SUBSTRATE AND SURFACE MICROMACHINED NICKEL STRUCTURES BY ELECTROLESS PLATING PROCESS

by

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"Substrate and Surface Micromachined Nickel Structures by Electroless Plating Process"

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ABSTRACT

Development of effective microelectromechanical systems (MEMS) requires integration of silicon micromechanical structures with suitable on-chip electronics for the appropriate actuation and detection mechanism. One of the limiting factor for the development of miniature MEMS is the incompatibility between silicon micromachining processes and Integrated circuit (IC) fabrication technology.

In this thesis, a novel, low-temperature process of electroless plating is proposed as a candidate technology for producing micromechanical structures for MEMS applications. Electroless plating is characterized by selective reduction of metal ions at the surface of a catalytic substrate surface. Continuous deposition is achieved through the catalytic action of the metal deposit itself via autocatalysis. One of the main advantage of electroless plating is that it does not require connection to an electrical source for the plating process. This feature and the low temperature process makes the technology very attractive and compatible with standard commercial CMOS process.

Electroless plating process has been studied and characterized for the fabrication of nickel micromechanical structures. Two types of micromachining processes, namely, substrate and surface micromachining processes have been demonstrated. The electroless plating process, characterization results and the fabricated micromechanical structures have been presented in this thesis.

To My Friends

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CHAPTER 1

Introduction

Since we have to deal with the analogue world we live in, there are various requirement for different kinds of sensors and transducers. The unprecedented growth of integrated-circuit technology and computing techniques has made sophisticated data processing accurate, economical, and widely available[1-2]. Today's electronic systems are capable of dealing with large numbers of physical input and output variables, but the sensors and transducers that provide the interface between the electrical and physical world are in many cases outmoded and dependent on hybrid-fabrication techniques. Recent developments in electronics technology and the availability of cheap microprocessors have led to an increased interest in electronic sensors and transducers, particularly the so-called microelectronic sensors, suitable for interfacing to microcomputer systems [3-4]. Many of the materials and processing used to produce integrated microcircuits can be employed in new ways to produce microsensors and

microactuators. These structures provide a means to produce new electronic systems.

1.1 Electromechanical Sensors Based on Micromachining Technology

During the past decade, there has been an expansion of interest in microelectromechanical systems (MEMS)[5]. Rooted in mature semiconductor process technology, micromachining is emerging as a new challenging area, which attracts more and more scientists and researchers[6-9].

As the key factor of the success of integrated circuits for the past forty years, microelectronics has a lot of mature miniaturized fabrication techniques. Apart from drastically reduced physical dimensions and low power dissipation, microelectronics technology has made batch fabrication of sensors possible. Micromachining technology utilizes mature fabrication techniques such as oxidation, diffusion, evaporation, chemical vapor deposition (CVD), epitaxy and etching in combination with silicon isotropic and anisotropic etching to produce precisely sculpted three-dimensional structures for transducer applications[10-17].

Taking the advantage of microelectronics, miniaturization of sensors to micron size become possible. It is expected that these kinds of solid-state sensor, which can sense much weaker signals in a much smaller area, will replace older designs in the future because of their small size, light weight, high performance, low cost and

high reliability[18][20]. Owing to the availability of batch-fabrication technology of sophisticated circuits technology, smart sensor systems which integrate transducers and signal processing circuitry in a single substrate (chip) can now be batch-fabricated. Because of these advantages, microelectronic processing is becoming a main technique for the fabrication of various electromechanical sensors and transducers.

Although electromechanical sensing parts and circuitry processing parts can be integrated, by utilizing mature microelectronic processing and micromachining techniques, sensor design still requires much more attention because the signal from the sensor is usually extremely weak and sensor fabrication may require special processing sequences to create three-dimentional structures which may conflict with standard IC(integrated circuits) bath processing which is fundamentally two-dimensional planar processing. To create integrated sensor systems, it is still necessary to develop sensor processing techniques which are compatible with conventional CMOS fabrication.

1.2 Nickel and Electroless Plating

Nickel is an attractive material for micromechanical structures fabrication due to the wide variation of the properties of its alloys such as hardness, melting temperature, magnetic permeability and reflectance. Nickel deposition, in combination with silicon micromachining, is capable of producing various micromechanical structures. Traditionally nickel has been deposited using sputtering or electroplating techniques.

In recent years the interest in producing smart sensor systems has increased and microstructure fabrication technologies that are compatible with standard IC fabrication processes are being seriously investigated. One such technique is CMOS-compatible micromachining technology[14]. Since CMOS processed wafers/chips cannot be post processed at high temperature, and very complicated metal deposition and pattering processes can reduce the yield and reliability of the final intended products, it is highly desirable to develop a simple low-temperature and highly reproducible post-processing technique.

In this thesis, electroless nickel plating has been utilized as a new process for micromachining. Its process characteristics, such as simple operation, low operation temperature, no electrical sources required, compatible with CMOS process, make it an attractive technology for micromachining.

1.3 Organization of Thesis

Chapter 2 describes the electroless nickel plating process. Chapter 3 and 4 detail the fabrication of micromechanical structures using substrate and surface micromachining processes. Chapter 5 gives some concluding remarks with an outlook on the future of this novel technology.

CHAPTER 2

Electroless Nickel Plating

2.1 Fundamentals

2.1.1 Electroless Plating

The term electroless plating was originally adopted by Brenner and Riddell to describe a method of plating metallic substrates with nickel or cobalt alloys without the use of an external source of electric current[21]. Over the years of development, the term has been subsequently broadened to encompass any process that continuously deposits metal from an aqueous medium without connection to an electric source.

The chemical deposition of a metal from an aqueous solution of a salt of said metal has an electrochemical mechanism, both oxidation and reduction, reactions involving

the transfer of electrons between reacting chemical species. The oxidation of a substance is characterized by the loss of electrons, while reduction is distinguished by a gain of electrons. Further, oxidation describes an anodic process, whereas reduction indicates a cathodic action. The simplest form of chemical plating is the so-called metal displacement reaction. For example, when zinc metal is immersed in a copper sulfate solution, the zinc metal atoms (less noble) dissolve and are spontaneously replaced by copper atoms from the solution. The two reactions can be represented as follows:

Oxidation:	$Zn^0 - - > Zn^{+2} + 2e^-$,	anodic,	$E^0 = 0.76mV$
Reduction:	$Cu^{+2} + 2e^- - - > Cu^0$,	cathodic,	$E^0 = 0.34mV$
Overall reaction:	$Zn^0 + Cu^{+2} - - > Zn^{+2} + Cu^0$,		$E^0 = 1.1mV$

where Zn and Cu represent Zinc and Copper respectively, E^o represents the electrochemical potential of the reaction

As soon as the displacement reaction begins, the surface of the zinc substrate becomes a mosaic of anodic (zinc) and cathodic (copper) sites. The displacement process continues until almost the entire substrate is covered with copper. At this point, oxidation (dissolution) of the zinc anode virtually stops and copper deposition ceases. Chemical plating by displacement yields deposits limited to only the surface. In order to continuously build thick deposits by chemical means without consuming the substrate, it is essential that a sustainable oxidation reaction be employed as an

alternative to the dissolution of the substrate. The deposition reaction must occur initially and exclusively on the substrate and subsequently continue to deposit on the initial deposit. The redox potential for this chemical process is usually more positive than that for a metal being deposited by immersion. The chemical deposition of nickel metal by hypophosphate meets both the oxidation and redox potential criteria without changing the mass of the substrate.

Reduction:	$Ni^{+2} + 2e^- - - > Ni^0$	$E^0 = -25mV$
Oxidation:	$H_2PO_2^- + H_2O - - > H_2PO_3^- + 2H^+ + 2e^-$	$E^0 = +50mV$
Overall:	$Ni^{+2} + H_2PO_2^- + H_2O - - > Ni^0 + H_2PO_3^- + 2H^+$	$E^0 = +25mV$

which is the summation of the oxidation and reduction equations. But this reaction representation is just a symbolic description. It does not represent the true electroless plating reaction, since electroless nickel deposition is accompanied by hydrogen evolution and not only pure nickel deposited from the solution. Fig 2.1 shows the difference between immersion and electroless deposition by comparing deposit thickness vs. deposit time.

In general, electroless plating is characterized by the selective reduction of metal ions only at the surface of a catalytic substrate immersed into an aqueous solution of said metal ions, with continued deposition on the substrate through the catalytic action of the deposit itself. Since the deposit catalyzes the reduction reaction, the term autocatalytic is also used to describe the plating process.

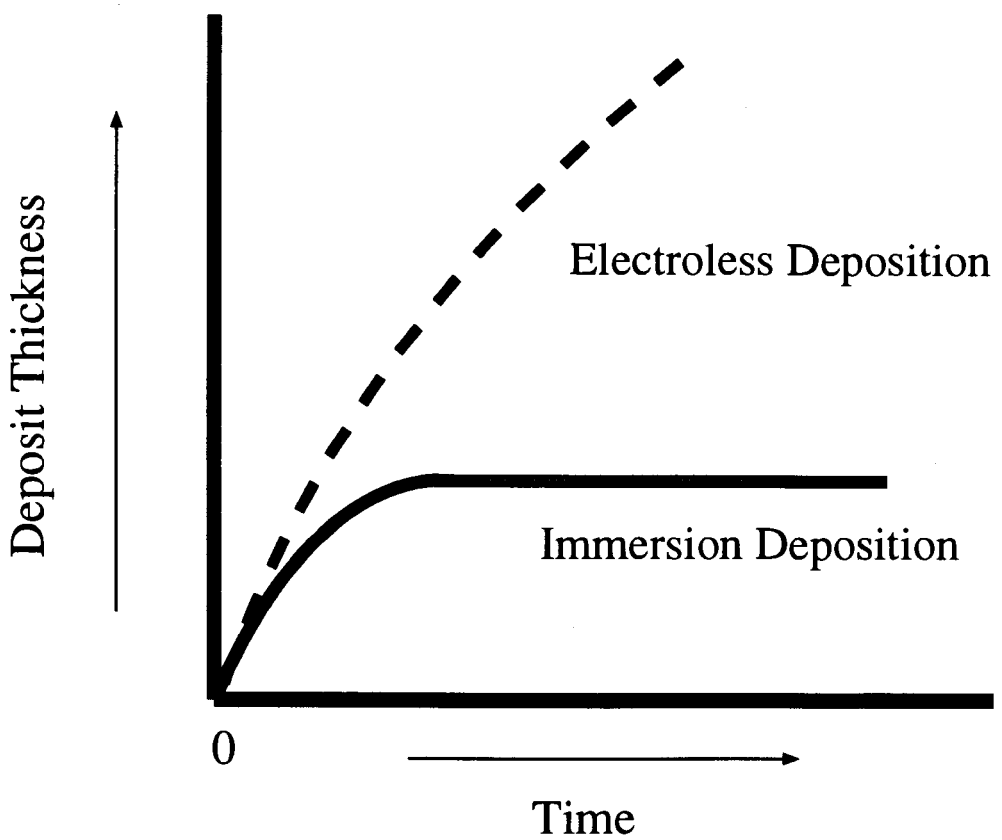

Figure 2.1: Comparison between electroless and immersion deposition

2.1.2 Electroless Nickel Plating

In 1844, Wurtz observed that nickel cations were reduced by hypophosphate anions[21]. However, Wurtz only obtained a black metal powder. The first bright metallic deposits of nickel-phosphorus alloys were obtained in 1911 by Breteau[21]. In 1916, Roux was issued the first patent on an electroless nickel plating bath[21]. However, these baths decomposed spontaneously and formed deposits on any surface that was in contact with the solution. Over the years of research and development by many researchers, this process has been developed as a mature technique being widely used in surface finish and printed circuit board fabrication.

As one of the most important catalytic plating processes in use today, the chemical and physical properties of an electroless nickel coating depends on its composition, which, in turn, depends on the formulation and operating conditions of the electroless nickel plating bath. Typically, the constituents of an electroless nickel solution are:

- A source of nickel ions
- A reducing agent
- Suitable complexing agents
- Stablizers/inhibitors
- Energy

2.1.2.1 Nickel Source

Usually, the plating source of nickel cations is nickel sulfate. Other nickel salts, such as nickel chloride and nickel acetate, are used for very limited applications. The nickel chloride can act deleteriously when electroless nickel plating is used to plate aluminum. The ideal source of nickel ions is the nickel salt of hypophosphorus acid, $Ni(H_2PO_2)_2$. The use of nickel hypophosphate eliminates the addition of sulfate anions and keeps to a minimum buildup of alkali metal ions while replenishing the reactants consumed during metal deposition.

2.1.2.2 Reducing Agents

There are four reducing agents that are used in the plating of nickel from aqueous solutions:

Sodium Hypophosphate	$NaH_2PO_2 - H_2O$
Sodium Borohydride	$NaBH_4$
Hydrazine	$N_2H_4 - H_2O$
Dimethylamine borane(DMAB)	$(CH_3)_2NHBH_3$

All the four reducing agents are structurally similar in that each contains two or more reactive hydrogens, and nickel reduction is said to result from the catalytic dehydrogenation of the reducing agent.

An explicit understanding of the reaction mechanisms that govern electroless nickel deposition is necessary from both theoretical and engineering viewpoints. Knowledge of the mechanisms of the reaction of a reducing agent can lead to the solution of a series of problems - methods to control nickel plating rate, the deposited nickel properties, the content of the deposit, especially as it relates to the reduction of phosphorus and boron, is extremely important. It is the inclusion of phosphorus and boron in the respective nickel alloys ($Ni-P$ and $Ni-B$) that determines the properties of each alloy.

2.1.2.3 Catalytic Surface

Each of the heterogeneous reaction mechanisms outlined below requires a catalytic surface on which the reaction sequence will proceed. Hence, electroless nickel plating occurs only on specific surfaces. The reduction reaction begins spontaneously on certain metals - almost all metals of Group VIII of the periodic table(Fe, Co, Ni, Rh, Pd, Pt) in active form. The active metals nickel, cobalt, palladium, and rhodium are considered to be catalytically active. Metals that are more electropositive than nickel, such as iron, will first displace nickel from solution of its ions as follows:

$$Fe + Ni^{+2} - - > Fe^{+2} + Ni^{0}_{cat}$$

forming the Ni^{0}_{cat} catalytic surface.

If the substrate is more electronegative than nickel, it can be made catalytic by

electrically depositing a thin nickel deposit on its surface or using activators or catalysts. Usually it is a solution that contains palladium salt. Electroless nickel plating has been used in surface finish on various materials such as plastics and polyamide via this method.

2.1.2.4 Stabilizers

An electroless nickel plating solution can be operated under normal operating conditions over extended periods; however, it may decompose spontaneously at any time. Bath decomposition is usually followed by an increase in the volume of hydrogen gas evolved and the appearance of a finely-divided black precipitate throughout the bulk of the solution. This precipitate consists of nickel particles, and either nickel phosphide or nickel boride, depending on the reducing agent used.

Fortunately, chemical agents called stablizers are available to prevent the homogeneous reaction that triggers the subsequent random decomposition of the entire plating bath. Research work shows that plating bath decomposition can be virtually eliminated by the addition of only trace amounts of stablizers, sometimes referred to as catalytic inhibitors to the plating bath[21]. Most of these so-called anti-catalysts (stablizers/inhibitors) are identical with the materials that prevent hydrogenation/dehydrogenation catalysts. The stabilizer concentration in the plating bath is very critical. The concentration of the stablizer depends most importantly on its structural class. Figure 2.2 show the relationship between deposition rate and

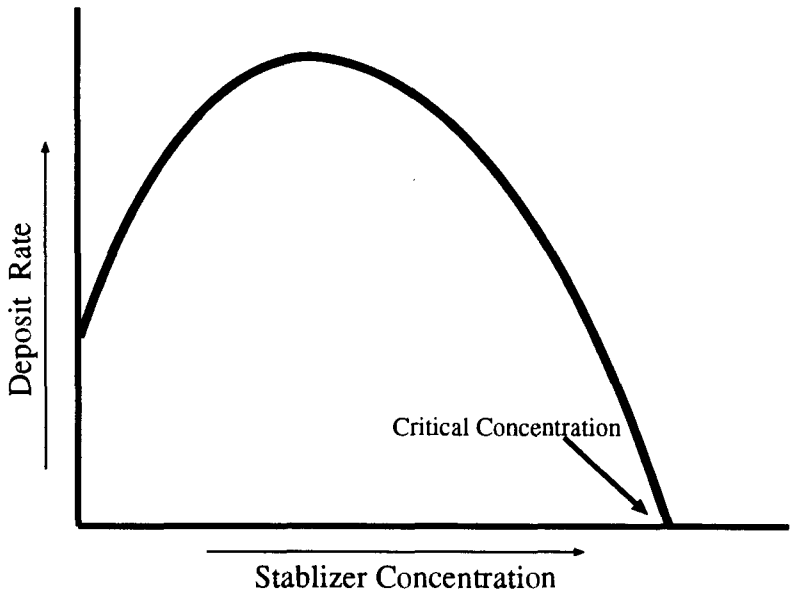

Figure 2.2: Effect of stabilizer concentration on electroless nickel deposition rate

stabilizer concentration.

2.1.2.5 Energy

Catalytic reactions, such as electroless nickel plating, require energy in order to proceed. The energy is supplied in the form of heat. Temperature as a measure of the energy (heat) content of the plating bath, is considered as an important bath variable. The quantity of energy required by the system or added to it is one of the most important factors affecting the kinetics and rate of the deposition reaction.

When operating an electroless nickel plating bath, it is necessary to know which

conditions yield deposits with the desired properties. The deposits should be produced with minimum cost and optimum efficiency. In some cases, it is more controllable to operate the plating bath at a temperature that does not give the maximum plating rate. The composition of Ni-P deposits can be altered slightly by merely raising(decrease P) or lowering (increase P) the temperature of hypophosphate reduced nickel plating solutions. The amine borane reducing agents are more temperature sensitive and will hydrolyze excessively at high temperatures, causing wasteful side reactions [23].

2.2 The Plating Process

The quality of the deposition is affected by many variables, such as temperature, pH balance, solution chemistry, etc. A good plating film is defined as a deposition with uniform thickness, smooth surface with uniform colour, good adhesion with substrate, and no porosity. Generally the colour of the plating is grey with different scale from dark to light depending on the deposit composition.

To get a good nickel film deposited from electroless baths, one has to optimize the variables which affect the depositing rate and composites. The deposition rate should be maintained low enough to make the whole deposition process controllable. Other than slowing down the deposition rate, concentration of hypophosphate ions and pH values should also be controlled if good material properties are expected.

2.2.1 Plating with Alkaline Hypophosphate

The rate of deposition in alkaline electroless nickel solutions is proportional to the hypophosphate concentration; the plating rate increases with increasing reductant concentration. Although raising the hypophosphate concentration improves the plating rate, the stability of the plating is lowered, necessitating the use of stabilizers.

Similarly, plating rate increases significantly with increasing nickel ion concentration in the hypophosphate solution[22]. It is obvious that both nickel and hypophosphate ion concentration have a pronounced effect upon the plating rate.

The rate of the deposition has an exponential relationship with temperature, and this is independent of the pH of the bath[23]. Generally, the plating rate is higher and initial temperature is lower for hypophosphate bath than those for acid plating bath.

The pH of an alkaline solution decreases as the plating reaction proceeds. So it is important to maintain the pH value in a certain range. Mostly ammonium hydroride is used to maintain the alkalinity of the solution. Fortunately, the plating rate is almost constant over a range of pH from 8.5 to 11.0. But there is a lower limit for pH. It is about 8.5[23].

It is found that the phosphorus and other components incorporated in the nickel deposit can be controlled by concentration of reducing agents as well as the pH of the plating solution[22-24]. The amount of impurities and phosphorus in electroless

plated nickel alloy are smaller in the range pH 9.5 - 11 than in the range pH 8.5 - 9.0. The amount of impurities was reduced by lowering the concentration of reducing agents. It is seen that for good nickel film lower NaH_2PO_2 concentration (lower than $20g/L$) and higher pH value (higher than 9.5) is necessary[25].

2.2.2 Plating With Amine Boranes

In general, the techniques of electroless nickel plating with hypophosphate as the reducing medium are applicable to plating with amine boranes. The substitute of water-soluable dimethylamine borane (DMAB) in a standard hypophosphate electroless nickel plating formulation will also yield a workable plating bath. The commercial electroless nickel plating solution Shipley 468 Niposit uses DMAB as reducing agent. Unlike the nickel film plated via alkaline hypophosphate bath, which is non-magnetic because of NiP_3 in Ni film, the nickel film plated via amine boranes solution is ferro magnetic which is useful in the fabrication of magnetically active microstructures.

The concentration of the reducing medium is an important rate-determining factor[21]. The rate of deposition increases linearly with increasing DMAB concentration until the concentration is about $0.06M$; above this point, increasing the DMAB concentration produces only small increases in deposition rate. DMAB-reduced nickel plating solutions show the same exponential dependence of plating rate on temperature as that of hypophosphate reduced nickel plating bath.

There is an exponential relationship of the deposition rate vs. temperature which

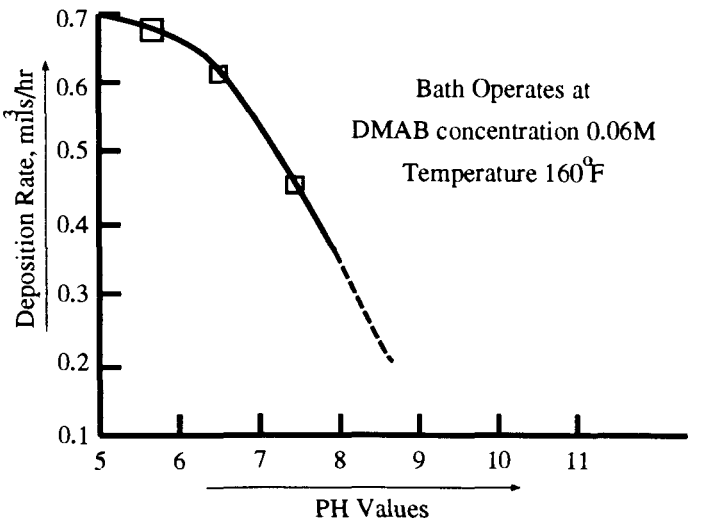

Figure 2.3: Effect of pH on deposition rate

is maintained until the temperature exceeds some crucial value that corresponds to the first inflection point on the curve. After this critical point, the rate of deposition becomes less dependent on temperature[21]. The reason is, at lower temperature, nickel reduction is the main reaction. When the temperature of the plating solution reaches a temperature that is equivalent to the first inflection point on the rate vs. temperature curve, the hydrolysis of DMAB becomes important.

The effect of pH on the plating rate in DMAB-reduced electroless nickel plating baths was found to be opposite to that observed in hypophosphate reduced plating solutions. The rate of plating increases when the pH of the plating bath is decreased. The relationship of rate vs. pH is symbolically illustrated in Figure 2.3.

Table 2.1: Typical alkaline nickel plating solutions[21]

Component	Concentration, g/L				
	Bath				
	A	B	C	D	E
Ni^{+2}	7.0	7.5	7.5	6.0	7.5
$NaH_2PO_2 - H_2O$	20.0	10.0	10.0	30.0	25.0
$C_6H_5ONa_3 - 2H_2O$	36.0	100.0	–	–	–
$C_6H_6O_7(NH_4)_2$	–	–	65.0	–	–
$K_4P_2O_7$	–	–	–	60.0	–
$C_6H_{11}O_7Na$	–	–	–	–	110.0
H_3BO_3	–	–	–	–	30.0
$C_{10}H_{10}N_2O_8$	–	–	5.0	–	–
NH_4C_1	–	50.0	50.0	–	–
PH with NH_4OH	8 9	8 9	8 10	10.0	9.0
Temperature, $°C$	90	90	90	75	80

At pH values below the first crucial point, the hydrolysis reaction is very temperature-dependent. Above that, the hydrolysis of DMAB does not become a major consideration until the temperature exceeds $70°C$. It has been shown experimentally that in the presence of reducible ions, such as Ni^{+2}, the hydrolysis of DMAB becomes more temperature-dependent, regardless of the pH.

Typically, the electroless nickel plating baths based on alkaline hypophosphate work at $70°C$ to $90°C$, pH values around 11. Electroless nickel plating baths based on amine boranes usually operate at $40°C$ to $65°C$ with a pH of 7. The relationship between the deposition rate vs. pH of DMAB based bath is illustrated in Figure 2.3. But stablizers should be used if a flat film is required. Table 2.1 and 2.2 gives the operating conditions of typical baths for both types of plating solution[21].

Table 2.2: Typical DMAB nickel plating solutions[21]

Component	Concentration, g/L			
	Bath			
	A	B	C	D
Ni^{+2}	6.0	11.0	7.5	7.5
Acetic Acid (88%)	30.0	25.0	–	–
Citric Acid	–	25.0	–	–
Sodium Succinate	–	–	20	–
Sodium Glycolate	15.0	–	–	–
Sodium Acetate	–	–	40	–
Sodium Pyrophosphate	–	–	–	60
DMAB	2.5	2.5	2.5	2.5
Thiourea, mg	1	–	2	–
Thiodiglycolic Acid, mg	–	70	–	50
$Pb(NO_3)_2$, mg	–	2	–	–
PH with NH_4OH	6.1	6.3	7.0	9.0
Temperature, ^oC	60	50	65	40

2.2.3 Solution Chemistry

The chemical reduction potential of the electroless nickel plating reaction can be affected by many factors. Proper chemistry treatment techniques are necessary to ensure that the plating process is reliable and consistent. One of the first and easiest bath parameters to be checked is pH. The pH is determined by pH meter or pH paper. The pH value of the solution may change because of evaporation of NH_4OH. It should be tested frequently and be maintained in its proper range. A high pH value of the plating solution can cause abnormally high plating rates that may lead to roughness, pitting, and/or cloudly deposits. Too low a pH will cause slow deposition rates and dull finishes.

Chemical imbalance of nickel and/or reducing agents concentrations may give slow deposition rates, poor coverage, and dull deposits. So a nickel plating solution cannot be used for longthy plating without good replenishing maintenance.

To get good plating, bath solutions must be kept clean enough. Since electroless metal plating process is a chemical process, it is affected by many impurities. Even very small contamination of impurities may lead to poor results.

2.2.4 Temperature

The temperature of the plating solution should also be closely monitored for consistent high-quality deposits. Accurate temperature controllers are necessary. Unchecked temperatures can lead to high deposition rate, or even decomposition of plating bath for too high a temperature, and no plating at all if the temperature is too low.

2.2.5 Surface Preparation

Proper preparation of the substrate to be plated is vital for quality results. Since oxide is not catalytic to electroless metal plating process, a properly prepared substrate is one whereby surface contamination is removed, leaving a clean, oxide-free surface. In order to get a uniform active surface, usually weak erosion is required. If the material of the substrate is more electronegative than nickel, a catalyst is

needed to active the surface, usually a Sn/Pd-based salt solution operated at room temperature. Poor surface preparation may cause lack of adhesion, deposit porosity, roughness, non-uniform coating and/or dark deposits.

In micromachining silicon is the material of substrate. Fortunately silicon is catalytic to electroless nickel deposition. Hence there is no need to active the bare silicon surface before deposition. The standard clean procedure used to clean silicon wafers is described in Appendix A. Since the silicon surface is extremely smooth and usually there is a thin native oxide layer, in order to get good deposition quality, a slight etch before the deposition is need to initiate the deposition process as well as to improve the adhesion of deposited Ni. Usually it is a mixture of HF and HNO_3.

With the use of Pd catalyst or Zinc oxide catalyst, many surface, such as aluminum, photoresist, can be activated to electroless metal plating. This implies a new approach to surface micromachining by depositing electroless nickel on activated photoresist surface, which will be presented in the fourth chapter in this thesis.

2.2.6 Selective Plating of Nickel Using Photoresist Mask

One other advantage of electroless plating process is that the plating takes place only on the catalytic surface. Hence, surface that are masked by a polymer, for example photoresist, will be not be plated. This gives an excellent masking technique for selective plating of nickel on a substrate. This technique is completely compatible with the standard microelectronics technology.

2.3 Electroless Plating for Microstructure Fabrication

The ease of electroless plating offers an economical means to deposit metal film on a substrate. Also the selective plating control using a photoresist opens up an exciting area of fabricating metal microstructures on a silicon substrate. This technique would be relatively easy, economical and technologically simple for the deposition of metal films on a silicon substrate, compared to sputtering or electroplating. Since nickel films are resistant to most of the silicon isotropic and anisotropic etchants, micromachining processes can be combined with the electroless plating process to fabricate micromechanical structures on a silicon substrate. Further, nickel would prove to be an attractive material for micromechanical structures due to its material properties with rich alloys.

Based on the discussion above, two kinds of micromachining processes have been proposed and set up to fabricate electroless nickel structures on silicon wafer.

Electroless nickel substrate micromachining is based on selective electroless nickel depositing on a masked silicon wafer which will be etched in anisotropic etching solutions EDP after the mask removal. A patterned photoresist layer can be used as depositing mask after hardbake. Since the silicon surface is itself catalytic to electroless nickel plating, nickel will deposit directly to areas with bare silicon. After electroless metal plating, photoresist can be removed by acetone. The whole chip can

be etched in EDP to anisotropically etch silicon. Since electroless nickel is safe to EDP etching, after processing, there will be a free-standing electroless nickel cantilever or beam over the anisotropic silicon cavity.

Electroless nickel surface micromachining is based on a selectively electroless nickel depositing on selectively activated photoresist surface which can be easily removed by acetone or photoresist thinner after depositing. A special thick photoresist layer has been used as depositing mask and surface sacrificial material. After patterning and developing, photoresist was selectively activated by Pd catalyst. Only the activated area is catalytic to electroless nickel plating. So nickel will only deposit on the selected area. After deposition, photoresist can be removed via acetone or photoresist stripper.

CHAPTER 3

Substrate Micromachined Nickel Structures

3.1 Substrate Micromachining

In substrate micromachining the microstructures are formed by the following methodology. The desired shape for the microstructure will be patterned on a silicon substrate using oxidation, lithography and sometimes diffusion processes. Normally boron is used as the diffusing species because silicon doped with boron in excess of $7X10^{19}/cm^3$ cannot be etched with anisotropic etchants[2]. After the patterning, a portion of the bulk silicon is etched using either an isotropic or anisotropic etchant to release the free-standing microstructure on the top surface of the substrate[26-31].

Table 3.1: AZ1312-SFD photoresist coating parameters

Sample	A	B	C	D	E	F
Speed/period (rpm/seconds)	4000/30	3000/30	2000/30	1000/30	1000/20 3000/10	1000/20 4000/10
Coating Quality	good	good	fair	poor, too thick at edges	fair, thick rings at corner	good
Thickness	1.0 μm	almost same	thicker	thicker poor uniformity	thicker poor uniformity	thicker good uniformity
Exposure (seconds)	60	60	60	-	-	100
Developer AZ351:Water	1:1	1:1	1:1	-	-	1:1
Developing (seconds)	50	60	90	-	-	90
Results	good	good	good	-	-	good

3.2 Nickel Microstructure Fabrication

In order to fabricate micromachined nickel structures the following procedure was adopted. 3-inch n-type silicon wafers were cleaned (Appendix A) and spin coated with AZ1312 positive photoresist and softbaked for 30 minutes at $100^{o}C$. The photoresist is used as a polymer coating to selectively expose the catalytic surface for plating. Table 3.1 gives the photoresist coating parameters used for this experiment. The ideal coating parameters derived from this experiment are as follows: spin speed 4000 RPM, exposure 100 seconds, developer dilution 1:1 and developing time 90 seconds.

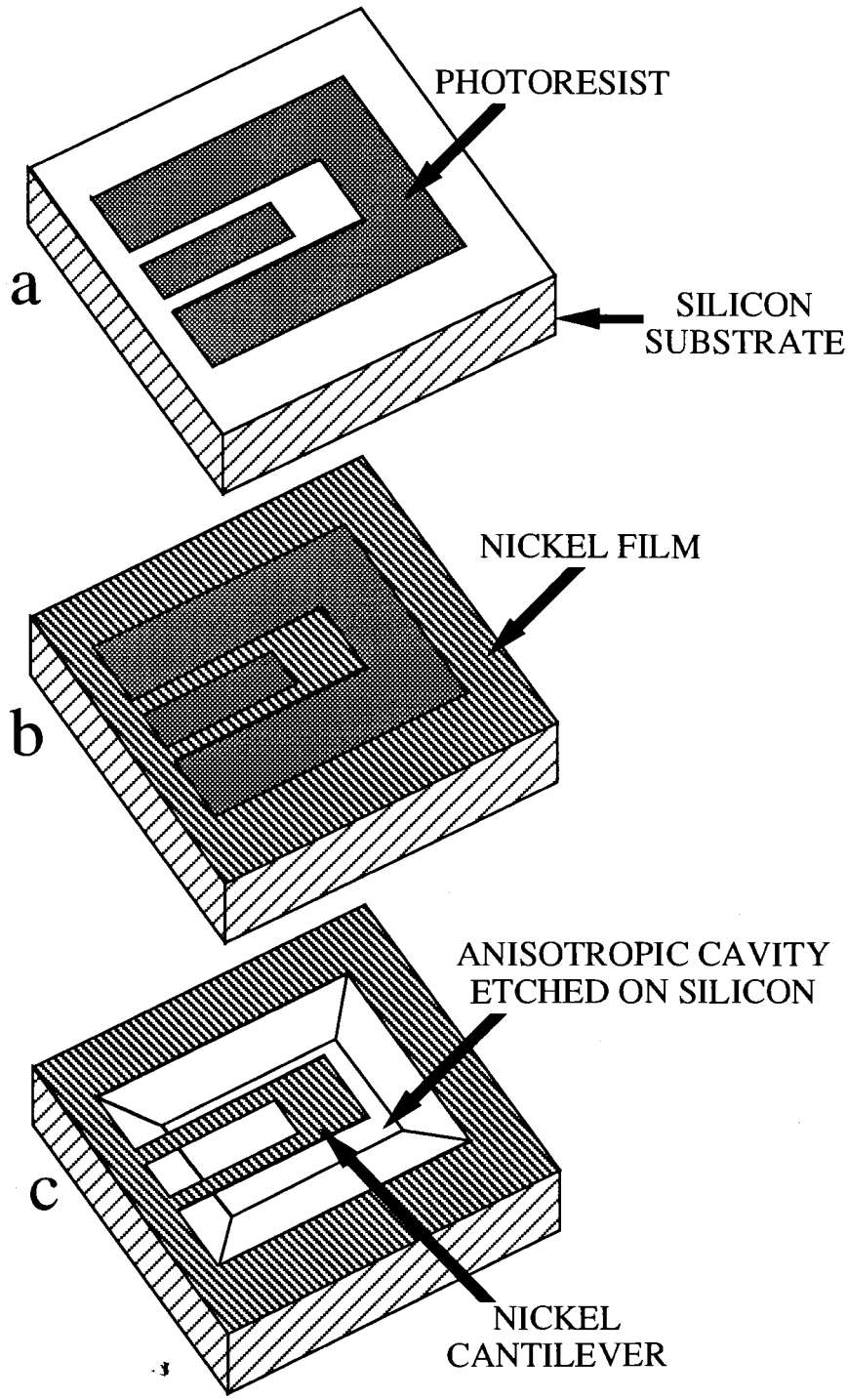

Figure 3.1: Illustration of substrate micromachining process

Table 3.2: Alkaline electroless nickel plating bath composition[32]

Component	Concentration, g/L
$NiSO_2.6H_2O$	33.58
$NaH_2PO_2.H_2O$	10.00
$C_6H_6O_7(NH_4)_2$	65.00
PH (with NH_4OH)	8.5 10.5
Temperature, ^oC	90

Wafers with patterned resist (Figure 3.1a) were then hardbaked for 50 minutes at 120^oC. To get good coverage, it is necessary to dip sample in BOE for 15 seconds to remove thin native oxide before putting the sample in an electroless nickel plating bath. After surface preparation, the sample was immersed in the aqueous electroless plating bath containing a nickel salt and a reducing agent for 20 minutes. Table 3.2 gives the plating solution composition. Nickel plating was systematically performed and this result is presented in Table 3.3.

During the plating process nickel was deposited only on the exposed silicon surface, as illustrated in Figure 3.1b. The resulting thickness of the nickel film was measured to be approximately $4\mu m$(inspected under microscope with focus difference). The photoresist was subsequently stripped with photoresist stripper acetone. The quality of the plating was measured. With the inspection under microscope, a good deposition is a deposition with uniform thickness, uniform color, and a smooth clean surface. The porosity and adhesion were determined by washing with DI water and acetone. A deposition with bad adhesion or porosity may form bubbles because of the penetration of DI water or Acetone. In order to get a stress-free microstructure, the wafers were

Table 3.3: Nickel deposition parameters for substrate micromachining

Sample	A	B	C	D	E	F
Photoresist	AZ1312 SFD	AZ1312 SFD	AZ1312 SFD	AZ1312 SFD	AZ1312 SFD	AZ1312 SFD
Nickel Bath	Alkaline Ni-P	Alkaline Ni-P	Alkaline Ni-P	Alkaline Ni-P	Alkaline Ni-P	Alkaline Ni-P
Temperature ^oC	70	80	90	90	90	90
pH	11.0	11.0	8.0	9.0	10.0	11.0
Plating Results	Poor Coverage	Not Shining	Hard to Start	Good	Good	Good
Immersing in DI Water	-	-	2 hours	No	2 hours	2 hours
Annealing at 120^oC	-	-	2 hours	No	2 hours	4 hours
EDP Etch ^oC/hrs	-	-	105/2	105/2	105/2	105/2
Resulting Microstructure	-	-	Flat	Curl up	Flat	Flat

heated at $120°C$. The appropriate annealing temperature is systematically recorded and given in the table 3.3. for one hour in nitrogen ambient. Exposed silicon was then anisotropically etched using Ethylene Diamine Pyrocatechol (EDP) in water, for 120 minutes, without any further masking step (Figure 3.1c). The resulting anisotropic cavity depth was measured to be approximately $75\mu m$ deep.

3.2.1 Stress Relief

Because the electroless Ni was deposited at a fairly low temperature, the polycrystalline Ni films exhibited residual stress. After the anisotropic etching all the structures exhibited an upward curl. In order to reduce this stress, the annealing step described in the previous section was incorporated. Literature reports that the pH of the plating solution has a strong effect on the magnitude of the residual stress[33-36]. Residual stress in unannealed films as high as 110MPa (tensile) has been observed[23]. Appropriate annealing procedures to relieve the stress can be performed and we have incorporated such an annealing procedure in our experiments.

With the processing procedure presented above, a few microstructures were fabricated, such as cantilevers with different length and width, microcoils, plates with various number of supporting arms. A minimum dimension in our experiments is $5\mu m$. Figure 3.2 shows an SEM photograph of a free-standing Ni cantilever fabricated using the technique described above. The dimensions of the free-standing cantilever are: each support arm is $15\mu m$ wide and $200\mu m$ long. The square plate structure at the

tip is $50\mu m$ on each side. The thickness of the structure is $4\mu m$(measured under microscope with focus difference).

If impurities or dust are present during the plating process, occasionally globular structures form on the microstructures and one such example is illustrated in Figure 3.3. Hence it is essential to maintain and clean environment as well as working solution to obtain good results.

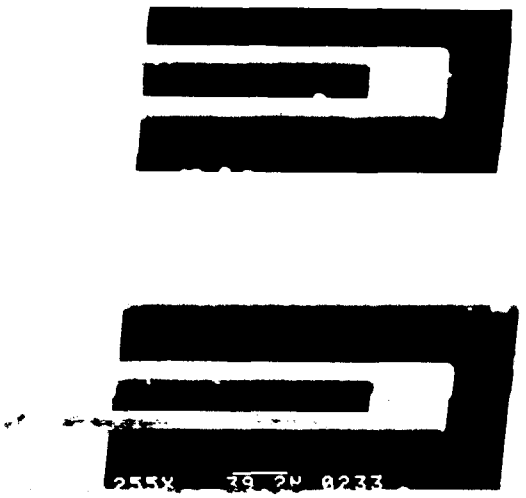

Figure 3.2: SEM photograph of a substrate micromachined nickel cantilever

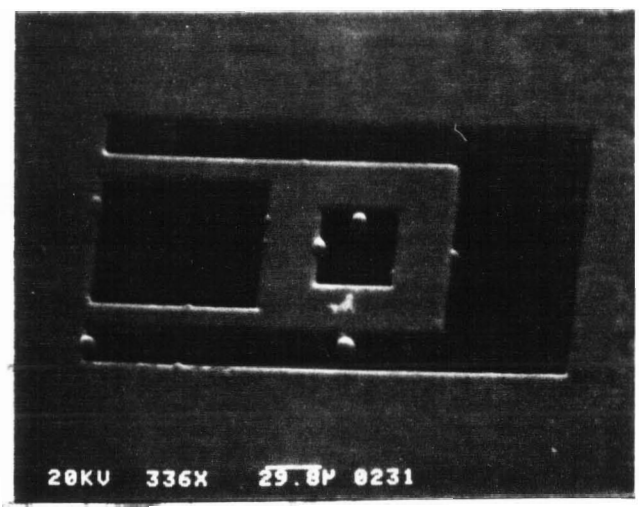

Figure 3.3: SEM photograph showing the effect of impurity during the plating process

CHAPTER 4

Surface Micromachined Nickel Structures

Surface micromachining is characterized by the fact that it uses a sacrificial layer to form microstructures on a substrate[37]. This technique is called as "lift-off" since the sacrificial materials will be removed finally. In this technology the required microstructure material is deposited on top of a sacrificial layer [38]. Usually silicon dioxide is used a sacrificial material[39-40]. In many cases a special photoresist can also be used as a sacrificial material[41-46]. The sacrificial material serves as a support to the microstructure during the fabrication process. At a last step of the fabrication process, the sacrificial material is chemically removed to form the desired free standing microstructure.

4.1 Metalizing Photoresist: a new approach to surface micromachining

Electroless metal plating has been used in printed circuit board fabrication for a long time, in which electroless copper is selectively plated on plastic surface for electric connection. In the last few years, several researchers have studied the characteristics of oxidized polyamide materials in electroless metal plating[47-48]. Photosensitive polyamide has also been used as an alternative to photoresist. With these alternatives, it is expected to get better coating quality on the special unplanar surfaces during micromachining fabrication [44].

In this section, a novel approach to surface micromachining will be described. It utilizes photoresist or other polyamide as the sacrifice layers, using electroless metal plating technique to fabricate surface microstructures.

This new process uses positive photoresist masks patterned by conventional lithography, which has the same geometric flexibility and is suitable for the deposition of metallic three-dimensional microstructures. Conventional near-UV lithography has been extended to thick resist layers (4 to 80 microns)[43] maintaining the resolution in the micron-range. This process offers various possibilities for applications. Especially the third dimension is taken advantage of the increasing performance of conductive structures. Because of the relatively simple and inexpensive processing, positive photoresist masks patterned by depth lithography seem to be particulary

suited to deposit conductors of high depth-to-width ratio in sensor and actuator applications.

As a novel process, its advantages is obvious. The operating temprature range is low, from room temperature to 120 ^{o}C. No electrical source is needed during processing. Therefore as a post micromachining process, it is completely compatible with traditional IC technology, especially CMOS technology. Novel mechanical components can be fabricated on a CMOS chip without any damage of circuitry on CMOS chips. Via this process, it is expected to be possible to build smart sensor systems more simply and low costly. A microbridge fabricated via this surface micromachining technique is simply shown in Figure 4.1

4.1.1 Deposition of Thick Photoresist Layer

Begin with a silicon wafer sample which has been cleaned by using standard clean and degrease process (Appendix A). A positive photoresist of high transparency and high viscosity (AZ 4620, Hoechst) is used for printing the thick resist layers. The solid content of AZ 4560 is 41.7 percent, and the viscosity is (300 - 420)cSt at 25^{o}C. The resist is deposited by spin coating. A photoresist layer with 7μm(inspected under microscope using focus difference) thickness is obtained with single coating at 3000RPM. A thickness of 4μm to 45μm can be achieved with varying speed spin or double spin coating[45]. A carefully carried out baking procedure at temperatures of 80^{o}C and 100^{o}C with a final slow cooling down process were necessary for stablizing

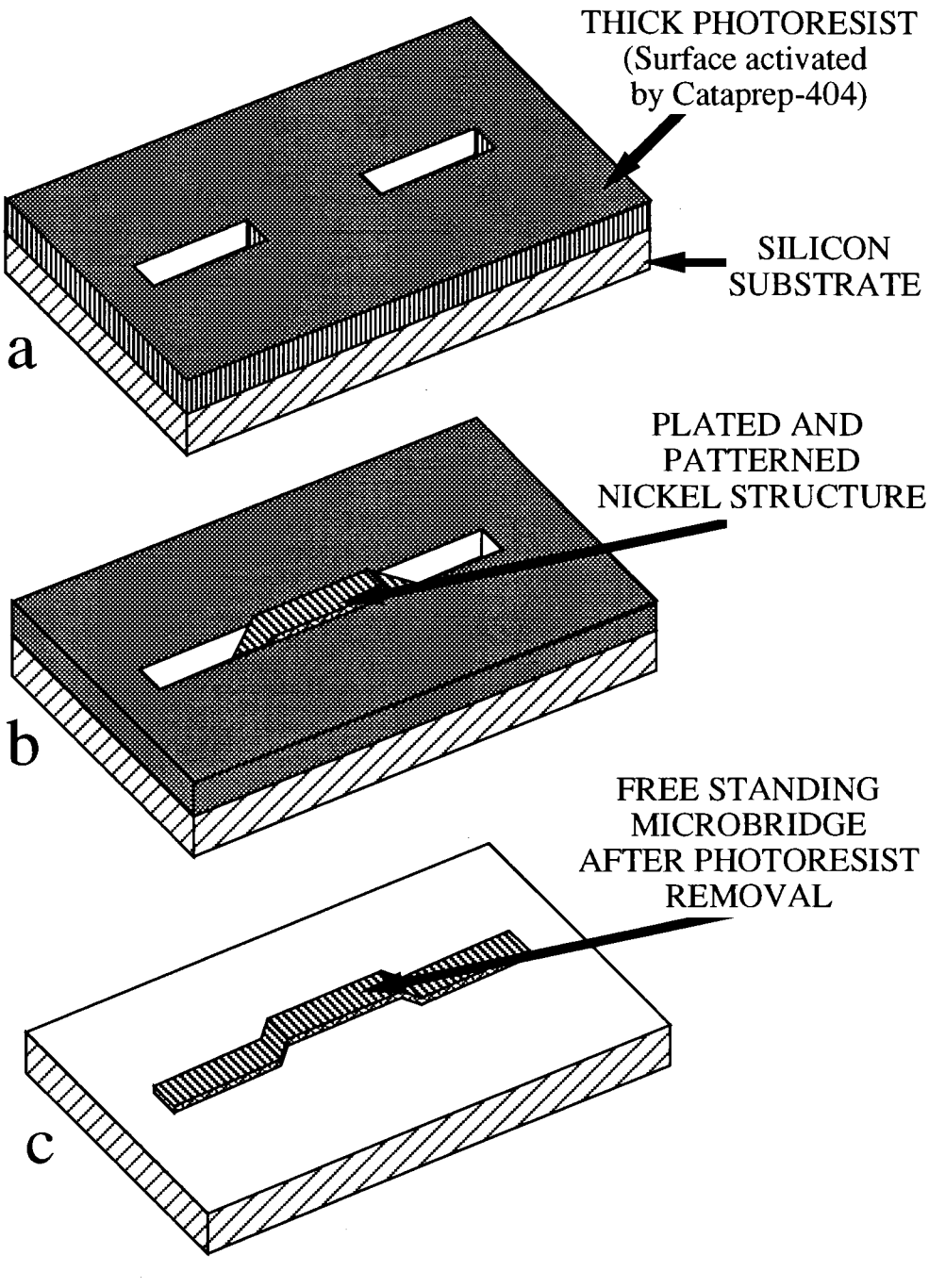

Figure 4.1: Illustration of surface micromachining process

Table 4.1: Photoresist AZ 4620 coating parameters

Sample	A	B	C	D	E	F
Spinning (rpm/seconds)	6000/30	4000/30	3000/30	2000/30	1000/60	1250/30
Softbake mins/100°	30	30	30	60	60	60
Thickness	5 μm	6 μm	7 μm	10 μm	12 μm	25 μm
Coating Quality	good	good	good	good	good	good

the samples after coating. As the prebake determines the photosensitivity of the resist, to maintain a good photosensitivity, temperature can not be over 130^oC. But hot plate baking is a very effective method of baking thick resists due to the efficient solvent removal and minimal sensitizer degradation it provides. A temperature of 120^oC for one minute was used to bake resists thicker than 7 μm. Table 4.1 shows the experimental records of photoresist AZ 4620 coating.

4.2 Nickel Microstructure Fabrication

The surface micromachined Nickel microstructures were produced by the following procedure. After a standard clean procedure, first a relatively thick layer of a high viscosity photoresist was spin-coated on 3-inch wafers. SPR2-1.8M and AZ4620

photoresist is needed in this case because of the relatively thick layer of photoresist required as the sacrificial layer. The experiments conducted on the AZ 4620 photoresist to determine the film quality is presented in Table 4.1.

4.2.1 Activation of Photoresist

Normally the photoresist behaves as a noncatalytic surface and electroless plating process does not deposit the film on top of the photoresist. This is the technique used in the substrate micromachining process. In surface micromachining technique, however, it is essential to deposit the nickel film on top of the photoresist. Hence the photoresist surface has to converted to a activated surface which will be amenable for nickel deposition.

This process of activation has been reported in literature [47 -51]. Using the activation chemical produced by SHIPLEY, CATAPREP 404 and CATALYST 44, the surface of the photoresist can be easily activated. The activation process was systematically performed and the observed results are given in Table 4.2, 4.3, 4.4. A recommended operation procedure are 3 minutes preparation at room temperature, 3 minutes activation at room temperature, 3 minutes of acceleration at 9.801%(0.25M in volume) HCl solution.

Due to activation the photoresist surface is now amenable to catalytic Ni deposition. Using the nickel plating solution, approximately $1\mu m$ thick Ni was deposited

Table 4.2: Photoresist activation parameters with CATAPREP 404

Sample	A	B	C	D	E	F
First Exposure	90sec	90sec	90sec	90sec	90sec	90sec
Developer AZ351:Water	1:1	1:1	1:1	1:1	1:1	1:1
Developing (Seconds)	120	120	120	120	120	120
Softbake mins/100°C	10	10	10	10	10	10
Second Exposure (Seconds)	50	50	50	50	50	50
Cataprep activation (Minutes)	*0.5*	*1*	*2*	*3*	*4*	*5*
Catalyst Action (Minutes)	3	3	3	3	3	3
Second Developer AZ351:Water	2:5	2.5	2:5	2.5	2:5	2.5
Developing (Minutes)	1	1	1	1	1	1
Nickel Plating Results	*No Plating*	*No Plating*	*No Plating*	*Selected Plating*	*Selected Plating*	*Selected Plating*

Table 4.3: Photoresist activation parameters with CATALYST 44

Sample	A	B	C	D	E	F
Photoresist	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M
First Exposure	90sec	90sec	90sec	90sec	90sec	90sec
Developer AZ351:Water	1:1	1:1	1:1	1:1	1:1	1:1
Developing Period sec.	120	120	120	120	120	120
SoftBake ^{o}C/min.	100/10	100/10	100/10	100/10	100/10	100/10
Second Exposure (Seconds)	50	50	50	50	50	50
Immersing in Cataprep (Minutes)	3	3	3	3	3	3
Immersing in Catalyst (Minutes)	*0.5*	*1*	*2*	*3*	*4*	*5*
Immersing in Accelerator (Minutes)	3	3	3	3	3	3
Second Developer AZ351:Water	2:5	2.5	2:5	2.5	2:5	2.5
Immersing in Developer (Minutes)	1	1	1	1	1	1
Nickel Plating Results	*No Plating*	*No Plating*	*No Plating*	*Selected Plating*	*Selected Plating*	*Selected Plating*

Table 4.4: Photoresist activation parameters with HCL based accelerator

Sample	A	B	C	D	E	F
Photoresist	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M	SPR2 1.8M
First Exposure	90sec	90sec	90sec	90sec	90sec	90sec
Developer AZ351:Water	1:1	1:1	1:1	1:1	1:1	1:1
Developing Period sec.	120	120	120	120	120	120
SoftBake ^{o}C/min.	100/10	100/10	100/10	100/10	100/10	100/10
Second Exposure (Seconds)	50	50	50	50	50	50
Immersing in Cataprep (Minutes)	3	3	3	3	3	3
Immersing in Catalyst (Minutes)	3	3	3	3	3	3
Immersing in Accelerator (Minutes)	*0.5*	*1*	*2*	*3*	*4*	*5*
Second Developer AZ351:Water	2:5	2.5	2:5	2.5	2:5	2.5
Immersing in Developer (Minutes)	1	1	1	1	1	1
Nickel Plating Results	*No Plating*	*No Plating*	*No Plating*	*Selected Plating*	*Selected Plating*	*Selected Plating*

on exposed silicon as well as on the activated photoresist. The wafers were subsequently heated at 120^oC for two hours in nitrogen ambient to relieve the residual stress. The Ni film is then patterned by photolithography as illustrated in Figure 4.1(b). In the end, photoresist was dissolved using a standard photoresist stripper leaving free-standing Ni microstructures on top of silicon surface as shown in Figure 4.1b. Figure 4.2 shows an SEM photograph of a Ni microbridge fabricated using surface micromachining technique. The dimensions of the microbridge are: width $5\mu m$, length between the two support locations $50\mu m$ and the thickness of the Ni film is about $1\mu m$.

Electroless plating technique, according to literature, should offer conformal coverage on the side walls. In our experiments however, for structures thicker than $3\mu m$, the side walls appeared to be slightly thinner.

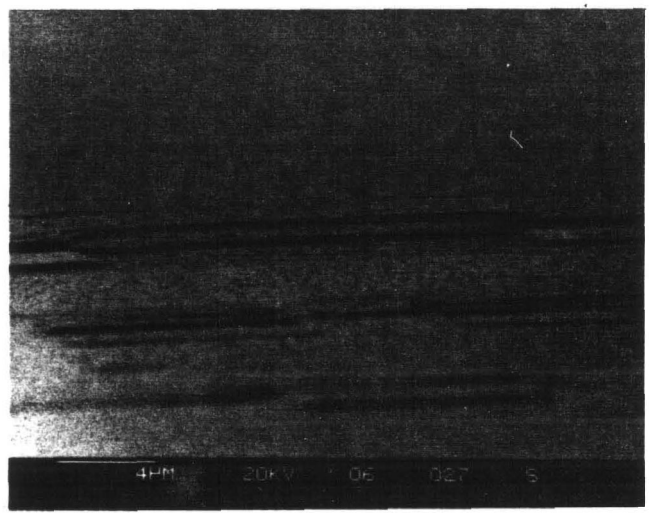

Figure 4.2: SEM photograph of a surface micromachined microbridge

CHAPTER 5

Conclusion

The major goal of this thesis is to develop a simple, economical and efficient micormachining process for the fabrication of microstructures for sensor and actuator applications on silicon chips. The purpose behind this aim is to realize the possibility of fabricating smart sensor systems with integrated sensors and actuators in an inexpensive manner. Several processes utilizing the electroless nickel as the constructing material has been presented. With their compatibility with standard CMOS IC technologies, they give rise to many useful features for micormachining development. This chapter summarizes their significant aspects and the main contributions of this research.

5.1 Contributions

With the careful study of electroless metal plating, it is introduced in the micromachining area as a new process. Photosensitive polimide has been used as the sacrificial material and mask to selectively plating metal on a selected area. This research provided the basis to easily fabricate 3D metal structures on a fabricated IC chip. Consequently, several processes have been set up whereby many fundamental building blocks for the fabrication of sensors and actuators can be obtained simply by photoresist lithography and electroless metal plating. These new processes not only introduce new materials to construct micromechanical structures but also provide a set of new methods in the design of micromachining systems.

Electroless plated metal alloy has been used as a new material in sensor fabrication. With various plating environments, various metal alloys can be deposited. The material properties can be controlled in a wide range, from good conductor to semi-insulator, from magnetic, ferromagnetic, to nonmagnetic, with various elasticity, hardness and sensitivity.

As a novel approach to surface micromachining, the use of photosensitive polymide sacrificial layers in the fabrication of surface-micromachined structures is presented. It is based on electroless metal plating processes. Utilizing electroless metal plating has several advantages: Since it does not damage any structures formed by conventional CMOS processes, as a postprocess of micromachining technique, it is completely compatible with CMOS process; there are many kinds of electroless metal plating bath

and it is easy to make in common chemical laboratories, so it can be carried out easily; its composition can be easily controlled, thus its material properties can be easily controlled to meet various requirements. A polymide sacrificial layer offers two benefits: first, structures suspended several tens of microns above a substrate can be fabricated due to the ability to coat relatively thick polymide layers; secondly, the selective deposition of electroless material only on activated catalytic polymide surface allows three-dimensional structures to be fabricated in a self-aligned process. All the structures are fabricated using the same process, using thick photoresist as the sacrificial layer and electroless nickel alloy as the structural material, although other material combinations can also be used(Cu, Co, Fn, **etc**).

Many of the same types of micromechanical devices previously fabricated from polycrystalline silicon are expected to be fabricated from electroless metal alloy. It provides a new approach in which many kinds of microstructures, even fully released components, can be designed.

5.2 Outlook

The combination of commercial IC processes and micromachining post-processing for solid-state sensor fabrication has significant merits and potential. Unlike conventional micromachining technology, besides the process-layers offered by CMOS process, the new processes offer a way of adding new layers and materials of electroless metal alloy. Since commercial CMOS processes are two-dimensional planar processes, using the

approaches reported in this thesis, combining electroless metal plating and CMOS process-layers, fabricating of novel three-dimensional sensor structures on the surface of a CMOS chip becomes possible.

Taking advantage of the processes presented in this thesis, a electroless metal plating based process for the fabrication of high-aspect-ratio microstructures is possible by using the thick photoresist via conventional ultra-violet exposuring equipment. Such process exploits the sharp-sidewall characteristics of photosensitive polyimide to create the electroless metal plating form through which the high-aspect-ratio structures are electrolessly plated. Although the resolution of this process is inferior to the X-ray-based process LIGA[43], this process has several advantages: it is simple and can be carried out using commercially available materials and common clean room equipment; it is operated at a low temperature and no electrical voltage is needed; the excellent chemical and thermal resistance of photoresist allows plating to be controled in a variety of variables; and multiple coats of polyimide can be used to fabricate vertically integrated structures which have variation in the third dimension. The process is completely compatible with surface micromachining sacrificial layer techniques to create released electroplated microstructures. It is expected to fabricate micromechanical structures in various physical size, from gears to cantilevers, membranes. All of these offer a wide selection in the design of new kinds of sensors and actuators.

A

Standard Clean Procedure for Silicon Wafer

All solutions should be mixed just prior to use; do not premix!

Step One

Solution:

50 ml DI Water : 20 ml H_2O_2 Peroxide : 10 ml NH_4OH (Ammonium Hydroxide)

Operation:

Heat solution to 80^o - 90^o.

Boil wafers at this temperature for 5 minutes.

Rinse in DI Water(1 min. minimum).

Step Two

Solution:

50 ml DI Water : 20 ml H_2O_2 Peroxide : 10 ml HCl (Hydrochloric Acid)

Operation:

Heat solution to 80° - 90°.

Boil wafers at this temperature for 5 minutes.

Rinse in DI Water(1 min. minimum).

Ultrasonic Degrease

Step 3	$(CH_3)_2CO$ (Acetone)	5 min. Ultrasonic Agitation
Step 4	$CICH : CCl_2$ (Trichloroethylene)	5 min. Ultrasonic Agitation
Step 5	CH_3OH (Methanol)	5 min. Ultrasonic Agitation

Rinse in DI Water(1 min. minimum).

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