POST-PROCESSING TECHNIQUES FOR THE INTEGRATION OF SILICON NANOWIRES AND MEMS

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ABSTRACT
Three post-processing techniques key for the integration of nanostructures and MEMS (Microelectromechanical Systems) are presented. The objective is to develop a toolset for integrated NEMS (Nanoelectromechanical Systems) design and processing. More specifically, experimentation is focused on (1) local contact metallization, (2) global metallization for rapid system functionalization and (3) aqueous treatment of self-assembled and suspended silicon nanowires between two MEMS bridges. These techniques are evaluated for their effectiveness and compatibility with integrated NEMS. It is found that local contact metallization effectively alleviates inherent problems at the nano-to-micro contact, while the aqueous treatment confirms that nanoscale components of the system exhibit similar response as their microscale counterparts. Further, the global metallization process enables rapid functionalization as demonstrated in a hydrogen sensing experiment.

1. INTRODUCTION
Nanoscale and NEMS based applications and devices have shown significant promise as a wide range of needs may be met along with improved performance and reduced costs when compared to their microscale counterparts [1, 2]. Post-processing techniques are indispensable steps toward the functionalization, realization and operation of various working NEMS, such as gas sensors, biomedical detectors, electrical interconnects and switches. Here we explore various post-processing techniques and assess the effectiveness of traditional microscale processes on a nano-to-micro integrated system.

Previously, our group has demonstrated localized synthesis and electric-field controlled self-assembly of silicon nanowires with MEMS bridges to yield a two-terminal NEMS device [3, 4]. In contrast to more traditional approaches for integrated nanoscale applications [1, 5, 6], the utilization of localized heating and thus localized nanowire synthesis permits the formation of the nano-to-micro contact in-situ yielding a CMOS compatible platform for NEMS fabrication and functionalization. This work presents key post-processing techniques required towards the development of a functional NEMS sensing system, and to this end we present results on hydrogen sensing capabilities as a demonstration.

2. EXPERIMENTAL
The NEMS studied in this work is fabricated following the process previously presented by our group [3, 4]. Briefly, two suspended MEMS bridges are positioned in close proximity to each other and the synthesis of silicon nanowires using the VLS growth mechanism is initiated locally as a result of the resistive heating of a single bridge. In the presence of a locally acting electric-field, constructed solely between the two bridges, the silicon nanowires follow electric-field lines and link together the two MEMS bridges. The first bridge on which the nanowire synthesis process originates is designated as the growth structure while a second bridge, located nearby, is designated as the bias structure. The two-terminal, self-assembled silicon nanowires based system is schematically illustrated in Figure 1. Post-processing techniques are required to enable and improve the functionality of such NEMS. Here we present our NEMS response to various post-processing steps where the selection of these processes focused on minimizing the complexity of the post-processing step and seeking compatibly with conventional processes, including local contact metallization, global metallization and aqueous treatment. The benefit and impact of these processes are evaluated.

Figure 1: Schematic of the integrated micro-to-nano system using silicon nanowires to link two MEMS structures to yield a two-terminal NEMS by means of localized synthesis and electric-field assisted self-assembly.

Electrically unreliable contacts, and high contact resistances have been consistently plaguing nanoscale-based systems [7, 8]. In an attempt to mitigate these inherent difficulties, a localized metallization step, or a ‘patch’ step was performed following the NEMS self-assembly process. More specifically, in order to improve the electrical properties at the micro-to-nano contact, localized platinum deposition was performed using an in-situ SEM deposition tool (GIS). Approximately 20-40nm-thick platinum covering various cross-sections was deposited locally along the MEMS structures at locations where the nanowires contact the MEMS structures. The localization of the Pt deposition to the respective MEMS structure was verified using an EDX tool and thus ensured that the Pt deposition regions are indeed isolated from each other. Figure 2 illustrates a few examples of the localized Pt deposition. In Figure 2(a) the localized Pt deposition is seen at the nano-to-micro contact at both the growth and bias structures. In the case of the
growth structure (top of the figure), where the exact location of the nano-to-micro contact is difficult to locate (typically in dense growth regions), a wider deposition area is utilized and the presence of platinum deposition is evident by the larger and brighter appearing nanowires. Figure 2(b) shows the close view of the bias bridge in Figure 2(a) and two slightly raised, rectangular regions are shown as the localized deposition regions. The left-hand side region spans approximately 2μm in length and 1μm in width. Figure 2(c) illustrates an enlarged view of the nano-to-micro contact region and a 90nm diameter silicon nanowire is seen to increase in diameter to approximately 130nm as deposition region extends slightly into the gap. The nano-to-micro contact is clearly observed and the boundary of the deposition region of 2.3μmx1.7μm is clearly defined. In order to determine the role of the platinum patch in transport properties of the system, the electrical characteristics of the NEMS are evaluated before and after the localized deposition process as discussed in the following section.

In another approach, a global metallization process was tested as a method for rapid system functionalization. Here, the global metallization step was achieved via the mask-less thermal evaporation of 20nm of palladium onto the as-assembled system. While many metals are compatible with the system, palladium is chosen as it is suitable for hydrogen sensing applications [9]. The thickness of the metallization layer was optimized to permit sensing application while maintaining the individual entity of each nanowire and preventing crosstalk or electrical shorts through or around the MEMS structures or the substrate (as described in [3], the MEMS structures are isolated from the substrate with a 2μm thick oxide). Figure 3 shows the NEMS before and after the global palladium deposition process. The integrity of the self-assembled system appears to be preserved after the deposition process and the micro-to-nano contacts remain intact. The expected increase in nanowire diameter of about 40nm is also observed.

![Figure 2: Nano-to-micro contact regions following the localized platinum deposition process (a) Localized deposition along both MEMS structures. (b) Enlarged view on the localized deposition along the bias bridge. (c) Close-up view of an isolated 2.3x1.7 μm² platinum patch.](image)

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![Figure 3: (a) As-assembled NEMS (the growth structure is the top structure). (b) NEMS following the global deposition of 20nm of palladium for hydrogen sensing applications. (scale bars are 5μm).](image)

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The integrity of the NEMS was further evaluated by subjecting the system to an aqueous treatment, a key component in the functionalization of nanostructures for biological sensing applications [6, 10]. This post-processing step evaluated the ability of a system constructed from suspended nanostructures to survive exposure to an aqueous environment. The experiment involved covering the NEMS and its surroundings with a few milliliters of a low surface tension liquid, isopropyl alcohol (IPA), followed by an accelerated drying step in air inside a 90ºC oven. Figure 4 illustrates the NEMS response to this treatment. It appears that strong surface tension forces make the well-distributed nanowires coalesce together and break most nano-to-micro contacts. The appearance of a fatter growth structure in Figure 4(b) is a result of surface tension effects which force
all nanowires, throughout their length, to hang down around the growth structure. However, by following the aqueous treatment step with a critical point drying step (CPD), it is possible to ensure that most nano-to-micro contacts survive this post-processing treatment as seen in Figure 5.

3. RESULTS AND DISCUSSION

The exposure of this self-assembled NEMS to various post-processing steps provides us with considerable information about the system and its characteristics.

Figure 6 shows the I-V characteristics of the two-terminal NEMS before and after the localized platinum contact metallization. The measurement was taken from a system of only two interconnected nanowires. The resistance to current flow in the system is comprised of the high resistance nanowires as well as the contact resistance at each nano-to-micro interface. Since the localized Pt deposition was isolated to the contact region, the improvement in the current carrying capacity after the localized platinum deposition may be attributed to resistance reduction at the contacts. The high contact resistances are at least partially attributed to the small contact area between the nanowires and the MEMS structures and hence the localized deposition at the contacts assists in increasing the contact area. Following the localized contact metallization, the electrical characteristics show an up to 30pA improvement in the current carrying capacity of the NEMS under an input voltage of 1V. The measurements were conducted using an Agilent 4155 parameter analyzer with fA resolution. The nonlinearity of the I-V characteristics suggests the presence of a non-ohmic contact and requires further investigation.

While the localized patching technique subjected only isolated regions to metal deposition, the global metallization experiment exposed the NEMS and its surroundings to full palladium deposition. It is seen that the state of the system remains unaltered and the nano-to-micro contacts are intact. Curling and increased bending of the nanowires is observed and is primarily due to the thermal expansion coefficient mismatch between palladium and silicon. The evaporation process, however, exposed the NEMS to relatively low temperatures (~100ºC). With the system intact, it was possible to test its performance in a functional application. Here we target a hydrogen sensing system by monitoring the resistance across the NEMS as hydrogen (100ppm in N₂) periodically enters a test chamber. Based on thin film
sensing mechanism, an increase in resistance is expected upon the exposure of a palladium film to hydrogen as the dissociation and diffusion of hydrogen onto the surface promotes increased scattering. Figure 7 shows preliminary hydrogen sensing demonstration in terms of changes in resistance as a function of time upon the periodic exposure of the system to hydrogen gas. The changes in resistance are consistent with theory. Upon the initial exposure to H₂, the resistance across the system shows an instantaneous increase and then proceeds to increase at a slower rate. A gradual decrease in resistance is observed when the hydrogen flow is stopped. It appears that the system reaches a steady state resistance after approximately 5 minutes. With a subsequent exposure to H₂, we note an instantaneous change in resistance approximately half the magnitude of the initial resistance increase followed by the gradual increase in resistance period. The difference in the magnitude of the instantaneous resistance change is attributed to the poor reversibility of the sensor. A more comprehensive cleaning process is required and exposure of the sensor to a hydrogen gettering environment or slightly elevated temperature would ensure a clean reaction surface and better sensor performance.

In another critical NEMS integrity test, the aqueous treatment experiments suggest that the system does maintain its structural integrity upon exposure to an aqueous environment and that the loss of contact occurs only during the drying process due to surface tension effects. Hence, the utilization of the CPD step makes the system compatible with necessary aqueous functionalization environments. In fact, our results show that over 95% of the original nano-to-micro contacts are present following the CPD step. The use of the CPD concept for the elimination of surface tension effects appears as effective with nanoscale components as with it is with microscale components. This simple experiment also provides additional information about the mechanical characteristics of the system. Most importantly, since it appears that the loss of contact to the bias structure occurs most often, we may deduce that the nano-to-micro contact at the growth structure yields a more robust mechanical bond than does the nano-to-micro contact at the bias structure. Synthesis conditions and specifically local temperature are believed responsible for this behavior and could be adjusted to mitigate this shortcoming.

4. CONCLUSIONS

Three post-processing techniques are used to examine the behavior and characteristics of a self-assembled NEMS. This unique silicon nanowire based NEMS is found to be highly compatible with conventional microscale processing techniques exercised here. This feature makes our synthesis and fabrication approach an attractive option for large scale integration as well as parallel nanoscale manufacturing efforts. Furthermore, the mechanical robustness of the system clearly stands out through the experiments while the hydrogen sensor demonstration illustrates a potential niche as well as a processing strategy for the system at hand. In addition, we successfully mitigated high contact resistance problems at the nano-to-micro interface, although while utilizing a serial processing step. Finally, the importance of these experiments transcends beyond this specific silicon nanowire based NEMS as the use such post-processing techniques introduces a wide range of functional opportunities for NEMS integration and manufacturing.

5. ACKNOWLEDGMENTS

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6. REFERENCES