Dual exposure glass layer suspended structures: A simplified fabrication process for suspended nanostructures on planar substrates

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We have developed and demonstrated here a simplified flexible fabrication process for glass nanomechanical systems. This process uses a single layer of spin on glass (SOG) material with two negative tone electron beam exposures at two different exposure energies to define the suspended and support structures, respectively. After development the SOG can be converted into glass. The process is additive and can be applied to any flat substrate. We have fabricated a variety of glass nanomechanical oscillators and measured their mechanical resonances using a mechanical piezoelectric driving force and optical interferometric detection. Suspended structures were fabricated with thickness of less than 50 nm and lateral dimensions of less than 100 nm supported anywhere from 150 to 800 nm above the substrate. Resonance frequencies for glass wires with both ends fixed (cross section 110 nm×180 nm) and lengths of 4–9 μm range from 7 to 30 MHz, with quality (Q) factors of over 1000. Annealing the structures in an oxygen ambient roughly doubles both the frequencies and the Q factors.
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I. INTRODUCTION

Nanoscale mechanical systems have potential to be very high quality factor (Q) oscillators and to be used in a variety of sensor applications. Currently, many materials have been used to fabricated such systems as well as larger micromechanical systems.[1–3] Typical fabrication requires a specialized substrate with two distinct layers. The upper layer becomes the freely suspended dynamic structure and the lower layer is a sacrificial layer that is used to undercut the suspended structure while still supporting it at large fixed points. The pattern is transferred into the upper layer by an anisotropic etch (typically reaction ion etching) and released by an isotropic etch with high selectivity of the sacrificial layer. This requires the geometry of the supporting layer to have features significantly larger than the dynamic structure.

We have developed a new far more simplified flexible fabrication process for nanomechanical systems. The dual exposure glass layer suspended structures (DEGLaSS) process uses a single layer of spin on glass (SOG) material with two negative tone electron beam exposures at two different exposure energies to define the dynamic and support structures, respectively. A low energy exposure has a very short penetration depth into the SOG, similar to a top surface imaging process, and defines the dynamic suspended layer. A second exposure is performed with a higher energy that penetrates through the entire glass layer thickness, resulting in a support structure for the dynamic layer. There is no processing between the exposures, so the order of the exposures does not matter. Both patterns are developed simultaneously in an aqueous base which removes all the unexposed material, including the material beneath the suspended layer, thus releasing the structure. Samples are transferred through an ethanol bath to a critical point CO_2 dryer to prevent collapse due to surface tension. After development the three-dimensional cross-linked SOG structures can be densified by converting them into an amorphous glass structure. Macroscopic amorphous glass structures are known to make extremely high Q resonators with Q above 50 million,[4] and should be appropriate for the development of high sensitivity sensors.

In this article we demonstrate that a wide variety of nanomechanical structures can be fabricated using the DEGLaSS process. An unusual, useful feature of the process is that the support structure can have very small lateral dimensions that are comparable to those of the suspended structures. Measurements of suspended structures that are clamped on both ends indicate that both material stiffness and tensile force are significant in determining the motion of these structures. Oscillators with Q factors greater than 10^3 are easily obtained.

II. FABRICATION PROCESS

We have demonstrated the DEGLaSS process using low energy electron beam exposures of hydrogen silsesquioxane (HSQ). HSQ was chosen for its high resolution (15 nm) and sensitivity in negative tone electron beam lithography,[5–7] well characterized properties,[8] availability,[9] and its chemical structure ($HSiO_{3/2}$) which should allow nearly complete conversion to SiO_2 with a minimal amount of impurities. Variable energy electron beam lithography allows control of the electron penetration depth in HSQ and in similar polymers over three orders of magnitude, from less than 10 nm to

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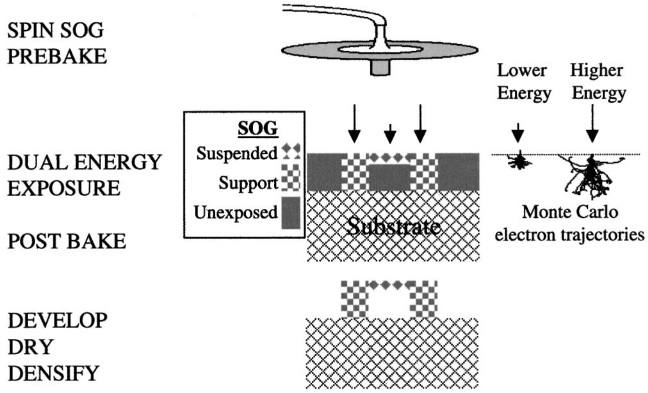

FIG. 1. Schematic of the DEGLaSS process for making a suspended glass nanostructure. Monte Carlo data are shown to demonstrate the variable electron penetration depth of the two separate exposures.

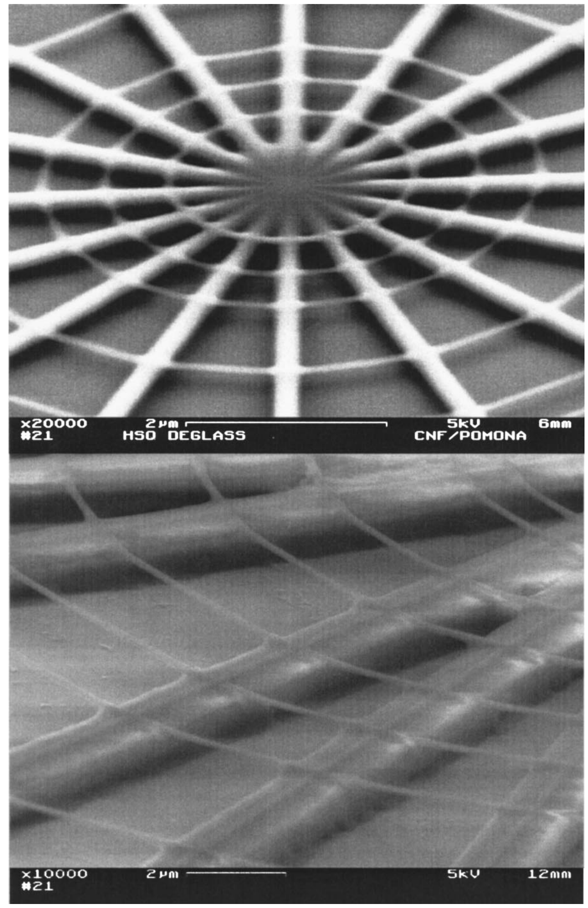

FIG. 2. Web structures fabricated from both thin (200 nm) and thick (850 nm) films of HSQ. In both cases the rings are suspended above the substrate by the radial support structures. See the text for details of the exposures.

greater than 10 μm with a single exposure tool with beam energies from 200 eV to 30 keV. Our exposure tool is a LEO 982 scanning electron microscope (SEM) with the Nabity NPGS pattern generator. Each exposure can be defined in a separate layer in a computed-aided design (CAD) system, and the beam energy, focus, and alignment are adjusted between exposures of the layers. Since the electrons used to expose suspended features cannot penetrate the entire layer thickness, alignment marks must be on the top surface of the resist film.

Because the exposed HSQ will become the final structure, it is critical that the process does not cause significant stress or swelling in the suspended films that would result in either cracking or pealing. Any post-apply bake or post-exposure bake must be done at sufficiently low temperatures (below 300 °C) to avoid thermal crosslinking of the underexposed regions. After experimenting with a variety of processing conditions we have developed the process flow shown in Fig. 1, which has been very reliable for thin films (<0.5 μm) of HSQ. Following standard spin coating to obtain the desired film thickness, the HSQ coated substrates (we used silicon substrates, but this is not necessary) are pre-baked at 250 °C for 2 min on a vacuum hot plate to remove residual solvents. An alignment mark is then added on top of the film and the sample is loaded into the vacuum system of the e beam exposure tool. Typical exposure energies for suspended layers are 1–3 keV, and typical exposures for support structures are from 3 to 20 keV. Optimal doses depend upon the beam energy and resolution desired, but the typical single pass line doses are on the order of 1 nC/cm, with area doses on the order of 10 μC/cm^2. A 250 °C post-exposure bake enhances contrast in the process. Films are then developed in standard aqueous base developer (0.26 N TMAH) for 2 min. While some structures can be removed from the developer and placed in an ethanol or isopropanol bath and air dried, more delicate structures benefit from transfer to ethanol followed by supercritical CO_2 drying.[10] At this point the fabrication process is complete, although further annealing, oxygen plasma, or ultraviolet (UV) ozone exposure can be applied to further densify the structures and to modify their mechanical properties. Thick films (>0.5 μm) develop more easily and cleanly if there is a brief pre-development step (20 s dip in TMAH) before the post-exposure bake. This additional step clears all the large unexposed open areas but does not release the suspended structures and allows thick films to have reasonable development times.

Figure 2 shows two examples of web structures fabricated using this process in thin and thick films. In each case the circular arcs are suspended above the substrate and were exposed with a low beam energy while the radial lines are the support structure extending the full depth of the initial spin cast HSQ film and were exposed with a higher beam energy. It should be emphasized that these are structures as fabricated with no etching or metallization applied to enhance imaging of the structures. In Fig. 2(a), the suspended rings were exposed with a 1 keV electron beam and are 40 nm thick and 100 nm wide. The supporting beams were exposed with 3 keV electrons and are 200 nm high and 220 nm wide. In Fig. 2(b), the suspended rings were exposed with 2 keV electrons and are 110 nm thick and 150 nm wide while the support structure was exposed with 10 keV electrons and is 850 nm tall. While the support structure in this case is quite wide, similar support structures have been fabricated with widths as small as 300 nm. Fully suspended structures 120

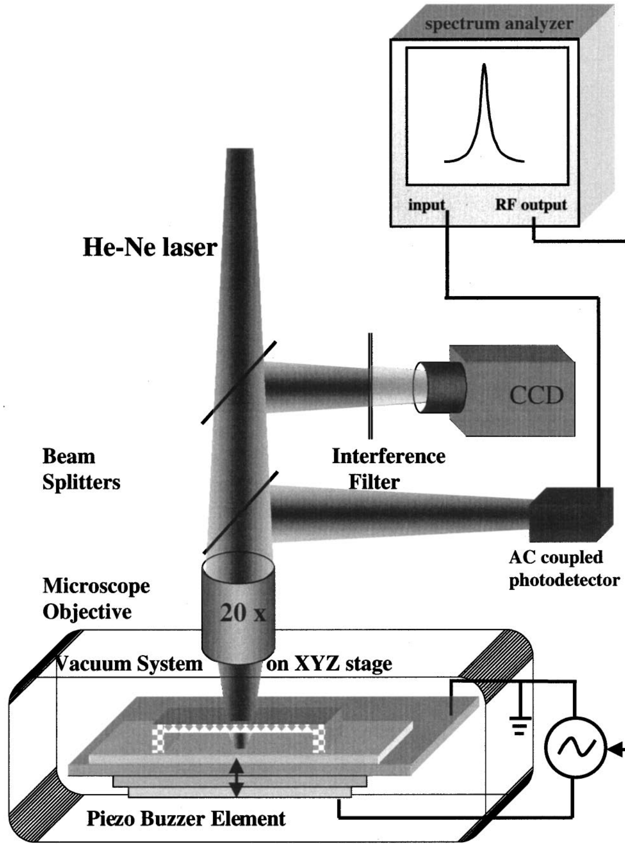

FIG. 3. Schematic diagram of the measurement apparatus used to record the resonance data.

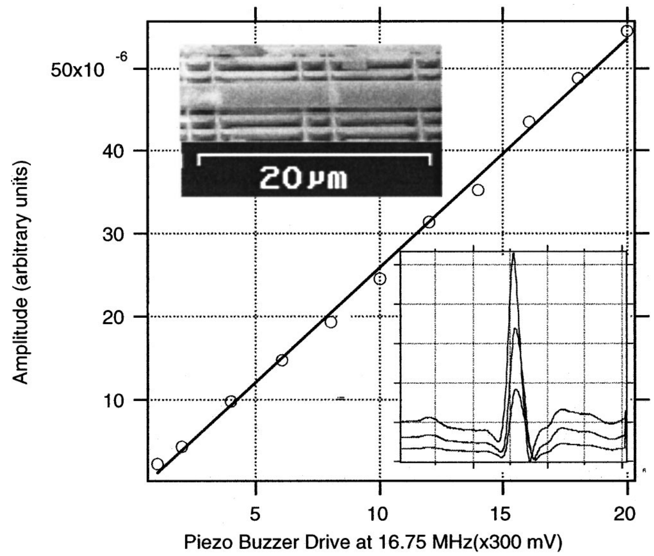

FIG. 4. Plot of the amplitude of the optical signal as a function of the drive voltage applied to the piezoelectric element. The upper inset shows the structure being measured and the lower inset shows the spectra for three of the data points.

nm thick have been formed with spans up to 15 μm long above 0.8 μm supports. When the structures fail they appear either to have cracked or become pinned on the surface due to surface tension in the drying process, rather than simply sagging. This suggests that fully suspended structures are under tensile stress.

III. EXPERIMENTAL MEASUREMENTS

In order to observe the mechanical properties of the suspended structures they are mounted in a high vacuum cell ($P \sim 10^{-7}$ Torr) and driven into resonance. The relative motion of the substrate and the structure acts as a Fabry–Pérot cavity, and laser light focused through a microscope objective onto the structures is modulated as the cavity size changes. This is similar to the optical detection arrangement we have used to measure silicon nanostructures.[11] The major difference in this case is that, since the structures are fabricated without a conductive coating, they are not driven electrostatically. Instead, our samples are mounted on a low cost piezoelectric buzzer element that can be driven by the amplified tracking generator output of a rf spectrum analyzer. The piezo has strong resonances in the low kHz regime, but still has useful response up to 50 MHz. The reflected laser light is directed to an ac coupled photodiode whose output is used as the input for the spectrum analyzer. In addition, a video camera is focused through the microscope objective, enabling the focused laser spot to be imaged and positioned on the specific structure to be measured. In this article all measurements were done at room temperature. A schematic diagram of the measurement apparatus is shown in Fig. 3.

Measurements were made on structures similar to those presented previously using other materials systems and fabrication techniques. These included membranes, mesh structures, torsional paddle oscillators, and wires with varying lengths in harp-like structures. It should be noted that the optical absorption and reflectivity of these thin (~100 nm thick) glass structures are fairly weak, so the signals detected are small and there are no signs of thermally induced nonlinear effects, as was recently observed for silicon structures.[12] An example of the linearity of the response as a function of the drive voltage on the piezo element is shown in Fig. 4. These data were taken on a doubly clamped ribbon-like membrane 7 μm long and 5 μm wide with a thickness of 110 nm whose fundamental resonance was observed at 16.75 MHz with $Q \sim 1500$. A SEM image of this structure is shown as an inset in the upper left portion of Fig. 4, while three of the measured resonance peaks are shown in the lower right inset.

The doubly clamped beam is one of the most studied structures due to its analytical simplicity and ease of fabrication (particularly in contrast to comparably long cantilever beams). Figure 5 is a SEM image of one of a series of harp-like structures that consist of sets of doubly clamped beams with varying lengths. In this case each step in length consists of eight nominally identical wires fabricated in HSQ. Unfortunately the shortest of these wires has resonances too high to be driven with the pizeoelectric element. However, we have recorded extensive measurements on similar wires ranging from 4 to 9 μm in length with widths of 200 nm and

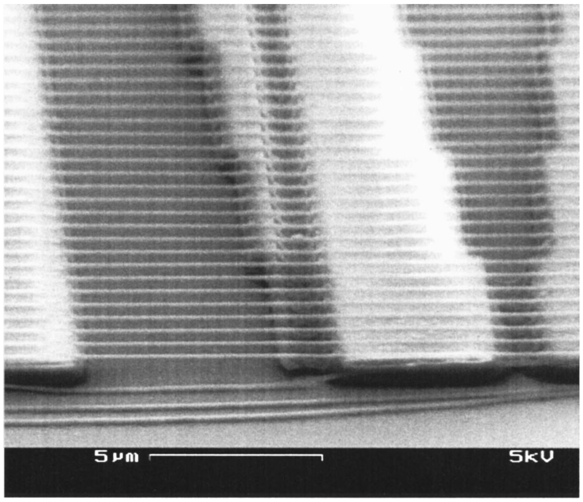

FIG. 5. Series of wires in one of the harp-like structures fabricated in an 850 nm thick film of HSQ. The wires are 120 nm thick and 200 nm wide and have 1 μm spacing between adjacent wires.

thicknesses of 120 nm. After measuring these wires we subjected them to an oxygen anneal at 1000 °C for 30 min. We then repeated the measurements on the same set of wires. (It should be noted that resonant frequencies and amplitudes of adjacent wires with the same nominal dimensions were discernable, but small, typically less than 2%.) One example of this type of data is shown in Fig. 6 in a log–log plot with a power-law fit. In this case the fit is fairly poor, in marked contrast to similar data for silicon wires. In collecting data from a large number of measurements of different wires fabricated by the DEGLaSS process, we found that the average Q of these wires is ~1100 after fabrication and ~2300 after annealing at 1000 °C in an oxygen rich environment. This doubling of the Q factor is a direct result of the doubling in the frequencies seen for individual wires in Fig. 6. Images of

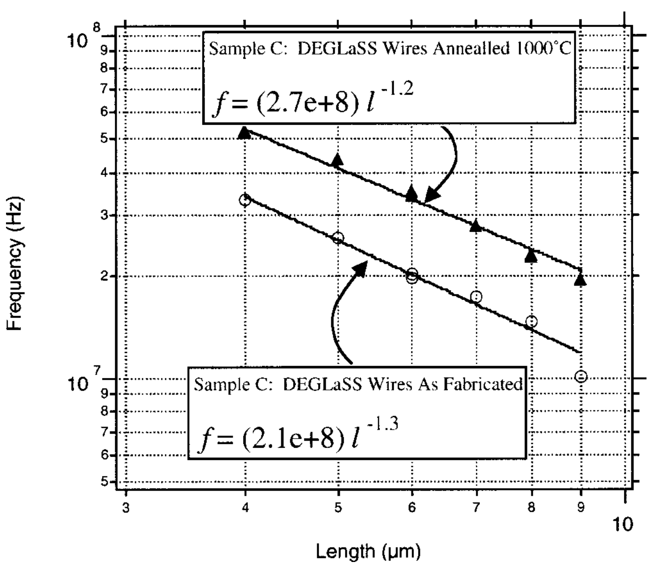

FIG. 6. Plot of the resonance frequency as a function of the wire length for wires similar to those shown in Fig. 5 before (lower) and after (upper) annealing at 1000 °C.

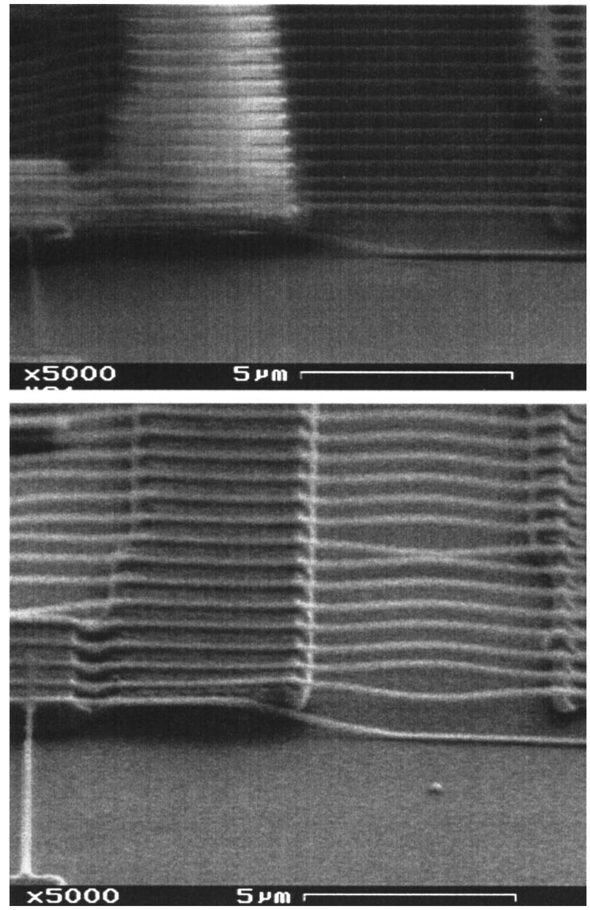

FIG. 7. Images of the wires before and after the annealing process showing a change from tensile stress to compressive stress in addition to densification.

these structures before and after annealing are shown in Fig. 7. The wires appear to be under compressive stress following this high temperature anneal in contrast to the tensile stress observed before annealing. Compressive stress normally would lower the resonant frequency of the structures, but it is clear that the material's density and stiffness have also changed in the annealing process and, even though the wires appear slightly buckled, the resulting resonance frequencies and Q factors have increased.

IV. DISCUSSION

There are two simple limiting cases for the behavior of doubly clamped beams or wires. In the first case the tension force on the wire is considered to be negligible compared to the stiffness of the wire. Under this condition, the fundamental resonance of the wire can be described by

$$f=\frac{\pi}{2l^2}\beta^2\frac{t}{\sqrt{12}}\sqrt{\frac{Y}{\rho}}, \quad \text{with} \quad \beta=1.505, \tag{1}$$

where Y is the Young's modulus, ρ is the density, t is the thickness of the wire, and l is the length of the wire. The power law is predicted to be $f\propto l^{-2}$. Clearly we do not see this behavior in the HSQ wires although it has been observed in both single crystal silicon wires[13] and silicon nitride wires.[14] The other limiting case is that the tension forces dominate over the stiffness in determining the motion. This is analogous to the resonance of a string, and can be described by

$$f = \frac{1}{2l}\sqrt{\frac{T}{wt\rho}}, \tag{2}$$

where T is the tension, and w is the width, resulting in a power law of $f \propto l^{-1}$. Again it is clear this is also not a good fit to the data. Incorporating both the tension and rigidity to model the data is clearly necessary, and it does not result in a simple power-law relationship. Attempts to model the data incorporating both effects have yet to produce a fit to the data that is suitable for the extraction of material properties such as the Young's modulus. One approach to simplify this problem is to study a set of cantilever beams in which tension is not an issue. It should be emphasized that the fabrication process enables the creation of significantly higher frequency oscillators than those reported here. Changing the piezoelectric drive element used should enable these higher frequency structures to be observed.

One of the advantages of the DEGLaSS process is that the structures can be produced with only low temperature processing (250 °C) and still achieve reasonable Q factors in a fully additive process. While high temperature oxygen annealing had a significant effect on the structures, it is not necessary for use in the process. A more complete characterization of the state of the material after fabrication and after different annealing conditions would be desirable. A comparison between thermal annealing and exposure to oxygen plasma curing and UV ozone exposure would be beneficial, since each of these might allow densification of the glass structures while maintaining a low thermal budget. Lower temperature densification may also reduce the stresses built up in the suspended structures due to thermal expansion differences between the structures and the substrates.

V. SUMMARY AND CONCLUSIONS

The ease of controlling the electron energy (and consequent penetration depth) make the DEGLaSS process ideal for working with low energy electron beam lithography. In fact, the potential exists to fabricate structures with more than two exposures, allowing suspended structures to be patterned with a variety of thicknesses. Such structures might be employed as movable diffractive optical elements. The relatively low throughput of electron beam lithography should not really be a concern since the density of suspended features is not likely to be very high in most sensor applications. However, the process could be achieved with photolithography if the wavelengths of the exposing photons are chosen carefully. HSQ has previously been combined with photobase generators to allow negative tone deep UV (DUV) lithography[15] that is suitable for creating support structures. A second exposure at a wavelength such as 193 or 157 nm (either of which is likely to be absorbed in the top 200 nm of the HSQ film) would define the suspended structures.

The process was demonstrated to create nanomechanical suspended structures. The structures were demonstrated to have reasonable Q values and should be viable as elements in sensors. The process is extremely simple and can be fully implemented to design arbitrary suspended structures using only a high quality electron microscope controlled by a pattern generator. The process eliminates the need to do reactive ion etching and can be performed on any planar substrate, even above existing integrated circuits, since both the dynamic and supporting structures are fabricated from the same SOG. The pattern generation and the computer aided design are done on a single computer, allowing very rapid prototyping of new structures without the delay of creating masks for optical lithography. The only chemical exposure is to TMAH, in contrast to the HF used in many processes. Finally, the process allows the support structure to have an arbitrary size scale independent of the dynamic structures.

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