

ELECTROLESS NICKEL PLATING ONTO SILICON

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Abstract

Contact metallization and interconnectivity of conductors are very important factors in the continuous advancement of very large silicon integrated technology. With size of devices shrinking to submicron devices, normal metal soldering method has been obsolete at fabrication stage. The need for good electrical contact onto silicon is discussed. A simple but effective method of plating nickel onto silicon is presented and experimental results for its conduction through the junction so formed are shown at different temperatures.

Keywords: *Metallization, soldering, diffusion, electroless, plating solubility, deposition, etchants, mask, mactape.*

1.0 Introduction

Semiconductor devices need leads via which current can flow through them from external connections for their operations. Electrical connections can be made onto germanium by simple soft soldering to give good reliable contacts. Unlike germanium it is found difficult to put electrical leads onto silicon by simple soldering. This is because of the presence of an oxide film on the surface. Normally silicon has great affinity to oxygen. As a result a number of different types of techniques have been devised for use on silicon [1]. Each method has its own merits and disadvantages and the final choice is usually a compromise. Methods as impurity diffusion that are not electroless have been used [2,4].

The methods usually used to put contacts unto silicon include alloying and diffusion. Some of the metals used as contact materials include silver, aluminum and gold; all of which are quite soluble in silicon and alloy deeply into it depending on the temperature and time allowed. This solubility can be a disadvantage as often one would like to control the depth of penetration into the silicon by the metal. Also these alloys are rather brittle and the over all mechanical properties may not be very good. The alloy method also needs high fabricating temperatures. Gold [3,5] has been found to be good for the process as its diffusion length in silicon is short. This paper

discusses the deposition of nickel onto silicon by simple electroless plating.

Electroless nickel plating onto silicon is based on a technique developed by [2] and involves the catalytic production of nickel in a system which is also liberating hydrogen. The process adopted here follows closely to the one reported by Sullivan [5,6].

1.1 Experimental details

1.1.1 Sample Preparations

As is the case in most contacting processes, the surface should be very clean. This can be achieved by chemical etching. There are different etchants that are used to clean silicon and the choice again depends on the required result. A list of etchants for cleaning and polishing silicon free of the oxide layer can be found in *Electrochemistry of semiconductors* by [3]. Whether or not any deposition of any impurities that might be produced during the etching will affect the silicon should be taken into account in choosing the etchant. Newman R.C. and Holms D.T.C. pointed out that etching silicon in hot potassium or sodium hydroxide (KOH or NaOH respectively) before plating (a method originally suggested in [7,8] will lead to deposition of iron crystallites (~100Å⁰ in size), although this impurity which is contained in commercial KOH or NaOH can be removed by a membrane.

The procedure used in the present work was as follows:- the silicon was first polished with grade '180' carborundum paper. This roughed the surface and then smooth polishing was carried out using grade 3/0 emery polishing paper. The sample was then degreased with acetone and was thus ready for etching. The etching solution used was a mixture of 40% by volume of 40% concentrated hydrofluoric acid, HF and 60% by volume of nitric acid, HNO_3 . The etching time was one minute, other etching concentrations were however tried before the above solution was found best to not only remove the oxide but also clean the surface well. It was also found that should the carborundum paper not be available, the emery paper and etching solution would be enough to give the silicon good clean oxide free surface within one minute. After etching the specimen was thoroughly rinsed in isopropyl-alcohol. This avoids use of water which would produce an oxide.

2.0 Plating Bath

Different plating baths could be used for silicon (Brenner, 1941). The solution used here has composition as follows:

Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) 30g/L

Sodium hypophosphite ($\text{NaH}_2\text{P}_2\text{O}_7 \cdot \text{H}_2\text{O}$) 30g/L

Ammonium citrate ($(\text{NH}_4)_2\text{HC}_6\text{H}_5\text{O}_7$) 65g/L

Ammonium chloride (NH_4Cl) 50g/L

Where the symbol g/L indicates the weight in grams per litre of water to make up the solution. Distilled water was used.

The solution which was filtered could be ignored if one feels that it was clear enough and no foreign materials were present. The colour of the solution was green. After filtering sufficient ammonium hydroxide (NH_4OH) was added to the solution until it turned from green to blue giving a pH value of 9 or 10. The base solution and the additions depend on the needed volume of the mixture. This addition was made in the cupboard as ammonia in air is dangerous. Prepared solutions can be stored in bottles and can last for months.

2.1 Plating

The solution was heated in a beaker using a small heat and when it was just boiling ($\sim 97^\circ\text{C}$) the silicon was put into it. Care was taken to make sure that the transfer of silicon from etchant to plating baths did not take long as oxide forms on a clean silicon surface within a few tens of seconds. In the present work, it took approximately twenty seconds to do so. As the silicon was introduced into the plating solution, rapid evolution of hydrogen was observed and the plating could immediately be seen to occur. During the plating, the solution becomes depleted of nickel, hypophosphite and hydroxyl ions. The colour of the solution is seen to turn from blue to bluish green, this is reversed by addition of more ammonium hydroxide. Thus as the process progressed, there was the need to keep adding ammonium hydroxide into the plating solution.

2.2 Contacting

The plating took a few minutes. To use the plated nickel as a contact surface onto silicon, the nickel had to be diffused into the silicon. This was done using a vacuum chamber. The diffusion temperature which was monitored using a chromel/Alumel thermocouple was 400°C and the diffusion time was about one minute. The heating element was made from a strip of niobius. For mechanical strength and ease of subsequent soldering a second thinner plating of nickel was applied.

2.3 Results

After the second plating, contacts were put on by soldering copper leads into the nickel surface with soft solder. The contacts were found to be mechanically strong. This was observed from the fact that when the contact was broken, the leads broke off not from the solder but by breaking off the edge of the silicon. The characteristics are shown in fig. 1.

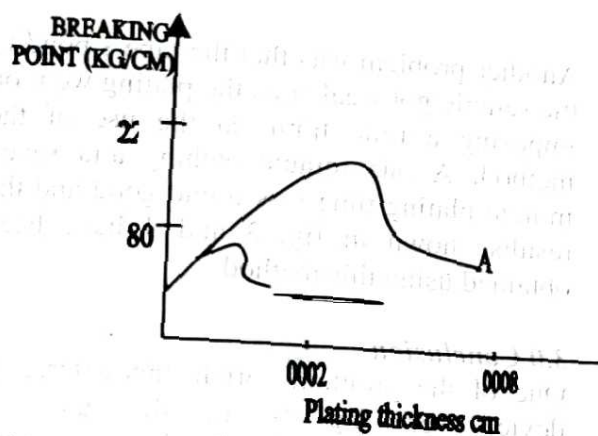


Fig. 1: Adhesion strength of Nickel into Silicon

After the leads have been connected, the conducting properties of the contact were checked by measuring its current-Voltage characteristics. The I:V plots were made at liquid helium temperature as well as room

and liquid nitrogen temperatures. At room and liquid nitrogen temperature, the current-voltage characteristics showed a linear or ohmic relationship as can be seen from the plots (a) and (b) of fig.2. Non ohmic behaviour was noticed at liquid helium temperature and plots of two contacts of different initial plating times are shown in fig. 3 and 4. The linearity of the contacts would be good enough even at liquid helium temperatures for most device operations.

One feature that was observed during plating was that it was not uniform. This was estimated from the evolution of hydrogen during plating. The rate of plating was initially high and reduced with time. The rate depended also on temperature. The hotter the plating bath the faster the rate of plating at least up to the boiling point of the solution.

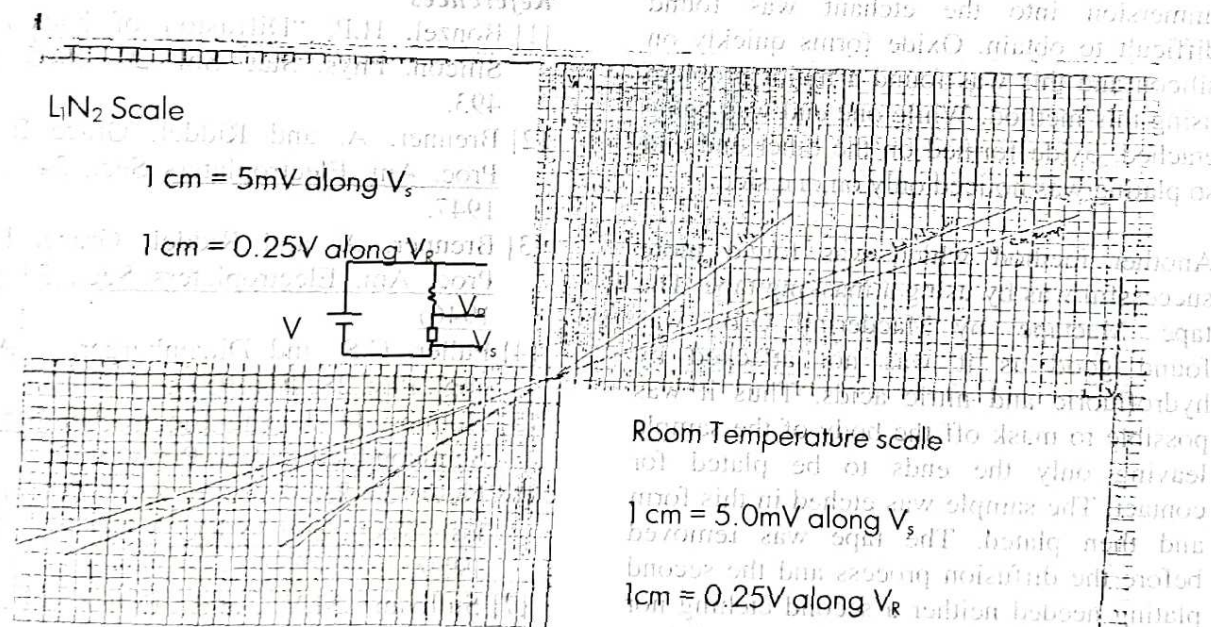


Fig. 2: Current Voltage Characteristics of the Contact

To be able to plate on only the ends to put the electrical leads, it is necessary to mask off the other parts of the silicon. It was found difficult to do this mainly because of the need for an oxide free surface.

Different methods as plating the samples and then stripping off the nickel at the unwanted

parts using fuming nitric acids was tried. Nitric acid was used as it attacks nickel but not silicon. This was however unsuccessful due to its unevenness. Two main methods were found fairly reliable. First, only the ends or parts to be plated were etched and then plated.

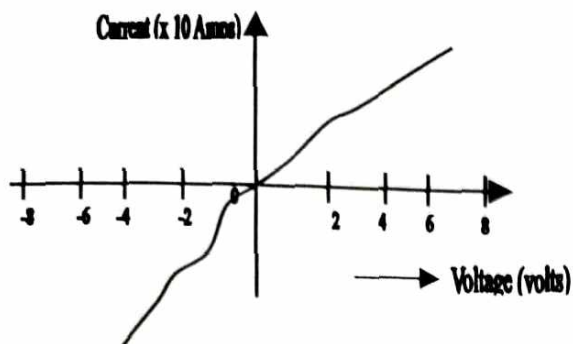


Fig. 3: Current-Voltage characteristics of contact (Plating time is 7mins)

The etching was done by dipping the ends of the sample to be plated into a solution of the etchant one after the other.

This would make the area to be plated small but not easily controlled as a uniform immersion into the etchant was found difficult to obtain. Oxide forms quickly on silicon and this was found a major problem using this method. While one end was being etched, oxide formed on the other end and so plating was noticed only on one side.

Another method which was found more successful was by using a masking tape. The tape 'Mactape' by Macdermil G.B., was found good as it was not attacked by hydrofluoric and nitric acids. Thus it was possible to mask off the body of the sample leaving only the ends to be plated for contact. The sample was etched in this form and then plated. The tape was removed before the diffusion process and the second plating needed neither a second etching nor the masking. Although the above conditions were met, it was found that the nickel plated over-lapped to the sides of the sample by about 0.5mm; and this overlap was not very uniform as illustrated below in fig. 5.

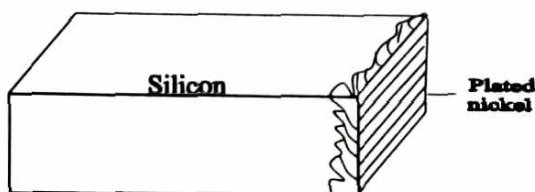


Fig. 5: Nickel Plated Onto Silicon Showing Possible Overlap

Another problem was that the tape's bond to the sample got weaker as the plating went on imposing a time limit on the use of the method. A one minute etching and seven minute plating time was found good and the results shown in fig. 3 and 4 have been obtained using this method.

3.0 Conclusion

One of the problems often encountered in device servicing is in the area of interconnectivities. Usually lead soldering has always been the technique but the difficulty of cold junctions is common. From the discussions an inexpensive and easy method that would enable electrical interconnections to be made has been presented. By this technique, interconnections would be very precise.

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