# Performance Improvement of an Electrothermal Microactuator Fabricated Using Ni-Diamond Nanocomposite

Li-Nuan Tsai, Guang-Ren Shen, Yu-Ting Cheng, Member, IEEE, and Wensyang Hsu

Abstract—In this paper, a low-temperature stress-free electrolytic nickel (EL) deposition process with added dispersed diamond nanoparticles (diameter  $< 0.5 \ \mu m$ ) is developed to synthesize Ni-diamond nanocomposite for fabricating electrothermal microactuators. Device characterization reveals dramatic performance improvements in the electrothermal microactuator that is made of the nanocomposite, including a reduction in the input power requirement and enhanced operation reliability. In comparison with the microactuator made of pure nickel, the nanocomposite one can save about 73% the power for a 3  $\mu m$ output displacement and have a longer reversible displacement range, which is prolonged from 1.8  $\mu$ m to more than 3  $\mu$ m. Furthermore, the nanocomposite device exhibits no performance degradation after more than 100 testing cycles in the reversible regime. The enhancements increase with the incorporation of the nanodiamond in a nickel matrix, so the Ni-diamond nanocomposite has potential for application in MEMS fabrication. [1610]

#### I. INTRODUCTION

**E** LECTROTHERMAL microactuators have been widely utilized for the fabrication of microgrippers [1], microswitches [2] and micromanipulators [3], with electrical, optical and biomedical applications [4]–[6]. The microactuators provide microscale mechanical movements with an electrical energy input, that is based on the thermal expansion property of a material. The device can be fabricated using existing integrated circuitry (IC) manufacturing infrastructures and processes, and so has drawn much research attention. A variety of electrothermal microactuators have been developed recently [7]–[9]. Some of the devices have been made of polysilicon using silicon-based micromachining processes [10], [11] and others are made of electroplated metal. Metal-based electrothermal microactuator has several advantages over polysilicon-based one: they can provide a larger output displacement with a smaller input voltage, can easily achieve

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thick structure by electroplating (~ 16  $\mu$ m/hr) for large output force, and can be fabricated at low temperature (~ 50 °C) without having a high-temperature polysilicon doping step. For low-cost and CMOS-compatible applications, such metal-based technology provides an alternative means of manufacturing MEMS-based microactuators. However, the metal-based electrothermal microactuator generally suffers from mechanical deficiencies, such as fatigue and aging [14], [15]. In general, at temperatures higher than 350 °C, nickel structures will degrade and have an irreversible darkening on the surface along with the actuator stroke due to surface oxidation [16]. Although low operational temperature can effectively prevent the actuator from oxidation, it could result in poor performance including smaller output force and displacement.

In 2002, Teh et al. [17] proposed the incorporation of diamond nanoparticles into an electroless nickel matrix to enhance greatly the overall stiffness of the nickel film due to the extreme hardness, stiffness, and temperature resistance of the diamond particle. As more diamond nanoparticles are incorporated into the matrix, the residual stress in the composite film is reduced, making it more suitable for fabricating suspended microstructures. In comparison with the electroless plating technique, electrolytic nickel (EL) plating has been a very important fabrication technique for 3D-MEMS due to the characteristics of lower processing temperature and better process stability and controllability. Diamond has also much higher hardness and Young's modulus than the EL. They are 75 and 1050 GPa for diamond and 6.7 and 200 GPa for EL, respectively. Thus, this paper is aimed to present a newly developed CMOS-compatible electrolytic Ni-diamond nanocomposite synthesis for fabricating the electrothermal microactuators. Effects on the performance of electrothermal microactuators resulted by the incorporation of diamond nanoparticles are also further investigated in terms of the mechanical strength, coefficient of thermal expansion (CTE) of the nanocomposite material, and the improvements of power consumption and operational reliability in the devices.

### II. 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  $\mu$ m wide, 6  $\mu$ m thick cantilevers shown in Fig. 1 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  $(E/\rho)$  ratio. The beam lengths are

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Fig. 1. Schematic diagram of cantilevers for material characterizations.



Fig. 2. Fabrication process flow of cantilevers: (a) 1  $\mu$ m FH-6400 PR deposition and patterning. (b) sputtered Ti/Cu (100 Å/1000 Å) as an adhesion and seed layer. (c) 10  $\mu$ m AZP-4620 PR deposition and patterning as the molding structure. (d) electrolytic nanocomposite plating. (e) structure releasing.



Fig. 3. EDS spectra of the (a) pure Ni film and (b) the nickel-diamond nanocomposite film.

designed as 150  $\mu$ m, 250  $\mu$ m, 350  $\mu$ m, and 450  $\mu$ m, respectively. Fabrication of the cantilevers is schematically illustrated in Fig. 2. The fabrication starts with a 1  $\mu$ m thick photoresist (PR) deposition, which is patterned as a sacrificial layer on a 4" n-type silicon substrate [see Fig. 2(a)]. A thin 100 Å Ti/1000 Å Cu film is then DC-sputter deposited as an adhesion and

plating seed layer [see Fig. 2(b)]. Another 7  $\mu$ m PR is coated and patterned to form the electroplating mold [see Fig. 2(c)] and electrodeposition of Ni or Ni-diamond nanocomposite is performed to construct the structural layer of the cantilevers at 50 °C [see Fig. 2(d)]. The plating current density is controlled at  $10 \text{ mA/cm}^2$ . Finally, the beam structures in Fig. 2(e) are then released after sequentially stripping the sacrificial layer by acetone (ACE), rinsing in isopropylalcohol (IPA) and deionized (DI) water, as well as drying by compressed air. Once the Cu seed layer underneath the released beams is removed by immersing in a diluted  $H_2SO_4$  solution for 5 s, the cantilevers are completely fabricated for following material characterizations. In the experiment, the nanocomposite films are synthesized in different plating solutions, which have different weight fractions of the diamond nanoparticles ranging from 0 to 2 g/L in the nickel baths. Table I lists the composition of the nickel-plating bath used in the experiment. The average size of the added diamond nanoparticles is below 500 nm in diameter.

The composition of the electroplated nanocomposite layer is first identified by energy dispersive spectrum (EDS). Fig. 3(a) shows the EDS of the cantilever beam made of pure Ni indicating that the electroplated layer contains very little carbon (C) in addition to Ni, Si, and O, which could be attributed to surface contamination. As compared, the stronger carbon signal in the EDS of the Ni-diamond nanocomposite shown in Fig. 3(b) indicates the existence of large amount of carbon content inside the film. In order to further verify that the carbon signal is originated from the diamond nanoparticles, X-ray diffractometer (XRD), Philips X'Pert Pro (MRD), is then utilized to compare the pure Ni and Ni-diamond nanocomposite films. The X-ray source is a Cu- $k\alpha$  line with the wavelength of 0.154 nm. The detection is made at a low angle  $(2\theta)$  in the range of  $20^{\circ}$  to  $60^{\circ}$ . Fig. 4(a) shows the diffraction spectra of the pure Ni film. The Ni (111) and Ni (200) peaks appear at the angles of  $45^{\circ}$  and  $53^{\circ}$ , respectively. The corresponding interlayer spacings of  $d_{111}$  and  $d_{200}$  are 2.012 Å and 1.725 Å, respectively. The same Ni peaks are also found in the XRD spectra of the Ni-diamond nanocomposite as shown in Fig. 4(b). In addition, the C (102) peak around  $2\theta = 50.5^{\circ}$  with corresponding 1.801 Å interlayer spacing indicates the existence of crystalline diamond in the composite film. Once the inclusion of nanodiamond in Ni matrix is verified, the nanocomposite films and cantilevers are used for the following mechanical property characterizations.

The hardness of the nanocomposite film is characterized by the nanoindenter [18], XP system of the America MTS Company. Fig. 5 shows the hardness increases with the concentra-

Composition	Quantity
Nickel Sulfamate, Ni (SO <sub>3</sub> NH <sub>2</sub> )·4H <sub>2</sub> O	400 g/L
Nickel Chloride, NiCl <sub>2</sub> ·6H <sub>2</sub> O	5 g/L
Boric Acid, H <sub>3</sub> BO <sub>3</sub>	40 g/L
Wetting Agent	5 g/L

 TABLE I

 The Composition of Ni Plating Bath



Fig. 4. X-ray diffraction spectra of the electrolytic (a) pure Ni film and (b) the nickel-diamond nanocomposite film. The carbon peak C (102) indicates the existence of diamond crystalline.

tion 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  $E/\rho$  ratio of the films can be derived from resonant frequen-

cies of the cantilevers that are made of the EL with different amounts of diamond nanoparticle incorporations. The relationship between  $E/\rho$  ratio and resonant frequency is determined by the following (1) [19]:

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



Fig. 5. Hardness and  $E/\rho$  ratio of the EL films plated with various nano-diamond concentrations.

where  $E, \rho, h$ , 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  $E/\rho$  ratio. Table II lists one set of frequency measurement results of 350  $\mu m$  long, 50  $\mu m$  wide cantilevers. The actual thicknesses of the beams plated in four different baths are 5.5  $\mu$ m (pure Ni), 5.08  $\mu$ m (0.5 g/L diamond), 5.06  $\mu$ m (1 g/L diamond), and 4.4  $\mu$ m (2 g/L diamond), respectively, instead of the original targeted 5  $\mu$ m thickness. In addition, before measuring, we used Tetramethylammonium hydroxide (TMAH) to further etch away the silicon underneath the cantilevers in order to prevent the beams being stuck to the substrate during vibration. Thus, the actual beam length is typically longer than its original design due to the undercut effect as shown in Fig. 6. Part of silicon under the beam anchor is etched, so the edge of the anchor pad is freely suspended and the stiffness of the structure is reduced [20]. The undercut effect results in the underestimation of  $E/\rho$  ratio. After taking both process variations into account, a more accurate  $E/\rho$  ratio is extracted and listed in Table II.

The derived  $E/\rho$  values corresponding to different concentration of nanodiamonds in plating bath are plotted in Fig. 5. Similar trend of hardness enhancement with increasing concentration is obtained. Total of 32 samples from eight different wafers were measured. Each data point presented is an average value obtained from the measurement of four samples on two different wafers, which were plated with the same condition but in two individual runs. The error bars represent the process variation between two identical plating conditions. The nonlinear behavior is most likely induced by process variation and can be eliminated by taking averages of more measured data. The cantilever fabricated in a plating bath with 2 g/L diamond nanoparticles has about 1.29 times higher  $E/\rho$  ratio than the one made of pure nickel. The mechanical property enhancements due to the incor-



Fig. 6. Cross-sectional SEM photograph near the edge of anchor pad with the undercut effect.

poration of diamond nanoparticles into nickel matrix ensure the electrolytic process for the further fabrication of electrothermal microactuators.

## III. DESIGN, FABRICATION, AND MEASUREMENT OF THE ELECTROTHERMAL MICROACTUATOR

The electrothermal microactuator with long-short beam configuration [22] as shown in Fig. 7 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  $\mu$ m gap spacing between them. Both beams have the same width and thickness. The longer beam is about 800  $\mu$ m long, 10  $\mu$ m wide, and 9  $\mu$ 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. Fab-

 TABLE
 II

 Measured Frequencies of the Designed 5  $\mu$ m Thick, 350  $\mu$ m Long Cantilevers Plated With Various Ni-Diamond Nanocomposites

Concentration of Diamond Nanoparticles (g/L)	Measured Beam Thickness (µm)	Effective Beam Length <sup>1</sup> (μm)	Measured Resonant Frequency (KHz)	<i>E/ρ</i> ratio <sup>2</sup> (N·m/Kg)	<i>Ε/ρ</i> ratio <sup>3</sup> (N·m/Kg)
0	5.5	535	14.031	3.6x10 <sup>6</sup>	2.04x10 <sup>7*</sup>
0.5	5.08	409	22.436	$1.12 \text{x} 10^7$	$2.09 \times 10^7$
1	5.06	372	27.367	$1.32 \times 10^7$	2.15x10 <sup>7</sup>
2	4.4	352	29.45	$2.57 \times 10^7$	$2.64 \times 10^7$

 $^1\text{The}$  effective beam length is equal to the designated beam length (350  $\mu\text{m})$  plus the undercut length.

Before considering the process variations.

<sup>3</sup>After considering the process variations.

\*Typical  $E/\rho$  ratio is about 2.02x10<sup>7</sup> N·m/Kg (=180GPa/8908Kg/m<sup>3</sup>). [21]



Fig. 7. Schematic diagram of the electrothermal microactuator with a long-short-beam design.



Fig. 8. SEM photograph of the fabricated electrothermal microactuator using Ni-diamond nanocomposite plated with the concentration of 2 g/L nanodiamonds.

rication of the electrothermal actuator is similar to that of the cantilevers (described in Section II) with the exception that a  $2-\mu m \operatorname{SiO}_2$  film is used as a sacrificial layer instead. The sacrificial oxide layer is removed by etching it in HF solution for 10 s. A SEM photograph of the completed electrothermal actuator is shown in Fig. 8. Device testing is conducted on a probe station with an automated CCD image system with 0.2  $\mu m$  resolution. Fig. 9 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- $\mu m$  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  $3-\mu m$  displacement, the microactuator made of the Ni-diamond nanocomposite plated in a bath with 2 g/L concentration of nanodiamond 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 3  $\mu m$ . In comparison with the pure nickel one, which exhibits irreversible deformation once the output displacement is over 1.8  $\mu$ m, the nanocomposite actuator can indeed provide a better reversible characteristic. Fig. 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  $\mu$ m. 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  $\mu m$  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.

#### **IV. DISCUSSIONS**

The CTE measurement and ANSYS simulation are performed to investigate the effects that the nanocomposite may have on electrothermal actuator performance. Equation (2) shows an analytical relation between the output displacement



Fig. 9. Input power versus displacement of the microactuators made of the nanocomposites plated with different nanodiamond concentrations.



Fig. 10. Cycling tests of the microactuators made of pure Ni and the nanocomposites plated with the concentration of 2 g/L nanodiamonds.

and the CTE of the structural material of electrothermal microactuator [9]

$$\delta = \alpha (\gamma_{\text{long}} \cdot \Delta T_{\text{long}} - \gamma_{\text{short}} \cdot \Delta T_{\text{short}})$$
(2)

where  $\delta$  is the displacement of beam tip,  $\alpha$  is the CTE, and  $\gamma_{\text{long}}$ ,  $\gamma_{\text{short}}$ ,  $\Delta T_{\text{long}}$ , and  $\Delta T_{\text{short}}$  are the conversion factors and average temperature changes of the long and short beams, respectively. Since the conversion factor is only a function of the device geometry which is independent of material properties, CTE

increase could be the major factor to enhance the device performance. In order to verify the supposition, an in-house apparatus shown in Fig. 11 is utilized for the CTE measurement of the electroplated Ni-diamond nanocomposite film. By measuring the elongation of cantilever in a heated chamber with temperature control, one can obtain the CTE by (3)

$$\alpha(T_1) = \frac{(L_1 - L_0)}{L_0} \cdot \frac{1}{\Delta T}$$
(3)

where  $L_1$  is the beam length at temperature  $T_1$ ,  $L_0$  is the initial beam length at temperature  $T_0$ , and  $\Delta T = T_1 - T_0$ . Fig. 12



Fig. 11. Schematic diagram of the CTE measurement apparatus.



Fig. 12. CTE values of the nanocomposites plated with various nanodiamond concentrations at 400  $^\circ\mathrm{C}.$ 

shows the CTE's of the nanocomposites with different diamond nanoparticle concentrations at 400°C. The CTE value increases with the concentration of diamond nanoparticle starting from the  $23 \times 10^{-6}$  °C of pure nickel to the  $50 \times 10^{-6}$  °C of the nanocomposite plated in a bath with 2 g/L of diamond nanoparticle. That is more than two-fold of increasing in CTE values contributed by the diamond particles. Once the CTE values are determined, the commercial finite element simulator, ANSYS 6, is then utilized to simulate the electro-thermo-mechanical behavior of the microactuator in which the element type of solid 5 is applied for the analysis. TEM analysis of the nanocomposite plated with the condition of 2 g/L nano-diamond concentration was performed. Fig. 13 is a dark field image taken from the nanocomposite film and it indicates that most of the diamond nanoparticles distribute in the grain boundary regions of the nickel matrix. On the other hand, according to the elemental analyzer (EA) measurement, the diamond content of the corresponding nanocomposite film is only 0.185% in weight fraction and 0.47% in volume fraction, which are calculated based on the density of nickel (8.908  $g/cm^3$ ), the density of diamond particle  $(3.51 \text{ g/cm}^3)$  and the assumption of no existence of voids



Fig. 13. Dark field TEM image of the fabricated electrolytic Ni-diamond (2 g/L) microactuator, indicating diamond nanoparticles distribute in the region of grain boundary.

inside the films. Thus, the material properties of the nanocomposite for the simulation, such as the thermal conductivity and specific heat capacitance, are assumed to be the same as that of pure nickel for the simulation due to such small amount of diamond incorporation. Meanwhile, according to the rule of the mixture [23], [24] and the previous measurement result of the  $E/\rho$  ratio, the effective Young's modulus and density for the Ni-diamond nanocomposite plated in a bath with 2 g/L concentration of nano-diamond can be derived as 233.5 GPa and 8.882 g/cm<sup>3</sup>, respectively. Table III lists the related data about the mechanical and thermal properties of the Ni and Ni-diamond nanocomposite for finite element analysis [25], [26].

In the analysis, only conductive heat transfer mode is considered since heat convection can be neglected at moderate temperature [27], [28]. Most of the heat generated in the electrothermal actuator will be transferred to device's anchor pads or through air gap to silicon substrate by conduction mechanism. Besides, the anchors were assumed at a constant temperature 25 °C because the anchor pad is much larger than the heating element of the actuator in the design. Fig. 14 shows the simulation results.



Fig. 14. Comparison of the experimental data with the ANSYS analysis in terms of the output displacements of the microactuators at different applied voltages.

TABLE III					
MATERIAL PROPERTY LIST FOR ANSYS SIMULATIONS					

	Pure Ni	Ni-diamond (2g/L)	
Modulus of Elasticity (GPa)*	181.7	233.5	
Density (kg/m <sup>3</sup> )*	8908	8882	
Thermal Expansion Coefficient (10 <sup>-6</sup> /°C) at 400°C	23	50	
Poisson Ratio	0.31		
Thermal Conductivity (W/mK)	90.5		
Specific Heat (J/Kg-K)	443.08		
Resisvitity (μΩ-cm)	13.5		

\*The data are calculated based on the rule of mixture [23,24] and the experimental results of the EA analysis and  $E/\rho$  measurement.

The relation between output displacement and input voltage is closely fitted with experimental data in the low input voltage region (< 0.4 V), indicating that CTE has evident effect on the displacement of electrothermal microactuator. With the incorporation of diamond nanoparticles in the nickel matrix, the CTE increases as well as the output displacement of electrothermal microactuators. Regarding the deviation between the simulation and experimental results, it could be attributed to the temperature dependence of the electrical resistance of pure nickel and nanocomposite and the mentioned neglect of heat convection mechanism in the thermal analysis.

In addition to displacement, the output force of the actuator is also analyzed using ANSYS simulator with the same boundary conditions. Fig. 15 shows the comparison of the output forces between the actuators made of pure Ni and Ni-diamond nanocomposite at different input voltage. The result shows the electrothermal actuator made of Ni-diamond nanocomposite can provide larger output force than that made of pure Ni while both devices are operated with the same input voltage. Furthermore, from Figs. 9, 14, and 15 it is found that the incorporation of diamond nanoparticles into Ni matrix can enhance the output force and displacement of microactuator simultaneously. For about 0.4 V input, the actuator made of Ni-diamond nanocomposite has 2.23  $\mu$ m displacement and 10  $\mu$ N output force, which are larger than that made of pure Ni only with 0.27  $\mu$ m displacement and 1.9  $\mu$ N output force. The result infers that the nanocomposite has better energy conversion than pure Ni which is resulted by the incorporation of diamond nanoparticles. Further investigation is required.

#### V. CONCLUSION

A low-temperature stress-free deposition process of the ELdiamond nanocomposite for fabricating cantilevers and electrothermal microactuators has been successfully demonstrated here. Test results indicate that the incorporation of diamond



Fig. 15. Comparison of the output force of the actuators made of pure Ni and Ni-diamond nanocomposite respectively at different applied voltages.

nanoparticles effectively modifies the material properties of pure EL film, such as hardenss,  $E/\rho$  ratio, and CTE, making it a more suitable candidate as structural materials for electrothermal microactuators. Furthermore, the performance enhancements in the electrothermal microactuator fabricated using Ni-diamond nanocomposite show the potential applications of the process for improving many MEMS devices, especially in terms of the characteristics of stronger mechanical strength and better electrical-to-mechanical energy conversion.

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