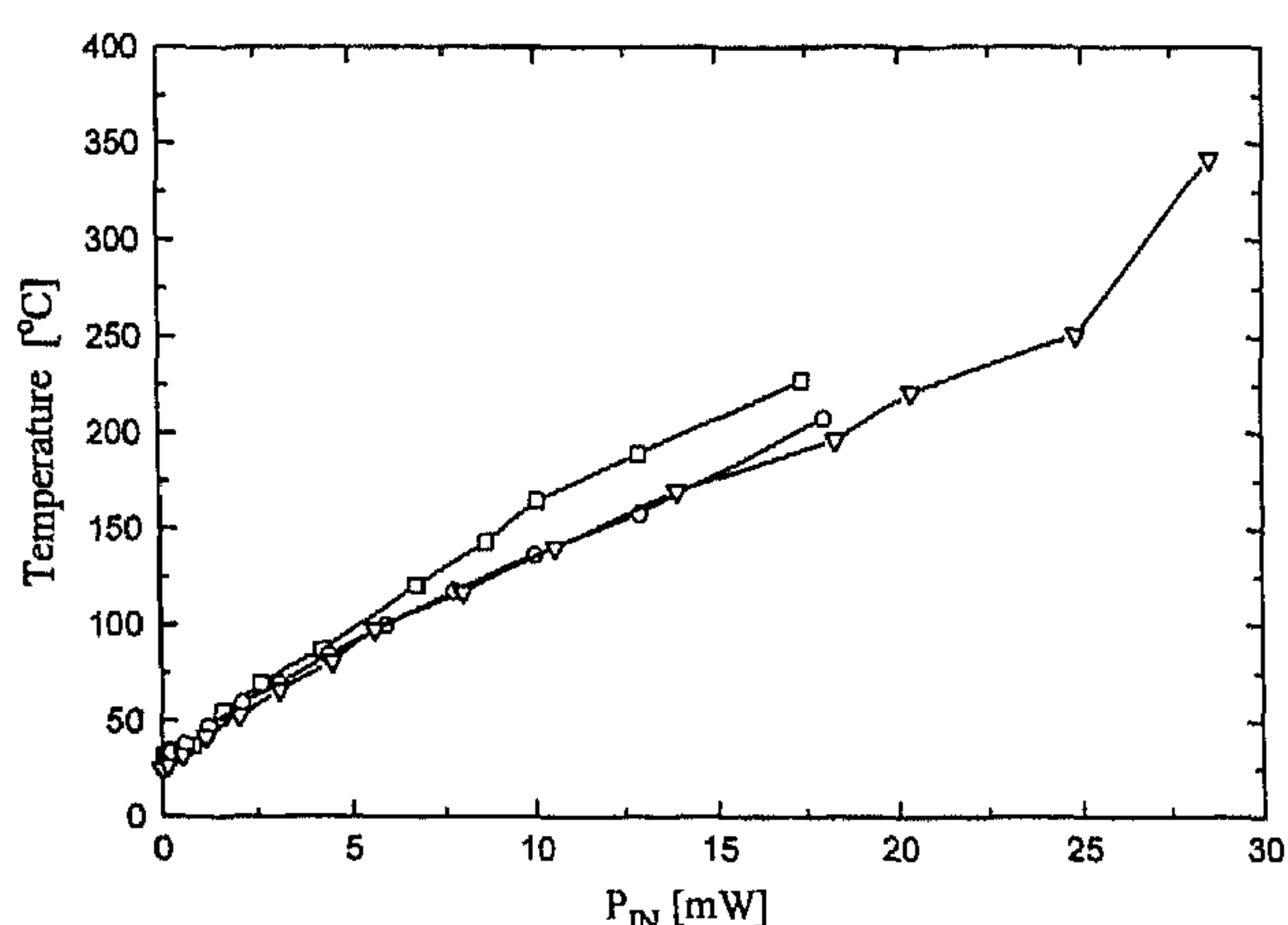


Fig. 7. Temperature of micro-hotplates of different geometries vs. heating power: □, membrane size  $70\ \mu\text{m} \times 70\ \mu\text{m}$ , suspension length/width  $110/20\ \mu\text{m}$ ; ▽, membrane size  $100\ \mu\text{m} \times 100\ \mu\text{m}$ , suspension length/width  $110/20\ \mu\text{m}$ ; ○, membrane size  $100\ \mu\text{m} \times 100\ \mu\text{m}$ , suspension length/width  $110/10\ \mu\text{m}$ .

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