Fabrication of planar semiconductor diodes : an educational laboratory experiment

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Fabrication of Planar Semiconductor Diodes, an educational laboratory experiment

By

C.J.H. Heijnen, H.A. Jansen,
J.F.G.J. Olijslagers and W. Versnel

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Eindhoven The Netherlands

FABRICATION OF PLANAR SEMICONDUCTOR DIODES, AN EDUCATIONAL LABORATORY EXPERIMENT

By

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EUT Report 81-E-119
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Contents page

Abstract.

A description is given of a laboratory experiment, in which students themselves manufacture semiconductor diodes and resistors in half a day. The process used is characterized by diffusion of impurities from an oxide layer into silicon and is not critical in this case. The oxide with impurities is formed at a temperature of 350°C by chemical vapour deposition of silane, oxygen and phosphine.

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1. INTRODUCTION

The idea of this experiment is to enable students to make their own semiconductor devices on which they finally perform a number of measurements.

Many strictly prescribed operations have to be carried out in making a semiconductor device. This is contrary to the now well-established practice in student experiments, where the instructions are so formulated that the students are confronted with a number of problems which they have to solve themselves. The intention was here to make that part of the experiment in which no deviations from the instructions are allowed, i.e. the fabrication, as short as possible. In the experiment described here that takes half a day (i.e. four hours).(See [1] for a one-year semiconductor technology course). This has been achieved first by making only simple devices, diodes and resistors of comparatively large dimensions, and second by using the technique of diffusion from doped oxide layers [2] obtained by chemical vapour deposition (shortly CVD) instead of the usual technique of gas diffusion [3].

The fabrication is described in this report. The measurements to be performed subsequently and that takes two more half days, are only shortly summarized (section 7), as they have been dealt with extensively elsewhere [4].

In Sections 2 to 6 the experiment is described as presented to the students. A drawing of the CVD reactor is given in appendix 1. In appendix 2 the lay-out and dimensions of the room in which the students do the experiment is shown.

2. GENERAL SURVEY OF THE EXPERIMENT

The original slice of p-type silicon has a diameter of 5.7 cm (2¼ inches) and a thickness of 400 μm. The specific resistance of the slice is $\rho = 1.8 - 2.4\ \Omega\text{cm}$.

At certain positions n-type regions will be formed by diffusion. The depth of the n-type diffusion is of the order of 1 μm. The configuration obtained is shown in Fig. 1.

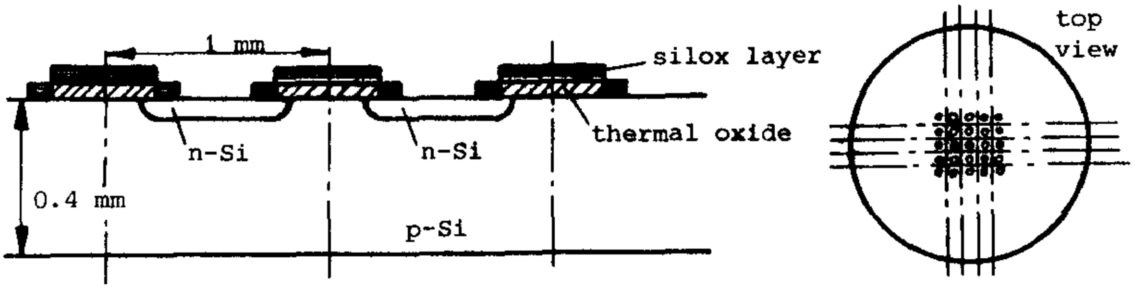

Fig. 1 *Configuration of slice with diffused n-regions in a p-substrate*

After all the actions have been performed, the slice is cut along the broken lines (see Fig. 1), resulting in a large number of diodes and resistors (more than 4000).

Slice cutting will not be done by the students. They just measure the electrical behaviour of the diodes and the resistors on the slice itself.

In the next section the steps that lead to a semiconductor device will be treated summarily, and the separate process steps are described in more detail in section 4. In Section 5 an introduction is given on the diffusion equation and on impurity profiles. In Section 6 we deal with the applied masks. Several measurements can be carried out on the devices obtained. They are summed up in Section 7.

3. THE STEPS IN THE FABRICATION OF THE DEVICE

In this section a general description will be given of the steps that have to be taken to obtain a semiconductor device.

(1) Thermal oxidation

Silicon reacts with oxygen at temperatures above 900°C [3]. The outer layer of the slice oxidizes, so that the slice is covered by a layer of SiO_2. An oxide produced in this way is called a thermal oxide.

The oxide has the following advantages:

1. The oxide layer acts as a protecting mask against penetration of impurities.
2. The layer is an insulator. This makes it possible to put conducting strips on the oxide which make contact with the silicon only at those places where windows are present in the oxide layer.
3. The crystal lattice of silicon dioxide matches fairly well with the crystal lattice of silicon. This implies a kind of stabilization of the silicon surface: foreign atoms can easily attach themselves to the surface of uncovered silicon.

(2) Photographic process

In order to obtain windows in the oxide a photographic process is used. The oxide is covered with a thin, uniform layer of photoresist, a light-sensitive material. By means of a mask, i.e. a photographic (glass) plate with a black and white pattern for the windows, the photoresist is partially exposed to light. Polymerization occurs at the exposed regions.

By means of a developer the photoresist at the unexposed regions[*] is removed. Then, the slice is placed in a bath filled with a silicon etch. The oxide will be etched away only at those regions where the photoresist layer has been removed (Fig. 2).

(3) Oxidation by the CVD process

In addition to the thermal oxidation process another oxidation process is applied in the semiconductor technology, viz. by chemical vapour deposition (CVD). In the latter process the gaseous silicon compound silane SiH_4 is used. At 350°C silane already reacts with oxygen to form SiO_2 which we will call silox. This is done in a glass holder, the CVD reactor[**] (Fig. 3). We refer to [5] for a more professional equipment.

The slice is placed in the reactor whose heating-plate is at a temperature of 350°C. The silane is rarefied with argon. Together with oxygen it is made to flow along the slice so that the slice is covered with a layer of SiO_2 (Fig. 4). This process is called deposition. By mixing the gas with phosphine PH_3, a gaseous phosphorus compound, at the same time, phosphorus atoms can be built into the silox. Afterwards, the phosphorus atoms can be diffused from the oxide in the silicon where the silox layer is in contact with the silicon surface, i.e. in the windows made in the thermal oxide layer.

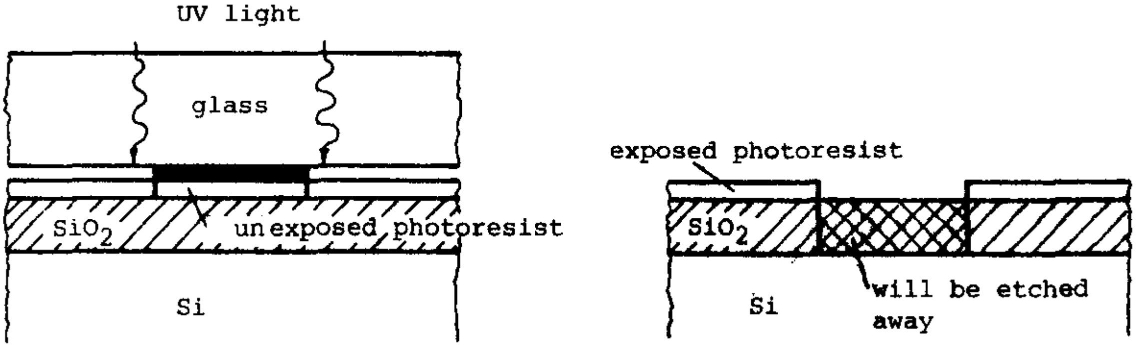

Fig. 2 *Photographic process*

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[*] This is true for so-called negative photoresist. Positive photoresist also exists. Then, the resist layer will remain at the unexposed regions.

[**] See Appendix 1 for more details. A drawing in A2 format (42 x 60 cm) is available on request.

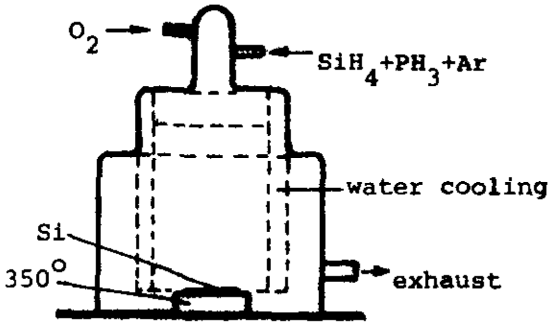

Fig. 3 *Silox reactor*

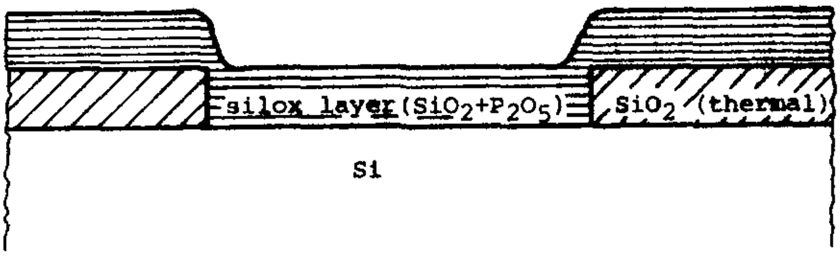

Fig. 4 *Slice covered with thermal oxide and silox layer*

(4) Drive-in diffusion

In the following it is assumed that phosphorus is present in the silox layer. The slice with the silox is heated in the oven at a high temperature (900°-1200°C) [2]. n-type regions are then formed. These n-type regions are slightly larger than the windows in the thermal oxide layer due to the fact that the diffusion also takes place parallel to the plane of the surface.

(5) Fabrication of contacts

After the diffusion, windows are etched in the silox (Fig. 5). Then, by evaporating aluminium, contacts are formed on the slice. Subsequently, the aluminium layer must be removed from places where no conduction should occur (Fig. 6). Again this is done by a photographic process. The remaining aluminium pads are suitable to bonding gold wires.

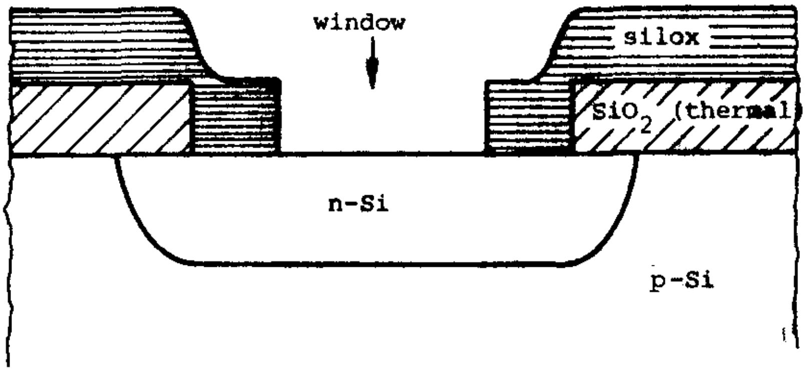

Fig. 5 *Windows in silox layer*

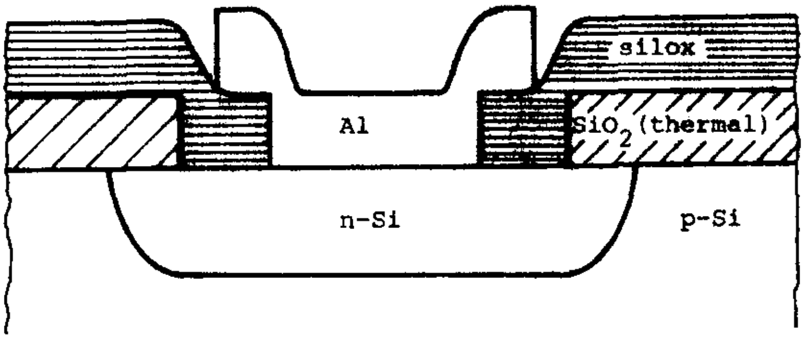

Fig. 6 *Aluminium pattern*

In the next section the process steps leading from slice to diode will be treated successively in more detail.

It should be noted that in the figures the dimensions in the vertical direction are considerably exaggerated to those in the horizontal direction.

4. MORE DETAILED DESCRIPTION OF THE PROCESS STEPS*)

The smooth surface of the silicon slice should be at the upper side.

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*) In Appendix 2 a sketch of the room with the necessary apparatus is given.

For the sake of economy only a quarter of a slice is used.

First step. Cleaning. This is necessary to remove organic and other contaminations from the surface. The slice is successively put into a solution of trichloro-ethylene, into propanol and into deionized water. Then the slice is placed in a boiling mixture of chloride HCl and nitric acid HNO_3 (ratio 3:1) for 10 minutes. It has to be rinsed again afterwards in deionized water. Finally the slice is dried by centrifugation.

Second step. Thermal oxidation. The slice is covered with a layer of SiO_2 3000 Ångströms (0.3 μm) in thickness. This takes two hours in a furnace at a temperature of 1200°C. The flow of oxygen is 1 l/min.

Note: The two steps mentioned above have been carried out beforehand. The next ones should be done by the students.

Third step. Etching of windows. Windows have to be etched in the silicon oxide at prescribed locations. For this purpose only one half of a quarter of a slice is used. At the same time the thermal oxide is removed completely from the other half. The latter is to be used for the measurements of junction depth and sheet resistance (see Section 7).

The etching of the windows comprises a number of separate actions.

3a. Applying photoresist. The slice is put into a spinner. It is maintained in position by underpressure, obtained by means of a vacuum pump. Five drops of negative resist HR 100 (Waycoat brand) are allowed to fall on the slice. Immediately afterwards the slice is rotated at a rate of 6000 revs. per minute. The slice is then covered by a uniform very thin layer of photoresist.

3b. Drying. The slice is now carefully placed on a hot metal plate the temperature of which is 90°C, and dried at this temperature for 5 minutes.

3c. Exposing. The slice is placed in an exposure apparatus. Use of a mask ensures that those areas of the slice where the windows should appear are not exposed in the oxide layer. The exposure time is 10 seconds.

3d. Photo developing. The photoresist is developed in xylene for 1 minute. Then the slice is successively put into propanol and deionized water, for 1 minute in each case. Afterwards, the slice is centrifugated and dried at 130°C for 5 minutes.

Where the photoresist has been polymerized, the silicon oxide is protected against the etching solutions (See 3g).

3e. Covering of the rear with photoresist. Evidently it is also necessary to protect the rear of the slice against the etching solution. Otherwise the oxide would disappear on that side during etching. This must be avoided because the silicon oxide acts as a protecting layer for the silicon.

The slice is put into the spinner upside down. Again, let a number of drops of photoresist fall on the slice. For 20 seconds it is centrifugated at a rotation velocity of 6000 revs. per minute.

3f. Drying. The slice is kept at 90°C for 5 minutes.

3g. Etching.Etching is carried out for about 3 minutes in a silicon-oxide etch (1:6 HF - NH_4F in water) at room temperature*). Then the slice is rinsed in deionized water and dried.

3h. Removal of photoresist. The slice is put into fuming nitric acid. Again it is rinsed in deionized water for 10 minutes and centrifugated.

Now the slice is provided with windows and is ready for the silox process.

Fourth step. Producing the silox layer. The slice is put into a silox reactor (Fig. 3) and kept at a temperature of 350°C for 2 minutes. The following gases are supplied to the reactor: O_2, SiH_4, PH_3 and Ar. The last one acts as carrier gas. The silane and the phosphine are highly rarefied.

A compound of SiO_2 and P_2O_5 is formed on top of the slice in the windows as well as on the thermal oxide.

Operating instructions of silox reactor.

At the beginning of the process the taps are in the positions as indicated in Fig. 7. Turning the power switch automatically opens tap C (see Fig. 7).

The switch clocks are adjusted as follows:

Clock 1	2 min.	(flow of nitrogen).
Clock 2	3 min.	(deposition).
Clock 3	2 min.	(flow of nitrogen).
Total time	7 min.	

Procedure is now as follows:

1. Push button "LIFT": reactor is lifted.
2. The cleaned slice is laid down on the hot plate. Then the button marked "VACUUM" is pressed: The air below the slice is sucked away. This ensures good heat contact between the slice and the hot plate.
3. Press "LIFT": reactor comes down.
4. Press "START": Clock 1 starts. Tap A (see Fig. 7) opens automatically.

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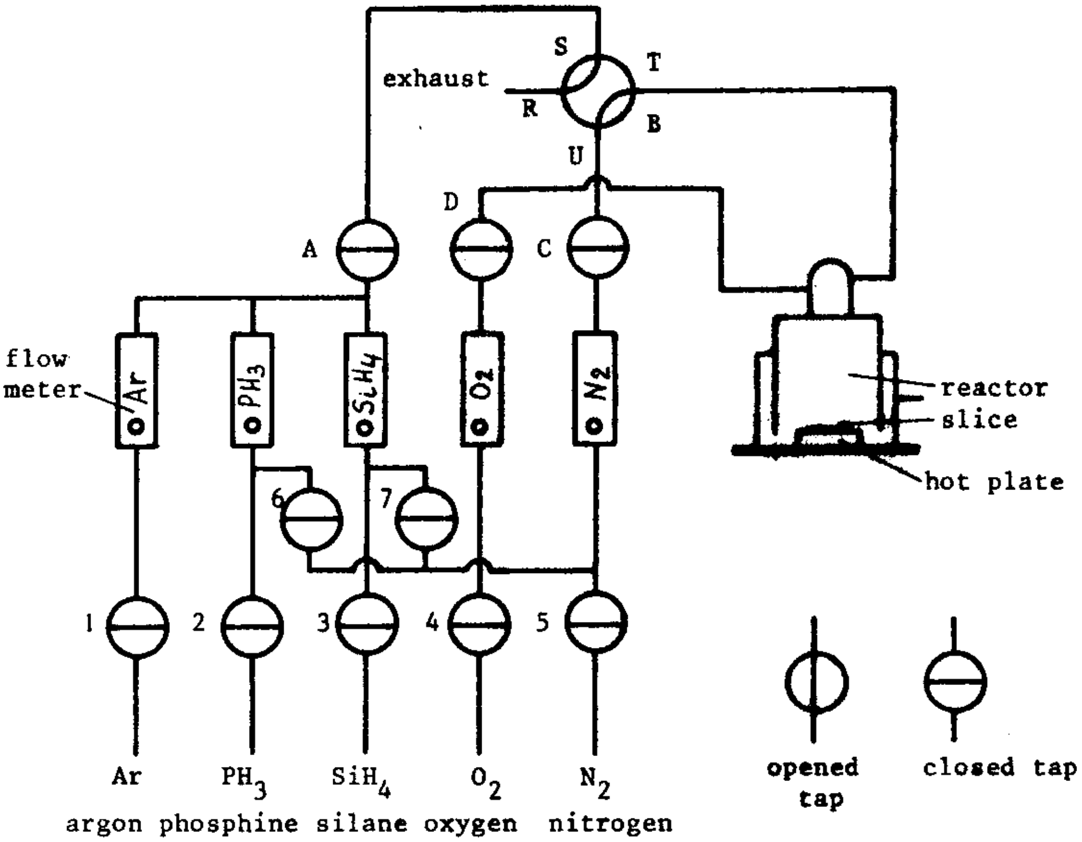
*) Be careful with the dangerous compound HF.



A, B, C, D: automatic taps
1 t/m 7 : normal taps

Fig. 7:*Sketch of silox reactor. The taps are drawn in the positions at the beginning of the process.*

The following taps are opened manually.

Tap 1, argon. Adjust flow of gas to 7 l/min.

Tap 2, phosphine. Adjust flow of gas as required.(For example, 0.3 l/min; 2000 ppm in argon).

Tap 3, silane. Adjust flow of gas to 0.7 l/min. (1% silane in argon).

Tap 5, nitrogen. Adjust flow of gas to 8 l/min.

5. After two minutes clock 2 will start. SiO_2 doped with phosphorus is then formed on top of the slice. Tap C is automatically closed. Tap B is automatically switched: R is connected to U and S to T. Tap D is also opened automatically (adjust the flow of oxygen to 0.1 l/min).
6. Close tap 2 after 2 min. (see clock 2). An additional layer of undoped SiO_2 is then formed.
7. After 3 minutes clock 3 is started. Tap B is automatically switched. Again R is connected to S, and T to U. Tap D is automatically closed. Tap C is automatically opened. Close taps 3 and 4. Open the two taps 6 and 7.
8. After 2 minutes clock 3 stops. Tap A is automatically closed. Close tap 1.
9. Press "LIFT": reactor is lifted.
10. Take the slice out of the reactor.
11. Press "LIFT": reactor comes down.
12. Adjust flow of nitrogen to 2 l/min.
13. Close the taps 6 and 7.

It is possible to interrupt the whole process by pressing :RESET". Nitrogen then flows through the reactor. After 2 minutes the process can be started again.

Fifth step. The SN diffusion (= shallow n-diffusion). The slice is put into a diffusion furnace for drive-in diffusion in an ambient of nitrogen for 15 minutes at a temperature of 1150°C. The nitrogen flow is 1 l/min.

Sixth step. Etching of contact windows. The slice is covered with negative photoresist HR 100 (Waycoat), placed in a photoresist spinner and gyrated at 6000 revs. per minute. Afterwards the slice is kept at 90°C for 5 minutes (steps 3a and b).

Then the slice is put into an exposure apparatus where, before exposure, the mask should be adjusted to the pattern of the windows already present.

After adjusting, the slice is exposed for 10 seconds (step 3c). The developing is done in xylene for 1 minute. Then, the slice is successively put into propanol and deionized water, for 1 minute in both cases. Further, the slice is centrifugated and dried at 130°C for 5 minutes. The windows in the oxide (at the front of the slice) for the contact regions are obtained by etching the slice in SiO_2 etch. At the same time the oxide layer at the rear is removed. Etching time has to be 6 minutes. Then, the slice is rinsed in deionized water (step 3g) and centrifugated. In order to remove the photoresist the slice is placed in fuming nitric acid. Again, one has to rinse in deionized water for 1 minute and centrifugate (step 3h).

Next, the slice is immersed in a HF dilution (4%) for 8 seconds and is rinsed in deionized water for 10 minutes. After centrifugating, the slice is ready for coating with aluminium by evaporation.

Caution: Touching the skin with fluor-hydrogen is dangerous. Should this happen, immediately rinse the skin thoroughly with water.

Seventh step. Applying aluminium by evaporation. A thin layer of aluminium (0.5 - 1 μm) is applied to the front of the slice. This is carried out in an evaporation apparatus.

Eighth step. Etching of the aluminium. The front of the slice is covered with positive photoresist AZ 1350 (Shipley). Positive means here that the photoresist disappears at exposed regions while developing.

To obtain a uniform layer the slice is put into the photoresist spinner, covered with a few drops of resist and gyrated at the rate of 4500 revs. per minute for 20 seconds. Then the slice is dried at 90°C for 5 minutes.

The slice is then placed in the exposure apparatus. The slice is adjusted and then exposed for 10 seconds. After developing for 1 minute in AZ developer, the slice is rinsed in deionized water for 1 minute and centrifugated.

The etching proper takes place in a etch bath for aluminium at a temperature of 40°C. This etch bath contains: phosphoric acid H_3PO_4 (80%), HNO_3 (65%), acetic acid (100%) and water (ratio 15:1:3:3).

Furthermore, the slice has to be rinsed for 1 minute and centrifugated.

Finally the slice is immersed in acetone for 5 minutes to remove the photoresist, after which it is rinsed in deionized water for 10 minutes and centrifugated.

Ninth step. Heating the aluminium. In order to obtain good ohmic contacts the slice should undergo a heat treatment. It is kept at 450°C for 15 minutes in wet nitrogen flowing along the slice at a velocity of 1 l/min.

After all these steps the manufacture of diodes and resistors is completed.

5. DIFFUSION EQUATION AND IMPURITY PROFILE

Consider a silicon slice with a silox layer which contains phosphorus atoms. During the diffusion phosphorus will enter the silicon as an impurity. After the diffusion the phosphorus has a certain impurity distribution in the silicon.

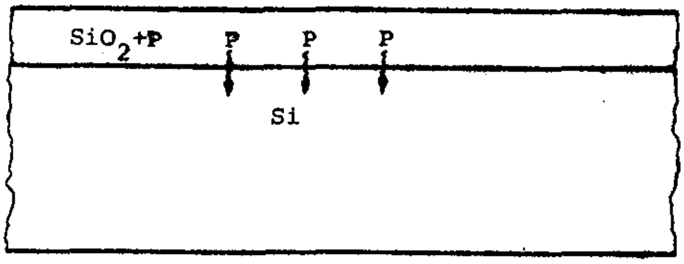


Fig. 8 *Diffusion of phosphorus from an oxide layer*

It is well known [3] that the diffusion is described by the equation

$$D \frac{\partial^2 N}{\partial x^2} = \frac{\partial N}{\partial t} \tag{1}$$

where D is the diffusion constant of the impurity considered, and the function N(x,t) is the concentration of the impurity at point x at time t. We assume that the concentration is constant at the interface between oxide and silicon, so that

$$N(0,t) = N_o$$

The solution of the differential equation (1) that satisfies this boundary condition is

$$N(x,t) = N_o \{1 - \mathrm{erf}(x/2\sqrt{Dt})\}$$

where

$$\mathrm{erf}(z) = (2\sqrt{\pi}) \int_0^z \exp(-\lambda^2)d\lambda.$$

It can be verified immediately by substitution in eqn. (1). Note that N(x,t) tends to zero if x tends to infinity.

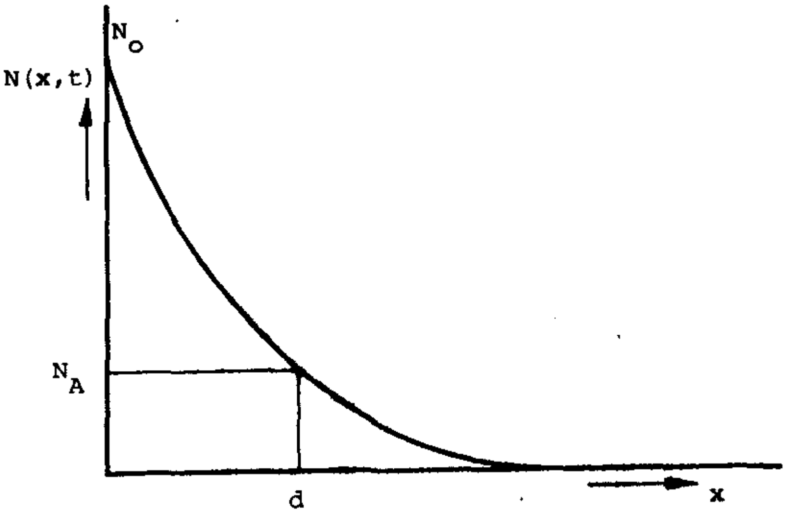


Fig. 9 *Impurity profile in silicon*

In Fig. 9 the impurity concentration is plotted versus the position x. When N_A is the acceptor concentration of the original slice then, after diffusion, the pn junction will be found at a depth d below the surface, which is determined by the equation

$$N(d,t) = N_A$$

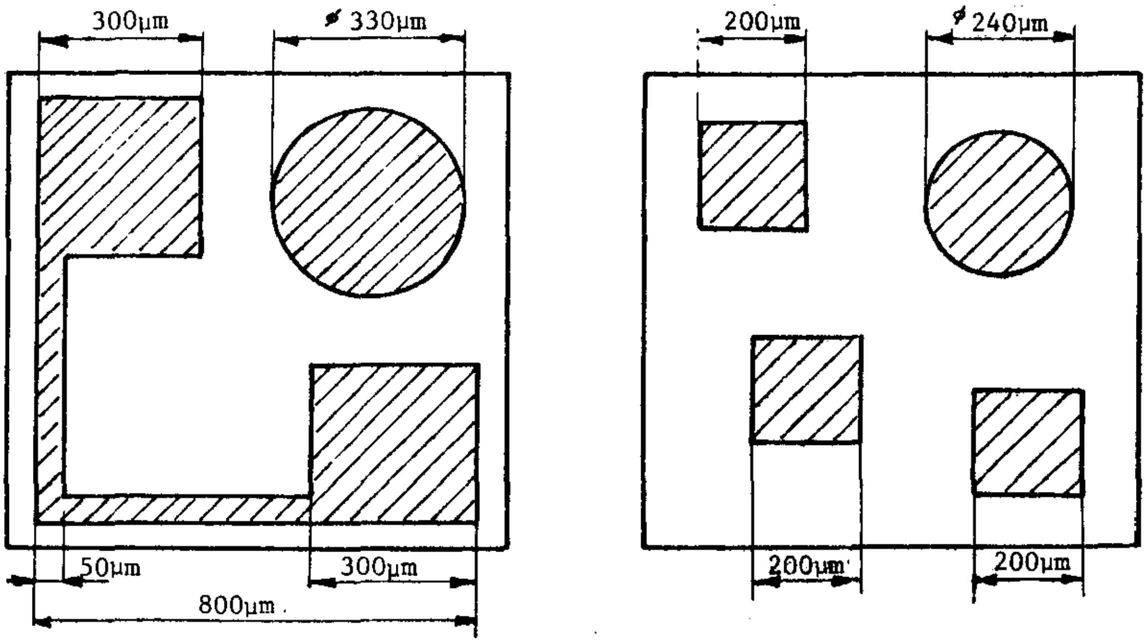
or

$$N_o \{1 - \mathrm{erf}(d/2\sqrt{Dt})\} = N_A$$

At the left of x = d one has n-type silicon, at the right of that plane p-type silicon. From the above theory it follows that, for known diffusion constant D, known diffusion time t and known background concentration N_A, the concentration profile is fixed completely, if N_o and d have been measured. Then, the electrical properties are determined as well.

6. APPLICATION OF MASKS

As has been observed already, the n-regions can be diffused at certain regions, defined by the available masks.



mask no.1

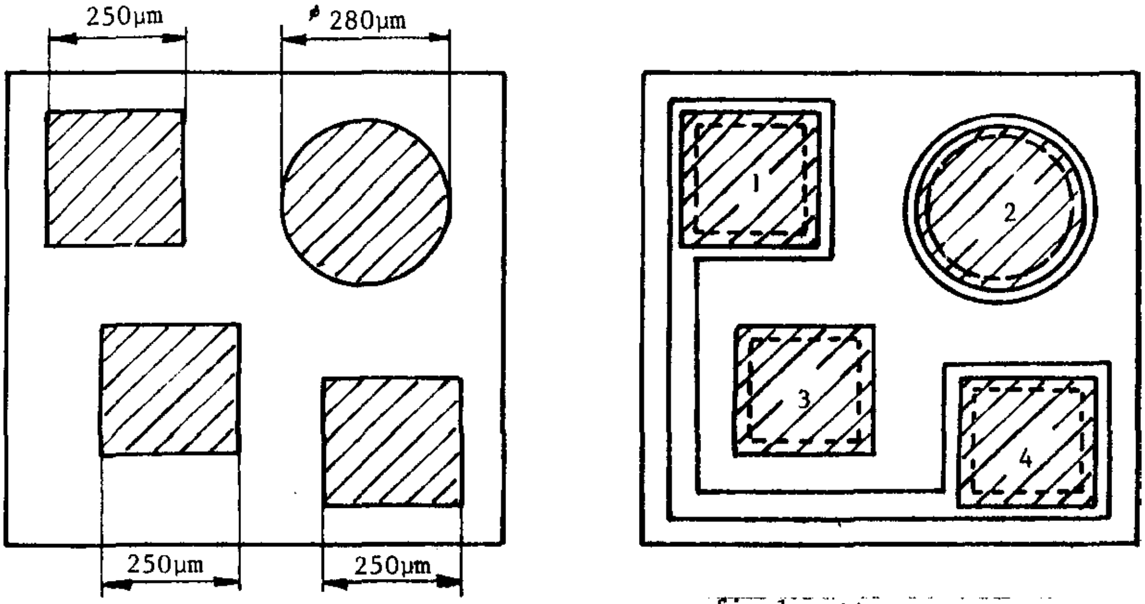
windows for diffusion

a

mask no.2

contact windows

b



mask no. 3
aluminium contact pads

c

final pattern

1-4 resistance
2 diode
3 substrate contact

d

Fig. 10 *Masks used in experiments*

Mask 1 is for making the windows in the oxide for the diffusion (Fig. 10a). By means of mask 2 contact windows can be etched (Fig. 10b). Mask 3 is applied in order to obtain aluminium contact pads (Fig. 10c)*). From Fig. 10d the final pattern can be read. After carrying out all the technological steps, we have a resistance between contacts 1 and 4, and a diode between contact 2 and the substrate. Contact 3 serves to supply a voltage to the substrate.

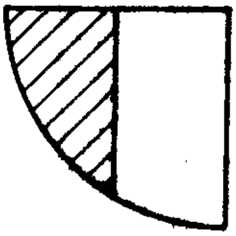


Fig. 11 *The hatched part of the slice is used for measurements of junction depth and sheet resistance. The other part contains the diodes and the resistors.*

The diffused diodes and resistors are located in a part of the slice. The other part is available for measurements of junction depth and sheet resistance (Fig. 11).

7. POSSIBLE MEASUREMENTS ON THE DEVICES

A number of measurements can be carried out by students. In our case the exercises concern

(a) determination of the junction depth of the diffused layer,

(b) measurements of the sheet resistance of the diffused layer,

(c) determination of the impurity concentration N_o at the surface,

(d) measurements on the current-voltage characteristic of the diodes and the influence of temperature on it,

(e) measurement of the diffused resistances and the influence of temperature on it,

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*) A drawing of the alignment equipment is available on request.

(f) measurement of the capacity of a diode and its dependence on voltage,
(g) measurement of built-in potential of a pn junction.

The theoretical background of these experiments is well known [4] and will not be described in this report.

8. DISCUSSIONS AND CONCLUSION.

We have shown that the silox process lends itself to education in the field of semiconductor technology. Until now four generations of students have passed. Students carry out the experiments in groups of two. Four students can participate in the experiments at a time. Assistance is given permanently by two persons of the staff of the university. Thanks to the chosen process the necessary technological steps can be carried out within half a day.

Acknowledgements

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Appendix 1

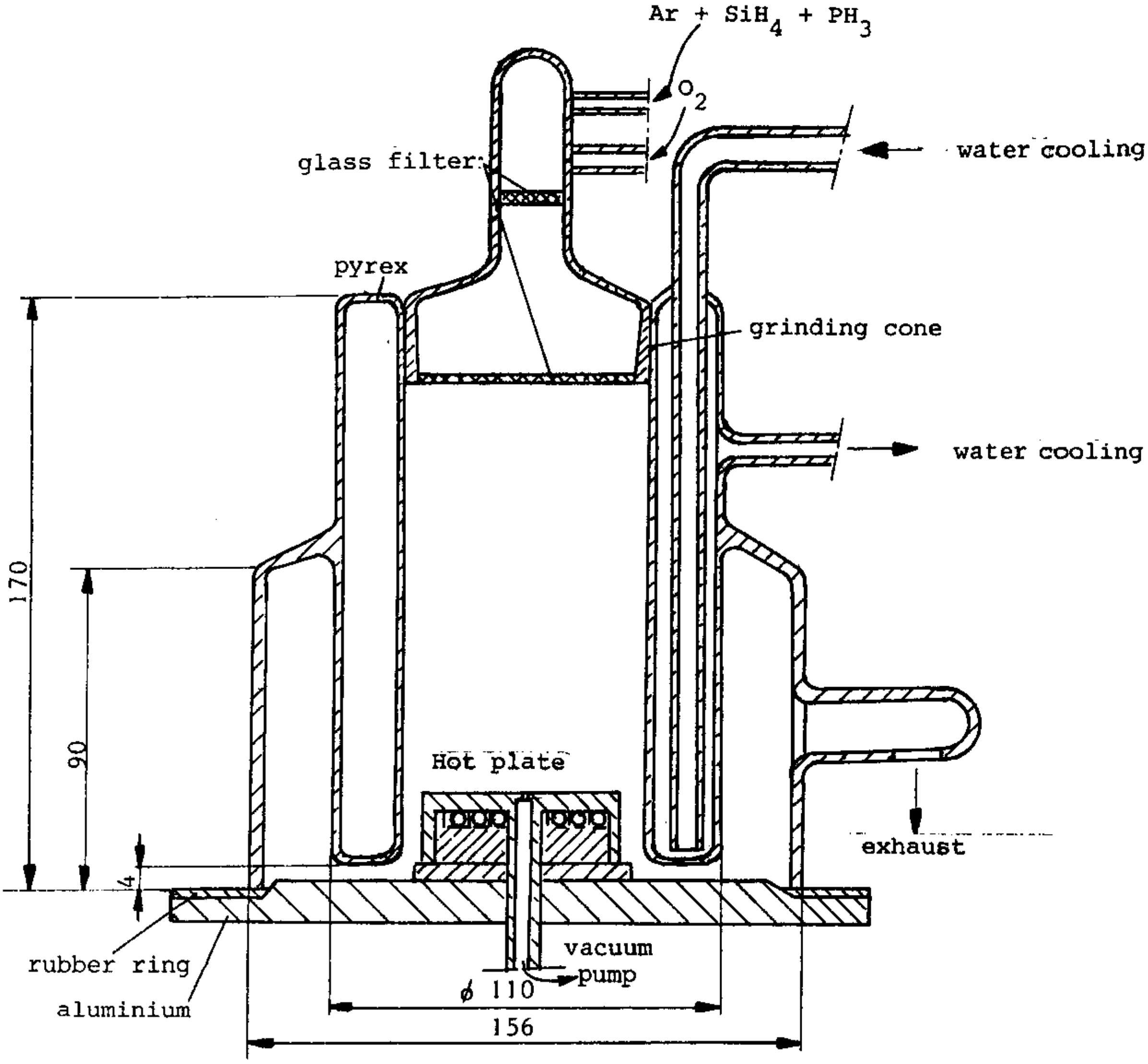

Fig. 12 *More detailed drawing of silox reactor. During experiments this reactor should be enclosed in a transparent cupboard with exhaust.*

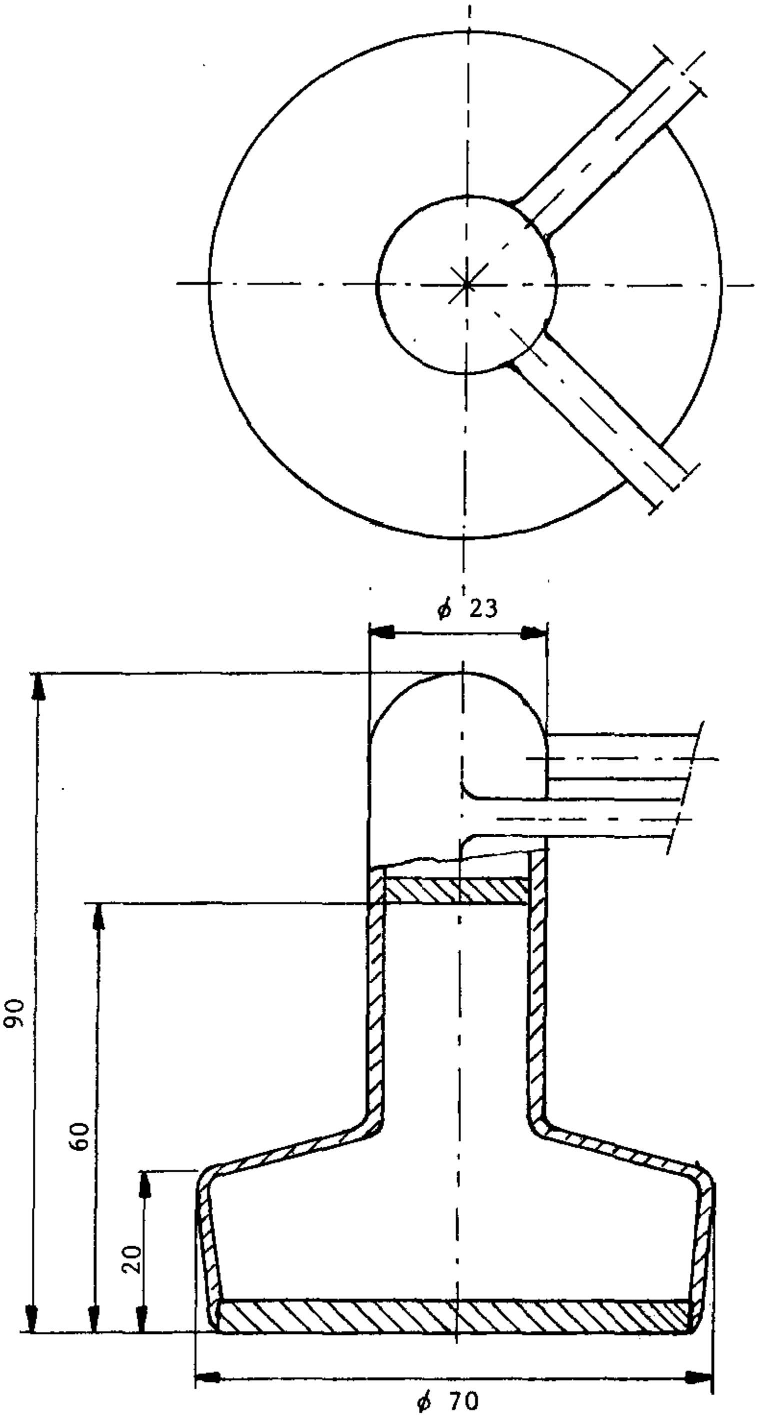

Fig. 13 *Detail of silox reactor (see Fig. 12)*

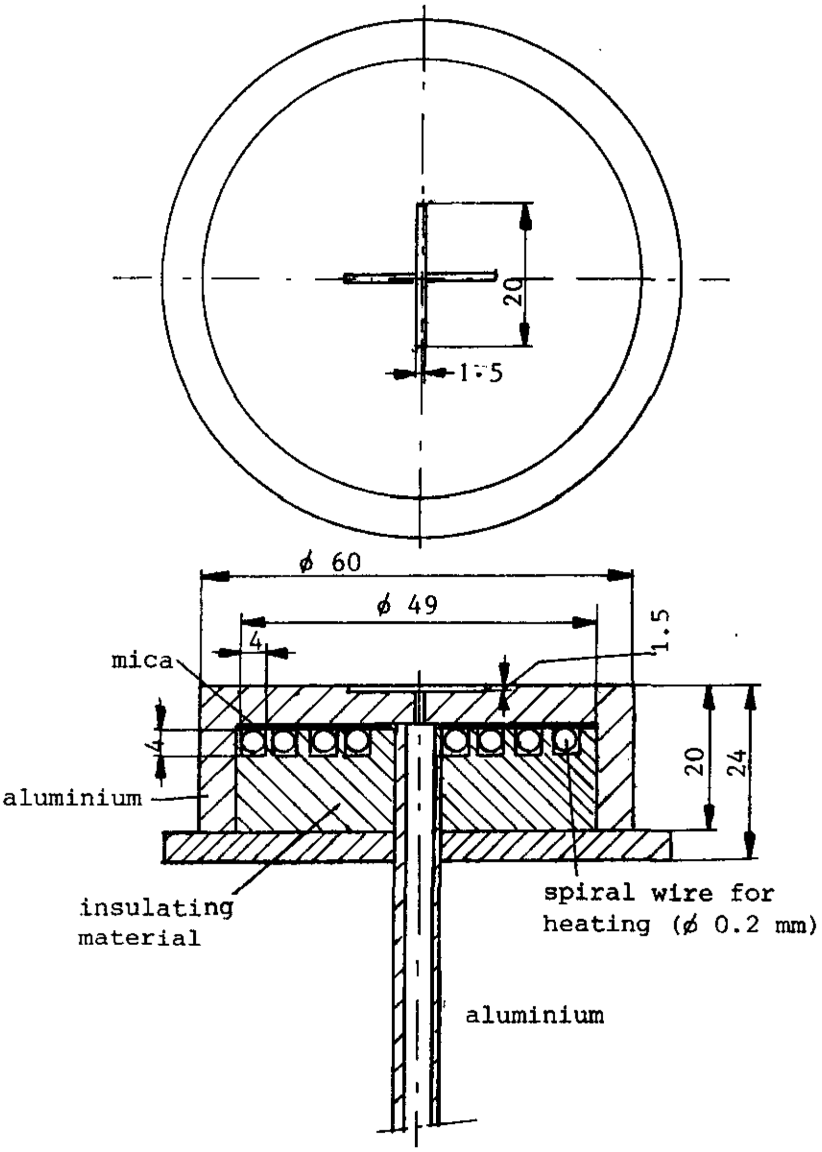

Fig. 14 *Detail of silox reactor (see Fig. 12)*

Appendix 2

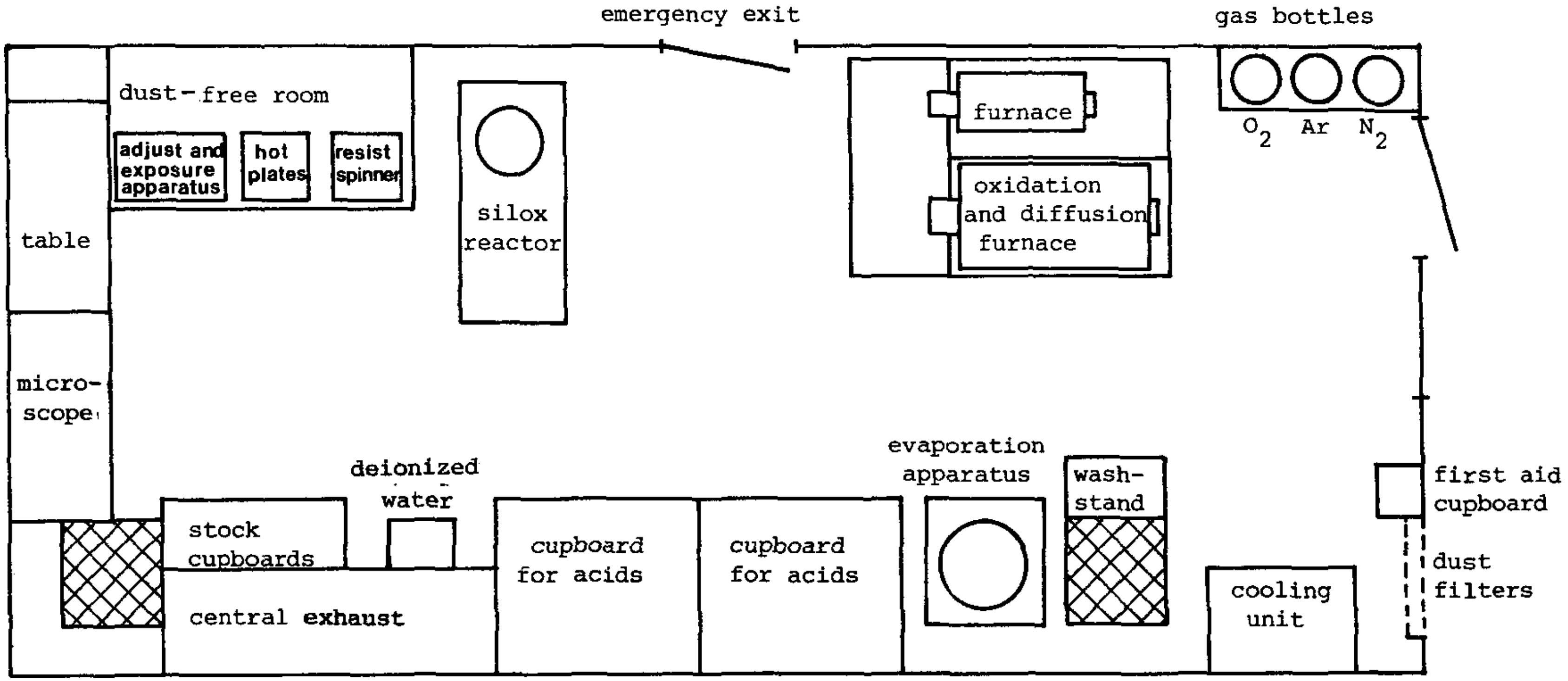

Fig. 15 *Laboratory (3.75 × 8.75 m^2)*

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