

# FAST AND VERSATILE FABRICATION OF PDMS NANOWRINKLING STRUCTURES

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## ABSTRACT

A benchtop process is reported to create and pattern nanowrinkling structures on the polydimethylsiloxane (PDMS) surface. Electrical discharge generates a rigid thin film on a uniaxially strained PDMS foundation using a hand-held corona tester. Subsequent relaxation leads to surface wrinkling with varied wavelength dependent on the exposure time. Those nanowrinkles are directed either parallel or orthogonal to linear PDMS microstructures by manipulating the strain field during wrinkling. Compared to E-beam evaporation, UV exposure, or focused ion beam for surface wrinkling, this wet bench process avoids the use of costly equipment and greatly reduces turn-around fabrication cycle.

## KEYWORDS

Surface wrinkling, electrical discharge, nanofabrication, PDMS

## INTRODUCTION

Periodic micro/nanoscale polymer structures are widely used in optical gratings, cell alignment guidance, and micro/nanofluidics. One popular method to create such structures is to utilize mechanical buckling instability. To this end, a rigid thin layer is often deposited on a solid support made of compliant materials such as polystyrene (PS), polydimethylsiloxane (PDMS), or shape memory polymers. Upon compression the thin layer undergoes buckling and self-organizes into microscale or nanoscale wrinkles. Effective approaches of micro/nano wrinkle formation include metal evaporation or sputtering on elastomeric films, UV-ozone irradiation or oxygen plasma treatment of mechanically stretched polymer substrates, and exposure of polymer substrates to focused ion beam (FIB) [1]. Special facilities are essential in these processes for creating the rigid thin layer on the solid foundation. The limited accessibility of these facilities and the associated long processing time, however, compromises the manufacturing efficacy. In this study we report an alternative method using a hand-held corona tester, which can create ordered micro/nanowrinkles within a few minutes on a wet laboratory benchtop. The need for cleanroom environment or expensive facilities is eliminated. This method also allows easy integration with conventional microfabrication to create hierarchical micro/nanostructures.

## FABRICATION OF NANOWRINKLES

The wrinkling process is depicted in Figure 1a. PDMS prepolymer is first mixed with the curing agent at 10:1 and spin-coated. After cross-linking, the PDMS thin film is cut into 55mm×10mm×1mm rectangular strips. They are elongated lengthwise using a tensile loading apparatus ((100Q250-6, Testresources, Shakopee, MN) and held at a strain that ranges from 5% to 70%. The strip is then exposed to electrical discharge generated by a hand-held high frequency corona tester (BD-20AC Electro-technic Products, Chicago, IL) (Figure 1b). During the exposure, the tip of the discharger is positioned 2 mm away from the PDMS strip. After treatment the PDMS strip relaxed to the original length at an unloading rate of 100  $\mu\text{m/s}$ . Low temperature electrical discharge is generated by approaching the discharge tip connected to the output A of the tester to the strip, while keeping another microelectrode B grounded (Figure 1c). The power transformer T1 sets up a high voltage that causes a spark gap to break down at the rate twice of the line frequency (100-120 Hz). The spark gap charges capacitors C1 and C2 that are connected to the primary windings of the resonator coil T2 with an air core. Because of the inductance of primary windings of T2 and capacitors, an oscillating current of very high frequency is set up in the circuit. The spark gap is adjusted to reach the resonant frequency of the circuit about 3.8 MHz. High voltage is thus induced in the secondary windings of T2.

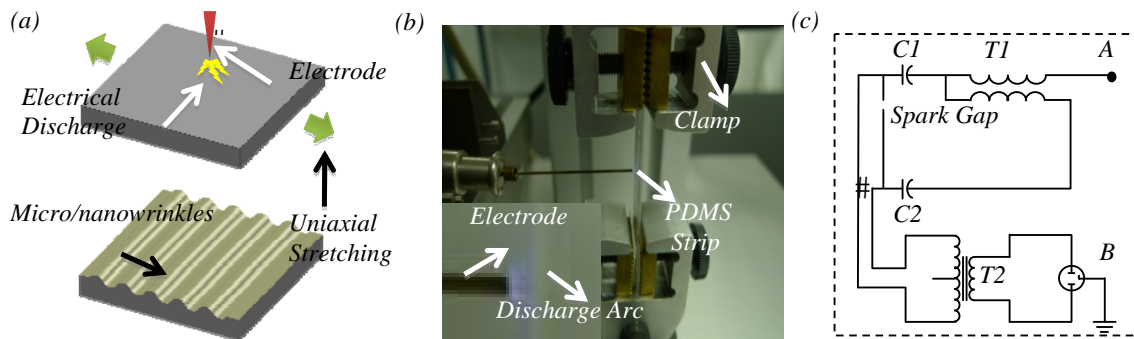


Figure 1 Benchtop wrinkling process. (a) Schematics of surface wrinkling; (b) Experimental setup; (c) Electrical diagram of the corona tester.

Figure 2a shows that wrinkle wavelength increases with the exposure time, while the strain magnitude plays a relatively minor role. For exposure time of 2 min to 10 min, wrinkle wavelength was from 500 nm to 1200 nm. Figure 2b&c shows a typical sinusoidal wrinkling pattern when the PDMS strip is subject to 70% strain and 2 min exposure time. The wrinkle has a 500nm wavelength and forms perpendicular to the strain direction. The depth is about 100 nm, yielding a height-to-wavelength ratio of 0.2.

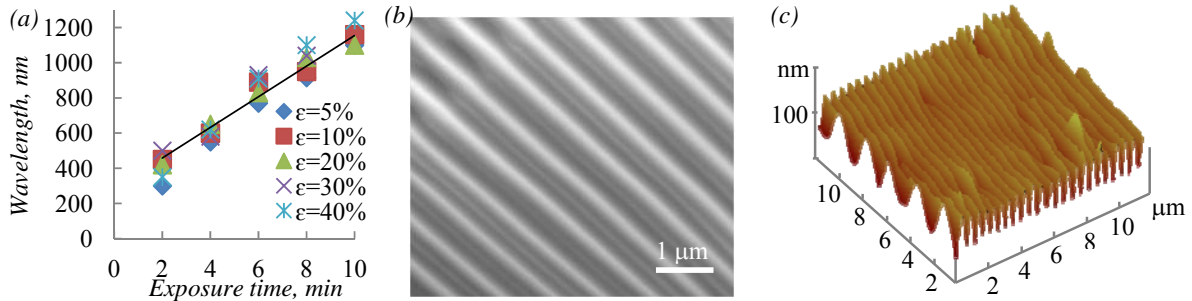


Figure 2 Characterizations of wrinkling features. (a) Wrinkle wavelength vs. Exposure time; (b) SEM micrograph; (c) AFM micrograph.

### PATTERNING OF NANOWRINKLES

Interfacing nanostructures with larger structures is a critical task of nanofabrication. Here, we demonstrate integration of nanowrinkles with linear PDMS microstructures. Figure 3 illustrates the fabrication process. First, an array of semi-cylindrical microstructures is fabricated by reflowing patterned AZ 9260 photoresist on a silicon wafer. The pattern is then transferred to a PDMS substrate by replica molding. Afterwards, the PDMS substrate is stretched uniaxially and held at 5% strain followed by 2 min exposure to the electrical discharge. Continuous nanowrinkles can thus be fabricated on the reflowed structures. The orientation of nanowrinkles is dependent on the stretching direction.

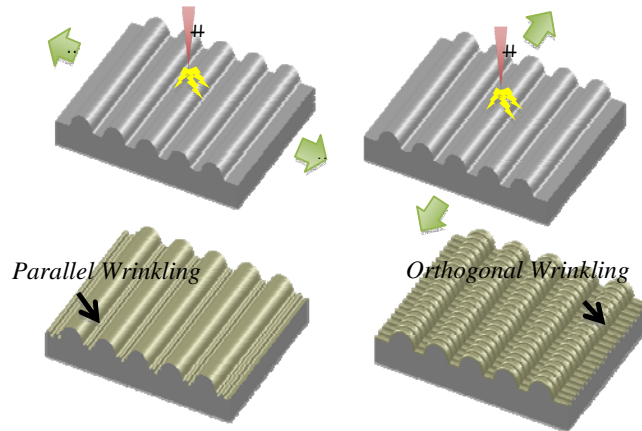


Figure 3 Fabrication of hierarchical micro/nanostructures by benchtop wrinkling.

Results show that for longitudinal stretching, linear wrinkles along the transverse direction form on the bottom surface, the crest and the slopes of microstructures; for transverse stretching, linear wrinkles formed only in the space between microstructures, but not on the crest and the slopes (Figure 4). In these structures, the characteristic dimension of the microstructures is one order of magnitude greater than that of the nanowrinkles.

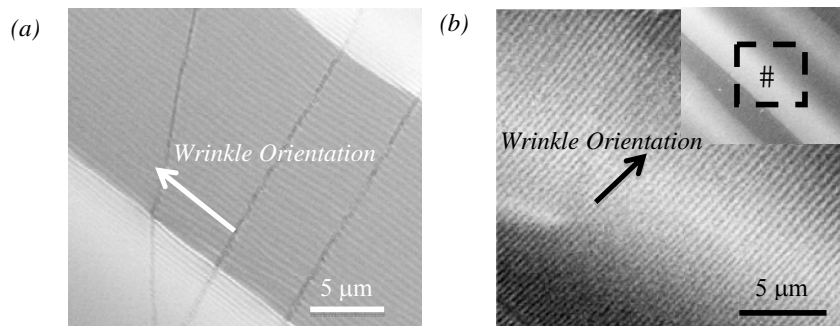


Figure 4 SEM of nanowrinkles (a) parallel and (b) orthogonal to the reflowed microstructures.

The spatial patterning of nanowrinkles with PDMS linear structures is achieved by manipulating the surface strain field and agrees with the finite element analysis (Figure 5). It shows that for a thin substrate with microstructures, the strain magnitude upon substrate stretching is dependent on topography of surface structures. In particular, for a substrate with a linear microstructure, the crest and slopes of the structure exhibited relatively lower strain magnitudes than that of the bottom surface if the uniaxial strain is applied normal to the longitudinal direction of the structure. On the contrary, the crest and the slopes of the structures and the bottom surface exhibited the same strain magnitude when the strain is along the longitudinal direction of the structure. In both scenarios, wrinkles occur only when the prestrain is greater than 3%.

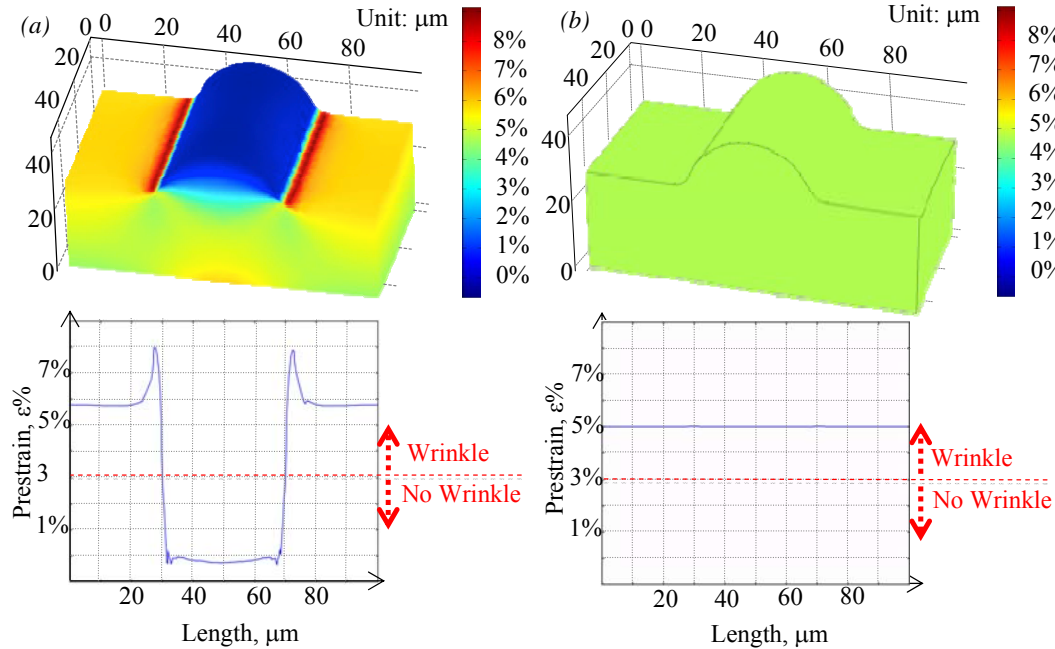


Figure 5 Strain distribution analysis of parallel wrinkling (a) and orthogonal wrinkling (b). Blue curve/line indicates the magnitude of the applied strain.

### CELL ALIGNMENT ON HIERARCHICAL STRUCTURES

Orthogonal microstructures/wrinkling features are especially useful for studying cellular mechanosensitivity. Skeletal myoblasts C2C12 (ATCC, MD) are cultured and seeded ( $1 \times 10^6$  cells/ml) on the pre-sterilized and pre-treated orthogonal micro/nanostructures. Results show that the transverse wrinkling structures, to some extent, disrupts cell alignment by the linear microstructures (Figure 6). The mechanism of such combined features on regulating cellular adhesion and migration deserves further investigation.

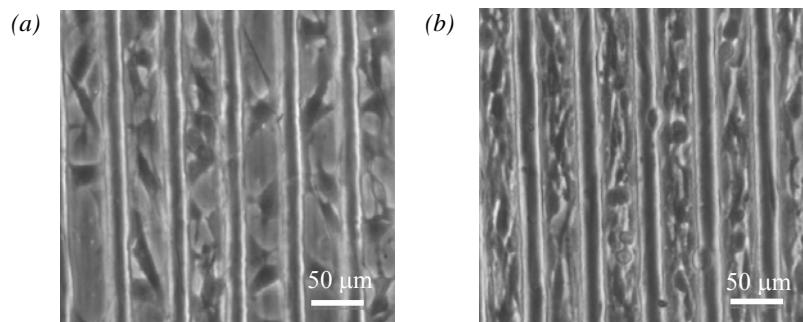


Figure 6 Cell alignment on microstructures (a) with and (b) without orthogonal nanowrinkles.

### CONCLUSION

Periodic micro/nanowrinkles are fabricated on benchtop with minimal cost and complexity. Spatial patterning of wrinkling structures is also achieved that facilitates the use of micro/nanowrinkles in broad applications.

### REFERENCES

[1] Alexandra S, et.al, *Controlled Wrinkling as a Novel Method for the Fabrication of Patterned Surfaces*, Advances in Polymer Sciences, 227:75-99, (2010).

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