Nickel Electroforming of LIGA Processed Structures

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Abstract

In this work we present some results for Nickel electroforming of LIGA processed microstructures intending to produce permanent metallic insertion mold. The chemical composition of the electrolytes as well as the bath physical parameters were adjusted to get satisfactory results of electroforming, as can be seen from our results. With our process it is possible to obtain high aspect ratios for metal deposition. We already control the process to avoid Ni delamination and are able to obtain good surface quality for our electroformed samples. However, some questions still remain to be clarified and will be discussed here in detail, such as the non uniformity of planar deposition near the borders. A set of "optimized parameters" must be taken into account in order to produce good electroformed structures.

I. Introduction

MICROELECTROMECHANICAL Systems or simply MEMS, Micro Systems Technologies (MST), Mechatronics, Micromanufacturing or Micromachining are synonymous identifying very small devices, varying from micrometers up to millimeters, fabricated for different functions and purposes normally mixing electronics and mechanical behaviors. Microsystems generally includes built-in circuitry to be connected to the macro-world [1].

The beginning of Micromachining occurred from the mid of 1960's, in general by adapting existing technologies in the field of precision engineering to the miniaturization of structures. In the late 1970's, at the Research Center Kalsruhe, German, a new process was developed, introducing the concept of the sacrificial layer. It was named **LIGA** as a German abbreviation of **LI**tographie (lithography), **G**alvanoformung (electroplating) and **A**bformung (injection molding) [2]. Their advantages are mainly to allow: the fabrication of any lateral shapes microstructures; the micro machining of high aspect ratio structures (heights of several hundred micrometers to lateral dimensions down to one micrometer); the possibility of a large scale reproduction of such Microsystems in different materials like plastics, metals and ceramics or any combination of them.

The **LIGA** process of microfabrication can be resumed as follows:

- First step (exposure): an absorber X-ray mask (consisting e.g. of a titanium beryllium thin membrane with gold as absorbers layers) is exposed to synchrotron X-ray radiation transferring its pattern, by shadow, into a thick layer of resist.
- Second step (development): the exposed resist layer, whose polymer chains were destroyed by radiation, is dissolved producing a template.
- Third step (electroforming and resist removal): metal is deposited by electrodeposition process filling the empties spaces of template up to the height of resist or even more. After this, the unexposed layer of resist is removed.
- Fourth step (injection molding): the metal pattern so obtained constitutes an insertion frame that can be filled by means of molding processes to allow mass production of the microstructure.

Scientific research has been carried out to serve chemical, medical and biological engineering, micro-mechanics, micro-optics and other areas of knowledge. There is also a great commercial interest in micro manufacturing technologies: pressure, gas and optical sensors, accelerometers, micro-actuators, micro-pumps, micro-valves, integrated microphones, micro-mechanical gears, etc [3,4].

In this paper we discuss our recent results concerning the electroforming of Nickel for microstructures obtained in SU-8 resine with the LIGA process.

II. Experimental Setup and Results

In Table 1 we show the optimized chemical reagent composition and physical parameters values for Nickel electroforming of microstructures. Since these parameters and the electrolyte chemical composition play a fundamental role in determining the final deposit quality, it is important to pursue their best values.

Some of the pieces we work on are composed of SU-8 resin LIGA processed over Si wafer covered with a (5,000 Å) Cooper layer. This metal layer is important to make possible the Ni electrodeposition. Nevertheless, another metal is necessary to promote the adhesion between Si and Cu. When it is used a (300 Å) Chromium layer Ni delamination we can observe that after some hours of Ni electrodeposition all the three metal layers loose adhesion to the Si wafer.

To test the bath composition intending to avoid this Ni delamination we introduced a new kind of substrate: plate for printed circuit or protoboard-type. This kind of plate is commercially sold and already comes covered with a Cooper layer, as find anywhere needed. It is an inexpensive material for substrate and worked out correctly.

Material	Weight
$NiSO_4 \bullet 6H_2O$(Nickel Sulfate)	340 g/l
$NiCl_2 \bullet 6H_2O$(Nickel Chloride)	45 g/l
H_3BO_3(Boric Acid)	3 ml/l
Wetting Agent Y - 17	3 ml/l
Additive M-901	6.0 ml/l
Additive M-902	1.0 ml/l
pH	3 - 4
Temperature	$55 - 65°C$
Direct Current Density	$4.0 - 4.5\ A/dm^2$

TABLE I
Principal reagents and external controls applied to the electrolytic bath used for this work

The two electroformed microstructures we show in Figures 1, 2 and 3 photographs are fabricated over this alternative substrate. It can be seen in these figures that the Cooper layer has

Fig. 1. Microstructure with $0.25mm^2$ of area, included in a silicon wafer. In this case the device is a capacitor that can be applied for different situations and goals.

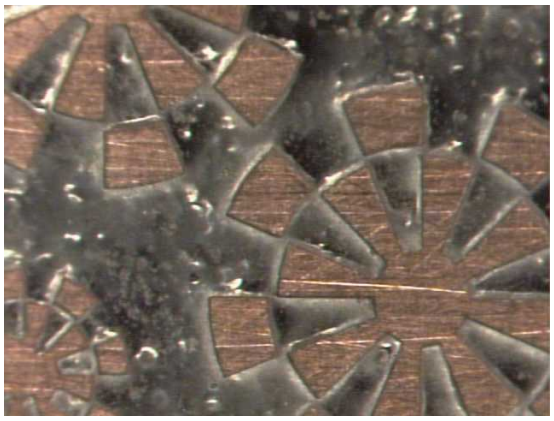

Fig. 2. Electroformed structure of Nickel. The substrate appearing was covered with Copper. Even tough the Nickel surface present irregularities the aspect ratio of the growth Nickel, which is the most important factor up to now, is of good quality.

some metallization failures due to its commercial characteristic. This characteristic, however, does not interfere in our results.

It can be seen from Fig. (1) a particular device chosen from our first substrate. The total device has $0.25mm^2$ of area surface. It acts as a capacitor and applicabilities for its usage are diverse. The Nickel electroformed presents a good degree of levelling nevertheless some occasional deposit's deformities, known as "pits", can be observed. Fig. (2) and Fig. (3), referring to micromechanical gears, were taken from another wafer of the same alternative kind of substrate used in piece 1.

Nickel electrodeposition were obtained with the following experimental setup: a Nickel bar acts as the anode in front of which the cathode is fixed and a moderate gas flux provides the bath agitation. The direct current intensity is kept constant by varying the applied bias voltage. The electrolytic cell is maintained over a hot plate that keeps constant the bath temperature. All the process occurs inside an exhaustion chamber.

III. Discussion

LIGA microstructure electroforming is different from electroplating of plain substrate because it presents cavities of high aspect ratios to be filled. In these narrow channels, mass transport is particularly important because it is difficult to fill a reduced hole with isotropic distribution. In consequence of the difficulty in the transportation of mass by convection to reach the bottom of deep gaps, the diffusive flow of mass becomes more important than the convective one. Therefore, limited current densities, lower than those to electroplating macroscopic films, have to be used. In our experiment, the range $4.0 - 4.5A/dm^2$ was good enough for the current density in order to fulfill the narrow deep gaps of microstructures [5,6].

Four points are influenced by the **pH** of the bath: the hydrogen discharge potential; the precipitation of basic inclusions; the composition of the complex or hydrate from which the metal is deposited and the extent of adsorption of addition agents. During the electrodeposition process, changes in **pH** can occur if there are inequalities in electrode efficiencies [7]. The co-deposition of hydrogen ions and the formation of adsorbed intermediate products causes an increase to the cathode **pH** meaning that its efficiency is lower than that of the anode. So, the **pH** for our electrolytic solution was controlled in order to be kept approximately constant between 3 and 4.

Deposit's surface quality, in other hand, can be endangered by pitting. A possible mechanism of pitting is the adhesion of hydrogen gas bubbles to the cathode surface. These bubbles locally prevent the plating making Nickel to deposit around them. Wetting agents are used to promote detachment of hydrogen gas bubbles, minimizing pits formation [7].

The internal stress of deposited Nickel is perhaps the most important cause for delamination as it causes the deposit to tear and roll up within a few hours. Incorporation of foreign material, such as occluded or co-deposited hydrogen, can obstruct normal lattice formation of Nickel deposits, originating stress and strain [8]. Supposing cathode in front anode, this tensor stress causes deposit to curve concavely in the anode direction.

The mismatch in the lattice can be avoided by organic stress reducers. Their action consists in entering the structure of deposited Nickel, altering the contraction of the lattice of nuclei at the moment when ions arrange themselves in regular lattice positions to form a metal. Stress reducers, however, act in proportion to quantity: diminish stress to a small addition, prevent it when in the "correct" amount and induce compressive stress if in excess. This kind of stress causes Ni deposit to curl convexly towards the anode [7,8].

In order to reduce internal stress we added organic additives (saccharin and coumarin) to the basic electrolyte chemical composition [9,10]. These additives also diminish Nickel grain size improving the hardness of mold inserts . Another providence to eliminate delamination was to change the first metal layer evaporated over Si substrate: instead of Chromium we employed Titanium. With this Si wafer no delamination is observed in the Ni deposit it does not matter the time the process takes. This results leads us to conclude that one reason for delamination is the poor adhesion of the Cu metal layer to Si wafer. So, we can see that the Ti metal layer provides a better adhesion between Cu and Si than does Cr.

The optimization of the referred parameters made possible to solve two fundamental problems: Nickel delamination and deposit's surface quality. Controlling additives, no further delamination was observed for optimized quantities; controlling current density, temperature, **pH** and electric field distribution, we improved the quality of surface. However, other questions still remain to be solved. The first one is the deposit's levelling over the surface, specially near its borders. Another is the uniformity of the metal grown up for very reduced dimensions' structures. For large areas the electroformed surface is almost homogeneous meanwhile for small dimensions we observe a lack of levelling near the borders.

To attack the lack of planarity and uniformity we are looking for the optimization of other parameters. The first step is to get the most possible homogenized spatial distribution for the electric field. To reach this point we created quasi-cylindrical anodes inside which we put the sample to be electroformed. Then, we set up a rotative system for the cathode with the double function of homogenizing any electric field difference in time as well as to modify the previous gas injection for bath agitation.

The steps we are working out actually are: to make a metallic sample carrier having the same dimensions of anode to mostly align the field lines;

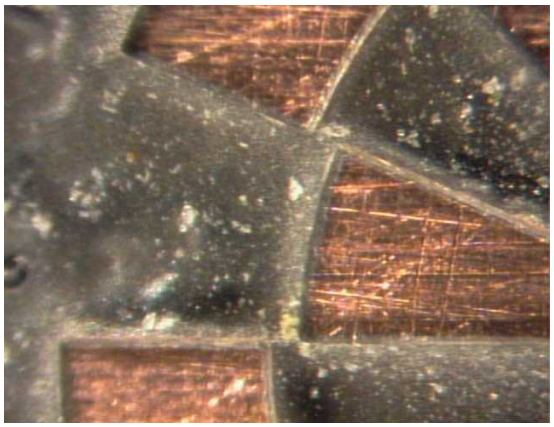

Fig. 3. Magnification of the preceding picture, in which it is possible to verify more precisely the good aspect/ratio of the electroformed Nickel.

to involve the anode bar in filters to avoid diffusion of mechanical anodic impurities; to provide continuous filtering of electrolytic solution instead of the actual filtering at the beginning of each electrodeposition process.

We hope to sensibly improve the deposit's levelling getting a good surface's planarity this way.

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