

POLYSILICON-FILLED CARBON NANOTUBE GRASS STRUCTURAL MATERIAL FOR MICROMECHANICAL RESONATORS

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ABSTRACT

Folded-beam capacitive-comb-driven micromechanical resonators constructed of polysilicon-filled carbon nanotube (CNT) grass structural material have been characterized via *electrical measurement* to extract numbers for mechanical properties. The process used here is based on that first demonstrated in [1], but improved by introduction of a guiding oxide mold and post-fabrication *in situ* localized annealing. Specifically, the measured frequency response for a 28.658-kHz *in situ* localized annealed version yields a Q of 3,230 twice as high as that of an un-annealed one; and an acoustic velocity of 9,042m/s slightly higher than the 8,024m/s of polysilicon, and inching towards the ~12,000m/s typical of SiC. This material not only facilitates fabrication of high aspect-ratio microstructures, but also shows potential (via its silicon-carbon makeup) for approximating SiC, for which theory predicts frequency and Q advantages over silicon for resonant devices.

INTRODUCTION

To date, capacitively-transduced micromechanical resonators have posted the highest Q 's among on-chip resonator technologies, with Q values exceeding 150,000 in the VHF range [2] and exceeding 11,000 in the GHz range [3]. However, the exceptional Q 's of these resonators are available in concert with the sub-200 Ω impedances desired by off-chip board-level applications only when technologies that achieve tiny high aspect-ratio gaps are used, such as that of [4] or [5]. Aside from impedance issues, there are applications that demand even higher Q 's at GHz frequencies [6], which means higher frequency- Q products are also desired. To this last point, theoretical studies in literature generally predict that the material Q of SiC should exceed that of silicon at the same frequency [7][8]. So far, however, actual SiC devices have yet to best silicon in this area. This combined with higher electrical resistance and greater difficulty depositing low-stress doped SiC makes this material much less popular than silicon or polysilicon for high Q , high aspect-ratio applications.

Recently, Hutchinson [1] demonstrated a polysilicon-filled CNT grass structural material that combines carbon and polysilicon on the nano-scale in a process that achieves high aspect-ratio microstructures more easily than conventional deposition and etch based methods. Although its combination of silicon and carbon are not at the molecular level, there is some interest in determining how closely this material mixture might approximate the material properties of molecular SiC. This work investigates this by first fabricating folded-beam capacitive-comb-driven micromechani-

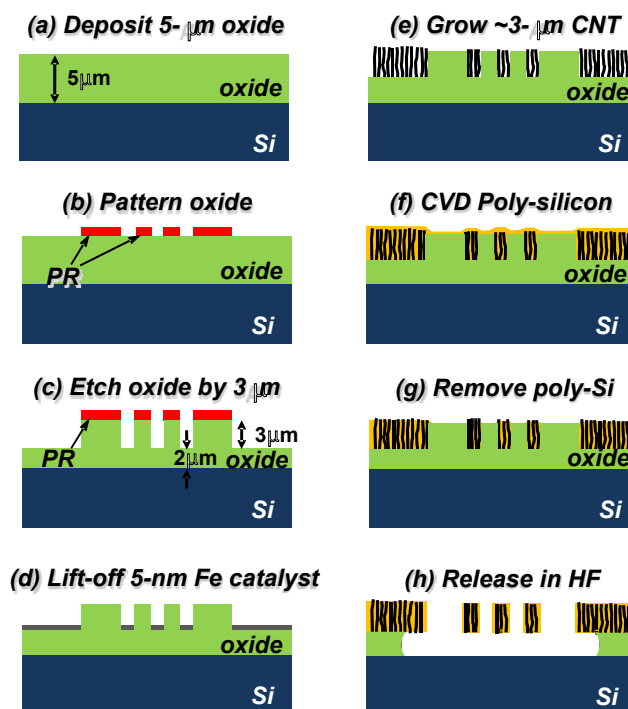


Fig. 1: Fabrication process flow for folded-beam comb-driven microstructures in polysilicon-filled CNT material.

cal resonators in polysilicon-filled CNT material using a modified process that insures straighter sidewalls and adds post-fabrication *in situ* annealing of structures to improve material properties; then extracting material properties, such as Q and acoustic velocity, from their electrically measured resonance curves. Using this approach, a 28.658-kHz *in situ* localized annealed version yields a Q of 3,230 twice as high as that of an un-annealed one; and an acoustic velocity of 9,042m/s slightly higher than the 8,024m/s of polysilicon, and inching towards the ~12,000m/s typical of SiC.

POLYSI-FILLED CNT PROCESS FLOW

Fig. 1 presents the fabrication process flow used to achieve devices in polysilicon-filled CNT structural material. This process is based on that of [1] but modified to include an oxide mold that prevents CNTs at the edges of structures from growing sideways. This in turn allows for smaller widths and gaps than those achieved in [1].

The process begins with heavy doping of the silicon substrate using POCl_3 , followed by a blanket deposition of 5 μm -thick silicon dioxide to serve both as an insulating anchor and as the aforementioned CNT-guiding mold. The silicon dioxide layer is then patterned and timed-etched to

Table 1: Deposition Rate and Resistivity Comparison Between CVD polySiC and polySi-filled CNT Grass

		PolySiC [9]	PolySi-Filled CNTs
Dep. Time	3 μm	7.5 hrs	2 mins+3 hrs
	60 μm	6.2 days	10 mins+ 3 hrs
Resistivity [$\Omega\text{-cm}$]		0.02	0.0016

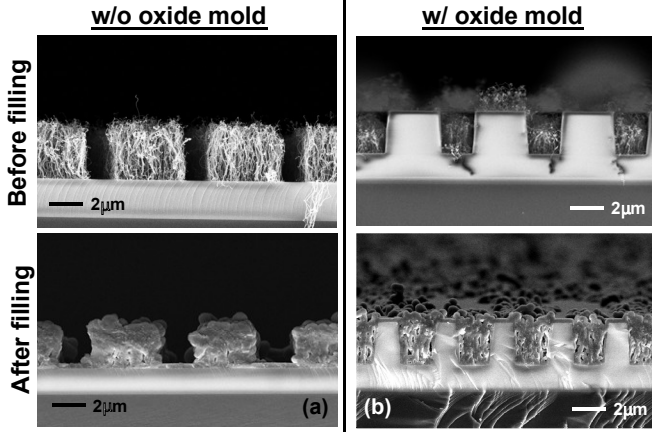


Fig. 2: SEMs of CNT structures grown (a) with and (b) without the oxide mold, before and after polysilicon-filling.

achieve the cross-section of Fig. 1(c), where battlement-like structures rise 3 μm above a 2- μm thick base of blanket oxide. Next, 5 nm of Fe seed layer (for later CNT growth) is evaporated and lifted off the tops of the battlements by removing the remaining photoresist to achieve the cross-section of Fig. 1(d). CNTs are then grown using a gas mixture of $\text{H}_2/\text{C}_2\text{H}_4$ at 720°C in a custom CVD furnace. The lengths of the CNTs, which determine the eventual thickness of the structural layer, are controlled via the growth time—in this case, only two minutes to attain a desired CNT grass thickness of 3 μm to achieve the cross-section of Fig. 1(e). The as-grown CNTs are then filled by depositing a thin layer of LPCVD doped polysilicon, followed by a timed dry etch of polysilicon to remove the polysilicon layer on the surface, but keep the polysilicon in the composite material, to yield the cross-section of Fig. 1(g). Finally, a timed HF dip releases the structures while preserving oxide mesas under large area features that serve as anchors.

It should be noted that the CNT growth rate is substantially faster than the deposition rate of conventional SiC CVD processes. To expand on this, Table 1 summarizes the required deposition time according to a reported deposition rate for polycrystalline SiC CVD [9] compared to the CNT growth time under the conditions described above, plus the fixed 3-hr deposition time for filling with polysilicon. Although the desired thickness for the devices designed here is around 3 μm , this process provides even greater benefits for thicker structures, e.g., 60 μm thick, for which the needed CNT grass requires only 10 minutes of deposition time under the growth conditions used here.

To gauge the efficacy of oxide molding, Fig. 2 compares SEMs of CNT structures grown with (right) and with-

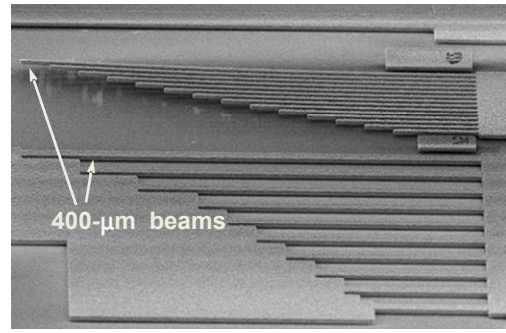


Fig. 3: SEM of polysilicon-filled CNT clamped-clamped and cantilever beam arrays immediately after release, showing little or no curling or buckling.

out (left) the oxide mold, before and after filling with polysilicon. Clearly, the sidewalls are uneven after polysilicon filling without the guiding oxide mold, while the oxide mold confines the growth towards straight sidewalls after filling.

This process not only provides a relatively fast method for fabricating high-aspect ratio structures with small gap structures, it also does so with little stress compared with alternative methods. Fig. 3 confirms this with a SEM showing released clamped-clamped and cantilever beam arrays constructed in polysilicon-filled CNT material with no apparent buckling and much less bending than other approaches. Thus, if this material does end up approximating SiC, it would do so while avoiding many of the stress issues often experienced by structures formed using CVD SiC. Indeed, stress control remains a major challenge for CVD SiC [10].

AS-FABRICATED POLYSILICON-FILLED CNT MATERIAL PROPERTIES

To allow characterization via electrical measurement, this work employs one-mask folded-beam capacitive comb-driven resonators [11], such as shown in Fig. 4(a), as measurement vehicles. As with previous one-mask devices, the anchors in this device are made much larger than any resonator features to allow complete undercutting of the resonator while retaining sufficient oxide under the anchors to hold the structures steadfast to the substrate.

Fig. 4(b) zooms in on the inter-digitated fingers of the device's transducer, showing the 1- μm gaps in \sim 3- μm thick polysilicon-filled CNT material achieved by this process. This SEM also reveals considerable surface roughness immediately after fabrication—a problem to be addressed.

To extract material properties, such as Q and acoustic velocity, the resonance behavior of this device must be measured, and this was done using conventional means, such as described in [12]. Fig. 5 presents the measured frequency response of a 28.482-kHz folded-beam comb-driven device using a 30V-bias under 10^{-4} Torr immediately after fabrication, i.e., with no post-fabrication annealing (to be used later). Here, a Q of 1,636 is measured, which is lower than the Q of \sim 10,000 exhibited by previous LPCVD polySiC comb-driven micromechanical resonators [13]. On first glance, the lower Q seems to arise at least partly from surface energy losses associated with the large surface rough-

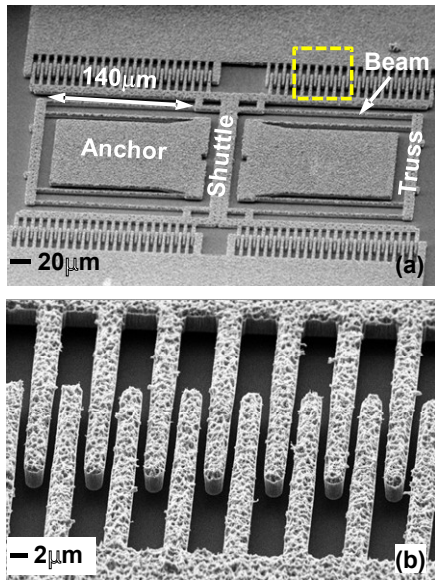


Fig. 4: (a) Overview and (b) zoom-in SEMs of a fabricated polySi-filled CNT comb-driven resonator.

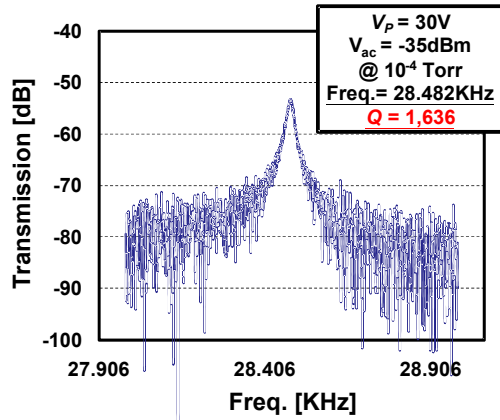


Fig. 5: Electrically measured frequency response of the resonator of Fig. 4 before *in situ* localized annealing.

ness of the structure shown in Fig. 4(b). One look at Fig. 6, however, which presents cross-sections of the polysilicon-filled CNT material, reveals voids that form during the polysilicon filling process and that could be major source of defect-based losses, where hysteretic movement of defects consumes energy, leading to loss and reducing Q .

Although quite porous, the polysilicon-filled CNT material seems to still post a very good resistivity of $1.6 \times 10^{-3} \Omega \cdot \text{cm}$, which is an order lower than reported for previous CVD polycrystalline SiC films [9].

To extract acoustic velocity, the well-known formula for the resonance frequency of a folded-beam comb-driven resonator is used, which can be expressed by [12]

$$\text{Freq.} = \frac{v}{2\pi} \sqrt{2 \left(\frac{W}{L}\right)^3 \frac{1}{A_{\text{equiv}}}}, \quad v = \sqrt{\left(\frac{E}{\rho}\right)} \quad (1)$$

where v is the acoustic velocity of the material governed by its Young's modulus E and density ρ W and L are the width and length of the supporting beams, respectively; and A_{equiv}

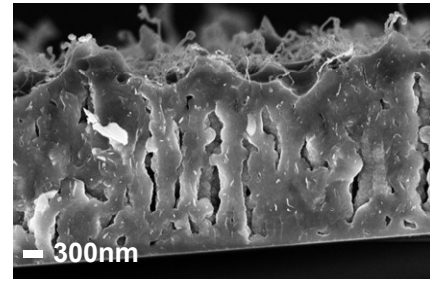


Fig. 6: Zoom-in SEM of the cross-section view of a released structure showing the voids inside the material.

Table 2: Summary Data for Devices Used in Acoustic Velocity Extraction

	Beam Size L/W [μm]	Equiv. Area $A_{\text{equiv.}}$ [mm^2]	Frequency [kHz]
1	140/2	0.0151	28.67
2	150/2	0.0152	25.95
3	160/2	0.0152	23.40

is the equivalent area of the shuttle, truss and beams, determined by considering their respective kinetic energies [12].

Table 2 summarizes measured frequencies for devices with varying beam lengths. By plotting measured frequency versus $1/\sqrt{E}$, as shown in Fig. 7, the acoustic velocity of this material is extracted to be 9,042 m/s from the slope. It should be noted that this direct electrical measurement of resonance provides a more accurate reading of material properties, especially for acoustic velocity, than the non-electrical, non-resonance methods used previously [1].

The fact that the measured value of acoustic velocity sits between the 8,024m/s of polysilicon and the 12,000m/s of SiC seems to suggest that the combination of silicon and carbon in the makeup of this material, although not at the molecular level, still approximates to some degree the behavior of SiC, at least from an acoustic velocity standpoint.

Q-ENHANCEMENT VIA IN SITU LOCALIZED ANNEALING

As already stated, the measured Q of 1,636 is not reminiscent of previous CVD SiC, as it is too low. To raise the Q of the as-fabricated polysilicon-filled CNT material, an *in situ* localized annealing step was applied, which has been demonstrated previously to raise the Q 's of nickel comb-driven micromechanical resonators from 4,103 to 14,172 [14]. Fig. 8 describes the annealing setup: a voltage difference V_a sends electrical current into one anchor, through the structure and its supports, then out the other anchor, all while the device is driven into resonance vibration via an ac voltage provided by a network analyzer. The electrical current Joule-heats the structure and its supports to temperatures exceeding 1300°C when $V_a=4\text{V}$ (predicted by finite element simulation), allowing annealing of the structure while it operates. Fig. 9 presents measured frequency characteristics before and after *in situ* localized annealing under $V_a=4\text{V}$ for one hour and cooling down to room temperature (i.e., $V_a=0\text{V}$), revealing a doubling in Q over the original un-

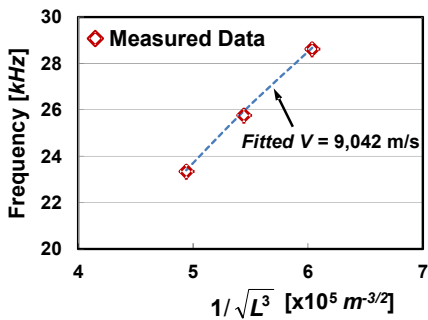


Fig. 7: Curve fitting to extract the acoustic velocity of polysilicon-filled CNT material from frequency information.

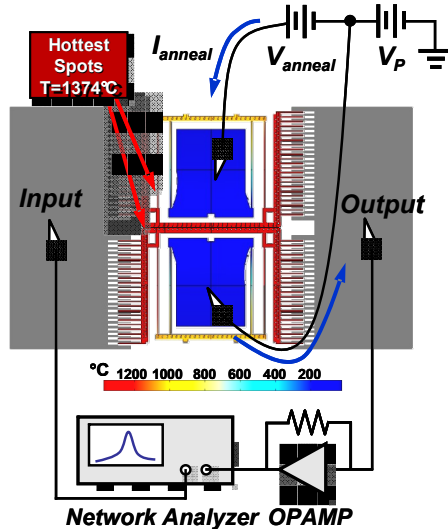


Fig. 8: Setup for in-situ localized annealing and measurement of frequency characteristics.

annealed Q to a value of 3,230.

To provide some insight into the mechanism behind the observed of Q increase, Fig. 10 presents SEMs showing the surface morphology before and after *in situ* annealing, revealing a much smoother surface afterwards. That the Q is higher when the surface is smoother bolsters the suspicion that surface roughness-derived energy losses play a key role in limiting the Q of this material. However, there are other plausible reasons, such as 1) removal of contaminants from the surface and voids of the polysilicon-filled CNT material; 2) hardening of anchors after high temperature treatment; and 3) removal of material defects, which occurs more rapidly when high temperature raises defect diffusivity during device operation help to push them out toward the material surfaces. Whatever the mechanism, the observed doubling in performance is quite welcome.

CONCLUSIONS

Although in no way polished at this time, polysilicon-filled CNT grass material seems to exhibit material properties commensurate with the needs of some lower end MEMS applications. In particular, the measured frequency response for a 28.658-kHz *in situ* localized annealed folded-beam comb-driven resonator in this material yields a Q of 3,230, which is not unusable; and an extracted acoustic ve-

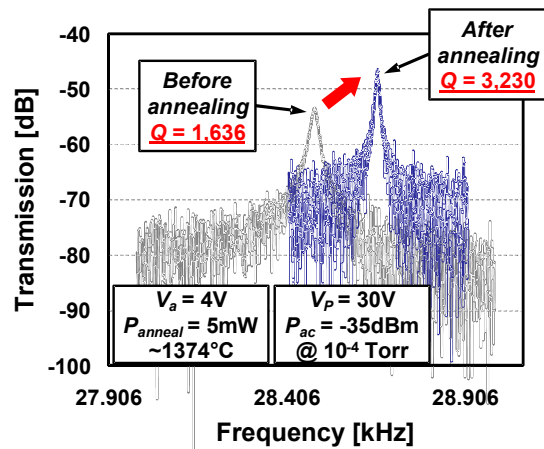


Fig. 9: Measured frequency characteristics before and after *in situ* localized annealing under $V_a=4V$.

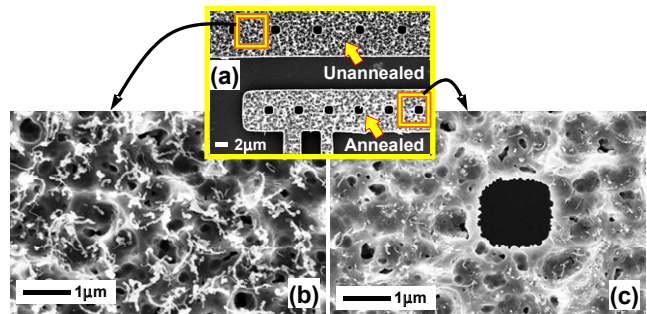


Fig. 10: (a) SEMs of unannealed and annealed parts; and (b)-(c) respective zoom-in views.

locity of 9,042m/s that sits right between the 8,024m/s of polysilicon and the 12,000m/s of SiC, suggesting that the makeup of this material, although not at the molecular level, might still approximate to some degree the behavior of SiC, at least from an acoustic velocity standpoint. If so, then it does so with the further benefits of a considerably faster fabrication process for low-stress high-aspect ratio structures with small gaps. Obviously, further improvements on film quality (e.g., its Q) are needed before any conclusions can be made on whether or not this material really approximates SiC, and how closely (if at all).

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