行政院國家科學委員會專題研究計畫 成果報告

含奈米粒子之金屬微元件的製程及機械特性研究

計畫類別: 個別型計畫

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<u>執行期間</u>: 94 年 08 月 01 日至 95 年 07 月 31 日 執行單位: 國立交通大學機械工程學系(所)

計畫主持人: 徐文祥

計畫參與人員: 蔡梨暖, 李毅家, 黃家聖

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中華民國95年8月9日

行政院國家科學委員會補助專題研究計畫 ■ 成 果 報 告 □期中進度報告

含奈米粒子之金屬微元件的製程及機械特性研究

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行政院國家科學委員會專題研究計畫成果報告 含奈米粒子之金屬微元件的製程及機械特性研究

計劃編號: NSC 94-2212-E009-016 .執行期間: 94年8月1日至95年7月31日 計劃主持人: 徐文祥 國立交通大學機械系教授 計畫參與人員: 蔡梨暖、李毅家、黃家聖

中文摘要

本計畫重點在探討含奈米鑽石粒子之鎮金屬微結構之材料性質變化及相關應用。在製程上是藉由低溫、低應力之電鍍製程並於鍍液中添加奈米鑽石粒子(直徑 < 0.5 μm),我們發現此奈米複材會隨添加鑽石奈米粒子濃度增加而提升硬度及楊式係數,最特別的是還可提升熱膨脹係數。因此我們以此奈米複材製作電熱式微致動器及梳狀共振器。

在電熱式微致動器部份,鑽石-鎮奈米複合材料減少了元件達相同輸出位移所需得輸入功率,並提供優於純鎳金屬元件的穩定性。實驗結果顯示, 3µm 的位移輸出下,鑽石-鎮奈米複合材料降低了純鎳電熱式微致動器 73%的功率輸入;可回復之變形量由 1.8µm 提升至 3µm,經過 100 次的測試,元件仍可恢復至原始位置。在梳狀共振器的部份,鑽石-鎮奈米複合材料的高楊氏係數使得共振器的共振頻率上升。以含 2g/L 奈米鑽石粒子之電鍍液製作之梳狀致動器,經頻譜分析後發現,共振頻率提升了 11.4%。

Abstract

In this study, a low-temperature stress-free electrolytic nickel (EL) deposition process with added dispersed diamond nanoparticles (diameter < 0.5 μm) is developed. It is found that mechanical properties, such as hardness, Young's modulus, and coefficient of thermal expansion (CTE), will be enhanced with the concentration of nano diamond particles. To demonstration the applications of this Ni-diamond nanocomposite, electro-thermal microactuators and microresonators are fabricated. For electro-thermal microactuators, device characterization reveals dramatic performance improvements, including a reduction in the input power requirement and enhancement on operation reliability. In comparison with the microactuator made of pure nickel, the nanocomposite one can save about 73% the power for a 3 μm output displacement and have a longer reversible displacement range, which is prolonged from 1.8 μm to more than 3 μm . Furthermore, the nanocomposite device exhibits no performance degradation after more than 100 testing cycles in the reversible regime. For the microresonator in comb structure, Ni-diamond nanocomposite is helpful in enhancing the resonant frequency because of the high Young's modulus of Ni-diamond nanocomposite. With 2g/L nanodiamond in the electrolytic nickel solution, an 11.4% improvement on the resonant frequency is measured from the frequency response analysis.

Introduction

Metal-based micromachining technologies have attracted lots of attentions recently due to their superior material properities in certain applications, such as RF components and electro-thermal microactuators. For electro-thermal microactuators, lots of operating principles have been proposed, some of them used

polysilicon as structural materials by silicon-based micromachining process [1, 2], and some used metal as the structural materials [3, 4].

Comparing with polysilicon, electro-thermally driven microactuators made of metal do not need doping process, are easier to achieve thicker structure by electroplating, and can provide larger output displacement with lower input voltage because of larger thermal expansion coefficient. However, the metal-based microstructure would suffer mechanical deficiency such as fatigue and aging [5, 6]. Also, it was found out that when operating temperature was higher than certain value, nickel structures would be degraded and an irreversible darkening on the device surface might occur [7].

Recent research in the nanocomposite synthesis, Teh et al. [8] have proposed the incorporation of nanodiamond particles into an electroless nickel can greatly enhance the overall stiffness of nickel film due to the extreme hardness, stiffness, and temperature resistance of the diamond particle [9]. Besides, it was found that the higher the concentration of nanodiamonds being incorporated into the matrix, the less residual stress can exits in the composite film, which is very suitable for the fabrication of microactuators. In order to examine the possibility to employ the low-temperature stress-free electrolytic nickel (EL) nanocomposite deposition process for future MEMS device applications. Adding the diamond nanoparticles into the structures could potentially improve and strengthen the material properties. In the future, Ni-diamond nanocomposite materials could displace polysilicon in certain applications, such as radio frequency (RF) MEMS metal switches [10] and optical micromirror arrays [11]. In this paper, the nanocomposite effects on the microactuators, cantilevers and electro-thermal microactuators, will be further investigated including effects on E/pratio, hardness, thermal expansion coefficient (CTE), power consumption, and reversible displacement range.

Characterizations of Nanocomposite Films

Before incorporating a nanocomposite material into MEMS devices, it is important to have a better understanding of the material characteristics and potential impact on device performance. To facilitate this, four kinds of as-fabricated 50 μ m wide, 6 μ m thick cantilevers are utilized for the examination of the material property enhancements in terms of static and dynamic characteristics, such as hardness and Young's modulus to density (E/ ρ) ratio. The beam lengths are designed as 150 μ m, 250 μ m, 350 μ m, and 450 μ m, respectively. Fabrication of the cantilevers is schematically illustrated in Figure 1.

The hardness of the nanocomposite film is characterized by the nanoindenter, XP system of the America MTS Company. Figure 2 shows the hardness increases with the concentration of the diamond nanoparticles in a plating bath. The hardness enhancement can be attributed to the incorporation of diamond nanoparticles. For the case of the nanocomposite film plated with the concentration of 2 g/L nano-diamonds, it can have 2.6 times larger hardness than the pure nickel one. On the other hand, a laser doppler vibrometer (LDV) is utilized to characterize the dynamic behavior of

the electroplated films. The E/ ρ ratio of the films can be derived from resonant frequencies of the cantilevers that are made of the EL with different amounts of diamond nanoparticle incorporations. The relationship between E/ ρ ratio and resonant frequency is determined by the following equation (1).

$$f = 0.1615\sqrt{E/\rho}(\frac{h}{L^2})$$
 (1)

where E, ρ , h, and L are the Young's modulus and density of the structural material and the thickness and length of the beam, respectively. Since the resonant frequency is very sensitive to the values of h and L, process variations should be considered for the accurate calculation of E/ ρ ratio. Table 1 lists one set of frequency measurement results of 350 μ m long, 50 μ m wide cantilevers.

The cantilever fabricated in a plating bath with 2 g/L diamond nanoparticles has about 1.29 times higher E/ρ ratio than the one made of pure nickel.

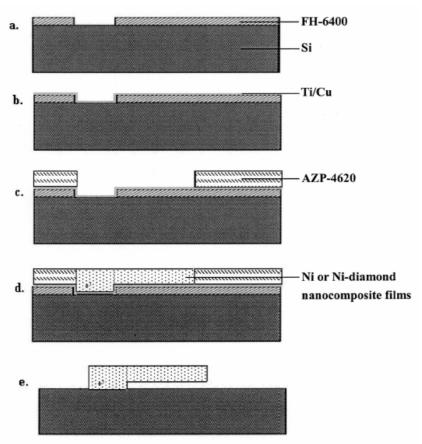


Figure 1 Fabrication process flow of cantilevers

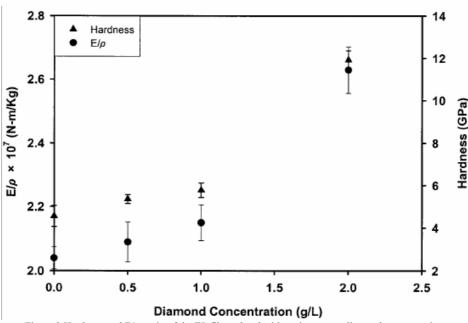


Figure 2 Hardness and E/ρ ratio of the EL films plated with various nano-diamond concentrations.

Concentr Diam Nanopa (g/l	ond rticles	Measured Beam Thickness (µm)	Effective Beam Length (µm)	Measured Resonant Frequency (KHz)	E/ρ ratio (N·m/Kg)
0		5.5	535	14.031	2.04x10 ^{7*}
0.:	5	5.08	409	22.436	2.09x10 ⁷
1		5.06	372	27.367	2.15x10 ⁷
2	·	4.4	352	29.45	2.64x10 ⁷

Table 1 Measured frequencies of the designed 5 μm thick, 350 μm long cantilevers plated with various Ni-diamond nanocomposites.

Electrothermal Microactuator

The electrothermal microactuator with long-short beam configuration as shown in Figure 3 is used for the experiment. The actuator has a pair of connected cantilevers that are made of the same material but with different actuating arm lengths. In the design, the two beams have a length ratio of 0.5 and a 10 μ m gap spacing between them. Both beams have the same width and thickness. The longer beam is about 800 μ m long, 10 μ m wide and 9 μ m thick. Via electrically resistive heating of these two beams, the arm tip curls toward the side of the shorter one due to the unequal thermal expansions of two beams. Fabrication of the electrothermal actuator is similar to that of the cantilevers (described in pervious) with the exception that a 2 μ m SiO2 film is used as a sacrificial layer instead. The fabrication result is as shown in Figure 4. Device testing is conducted on a probe station with an automated CCD image system with 0.2 μ m resolution. Figure 5 shows the displacement versus input power data obtained from the actuators plated in the baths with different nano-diamond concentrations. The microactuator made of the pure nickel provides a 3 μ m displacement with an input power of 0.924 W. Meanwhile, the required input power for the same output displacement decreases with the concentration increase of diamond nanoparticles. It indicates that the microactuator made of nanocomposite is more efficient than the one made of pure nickel. To produce a 3 μ m displacement, the microactuator made of the Ni-diamond nanocomposite plated in a bath with 2 g/L concentration of nano-diamond requires only 0.248 W, a 73% reduction in power consumption in comparison with that of a pure nickel device.

An electrothermal microactautor is normally operated in the region of reversible deformation in which the device must come back to its initial position while input power is removed. According to the actuation measurement, we found that the microactuator made of the Ni-diamond nanocomposite has a reversible output displacement over 3µm. In comparison with the pure nickel one, which exhibits irreversible deformation once the output displacement is over 1.8 µm, the nanocomposite actuator can indeed provide a better reversible characteristic. Figure 10 shows cycling test results. The tests are performed on both sets of actuators, which are made of the pure nickel and the Ni-diamond nanocomposite (2 g/L) films respectively, with the operational range from 0 to 3 µm. The hysteretic displacement and surface darkening occur in the pure nickel device during the first cycling test and the device is permanently deformed even after the power is reduced to 0 W. The resting position of the deformed device is 0.2 µm away from its designed origin. In contrast, no hysteresis is found in the nanocomposite device. The back and forth displacements are almost overlapped and no degradation has been found after 100 times cycling tests. The overall results indicate that the electrothermal microactuator made of the nanocomposite not only has better power efficiency but also has enhanced operational reliability by extending the reversible displacement range of the structure.

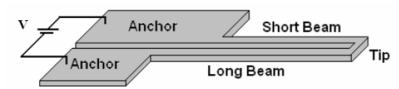


Figure 3 Schematic diagram of the electrothermal microactuator with a long-short-beam design.

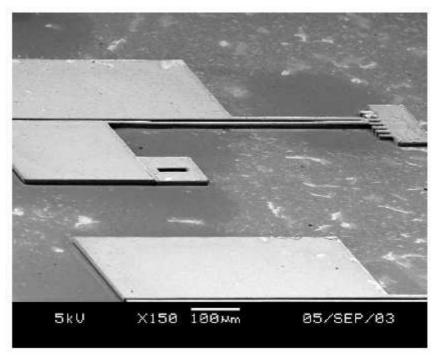


Figure 4 SEM photograph of the fabricated electrothermal microactuator using Ni-diamond nanocomposite plated with the concentration of 2 g/L nano-diamonds.

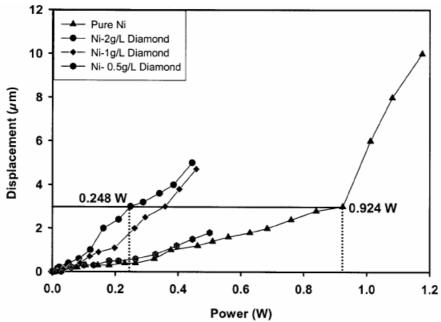


Figure 5 Input power versus displacement of the microactuators made of the nanocomposites plated with different nano-diamond concentrations.

Resonator

The three-mask fabrication process, including deposition of the pad layer, the sacrificial layer, and structure mold are proposed to fabrication comb drive. By controlling electroplating parameters which include current density at 1.2

mA/cm² and operation temperature at 35 °C, the comb drive made of pure nickel or nickel-diamond nanocomposite can be successfully released without bending, as shown in Figure 6.

The resonant frequency is measured by MEMS Motion Analyzer. This instrument uses both brightfield and interference based illumination modes combined with sophisticated machine vision algorithms to quantify target motions. Under 50V AC with 50V DC bias and the frequency from 5 kHz to 15 kHz, there is a peak for nickel and nickel-diamond comb drive, individually. The results of measurement are shown in Figure 5. The first mode of pure nickel comb drive with 300 μ m spring length occurs at 8.034 kHz (Figure 7(a)). The nickel-diamond one resonates at 8.949 kHz (Figure 7(b)). There is an 11.4% improvement in resonant frequency.

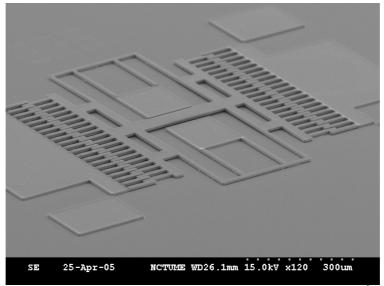
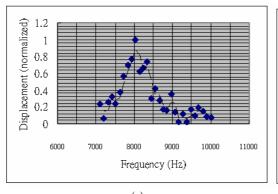


Figure 6 no-bending comb drive made by improved process (35 °C and 1.2 mA/cm²).



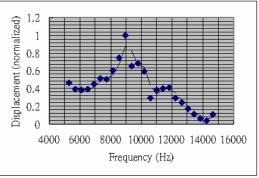


Figure 7 Measured frequency response of comb drive made by (a) pure nickel (resonant frequency=8.034 kHz); (b) nickel diamond (resonant frequency=8.949 kHz).

Discussions

A low-temperature stress-free electrolytic nickel (EL) deposition process with uniformly dispersed diamond nanoparticles (diameter $< 0.5 \mu m$) is proposed and successfully demonstrated here. The testing results show that the increasing diamond concentration can effectively enhance E/ρ ratio, rise hardness and increasing CTE.

In the application of electrothermal microactuator, it reduces power requirement of electro-thermal microactuator to achieve the same output displacement. Furthermore, the reversible displacement range of microactuator is also found to be enlarged.

Controlling the electroplating conditions under 1.2mA/cm² and 35°C, it is possible to get the structure with no-bending for pure nickel and nickel-diamond nanocomposite comb drive. By the test, it is found that there is an 11.4% improvement in resonant frequency.

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出席國際學術會議報告

會議名稱:2006年亞太地區傳感器暨奈微米科技研討會

APCOT2006

Asia-Pacific Conference of Transducers and Micro-Nano Technology

時間:2006年6月25日至6月28日

地點:新加坡 Singapore

報告人:交通大學機械系博士班研究生 李毅家

一、會議內容

亞太地區換能器暨奈微米科技研討會(APCOT)為亞太地區所舉辦之國際研討會,由 2002 年開始,每間隔兩年,由亞洲地區國家輪流舉辦。

此次為第三屆會議,由新加坡主辦。會議於 25 日報到,26 至 28 日進行會議,每日會議各由 Prof. C.-M. Ho(UCLA)、Prof. T. Laurell (Lund University)、Prof. H. Fujita (University of Tokyo) 作 Plenary Talk,之後則有四個 session 同時進行,會議內容主要分為以下主題

- 01 Theory, Design, Analysis of MEMS and NEMS
- 02 Materials and Device Characterization
- 03 Fabrication Technologies
- 04 Packaging and Assembly Technology
- 05 Mechanical and Physical Sensors
- 06 Chemical Sensors and Microsystems
- 07 Bio Microsystems and Fluidic Systems
- 08 Actuators
- 09 Optical MEMS and Nano-optics (PBG, QD)
- 10 RF MEMS
- 11 Sensing System, Algorithm and Sensor Networks
- 12 Nano Devices and Nano Technology
- 13 Special Session: MEMS/Nano Technology for Data Storage
- 14 Special Session: Power MEMS
- 15 Special Session: Nanowire, Nano-structures and CNT
- 16 Special Session: Packaging Reliability

由會議討論可發現微機電系統的研究趨勢,在尺度上,正逐步由微 米朝向奈米推進;在應用領域上,由傳統工程拓展至分子生物科學。

二、參加會議經過

此會議在Singapore西部的Marina Mandarin飯店舉行,由當地機場至會場約需半小時車程,往返之間交通方便,可搭乘交通車或捷運。主要與會者多半為亞太地區國家,根據會議統計總投稿數571篇論文中,亞太地區佔536篇,歐洲24篇,美洲11篇,而台灣以137篇位居投稿數榜首。

本實驗室在此會議共發表五篇論文,本人所報告的論文題目是 Performance Enhancement of Comb Drive Actuator Utilizing Electroplated Nickel-Diamond Nanocomposite,投於Materials and Device Characterization 的session中,報告之內容主要在於陳述以電鍍製作金屬微結構時,加入鑽 石奈米粒子,發現材料性質的變化,利用其中楊氏係數的提升,將之應用 於共振器(梳狀致動器)製作,並使其共振頻率提升。

此次投稿論文報告張貼時間為27日,過程中須由主講者於張貼之海報旁,並作解說,透過直接面對面的問答,促進彼此之間研究的交流,並且了解在不同國家,不同學校之間,對於微奈米機所著重的方向,以及研究的成果。

除了投稿論文報告外,主辦單位邀請了在微機電領域中著名的學者作 Plenary Talk,作為每天會議的開場,並於會議中穿差受邀請教授之專題 演說。除此之外,在報到當天晚上(25日)以及會議開始第二天(27日)晚上 各有Welcome Reception和Dinner Banquet的活動。

三、與會心得

藉由參與此次會議,了解目前在亞太地區各國發展微機電領域的主流。因應了會議名稱,研究的尺度,由微米朝向奈米推進,所謂朝向奈米尺度並非只是單純將元件製作至奈米等級,而是研究目前在人力所及之下,一些奈米尺度的因子對於元件造成的影響,例如奈米粉末對於材料性質的改變。

各門學科技術,除了學術上的研究外,還必須在產業界能有所應用, 奈微米機電技術也是如此。在奈微米機電技術應用中,最著名的當是加 速規、DLP以及噴墨頭的,這三者開發至今都有十幾年的歷史,在這十幾年間,雖然也有其他元件的開發以及應用,但都未能真正帶起奈微機電蓬勃發展。由三個 Plenary Talk 可發現,目前奈微米機電技術研發的方向已漸漸朝向分子生物科學以及醫療應用邁進,這說明接下來的奈微米機電技術的契機將會在生物科技應用上。

感謝國科會補助,讓作者能順利參與此次會議。此會議為作者首次參加之國際研討會,藉由此次會議,了解國際上奈微米機電的發展狀況, 獲益良多。

四、攜回資料

- 1. 論文集一本
- 2. 論文光碟一片

PERFORMANCE ENHANCEMENT OF COMB DRIVE ACTUATORS UTILIZING ELECTROPLATED OF NICKEL-DIAMOND NANOCOMPOSITE

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The comb drive is demonstrated by nickel electroplating. The nano diamond particles with particle size (diameter) less than 500nm are added into the electroplating solution. By pumping during electroplating, the diamond nanoparticles can be successfully dispersed in the electroplating nickel layers. By the experiment, electroplating is easy to cause bending comb structure. This problem can be solved by tuning the parameters of electroplating conditions which include current density and operation temperature. Under 1.2mA/cm^2 and $35\,^{\circ}\text{C}$ electroplating conditions, it is possible to get the structure with no-bending for pure nickel and nickel-diamond nanocomposite comb drive. It gets 11.4% increase for the first mode resonant frequency.

1. Introduction

For MEMS application, metal has certain advantages over polysilicon, such as high deposition rate, easy stress control, low deposition temperature, and low resistivity. Also by choosing the suitable fabrication process, it is well suited for post processing on preprocessed CMOS wafers.

For further improvement metal properties, Teh et al used the ceramic particles as the additive to electroplate the nickel ceramic composite film in 2002 [2]. It was found that composite film significantly reduced the mismatch of thermal expansion between nickel and silicon. Fortunately, the Young's modulus, Berkovich hardness, and electrical resistivity were maintained.

After nickel ceramic composite film, Teh et al used diamond and cordierite as the additive [3]. The nickel cordierite microresonator was residual stress free because of the better thermal compatibility with silicon. By adding various concentration of diamond particles, it was found that higher diamond concentration obtained the film with more compressively stress.

In 2003, Tsai et al followed Teh's research and used nickel-diamond composite as the material of electrothermal microactuators by electroplating [4]. The E/ρ ratio of microactuators could be enhanced 1.29 times with diamond concentration of 2 g/L. Comparing to device made of electroplated nickel, the electro-thermal microactuators with diamond concentration of 2 g/L could reduce 73% power requirement for 3 μ m output displacement.

According to the phenomenon in pervious researches, the nano diamond particles can increase the E/ρ ratio of nickel. This property is suitable for resonator made by nickel. Its own resonant frequency can be improved by the nano diamond particles. Here, comb drive is used as the tested device and demonstrating that nano diamond particles can improve the resonant frequency of comb drive made by electroplated nickel. Moreover, it must be concerned that diamond particles cause the compressively stress of nickel film. Therefore, a stress free electroplating process is needed to obtain nobending comb structure.

2. Fabrication

In the previous test by our group [4], it was found that the E/ρ ratio and hardness increased with the concentration of nano diamond particles as shown in Figure 1. The film with 2 g/L nano diamond particles has about 1.29 times higher E/ρ ratio than the one made of pure nickel. These properties are helpful to calculate the resonant mode of comb drive. By using FEM software, the resonant frequency can be computed easily.

2.1. Dimension and Simulation

The dimensions of comb drive are decided according to the location of the resonant frequency. By using Ansys, the resonant frequency of comb drive is computed easily. The material properties utilized in the simulation follow the results of previous test mentioned before. The model of comb drive by Ansys is shown in Figure 2.

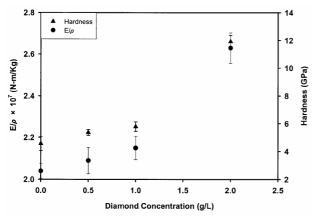


Figure 1. E/ρ ratio and hardness with different concentration of diamond nanoparticles.

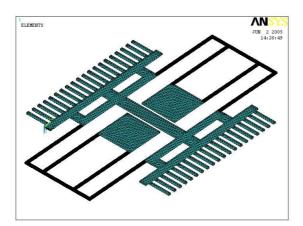


Figure 2. the model the comb drive by Ansys.

The length of spring is designed from 200 μm to 300 μm . By modal simulation, the fist mode is computed, as shown in Table 1. This table shows that there is about 10.2% improvement of the first mode resonant frequency.

Table 1. FEM simulation of the resonant frequency

Material	E (GPa)	ρ (kg/m³)	Spring length (μm)	1st mode Resonant frequency (Hz)
Pure	190	8908	300 250	6804 8052
Nickel			200	9921
Nickel-	230	8882	300	7497
Diamond			250	8872
(2g/L)			200	10932

2.2. Process Flow

The process flow is shown in Figure 3. There are five steps in the whole process.

Step1: After RCA cleaning, wet oxide and LPCVD nitride are deposited on the silicon substrate. FH6400 is spun on the wafer and patterned by Mask#1. Sputtered Ti and Ni are lifted off by FH6400. Here, Ti is the adhesion layer and Ni is the pad material.

Step2: FH6400 is spun on the wafer and patterned by Mask#2' (inverse of Mask#2). Cu is sputtered as the seed layer after hard bake.

Step3: AZ9260 is spun on the wafer and patterned by Mask#3. This AZ9260 layer is the mold of the Ni electroplating.

Step4: Ni and Ni-Diamond are electroplated by using the Cu as the seed layer. The diamond concentration is about 2 g/L in the electroplating solution.

Step5: AZ9260 and Cu seed layer are removed by ACE and the solution of ammonia plus hydrogen peroxide individually. Here the etching of the Cu seed layer must be well control. The structure may peel off because of the over etching. The FH6400 sacrificial layer is removed by PR stripper. At last, the sample is baked dry after IPA immersion.

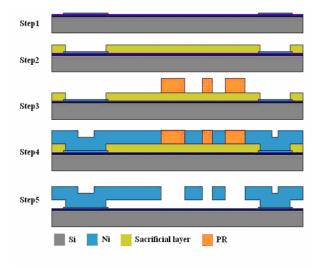


Figure 3. Process flow chart

2.3. Stress Control

There are many factors need to be controlled, the quantities of the compositions and pH level of the solution, current density, temperature, and ventilated system etc. In order to simplify the stress problem, only the current density and temperature are considered as the control variables.

The standard nickel electroplating process is operated at 50 $^{\circ}$ C. The deposition rate increases with the current density. Under 50 $^{\circ}$ C and 10 mA/cm², it is found that the spring of comb drive bends downward, as shown in Figure 4(a). According to the experimental results, it seems helpful to reduce the stress and get flatter structure by reducing the current density. Under the current density less than 1 mA/cm², the deposition rate is too slow. It makes the fabrication process inefficiently. So it is necessary to find another way to solve the stress problem.

Under operation temperature test, it is found that reducing the operation temperature is also helpful to solve the stress problem. Maintaining the current density at 1.2 mA/cm² and reducing operation temperature, it is found that comb drive can be free standing with electroplating temperature at 35 °C. The free standing comb drive is shown in Figure 4(b).

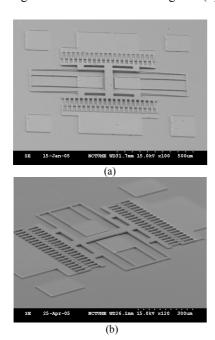
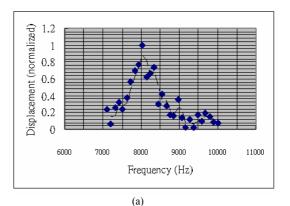


Figure 4. (a) comb drive ade by original process (50 $^{\circ}$ C and 10 mA/cm²). (b) no-bending comb drive made by improved process (35 $^{\circ}$ C and 1.2 mA/cm²).

3. Measurements

The resonant frequency is measured by MEMS Motion Analyzer. This instrument uses both brightfield and interference based illumination modes combined with sophisticated machine vision algorithms to quantify target motions.

Under 50V AC with 50V DC bias and the frequency from 5 kHz to 15 kHz, there is a peak for nickel and nickel-diamond comb drive, individually. The results of measurement are shown in Figure 5. The first mode of pure nickel comb drive with 300 μ m spring length occurs at 8.034 kHz. The nickel-diamond one resonates at 8.949 kHz. There is an 11.4% improvement in resonant frequency.



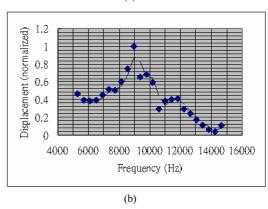


Figure 5. Measured frequency response of comb drive made by (a) pure nickel (resonant frequency=8.034 kHz); (b) nickel diamond (resonant frequency=8.949 kHz).

4. Discussion

Controlling the electroplating conditions under 1.2mA/cm² and 35°C, it is possible to get the structure

with no-bending for pure nickel and nickel-diamond nanocomposite comb drive. By the test, it is found that there is an 11.4% improvement in resonant frequency. This value is close to 10.2% which is gotten by simulation.

The mismatch of the first mode resonant frequency between simulation and measurement may result from the fabrication control ability. For example, the geometry of the PR mode, the sidewall of the PR mold is not easy to control as a perfect vertical sidewall. It results in the geometry mismatch between simulation and experiment.

Except the geometry problem, the unstable of material properties is another issue. It seems that different run obtains different material properties. The deviation of the material properties between different run may be caused from the quality of electroplating solution. Although it is unstable in material properties, the Young's modulus of nickel-diamond is always larger than nickel's.

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