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What is This?
Cobalt–nickel microcantilevers for biosensing

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Abstract
We present microcantilevers that utilize magnetic actuation for use as mass sensors for bioapplications. The microcantilevers are made of electroplated cobalt–nickel that has low coercivity and high saturation magnetization. The microcantilevers are actuated by applying magnetic fields, and the deflection is measured using a laser Doppler vibrometer. The microfabrication of the microcantilevers is based on two lithography steps, an electroplating step and a sacrificial layer etching step. The magnetic actuation and optical readout using the fabricated cobalt–nickel microcantilever were successfully demonstrated in air under atmospheric pressure and in deionized water. A feedback circuit is used to enhance the quality factor of the microcantilever. The quality factor increased from approximately 550 to 1600 in air and from 7.3 to 10.6 in deionized water. The microcantilevers can be readily functionalized with selective binding molecules and used as a biomass sensor.

Keywords
Cobalt–nickel, microcantilevers, Q factor, Q enhancement, feedback, magnetic actuation

Introduction

The growing demand for biosensing applications has resulted in a great interest in cantilever-based microresonators (Suter et al., 2011). Dynamic mode sensing is usually preferred as it provides a higher sensitivity in mass measurements compared to static deflection measurements (Lee et al., 2005; Wu et al., 2001). In this article, we present the fabrication, magnetic actuation, and optical readout of magnetic microcantilevers (Figure 1). The cantilever is actuated using electromagnets, and its dynamic deflection is detected by a laser Doppler vibrometer (LDV). The simple fabrication and wireless readout makes our microresonators low cost and passive.

In microsystem development, fabrication remains the main challenge. Several techniques, such as sputtering (Budde and Gatzen, 2006), screen-printing (Park and Allen, 1998), and microassembly (Cheng et al., 2009), have been used for the implementation of magnetic materials in micro- and nanodevices. However, obtaining thick, high-quality, well-controlled magnetic layers with these methods is challenging. Recently, electrodeposition has emerged as a reliable and simple method for the fabrication of thick-patterned microstructures (Ergeneman et al., 2012; Guan and Nelson, 2005). Since electrodeposition methods do not require the use of high temperatures and vacuum, they are inherently less expensive. Moreover, by controlling bath compositions and deposition parameters, it is possible to achieve a high level of tunability in the chemical composition and the mechanical and magnetic properties of electrodeposited materials (Myung et al., 2003). The magnetic properties of the microdevices are critical to overall system performance. In this study, electrodeposited cobalt–nickel (CoNi) alloys were used as they have excellent mechanical properties and can exhibit different magnetic behavior depending on both the

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composition of the alloy and the crystallographic structure (Ergeneman et al., 2011; Pellicer et al., 2011). The CoNi alloy used has low coercivity and high saturation magnetization. Although, other more widely used materials such as NiFe or CoNiFe exhibit lower coercivities and higher saturation magnetization values, the presence of Fe leads to poorer corrosion resistance in these alloys (Kola and Davis, 2010). The CoNi device layer is fixed on an SU-8 polymer anchor layer. The SU-8 is used widely in microsystems due to its stability and good mechanical properties. In Schmid and Hierold (2008), the anchor loss of a cantilever with SU-8 anchor is investigated. It is shown that the anchor loss for single-clamped polymer cantilevers with SU-8 anchors was negligible. The anchor loss is mainly important if the cantilever is used in low-environmental damping. Due to strong damping of water, the anchor loss should play a minor role for this application.

When used as a mass detector, the sensitivity of a microresonator depends on the spectral resolution, which is directly related to the quality factor (Q) of the resonance mode (Sandberg et al., 2005). Due to viscous damping mechanisms, all microresonators experience high damping in liquids. Typically, the Q in liquids is between 1% and 10% of that in air/vacuum. Many groups have investigated different methods to overcome this problem. In Schmid et al. (2008), a self-oscillating system with feedback loops was used to increase the Q of polymer cantilevers. In Vidic et al. (2003), the Q of silicon cantilevers was enhanced by adding a magnetic layer. Higher oscillation modes of the microcantilevers were also investigated to have higher Qs implying that the higher-order modes will yield higher sensitivities in sensing applications (Dohn et al., 2005; Sandberg et al., 2005). The feedback system designed and used for the actuation of the magnetic microcantilevers in this study results in a significant improvement in the Q.

Fabrication

The CoNi microresonators are fabricated using standard microfabrication techniques combined with enhanced electroplating techniques. The microfabrication of the device is based on two lithography steps, an electroplating step and a sacrificial layer etching step. The fabrication sequence is shown in Figure 2. Si wafers are used as substrate. They are cleaned by immersing in acetone and isopropanol under sonication and then in piranha solution. They are plasma ashed at 600 W for 10 min. A 25-nm adhesion Ti layer (not shown) and a 500-nm sacrificial Cu layer are deposited by e-beam evaporation (1). The Cu layer also acts as a seed layer for electrodeposition. A negative photoresist (AZ4562; Microchem Inc.) is used for the first photo lithography (2) and the device layer is formed by direct current (DC) electroplating of CoNi. After electrodeposition, the photoresist is removed (3). Adhesion of SU-8 to metals is generally weak; hence, the adhesion promoter Omnicoat (Micro Chem Inc.) is used to improve the adhesion between SU-8 and CoNi (not shown) (Dai et al., 2005; Nordstrom et al., 2005). A thin layer of Au (i.e. 10 nm) can be electroplated after CoNi electrodeposition to further enhance the adhesion between SU-8 and CoNi. A 90-μm-thick SU-8 layer is spin coated on the wafer (4) and exposed with the anchor mask. After SU-8 development, the anchor layer is formed (5). The wafer is diced, and the devices are released by etching the sacrificial Cu layer (6).

Characterization of the CoNi microcantilevers

The magnetic characteristics are crucial for the performance of the CoNi microcantilevers. Low coercivity and high saturation magnetization values are desired for actuation. The magnetization characteristics of the CoNi used to make microcantilevers are obtained using a vibrating sample magnetometer (VSM) (Micromag.

Figure 1. CoNi microcantilevers with SU-8 anchor. CoNi: cobalt−nickel.

Figure 2. Fabrication of the CoNi microresonators with SU-8 anchor. CoNi: cobalt−nickel.
an unoccluded optical path to the cantilevers. The two DC magnets are placed in a Helmholtz configuration to magnetize the microcantilevers. The electromagnet generates a gradient in both axial and radial directions of the coil. The size and the location of the coils were optimized by finite element method (FEM) simulations with COMSOL Multiphysics. The magnetic force, $F$ (N), on the microresonator depends on the magnetization of the CoNi beam, $M$ (A/m), and magnetic field gradient generated by the actuation coil (Abbott et al., 2007).

$$F = \mu_0 n (M \cdot \nabla)H$$

where $\mu_0$ (N/A²) is the permeability of free space, $n$ (m³) is the volume of the microresonator, $\nabla$ is the gradient operator, and $H$ (A/m) is the applied magnetic field. The magnetic torque that tends to align the magnetization of the object with the applied field is

$$T = \mu_0 \nabla \times (M \times H)$$

in units N m. The magnetic torque depends on the magnetization of the CoNi beam, $M$ (A/m) and on the magnetic field generated by the actuation and Helmholtz coils.

Due to the shape anisotropy of the microcantilevers, the magnetization remains in-plane. The microcantilever can be excited in its first mode (i.e. out-of-plane) with the magnetic force and torque.

The microcantilevers can be operated in two modes: (a) AC only and (b) AC and DC modes. In the AC only mode, the excitation is performed with the AC actuation coils without the presence of DC permanent magnets. The magnetization of the microcantilever and the magnetic field and, hence, the magnetic field gradient change sign simultaneously. In this mode, the magnetic force and torque are unidirectional as can be seen from equations (1) and (2). The magnetic force and torque signal on the microcantilever is a rectified sinusoid. This signal has its main frequency component at the second harmonic of the applied input frequency. This results in a frequency-doubling effect for the deflection of the microcantilever. When the AC signal is applied together with the DC magnetic field the frequency-doubling effect does not occur. Since the DC magnetic field is much stronger than the AC magnetic field component, the microcantilever is magnetized by the DC magnetic field. In this mode, the magnetization does not change sign with the applied AC field, and the magnetic force and torque are bidirectional. The deflection occurs at the same frequency as the applied AC frequency.

The initial load, ambient conditions, process, and material imperfections affect resonance characteristics of microresonators, and they are studied in detail in literature (Preidikman and Balachandran, 2006). The setup used in this work is designed to minimize these effects. Due to the DC magnetic field, a magnetic

3900; Princeton Measurements Corp.). Figure 3 shows the magnetization of CoNi film as a function of the applied magnetic field. The deposits showed a saturation magnetization of 680 kA/m (0.85 T) and a coercivity of 1.6 kA/m (20 Oe). The composition of the CoNi deposit was found to be 50% Co and 50% Ni and was analyzed by energy-dispersive X-ray spectroscopy (EDX).

**Actuation**

The two alternating current (AC) electromagnets and two DC magnets (i.e. permanent ring magnets) are utilized to actuate the CoNi microcantilevers. The setup to actuate the CoNi microresonator is shown in Figure 4. The deflection of the microcantilevers is detected with a LDV. The deflection occurs at the same frequency as the applied AC frequency.

The initial load, ambient conditions, process, and material imperfections affect resonance characteristics of microresonators, and they are studied in detail in literature (Preidikman and Balachandran, 2006). The setup used in this work is designed to minimize these effects. Due to the DC magnetic field, a magnetic
torque could act on the cantilevers. However, when the AC actuation is compared with AC + DC actuation, no significant change in resonance characteristics was observed.

In order to find the damped resonant frequency and the Q of the microcantilever, the excitation frequency is swept over a band that includes the resonant frequency of the microcantilever. At the resonant frequency, the deflection amplitude of the microcantilever is maximum, and the phase lag between excitation signal and the deflection signal changes distinctively. Either effect can be used to detect the resonant frequency.

Q enhancement
The mass sensitivity and the minimal detectable mass of a microresonator sensor depend on the Q. Positive feedback can be used to increase the Q of a microresonator working in gaseous or aqueous environments (Tamayo et al., 2001; Vidic et al., 2003; Schmid et al., 2008). For positive feedback, the readout signal is amplified, and the phase is shifted to maintain a phase difference of 90° compared with the drive signal (i.e. resonance) and added to the driving signal.

A feedback circuit is designed to be used for enhancing the Q of microcantilevers. The LDV is used for the detection of out-of-plane deflections of microcantilevers. The LDV outputs the velocity of the microcantilever with a 90° phase shift with respect to the deflection signal. Hence, the readout signal that is used for the feedback system should have no phase difference with respect to the input excitation signal. Figure 5 shows the schematic illustration of the positive feedback system for the Q enhancement. The readout signal is amplified with a variable gain amplifier, and the phase is adjusted to match the phase of the input signal with a variable phase shifter. The phase adjustment is necessary to correct for phase delays due to the electronic components.

The deflection of the microcantilever \( z = A e^{i(\omega t - \phi)} \) can be modeled as a damped harmonic oscillator driven by the input signal \( F_{in} = F_0 e^{i\omega t} \) and the feedback signal \( F_{fb} = G A e^{i(\omega t + \pi/2)} \) (Tamayo et al., 2001).

\[
m \frac{d^2z}{dt^2} + \gamma \frac{dz}{dt} + k z = F_0 e^{i\omega t} + G e^{i\pi/2} \tag{3}
\]

where \( A \) is the deflection amplitude, \( \omega \) is the driving frequency, \( \phi \) is the phase shift with respect to the input signal, \( G \) is the gain of the feedback signal, \( m \) is the effective mass of the cantilever, \( \gamma \) is the damping factor of the system, and \( k \) is the spring constant. The feedback force is proportional to the velocity of the cantilever

\[
\frac{dz}{dt} = j \omega A e^{i(\omega t - \phi)} = \omega A e^{i\pi/2}
\]

\[
F_{fb} = G \frac{dz}{dt}
\]

Equation (3) can be written as

\[
m \frac{d^2z}{dt^2} + \gamma_{eff} \frac{dz}{dt} + k z = F_0 e^{i\omega t}
\]

where \( \gamma_{eff} = \gamma - G/\omega \) is the effective damping resulting from the feedback control. The Q enhancement can be achieved controlling the effective damping of the system.

Results
A LDV (Polytec MSA-500) was utilized to detect the resonance behavior of the CoNi microcantilevers. The microcantilevers are operated in AC only mode and AC and DC mode. In the AC and DC mode, the magnetization of the microcantilevers is significantly higher than in the AC only mode due to the soft magnetic properties of the CoNi. Hence, the AC and DC mode was utilized in further experiments. The positive
feedback system was also utilized to enhance the Q of the microcantilevers.

Figure 6 shows the measurement from the microcantilever with dimensions of 205 \( \mu m \times 15 \mu m \times 4.5 \mu m \) in air under atmospheric pressure and room temperature. As the DC field was applied, the magnetization of the cantilever was induced by the DC field. Hence, the main vibration frequency is the same as the excitation frequency. Figure 6 shows the resonance characteristics when the excitation is performed in the open loop and closed loop. The resonant frequency was found to be 38.75 kHz. The Q increased from approximately 550 (open loop) to 1300 (closed loop) with the positive feedback system.

Figure 7 shows the measurement of the same microcantilever in deionized (DI) water and room temperature. The resonance characteristics were measured when the excitation was performed in the open loop and closed loop. The resonant frequency shifted to 26.5 kHz. The Q also dropped to 7.3 (open loop) and could be increased to 10.6 (closed loop) with the positive feedback system. Figure 8 shows the closed-loop resonance characteristics of this cantilever under atmospheric pressure and in DI water. The shift of the resonance frequency and the decrease in the Q due to damping can be easily seen.

**Conclusion**

A fabrication process was developed to make magnetic microcantilevers using an electroplated CoNi and the polymer SU-8. The Passive microdevices can be made using this process and can be fully released from the substrate to create disposable low-cost sensors. The problems associated with the substrate and device sticking can be avoided with this method. The device can be placed close to the readout or actuation systems,
optical readout can be made from the top or bottom sides. The electroplated CoNi has desirable magnetic properties for microactuators. The successful actuation and readout of the device was demonstrated in air and water. Using a feedback circuit, the Q of the microcantilevers was significantly improved. The microcantilevers can be readily used for biosensing applications. A variety of sensing applications using microcantilevers can be found in Lavrik et al. (2004) and Ziegler (2004).

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**References**


