# Semidry release of nanomembranes for tubular origami

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#### ABSTRACT

Three-dimensional tubular origami, fabricated by the self-rolling of functional nanomembranes, is of great interest due to its numerous opportunities for applications in photochemical sensing, intelligent actuators, microrobots, electronics, and many others. A continuing opportunity of this area is in the development of strategies for fabricating tubular origami, in solvent-free and low-cost conditions. This paper proposed a semidry release approach, allowing for the sacrificial layer-free, vapor-assisted self-rolling, and recyclable use of substrates, to create microscale tubular origami. Interface engineering designs that involve hydrophilic and hydrophobic material stacks are introduced to realize the semidry release of nanomembranes, which finally self-roll into multifunctional tubular structures. Systematic experimental and theoretical studies demonstrate the controllability of their dimensions. Finally, a bioresorbable microtube with potential for transient implantable devices is demonstrated. Our present work adds to the portfolio of routes for the construction of tubular origami, which can be utilized as functional platforms for fundamental studies and practical applications.

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Recent advances in materials science and nanofabrication technology have led to the development of three-dimensional tubular origami,<sup>1–6</sup> which can be exploited as functional platforms in applications such as radio frequency passive electronics,<sup>7,8</sup> micromotors,<sup>9</sup> microactuators,<sup>10</sup> photonic mirocavities,<sup>11</sup> and many others. Strategies for creating these tubular structures lie in the mechanical deformation of planar origami via the release of the internal strain existing in them. For example, George *et al.* successfully demonstrated a single-layer fabrication route for creating submillimeter-sized polymer polyhedral structures via controlled cross-linking and capillary deformations.<sup>12,13</sup> One versatile route was demonstrated by the deposition of layer-stacked nanomembranes on a sacrificial layer.<sup>14</sup> The subsequent removal of the sacrificial layer will induce the release of nanomembranes, which finally self-roll into tubular structures driven by the

internal strain. However, the removal of the sacrificial layer normally involves a wet etching process with specific solvent and/or etchant, tending to bring complexities to the fabrication, inevitable damage to the created functional tubular origami, and unwanted collapse of the tubular origami. Therefore, the capability of creating tubular origami via a dry or semidry process becomes an important figure of merit to evaluate the fabrication strategy.

Although a previous study has demonstrated a metal particleassisted dry release method toward self-rolling nanotubes,<sup>15</sup> the involved and necessary high temperature annealing process for creating metallic droplets, which is crucial for the dry release process, makes this method only suitable for temperature-insensitive materials. Directly depositing nanomembranes on a template with coaxial porous structures, for example, Cyclopore polycarbonate membranes,<sup>16</sup> represents a straightforward way to create micro/nanoscale tubes without any solvent or etchant. However, the release of deposited tubes, which involves the wet etch of the template, is required for their future use. Moreover, the excellent homogeneous and conformal deposition is always necessary for this template-based method.

In this work, we propose a sacrificial layer-free, vapor-assisted self-rolling approach, with interface engineering designs, to fabricate tubular origami. Hydrophilic and hydrophobic material stacks can be easily broken to form an interfacial delamination in a humid environment, and the thickness-dependent delamination behavior is investigated. Systematic experimental results and theoretical calculations demonstrate the controllability of the dimension of self-rolled microtubes via tuning the thickness of planar nanomembranes. By utilizing the proposed approach, a set of functional tubular origami with different application prospects is demonstrated. Finally, an example of bioresorbable microtube and its time scale dissolution process are exhibited, suggesting great potential for application in bioimplantable devices and micromotors. The present study demonstrating a semidry fabrication route for creating tubular origami provides the following advantages to existing methods: (1) avoiding the unwanted collapse of the tubular origami, which is normally induced by capillary forces; (2) preventing the solvent/etchant-induced damage to the tubular origami; and (3) reducing the fabrication complexities because the sacrificial layer is not required.

Figure 1(a) shows the schematic illustration of the fabrication process and the key constituent materials. Here, Cr/Au (10/200 nm) is deposited on a cleaned silicon substrate via sputtering [Fig. 1(a-i)]. Then, e-beam evaporation is used to deposit the SiO<sub>2</sub> layer on it, followed by the deposition of nanomembranes [Fig. 1(a-ii)], and the material choice only depends on the expected functions of the finally



**FIG. 1.** (a) Schematic illustrations of the fabrication of three-dimensional tubular origami via the vapor-assisted self-rolling technique. Optical image (b) and SEM image (c) of a typical self-rolling Cu/SiO<sub>2</sub> microtube. Thicknesses of Cu and SiO<sub>2</sub> layers are 30 nm and 50 nm, respectively. All scale bars are 10  $\mu$ m.

formed tubular origami. Exposing the obtained nanomembrane stacks on silicon to a humid ambiance, interface delamination will occur between Au and SiO<sub>2</sub> [Fig. 1(a-iii)]. The corresponding mechanism will be discussed later. Since the internal strain can be introduced into nanomembranes during their deposition process,<sup>17,18</sup> the interface delamination arouses internal strain release, thus resulting in the bending or rolling of nanomembranes [Fig. 1(a-iv)]. Notably, the humidityinduced interface delamination is isotropic. However, during the rolling process, nanomembranes with rectangle shapes will preferably roll along the direction perpendicular to the long side.<sup>19</sup> Finally, the complete delamination induces a self-rolled microtube, as schematically shown in Fig. 1(a-v). The left-over Au/silicon substrate is recyclable for following uses as the temporary supporting substrate. It should be noted that delamination occurs at the interface between Au and SiO<sub>2</sub>, and thereby, the SiO<sub>2</sub> layer is also driven to roll, as the outer surface of the obtained tubular origami. To intuitively exhibit the capability of the proposed method for creating tubular origami, a nanomembrane with a trapezoid pattern is utilized. With this scheme, a Cu microtube with sharp and clear helical edges, which is induced by the self-rolling of the trapezoid-shaped nanomembrane, is obtained, as shown in Fig. 1(b). From the scanning electron microscope (SEM) result presented in Fig. 1(c), the Cu microtube has eight turns with the inner diameter and outer diameter of about 4.86  $\mu$ m and 7.68  $\mu$ m, respectively.

Key aspects of our approach are the selections of Au and SiO<sub>2</sub> to form the interface. On one hand, they are commonly used materials that allow for the micro/nano-fabrication as well as high temperature processing (below the melting temperature of Au, i.e., 1337 K). On the other hand, the adhesion strength between them is rather weak, due to their hydrophobic (Au) and hydrophilic (SiO<sub>2</sub>) characteristics.<sup>20</sup> Therefore, vapor can easily intercalate the interface, resulting in the delamination of the two layers, as schematically shown in Fig. 2(a). The thickness dependence of SiO<sub>2</sub> delaminated from Au is



FIG. 2. (a) Schematic diagram of the vapor-induced delamination of the SiO<sub>2</sub> nanomembrane from the hydrophobic gold surface. (b) Left panels: optical images of the as-deposited SiO<sub>2</sub> nanomembranes, with various thicknesses (30, 50, and 100 nm), on the Au/Si substrate; right panels: optical microscope images of SiO<sub>2</sub> nanomembranes on Au/Si substrates after being exposed to a 52% humidity environment. Scale bars for left panels, right panels, and right insets are 5 mm, 100  $\mu$ m, and 40  $\mu$ m, respectively. (c) Full delamination time of the SiO<sub>2</sub>/Au interface as a function of the SiO<sub>2</sub> layer thickness.

investigated. As shown in Fig. 2(b), full release of a centimeter-scale SiO<sub>2</sub> layer from the Au substrate, exposure to a 52% relative humidity condition, is revealed by the randomly arranged wrinkle structures. Upon further increasing the thickness of SiO<sub>2</sub>, such as 180 nm, immediate delamination occurs when taking the sample out from the ultrahigh vacuum environment. Previous investigation indicates that the energy release rate G, accounting for the breaking energy of the interface bond, increases with the thickness of films.<sup>21</sup> Considering that the energy release rate is related to the effective peeling force by<sup>22,23</sup>  $G \propto F$ , therefore, the effective peeling force of the SiO<sub>2</sub> layer with a thick thickness is larger than that of the thin SiO<sub>2</sub> layer, thus resulting in a much shorter delamination time,<sup>24</sup> as revealed in Fig. 2(c). It should be noted that in practice, however, other factors including the internal strain of the SiO2 deposition layer, microscopic morphologies of materials, and interface qualities may also affect the full delamination time.

The capability of easy delamination at the interface between Au and SiO<sub>2</sub>, which does not involve any sacrificial layer and wet etching process, enables the creation of tubular origami by various materials and material combinations with the material selection only depending on the practical applications. As shown in Fig. 3, examples include (i) Mg/SiO<sub>2</sub>, (ii) Fe/SiO<sub>2</sub>, (iii) Py/SiO<sub>2</sub>, (iv) Ag/Au/Ni/Ag/SiO<sub>2</sub>, and (v) Ag/TiO<sub>2</sub>/SiO<sub>2</sub>. Both Mg and SiO<sub>2</sub> are known as biodegradable and bioresorbable materials,<sup>21</sup> <sup>26</sup> and thereby, Mg/SiO<sub>2</sub>-based microtubes have potential for application in transient devices,<sup>27</sup> such as bioimplantable micromotors.<sup>28</sup> Fe and Py are known as magnetic materials, and measuring the rotational response of the corresponding magnetic microtubes to an external alternating magnetic field offers an opportunity for determining the viscosity and detecting organisms or particles in liquids.<sup>14</sup> Ag as a catalyst can accelerate the decomposition of H<sub>2</sub>O<sub>2</sub> into O2 and H2O, and therefore, our fabricated Ag/Au/Ni/Ag/SiO2 microtubes with the Ag layer at the inner surface can be utilized as micromotors driven by the O<sub>2</sub> bubble production.<sup>29</sup> The intercalated magnetic layer, herein, Ni, is used to guide the propelling direction of micromotors under an external magnetic field.<sup>30</sup> TiO<sub>2</sub> is widely used as the photocatalyst to purify the environment from various pollutants.<sup>31</sup> The present work also integrates TiO<sub>2</sub> within a micromotor, as shown in Fig. 3(v), and then the gathering of these functional micromotors will promote the sufficient mixing in water bodies.<sup>32</sup> It is

foreseeable that our proposed strategy is not limited to fabricating the above-discussed examples; other functional material-based tubular origami is also in prospect.

In terms of practical applications of tubular origami, the corresponding diameter plays vital roles in their performances. For example, the propulsion speed of tubular micromotors was demonstrated to strongly depend on their diameter,<sup>33</sup> and for a tubular optical microcavity, the quality factor and variation mode are also determined by its diameter.<sup>34</sup> Therefore, the ability to control the diameter of the fabricated tubular origami is important. Herein, a bilayer system, i.e., the top nanomembrane and the SiO<sub>2</sub> layer, is considered. Systematic experiments are performed via varying the thickness of both the nanomembrane and the SiO<sub>2</sub> layer, respectively. The thicknesses of the nanomembrane and the SiO<sub>2</sub> layer are kept at 40 nm and 100 nm, respectively. As shown in Fig. 4, either the increase in the thickness of the  $SiO_2$  layer [blue dots in Fig. 4(a)] or the increase in the thickness of the nanomembrane [red dots in Fig. 4(b)] will induce an increase in the tube diameter. Therefore, the diameter of the tubular origami can be scaled by tuning the overall thickness and/or the thickness ratio of the stacked layers.

In order to verify the above conclusion, theoretical predictions of the tube diameter are achieved by numerical calculations. According to the macroscopic continuum mechanical theory,<sup>35</sup> the diameter D of a bilayer tube can be calculated using the following equation:

$$D = \frac{h \left[ 3 \left( 1 + \frac{h_1}{h_2} \right)^2 + \left( 1 + \left( \frac{h_1}{h_2} \right) \left( \frac{Y_1}{Y_2} \right) \right) \left[ \left( \frac{h_1}{h_2} \right)^2 + \left( \left( \frac{h_1}{h_2} \right) \left( \frac{Y_1}{Y_2} \right) \right)^{-1} \right] \right]}{3\varepsilon \left( 1 + \frac{h_1}{h_2} \right)^2},$$
(1)

where *h* is the total thickness of the bilayer system and  $h_1$  and  $h_2$  are the thickness of the first and second layers, respectively.  $Y_1$  and  $Y_2$  denote the Young's modulus of the thickness of the first and second layers, respectively.  $\varepsilon$  represents the in-plane biaxial strain. The theoretical tube diameter varying with the thickness of the first layer (nanomembrane) or the second layer (SiO<sub>2</sub>) is calculated based on Eq. (1). As shown in Fig. 4, the theoretical predictions exhibit good



**FIG. 3.** SEM images of various functional microtubes obtained by the vapor-assisted self-rolling technique: (i) Mg/SiO<sub>2</sub>, 50/50 nm; (ii) Fe/SiO<sub>2</sub>, 200/100 nm; (iii) Py/SiO<sub>2</sub>, 200/ 50 nm; (iv) Ag/Au/Ni/Ag/SiO<sub>2</sub>, 10/10/10/10/50 nm; and (v) Ag/TiO<sub>2</sub>/SiO<sub>2</sub> 30/40/50 nm. Scale bars of (i), (ii), and (iv) are 20 µm and are 100 µm for (iii) and (v).



**FIG. 4.** (a) Experimental results and theoretical predictions of the diameter of selfrolling tubular origami as a function of the thickness of the silicon dioxide layer. (b) Experimental results and theoretical predictions of the diameter of self-rolling tubular origami varying with the thickness of functional nanomembranes, herein, using magnesium nanomembrane as an example for the illustration. Error bars represent one standard deviation of the mean.

agreement with the experimental results, indicating the superior controllability of the tubular origami diameter by our proposed approach.

Finally, a bioresorbable Mg/SiO<sub>2</sub> microtube is demonstrated, as shown in Fig. 5(a). Figure 5(b) shows the optical microscope image of a typical bioresorbable Mg/SiO<sub>2</sub> microtube after the immersion in phosphate buffer solution (PBS, pH = 7.4, at room temperature) for 1 h. Compared to the initial tube, the tube after 1-h dissolution becomes transparent. Because the dissolution rate of Mg is much faster than that of SiO<sub>2</sub>,<sup>26,36</sup> Mg is rapidly dissolved, leaving the transparent SiO<sub>2</sub> tube. Notably, because the SiO<sub>2</sub> tube loses the restriction of the Mg layer, the corresponding diameter becomes slightly larger than that of the initial tube. After the immersion in PBS for about 20 h, most of the SiO<sub>2</sub> tube is dissolved, as highlighted in Fig. 5(c).

Notably, the dissolution of the Mg layer, existing at the inner surface of the tubular origami, involves a hydrolysis reaction,  $Mg + 2H_2O \rightarrow Mg(OH)_2 + H_2$ , as shown in Fig. 5(d). Therefore,  $H_2$  generated within the tube can drive the movement of the tube, i.e.,



**FIG. 5.** (a)–(c) Optical microscope images of the dissolution of a bioresorbable Mg/ SiO<sub>2</sub> microtube after the immersion in phosphate buffer solution (PBS, PH = 7.4, at room temperature) at room temperature. (d) Optical microscope image of a bioresorbable Mg/SiO<sub>2</sub> microtube during the dissolution in PBS, with the hydrogen products (bubbles).

the so-called micromotor, until the complete dissolution of the Mg layer. Since the materials used here are biocompatible and bioresorbable, the easily fabricated Mg/SiO<sub>2</sub> microtube by our proposed approach has the potential for use as a bioimplantable transient micromotor,<sup>37</sup> with advanced capabilities, such as cargo transportation and/or assembly,<sup>38</sup> triggered drug release,<sup>39</sup> and dissolvability when it is no longer needed.

In summary, our present work demonstrates a sacrificial layerfree, semidry release, and vapor-assisted self-rolling approach for the fabrication of tubular origami. The semidry release process is facilitated by interface engineering design, consisting of hydrophilic and hydrophobic material stacks, which allows for the easy intercalation of vapor at the interface, thus resulting in delamination and rolling of the nanomembranes. With this scheme, various functional tubular origamis are created by utilizing different materials and material combinations, revealing the versatility of our proposed approach. Systematic experiments and theoretical calculations of a bilayer system demonstrate the capability of controlling the diameter of tubular origami via tuning the thickness of the SiO<sub>2</sub> layer and/or nanomembranes. Finally, a bioresorbable Mg/SiO<sub>2</sub> tubular origami, with potential for application in transient bioimplantable micromotors, is demonstrated. This work may provide a versatile and controllable strategy for the creation of tubular origami, which can serve as a multifunction platform for fundamental studies and practical applications.

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#### DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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