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ORIGINAL REPORT



Coupling of magnetostrictive FeGa film with single-crystal diamond MEMS resonator for high-reliability magnetic sensing at high temperatures

Zilong Zhang^{a,b}, Yuanzhao Wu^c, Liwen Sang^a, Haihua Wu^a, Jian Huang^b, Linjun Wang^b, Yukiko Takahashi^a, Runwei Li^c, Satoshi Koizumi^a, Masaya Toda^{b,d}, Indianto Mohammad Akita^d, Yasuo Koide^a and Meiyong Liao^{b,a}

^aNational Institute for Materials Science, Tsukuba, Japan; ^bSchool of Materials Science and Engineering, Shanghai University, Shanghai, People's Republic of China; ^cChinese Academy of Sciences Ningbo, People's Republic of China; ^dGraduate School of Engineering, Tohoku University, Sendai, Japan

ABSTRACT

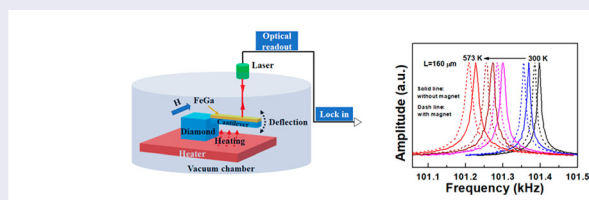
Micro-electromechanical systems (MEMS) magnetic sensing at high temperatures over 573 K has not been achieved due to the difficulty in designing the rational interface with strong coupling. Here, a magnetic sensor based on FeGa/single-crystal diamond (SCD) MEMS resonator is realized to circumvent the challenges in magnetic sensing. The present magnetic sensor exhibits a stable high-sensitivity of 7.3 Hz/mT at 573 K and an experimental noise level of $4\text{ nT}/\sqrt{\text{Hz}}$. The performance exceeds those of reported high-temperature magnetic sensors. This work offers a strategy for developing magnetic sensors with merits super to the existing ones.

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KEYWORDS

Single-crystal diamond; galfenol (FeGa); magnetostrictive effect; MEMS



IMPACT STATEMENT

The first high-temperature MEMS magnetic sensor over 573 K super to the present ones is realized by coupling a magnetostrictive FeGa film with a diamond MEMS resonator.

1. Introduction

Noncontact magnetic sensing has attracted broad attention due to the various applications in precise biomedical diagnosis, infrastructures maintenance, transportation management and wheel speed monitoring, etc [1,2]. The current magnetic sensors still have various prominent weakness, for example, high cost, low-temperature requirement, and complex infrastructure for SQUID, low sensitivity for Hall sensor, high volume and power consumption for fluxgate sensor, and restricted magnetic field magnitude for GMR sensor [3,4]. Micro-electromechanical systems (MEMS) magnetic sensors yield prominent advantages over aforementioned sensors with small dimensions, batch fabrication, low-power consumption, and facile integration with CMOS

technology [3,5,6]. However, the present MEMS magnetic sensors are at initial stage, having low sensitivity and poor reliability issues, especially under harsh environments.

Single-crystal diamond (SCD) presents a promising material for high performance and high-reliability MEMS super to other semiconductor materials in terms of its outstanding mechanical strength, as reported by our group and others [7,8]. Unfortunately, the practical applications of SCD MEMS integrating with other materials under harsh environments have not been achieved due to the lack of device concepts. Galfenol (FeGa) has a huge magnetostrictive coefficient and excellent thermal stability with an ultra-high Curie temperature of 675°C. In view of the excellent intrinsic thermal stability of SCD

CONTACT Meiyong Liao ✉ meiyong.liao@nims.go.jp National Institute for Materials Science, Namiki 1-1, Tsukuba, Ibaraki 305-0044, Japan; Jian Huang ✉ jianhuang@shu.edu.cn School of Materials Science and Engineering, Shanghai University, Shanghai 200444, People's Republic of China

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and FeGa, SCD MEMS coupling with a FeGa film based on the magneto-strictive effect provides an ideal system for magnetic sensing with high performance and high reliability.

Here, we successfully develop the thermally-stable high-temperature magnetic sensor based on the device concept of FeGa/SCD MEMS resonator. The magnetic sensor exhibits a high magnetic sensitivity of 7.3 Hz/mT at 573 K, a minimum detectable magnetic field of 159 pT, and an measured noise floor of $\sim 4\text{ nT}/\sqrt{\text{Hz}}$ at 573 K, super to the present high-temperature magnetic sensors. The coupling of SCD MEMS resonator with the magneto-strictive material provides a promising strategy for magnetic sensing with high sensitivity and high reliability under extreme environments.

2. Experimental

The SCD cantilevers were fabricated by a smart-cutting method, leading to a SCD-on-SCD structure (Figure S1, Supplemental Information) [7,9]. The as-fabricated cantilevers suffered from the bending due to surface stress, depending on the thickness (Figure S2). The FeGa films were deposited on the SCD cantilevers and substrates by the radio frequency magnetron sputtering with nearly uniform thickness. A series of FeGa/SCD samples were annealed at various temperatures for 1 h. The surface morphologies and microstructures of the FeGa films were examined by atomic force microscopy (AFM), transmission electron microscope (TEM), X-ray diffraction (XRD). The magnetic properties of FeGa films on flat SCD were measured by the vibrating sample magnetometer (VSM) and the physical properties measurement system (PPMS). The resonance frequency of SCD cantilever was measured optically by a Laser Vibrometry (LV 1710) system [7]. The magnetic sensing of the FeGa/SCD cantilever was based on the resonance frequency shifts with applying magnetic fields during heating-cooling process. The experimental details are described in Figure S1–S3.

3. Results and discussion

The FeGa film on SCD exhibits thermally-stable magnetic properties up to 573 K, which has not been investigated yet. It is known that the A2 and D0₃ phases can coexist in FeGa film with 19 at. % Ga [10]. Figure 1(a) shows the FeGa/SCD cantilever with unit cell structures for both materials. The cross-sectional bright-field profile of the as-grown FeGa film is shown in Figure 1(b), indicating the formation of the columnar nanograin with grain size around 20 ~ 30 nm (Figure S4) and thickness of ~ 90 nm. The FeGa film exhibits a smooth surface with a root-mean-square (RMS) value of 0.844 nm (Figure S4).

Figure 1(c) shows the element distribution of the FeGa film. The high-resolution TEM (HRTEM) profile of the as-grown FeGa film on SCD is shown in Figure 1(d). A disordered layer with a thickness ~ 0.5 nm between the FeGa film and interface is observed, as marked by the yellow dotted line in Figure 1(d). The interplanar spacing of as-grown FeGa film is 2.05 Å, which is the (110) plane of A2 or D0₃ phase [11,12]. The corresponding selected area electron diffraction (SAED) pattern is shown in the inset of Figure 1(d). The reflections marked by the yellow circles and arrows in the SEAD patterns also demonstrate the existence of D0₃ phase [11,13,14]. Therefore, a high (110)-textured FeGa film with bcc-phase of A2 and D0₃ is formed on SCD. The magneto-striction property of FeGa film can be enhanced due to the short-range interaction of Ga atom pairs as well as the coexistence of A2 and D0₃ phase [10,15]. A series of FeGa/SCD samples were subjected to annealing treatment (AT) (300 ~ 873 K) for 1 h. The stress release is confirmed by the HRTEM image of the FeGa film after annealing at 573 K (Figure 1(e)), with the interplanar spacing of 2.003 Å. The interface between FeGa and SCD is also thermally stable at 573 K disclosed by the HRTEM. The X-ray diffraction patterns of FeGa films on SCD annealed at various temperatures for 1 h are shown in Figure 1(f), illustrating the annealed FeGa films maintain highly (110)-textured orientation of bcc phase for the temperature lower than 573 K. Higher temperature annealing leads to the appearance of L1₂. In addition, the surface morphologies of FeGa films exhibit strong temperature-dependence (Figure S4). Highly (110)-textured FeGa film with a smooth surface was produced on SCD even under annealing at 573 K.

The magnetic properties of the FeGa film with different ATs were investigated to realize the thermal-reliability. Up to now, no research has been performed on the temperature-dependence of FeGa films [10,14,16]. Figure 2(a,b) show the M_s - T and H_c - T profiles of FeGa films on SCD measured by PPMS technology. The FeGa films exhibit weak temperature dependence of magnetization up to 600 K upon different magnetic fields (Figure S7). The H_c decreases from 75.0 Oe to 23.1 Oe due to the random field caused by thermal agitation [17]. The magnetization of the annealed FeGa films show only a slight degradation up to 573 K with excellent magnetization (Figure S5). In addition, the annealed FeGa films exhibited highly textured orientation and long-time magnetization stability at 573 K for 5 and 15 h (Figure S6). Figure 2(c,d) summarize the orientation angular, θ dependence of the reduced remanence (M_r/M_s) and H_c of the FeGa films under AT at 300 and 573 K respectively, showing the approximate uniaxial symmetry characterizations of $\theta = 0^\circ$ and $\theta = 90^\circ$.

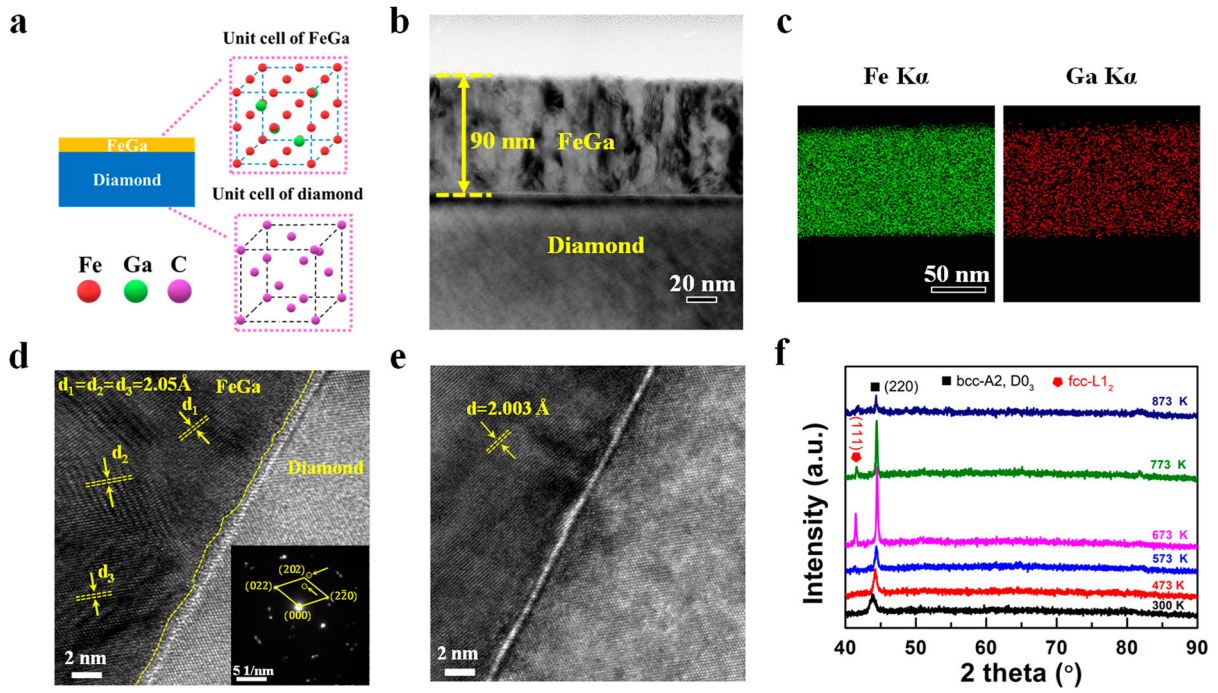


Figure 1. (a) Schematic diagram of galphenol (FeGa) film on SCD. (b) Bright-field image of as-grown FeGa film on SCD. (c) EDS images of Fe and Ga elements. (d) HRTEM image of as-grown FeGa film. The inset shows the SAED patterns of FeGa film. (e) HRTEM image of the FeGa film annealed at 573 K for 1 h. (f) XRD spectra of FeGa films annealed at various temperatures for 1 h.

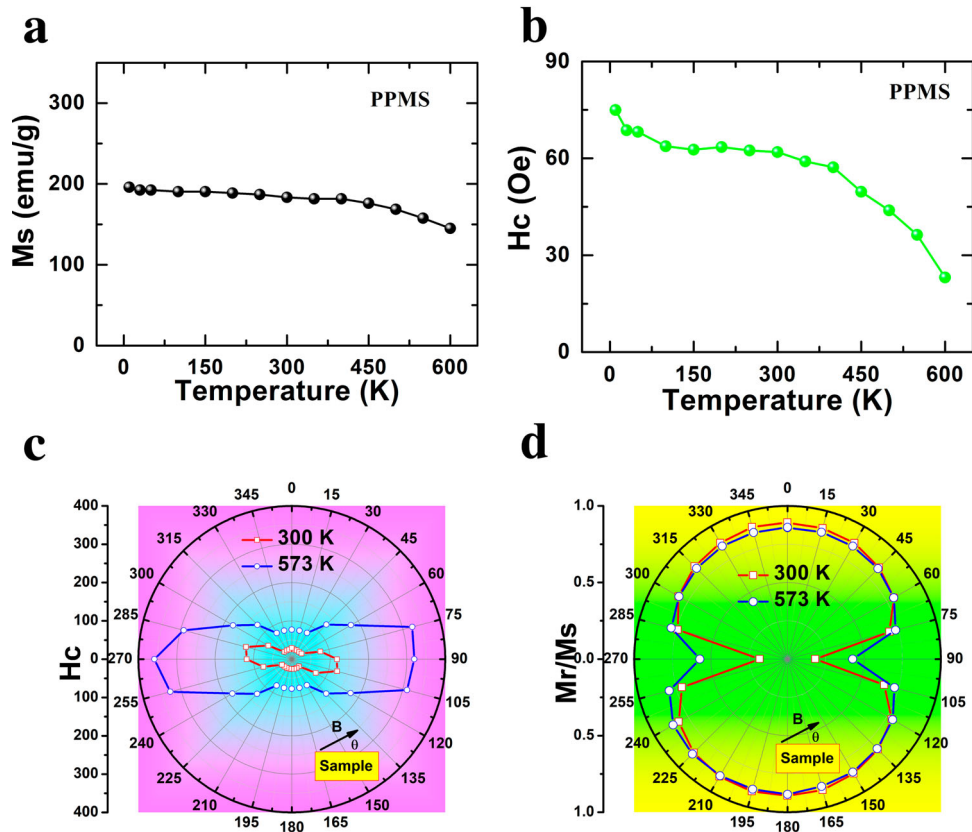


Figure 2. (a) and (b) Temperature dependence of the saturation magnetization, M_s and coercive field, H_c of the FeGa films measured at magnetic fields parallel to the sample surface, respectively. (c) and (d) Loop squareness the M_r/M_s and H_c of the FeGa films subjected to AT at 300 and 573 K.

The Mr/Ms reaches the maximal and minimal values at $\theta = 0^\circ$ and $\theta = 90^\circ$, respectively. The FeGa film with annealing at 573 K possesses better uniaxial magnetic anisotropy than the FeGa film without annealing due to the high symmetry characteristic (Figure 2(c)).

In general, the effective magnetostriction constant, λ_{eff} of a textured film at room temperature can be given by [12],

$$\lambda_{eff} = \frac{1}{5}\lambda_{100} + \frac{4}{5}\lambda_{111} \quad (1)$$

wherein the λ_{100} and λ_{111} represent the λ of (100) and (111) textured $\text{Fe}_{81}\text{Ga}_{19}$ composite, respectively. The value λ_{eff} is estimated as high as 75.1 ppm for the (110)-textured FeGa film. The magnetostriction of FeGa film shows temperature-dependent behavior. When $T < T_c$, the temperature dependence of magnetization of the FeGa film is described as [10],

$$M(T) = M_0 \left[1 - \left(\frac{T}{T_c} \right)^\alpha \right]^\beta \quad (2)$$

where M_0 is the low temperature saturation magnetization and T_c is Curie temperature set as 950 K of $\text{Fe}_{81}\text{Ga}_{19}$ film. α and β are critical coefficients. The values of α and β are calculated to be 2.20 and 0.64, respectively, as shown in Figure 2(a) (Figure S7). The temperature coefficient of magnetostriction is expressed as [10],

$$R_\lambda(T) = -2\alpha\beta \frac{(T/T_c)^\alpha}{T[1 - (T/T_c)^\alpha]} \quad (3)$$

The absolute value of $R_\lambda(T)$ exhibits a negative relation with T . According to Equation (3), the $R_\lambda(T)$ is obtained as $-3.02 \times 10^{-3} \text{ K}^{-1}$ at 573 K, less than the value of $-1.22 \times 10^{-2} \text{ K}^{-1}$ of bulk FeGa alloy. The lower R_λ of the FeGa film notably suppresses the reduction of magnetostriction constant at elevated temperatures. For Fe-based alloys, the phases of A2 and D0₃ possess positive magnetostriction compared to the negative magnetostriction of L1₂ phase [14,16,18]. The FeGa film can maintain the bcc phases of A2 and D0₃ below 573 K (Figure 1(f)), which ensures the high-thermal stability of the FeGa film on SCD. The change in magnetization and magnetostriction of FeGa film are determined by the comprehensive effects of bcc and fcc phases [12,16,18]. If there is no phase transformation of bcc phase to fcc phase, the magnetostriction enhancement of Fe-based alloys under thermal treatment can be attributed to the D0₃ nanoclusters embedded in matrix A2 phase [12,19]. Alternatively, the occurrence of bcc phase to fcc phase (L1₂) can lead to the magnetostriction enhancement of FeGa alloys after thermal treatment [14].

In general, the fundamental resonance frequency of a cantilever is described by the Euler-Berboulli beam

theory [20],

$$f = 0.162 \frac{t}{L^2} \sqrt{\frac{E}{\rho}} \quad (4)$$

wherein E is the Young's modulus, ρ is the mass density, t is the thickness, and L is the length of the cantilever. The high crystal-quality of the SCD cantilevers were confirmed by HRTEM and Raman mapping. The resonance frequency decreases with the temperature increasing due to the decrease in E (Figure 3(a)). The temperature coefficient of resonance frequency (TCF) of SCD cantilever is defined as $\text{TCF} = (\Delta f/f_0)/\Delta T$, where $\Delta f = f - f_0$. f_0 is the fundamental resonance frequency when $T = 300 \text{ K}$. Figure 3(b) displays the TCF variation as temperature (Figure S8). The TCF shows little dependence on the SCD cantilever length. The Q factors of SCD cantilevers can maintain above 3000 at 773 K (Figure 3(c)). The high-thermal stability of the resonance frequency and high Q factors of the SCD cantilevers guarantee the high performance. The resonance frequency spectra of a 160 μm -length SCD cantilever with FeGa deposition is shown in Figure 3(d), the inset of which illustrates the linear dependence of the vibration amplitude on the actuation voltage. The dynamic vibration of a bilayer beam system is described in considering the effective Young's modulus and mass density of the two coupling layers (Figure S9 and Equation S(4)). The resonance frequency dependence on the cantilever length with and without FeGa film is displayed in Figure 3(e), the inset of which shows the frequency shifts of SCD cantilevers with FeGa film. The strong coupling of SCD with FeGa film is demonstrated by the excellent consistence of the experimental resonance frequencies with those of theoretical calculation. Alternatively, the Q factors of FeGa/SCD cantilevers are still as high as 3000 ~ 7000 after depositing FeGa films, as shown in Figure 3(f).

Concerning about the high-temperature magnetic sensor, we note that magneto-resistive sensors and Hall sensors are also capable [21]. However, these sensors present fatal drawbacks with technological and physical limitations. For the magneto-resistive sensors, interfacial diffusion and scattering process strongly degrade the sensing properties under extreme conditions [22,23]. For the Hall sensor, the deterioration in carrier mobility and the high power consumption impair temperature-related sensitive resolution and prohibit a stable operation at high temperatures [21,24]. The present magnetic sensor utilizes a thermally stable SCD MEMS resonator coupling with a thermally stable magneto-strictive FeGa material. Upon external magnetic fields, the FeGa film induces an interface strain upon the MEMS resonator, which, in turn, leads to the resonance frequency shift

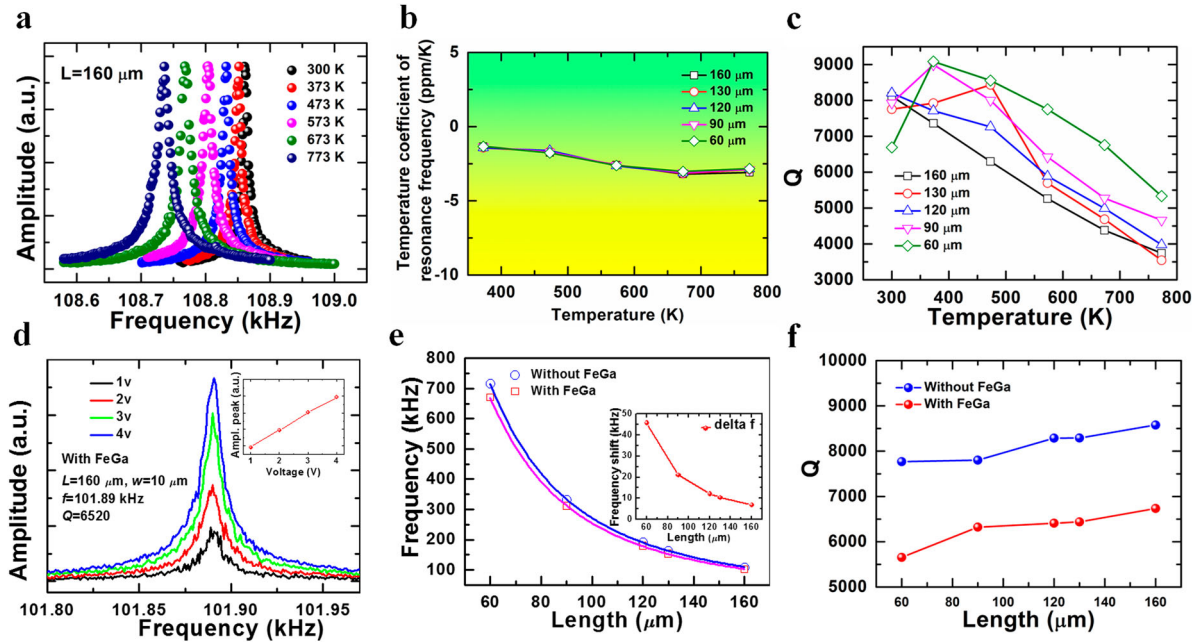


Figure 3. (a) The resonance frequency shift as measurement temperature of a bare SCD cantilever. (b) TCF of the SCD cantilevers. (c) Temperature dependence of Q factor of the SCD cantilevers. (d) The resonance frequency spectra of a FeGa/SCD cantilever. (e) The resonance frequency and (f) Q factor vs cantilever length without and with FeGa film.

of the resonator. The excellent thermal stability of the FeGa/SCD MEMS magnetic sensor is due to the (1) strong coupling of the FeGa with the resonator, (2) thermal-stability of the magneto-strictive coefficient of the FeGa film, (3) high Q factor of the resonator even at high temperatures, and (4) thermal-stable interface of the FeGa/SCD. Figure 4(a) shows the measurement setup of the magnetic sensor based on the FeGa/SCD cantilever. The response of the magnetic sensor is reflected by the resonance frequency shift of the FeGa/SCD cantilever as external magnetic fields. For the magnetic sensing at high temperatures, the resonance frequency was measured until the steady state reached. Figure 4(b) schematically illustrates magnetic sensing at different temperatures of the 160 μm -length FeGa/SCD cantilever without and with magnetic field. The resonance frequency shift induced by the magnetic field is defined as $\Delta f' = |f' - f'_0|$, which represents the sensing characteristic of the magnetic sensor. f'_0 and f' is the resonance frequency of FeGa/SCD cantilever without and with magnetic field. The dependence of the resonance frequency shift on the temperature during heating-cooling process (300 K to 573 K) is shown in Figure 4(c). The frequency shift is slightly enhanced with increasing temperature. Figure 4(d) shows the dependence of the frequency shift on the magnetic field at elevated temperatures, revealing that the frequency shift linearly increases with the magnetic field. The highest sensitivity of 7.3 Hz/mT is achieved at 573 K. The Q factor variation is in range of 3000–7000 during heating-cooling process (Figure S10).

The high Q factor of the magnetic sensor is beneficial to the high resolution. The FeGa/SCD cantilever showed a fast and stable response to the applying magnetic field even at 573 K (The Video, Supplementary Information). Other SCD cantilevers also show similar resonance frequency response to the external magnetic field (Figure S11). As aforementioned, the (110)-textured structure and smooth surface of the FeGa film are maintained up to 573 K, which indicates the upper-limit working temperature with high reliability of the FeGa/SCD cantilever magnetic sensor. The enhancement of the response to the magnetic field can be attributed to the improvement of the magnetostriction constant upon the high temperature treatment. This is consistent with the above discussion about the effect of the thermal treatment on magnetostriction of FeGa films.

Alternatively, we calculate the intrinsic magnetic noise (b_n) of the magnetic sensor, which is determined by the thermomechanical noise, as expressed by [25],

$$b_n = \frac{\mu_0}{2} \left(\frac{dH}{df} \right) \sqrt{\frac{2\pi k_B T f'_0}{QV\sigma}} \quad (5)$$

where the $\mu_0(dH/df)$ is the magnetic sensitivity, σ is the stress (Equation S(6)). The calculated noise floor of the intrinsic magnetic sensor noise of the FeGa/SCD cantilever is $\sim 589 \text{ pT}/\sqrt{\text{Hz}}$ at 300 K and $530 \text{ pT}/\sqrt{\text{Hz}}$ at 573 K. Furthermore, the magnetic noise was measured at 300 K, showing $\sim 4 \text{ nT}/\sqrt{\text{Hz}}$ (Figure S12). The estimated magnetic noise is close to that of the tests. The noise level

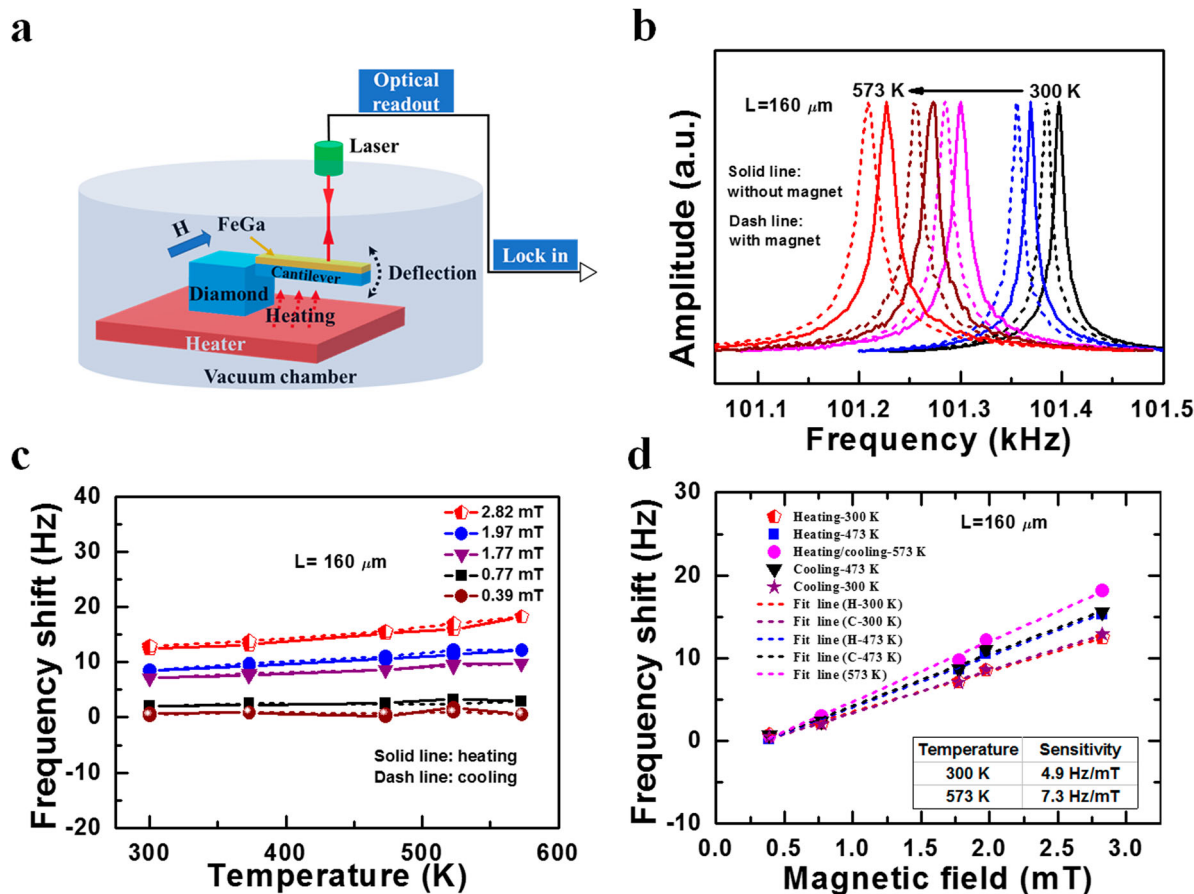


Figure 4. (a) Measurement setup of magnetic sensing at elevated temperatures. (b) Resonance frequency shift vs measurement temperatures of the FeGa/SCD cantilever without and with magnetic field. (c) Dependence of the resonance frequency shift on temperature during heating-cooling treatment. (d) The resonance frequency shift as a function of magnetic field at elevated temperatures.

could be greatly improved by structure design. For example, by inserting Ti layer between FeGa and diamond, the noise level was improved to be ~ 230 pT/ $\sqrt{\text{Hz}}$ at 300 K. The noise level could be improved further by geometrical design of diamond cantilever. For example, for a diamond NEMS resonator with length = $5 \mu\text{m}$, width = 50 nm, thickness = 10 nm and Q factor of $500,000$, the noise level was calculated to be 0.27 pT/ $\sqrt{\text{Hz}}$. Expectedly, the noise level can be strongly suppressed by increasing the Q factor and decreasing the resonance frequency. Based on the magnetic sensitivity, the minimum detectable magnetic fields are 132 pT and 159 pT at 300 K and 573 K, respectively (Figure S12). The magnetic sensitivity could be further improved if metalloid doped FeGa ternary alloy films are utilized [5,6]. The on-chip all electrical actuation and readout can favor the practical applications of the SCD MEMS magnetic sensors (Figure S13) [7].

4. Conclusion

We successfully developed the high-temperature magnetic sensor based on the galphenol/SCD MEMS resonator.

The high sensitivity and thermal stability operation of the magnetic sensor were achieved due to the outstanding thermal properties and strong coupling between FeGa and SCD. The current work overcomes the drawbacks of the existing high-temperature magnetic sensors, such as low high-temperature sensitivity and weak thermal stability at high temperatures.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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ORCID

Zilong Zhang <http://orcid.org/0000-0002-9759-9253>

Masaya Toda <http://orcid.org/0000-0003-3849-6948>

Meiyong Liao <http://orcid.org/0000-0003-1361-4266>

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