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Quantifying adsorbed water monolayers on silicon MEMS resonators exposed to humid environments

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ABSTRACT

This study investigated the influence of temperature and humidity on the adsorbed water layer on micron-scale monocrystalline silicon (Si) films in air, using a Si-MEMS kHz-frequency resonator. Both temperature and relative humidity induced a reversible change in resonant frequency, attributed to the temperature-dependent properties of Si and to a change in adsorbed water layer. The excellent precision in resonant frequency measurement (0.02 Hz, or 0.5 ppm) allowed precise calculation of the changes in adsorbed water layer thickness over the specimen surface. The increase in water thickness with relative humidity was a function of temperature and could not be described with simple multimolecular adsorption theories such as the BET theory. A likely explanation is the presence of hydrocarbon contaminants on the Si surface. Guidelines are provided to accurately measure the influence of temperature and relative humidity on the adsorbed water layer thickness on micron-scale Si surfaces, using this technique.

1. Introduction

Structural reliability concerns of Si-based MEMS devices in non-inert environments warrant extensive fatigue and fracture studies of micron-scale Si in harsh environments. Indeed, while Si films are immune to fatigue failure in ultra-high vacuum environments [1,2], the presence of water molecules in the environment leads to unambiguous degradation of the fatigue properties [1–8]. Several fatigue mechanisms have been proposed to account for these experimental results [9,10]. The reaction-layer fatigue (RLF) mechanism is based on stress-assisted oxidation of the Si surface [11], which has been experimentally observed in the high cycle fatigue/very high cycle fatigue regime [1,4,11]. Recently, static fatigue results in different humidity-containing environments suggested that time-dependent crack growth is likely to account for short fatigue lifetime tests [12]. The strength of Si films may also degrade with increasing humidity levels [13].

The exact role of the water molecules on the fracture and fatigue processes of micron scale Si is still unclear. For example, water molecules form a thin adsorbed film on the surface [14], whose thickness (~nm range) is commensurate with critical crack size in micromachined Si films (~10 s of nm). It is therefore worthwhile characterizing the adsorbed water layer thickness (h_w) directly on the surface of the Si MEMS devices in humid environments to further investigate its role in these degradation processes. The study of ambient thin water films on macroscopic surfaces usually relies on optical techniques, such as ellipsometry and infrared spectroscopy. However, these techniques may not be appropriate to study microscopic samples due to their minimum required sample size.

Accordingly, the present study focused on developing an experimental technique to measure the relative changes in h_w, Δh_w, as a function of temperature (ranging from 20 to 80 °C) and relative humidity (RH; ranging from 20 to 90%), directly on the surface of a Si-based MEMS resonator. The technique relies on the precise measurement of the MEMS' resonant frequency in controlled environments. The experimental results are compared to theoretical models for adsorption of gases in multimolecular layers [15,16], as well as thin film water studies on macroscopic Si surfaces at room temperature.

2. Experimental

The MEMS structures (10 and 25 μm thick) were fabricated with the 20th run of the SOIMUMPs™ process (see Fig. 1) [17]. The single-crystal Si film is patterned using a deep reactive ion etching (DRIE); after the etch, the wafers undergo a "Piranha" clean (7 parts of H_2SO_4 (98%): 3 parts of H_2O_2 (30%) at 125 °C for 10–20 min) and SC1 clean (5 parts of H_2O: 1 part H_2O_2: 1 part NH_4OH at 70 °C for ~10 min), followed by an ash, to remove all sidewall polymer and photoresist. The patterned film is then covered with a polyimide front side protection material (during patterning and etching of the wafer's back face), which is later removed using a dry ash.

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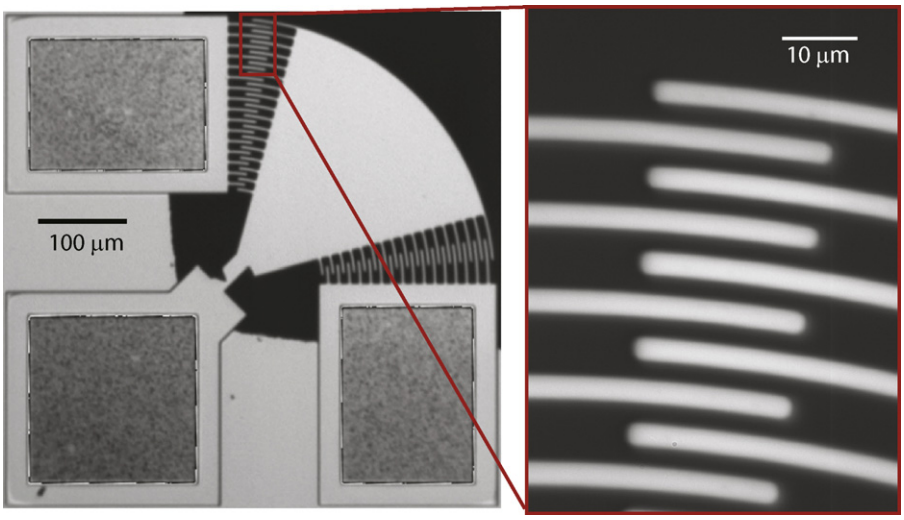

Fig. 1. Optical images of the monocrystalline Si micro-resonators used in this study.

The structures behave as linear rotational oscillators [18], with a natural frequency of ~40 kHz. The structures consist of a 40-μm long, 20-μm wide [1 0 0] oriented notched cantilever beam attached to a fan-shaped mass (60° span, 30-μm inner radius, 300-μm outer radius) with two comb drives (16 interdigitated fingers; see Fig. 1). The notch lies approximately at 9 μm from the base of the beam, is 13-μm deep and has a 1-μm root radius. The beam-mass system and each side of the comb drives are connected to metallic pads, providing electrical connections via aluminum wirebonds between the structure and a ceramic package in which the die is glued.

The testing principle consists of applying a sinusoidal signal (with no offset component [18]) on one comb drive to actuate near resonance the electrically grounded beam-mass assembly via electrostatic forces, and measure the second-harmonic, induced currents with the other comb drive. The input sinusoidal voltage is applied with a waveform generator (Agilent 33220A 20 MHz) and amplified with an AVTECH-110G high-voltage amplifier. A DC bias voltage (50 or 100 V) is applied with an Agilent E3612A power supply to the sensing comb drive. The induced currents (~nA level) generated during cyclic motion are amplified and converted to a voltage with a custom-made, off-chip, current–voltage amplifier circuit containing an OPA 128 operational amplifier. The distance between the circuit and the ceramic package is only a few inches, in order to minimize the electrical noise picked up by the connecting wires. The amplified signal is measured with a lock-in amplifier (SR830-100 kHz DSP synchronized with the waveform generator) at the second harmonic of the input sinusoidal voltage. These measurements are performed in a controlled environment using a temperature and humidity chamber (ESPEC SH-241 Benchtop type), with a 0.1 °C, 1%RH resolution.

This experimental setup was employed to measure f_0 as a function of environment at low, non-damaging maximum principal stresses (stress at notch root, $\sigma_1 < 1$ GPa) [6,18]. The f_0 was measured by recording the output voltage as a function of applied frequency around the frequency corresponding to resonance ("sweep" curve). f_0 was calculated by fitting a second-order polynomial equation to the sweep curve (frequency range ~8 Hz) and extracting the frequency corresponding to the maximum output value. These measurements were performed for a series of environments, starting at 20 °C, 50%RH, with incremental steps of 10%RH. Once the environment reached 90%RH, temperature was increased by 10 °C, and the RH level decreased to low humidity level (~30, 40, or 50%RH, depending on the control capability of the chamber). The measurements were repeated until 80 °C, 90%RH. For each environment, f_0 and Q were measured 6 and 5 times, respectively, 10 min after the targeted temperature and RH level was reached. In order to confirm the reversible nature of the change in f_0 and Q due to the environment, all the measurements were repeated as the environment was reversed from harsh (80 °C, 90%RH) to mild (20 °C, 50%RH).

The MEMS devices were tested as received: no specific cleaning step was performed to remove possible hydrocarbons accumulated on the surface during the ~1 year period between the fabrication of the devices and the tests.

3. Modeling

3.1. Environmental effects on resonant frequency f_0 and adsorbed water layer h_w

Previous experimental characterization of these MEMS resonators [18–20] (shown in Fig. 1) at room temperature revealed that its dynamic behavior is equivalent to a one-degree-of-freedom, simple harmonic rotational oscillator, whose governing equation is:

$$J_\theta \ddot{\theta} + b\dot{\theta} + k_\theta \theta = M_0 \sin(2\pi f t) \quad (1)$$

where J_θ is the mass moment of inertia ($J_\theta = 5.475\,10^{-17}$ kg m^{-2} [18]), b the damping coefficient (assumed constant for a given environment), k_θ the torsional stiffness, M_0 the amplitude of the applied moment, f is the frequency of the applied moment and $\theta(t)$ is the angle of rotation of the structure about the midpoint of the remaining ligament of the notched beam. The resonant frequency f_0 is defined as:

$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k_\theta}{J_\theta}} \quad (2)$$

The environmental effects on f_0 are due to the influence of temperature and RH on k_θ and J_θ (see Eq. (2)). The stiffness k_θ is mainly affected by temperature, due to softening of the elastic constants. The effect of RH (adsorbed water layer) on k_θ can be neglected as both the thickness ($h_{water} \sim$ nm) and the bulk modulus ($B_{water} = 2.2$ GPa) of the adsorbed water are negligible compared to the values for Si ($h_{Si} = 10$ or 25 μm, $B_{Si} = 97.84$ GPa).