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FULL PAPER

Template-Free Vibrational Indentation Patterning (VIP) of Micro/Nanometer-Scale Grating Structures with Real-Time Pitch and Angle Tunability

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A template-free, high-throughput patterning technique named vibrational indentation-driven patterning (VIP), which achieves continuous, period-tunable fabrication of micro/nanometer-scale grating structures, is reported. In VIP, a tilted edge of a hard material vertically vibrating at high frequency makes periodic indentations onto a moving substrate of any material softer than the tool, thereby continuously creating grating patterns at high speed. By modulating the tool vibration frequency, substrate feeding rate, and the tool tilting angle, the period-variable chirped gratings and angle-tunable blazed gratings can be easily achieved; they can be utilized in various opto-electronics and photonics applications. As an example, an infrared polarizer directly fabricated from the VIP-created blazed grating is demonstrated.

1. Introduction

Optical lithography has dominated the micro/nano-patterning for over half a century due to its excellent reproducibility and ability to stack multiple layers of complex geometry.[1] Nanoimprint lithography (NIL) that relies on direct mechanical stamping provides an unique solution to achieve sub-10 nm spatial resolution with high precision.[2] Roll-to-roll (R2R) NIL[3] has opened a way for continuous NIL processing over large area, where a roll bearing a flexible imprinting stamp prints the replica pattern on the substrate roll in a continuous fashion. For seamless and faster patterning, continuous dynamic nano-inscribing (DNI)[4] and nanochannel-guided lithography (NCL)[5] have been introduced to produce linear patterns that only require a thin slice of cleaved grating mold. Nevertheless, all these mechanical-based processes require the original stamps containing nanostructures which should be prepared by low-throughput processes such as electron beam (e-beam) lithography or laser interferometry.

One attractive strategy to overcome the aforementioned issues is to utilize direct mechanical indentation to the micro- and nano-scale patterning as long as an indenting tool is mechanically harder than the substrate materials. With repeated indenting by the tools having relatively simple shapes, the micro/nano-scale patterns can be obtained in a continuous and seamless manner. Indeed, nanohole arrays have been successfully fabricated by using atomic force microscope (AFM) for biomimetic dry adhesives[6] or ultrahigh density data storage applications.[7] However, these approaches require multiple times of tip indentations and still should be accompanied with the prerequisite steps to prepare nanopore membranes[6] or block copolymer templates.[7] A microindentation process using a pyramidal diamond tip has also been adopted to develop micro/nano hybrid patterns on an anodized aluminum substrate, which is however based on the destructive denting of nanostructures under heated conditions.[8] Therefore it is still demanding to capitalize the mechanical indentation to more scalable and controlled patterning at ambient conditions.

Here we present a highly versatile and more practical patterning method, vibrational indentation-driven patterning (VIP), to continuously create micro/nanometer-scale gratings on various materials with real-time tunability of grating periods and profiles. In VIP a tilted edge of a hard material (e.g., a Si wafer) is set to high-frequency vertical vibration, where the tool edge makes sequential indentations onto a moving substrate and continuously create grating patterns at high speed. VIP can make perfectly straight and multidimensional grating patterns onto any substrates softer than the tool material. The grating periods and depths can be easily controlled simply by modulating the tool vibration and/or substrate feeding rate. As a result, the period-variable chirped gratings can be easily achieved. Furthermore, controlling the tool tilting angle leads to the fabrication of angle-tunable blazed gratings. Such structure can be further configured to become optical polarizers after angled-deposition of metal on one side of the blazed planes.

Additionally the nature of sequential indentation of VIP process enables the creation of nano-scale grating pattern over

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infinitely long flexible substrate, by using a finite size indenting tool. Such a utility makes it very attractive to use VIP to produce flexible template for high-throughput R2R NIL.

2. Principle and Design of Vibrational Indentation-Driven Patterning

2.1. Basic Principle of VIP

The working principle of VIP is simple and straightforward. A well-cleaved Si wafer edge (or a flat edge of any hard materials) tilted at a proper angle and vibrating at a desired frequency sequentially indents into a moving substrate to generate V-shaped periodic gratings (**Figure 1**a). In one embodiment, the vibration is generated by a high-speed servo motor controlling a rotation with a mass eccentrically mounted on the head. The period λ of resulting pattern is simply given as $\lambda = V/f$, where f is vibration frequency of the tool and V is the substrate feeding speed. Assuming the edge is perfectly cleaved to 90° and the elastic recovery of a substrate after deformation is negligible, the indented trench width s becomes $s = h(\tan\theta + \cot\theta)$, where h is indented depth and θ is tool tilting angle. Therefore, the period of grating patterns can be easily controlled by modulating the vibration frequency and/or substrate feeding speed. As will be demonstrated later, the real-time modulation of tool-substrate motion and tool tilting degree during a single VIP process can create the grating patterns having varying periods and angles. Also, the trench geometry can be readily controlled by the indenting force (vibration amplitude) and tool tilting angle. Here, VIP has been performed at 45° tilting angle to achieve the symmetric V-shaped patterns unless otherwise specified; later, the tilting angle will be varied to demonstrate the fabrication of asymmetric blazed gratings with different blazed angles.

2.2. Additional Design Considerations

Several design aspects may be taken into account to generate more reliable vibration in VIP. Stable vertical oscillations of the tool at a consistent frequency and with sufficiently high amplitude are of great importance in VIP to achieve uniform and faithful patterns. Vibration force (F) generated from an eccentric mass (m) with the distance from mass center (d) at the rotating speed of ω (see Figure 1b for notations) is given as a function of time t as,

$$F_e(t) = md\omega^2 \sin\omega t \tag{1}$$

The corresponding vertical response z of the total system (motor-assembled tool) with the mass of M is

$$z(t) = \frac{m}{M} d \left(\frac{\omega}{\omega_n}\right)^2 |G(i\omega)| \sin(\omega t - \phi) \tag{2}$$

where ω_n is a natural frequency given as $\omega = \sqrt{k/m}$, and $G(i\omega)$ is defined as $G(i\omega) = (1 - (\omega/\omega_n)^2 + 2i\zeta(\omega/\omega_n))^{-1}$ where ζ is a damping ratio of system written as $\zeta = c/(2\sqrt{mk})$ with arbitrary constant of c. An excitation frequency can be chosen to be away from the system's resonance frequency to avoid the resonance or by increasing the damping factor (e.g., critical damping, $\zeta = 1$). In this work we adopt the former, as it is experimentally more reasonable to implement.

Since the vibrating motor uses a single eccentric mass, there are other degrees of vibrating motion apart from the vertical direction. To enable vertical indentations (i.e., along the z-axis as specified in Figure 1) with minimal lateral (i.e., x- and y-axes) perturbations, the tool mounting setup is designed to have much higher stiffness in the x- and y-directions.

Similar to our previously demonstrated techniques such as DNI and NCL that also use tilted edges, conformal contacting to the substrate is a key criterion for reliable patterning in VIP. Hence, polymer substrates having a certain level of compliances are desirable to accommodate conformal contact. This also helps extend the tool life.

3. Results and Discussion

3.1. Fabrication of Uniform Period Gratings with Easy Control of Pattern Period and Depth

The micro/nano-scale grating structures of arbitrary periods can be fabricated by VIP without prefabricated stamps. The grating period can be easily controlled by modulating vibration frequency and substrate feeding speed. **Figure 2** shows scanning electron microscope (SEM) images of grating patterns having uniform periods fabricated on polyethylene terephthalate (PET) substrates. With the vibration frequency fixed vibration at 150 Hz (corresponding to ≈9000 rpm motor rotation), a 250 µm s^{-1} substrate feeding speed produces ≈1.6 µm periodic pattern (Figure 2a). The same vibration frequency with 100 µm s^{-1} feeding speed yields ≈660 nm periodic pattern as shown in Figure 2b. Likewise, the period can also be controlled by adjusting the vibration frequency at constant feeding speed.

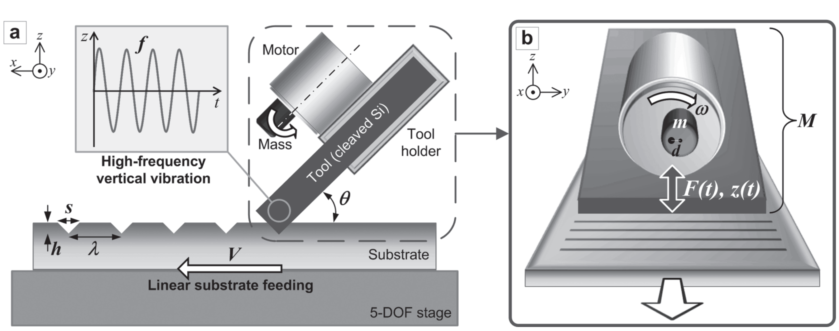

Figure 1. Schematics of VIP with the process parameters indicated: a) side view and b) perspective view. A mass eccentrically mounted to the high-speed motor generates high-frequency vertical vibration at the tool tip, creating periodical indentations into a moving substrate to continuously produce micro/nano-scale grating structures with V-shape trench profile.

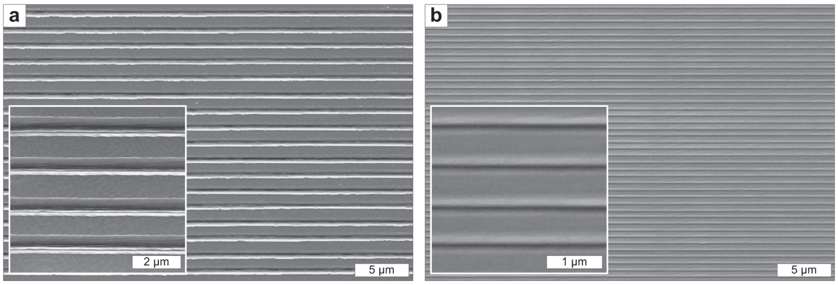

Figure 2. SEM images of micro/nanometer-scale gratings fabricated on PET substrates by VIP. Different substrate feeding speeds of a) 250 µm s^{-1} and b) 100 µm s^{-1}, under the constant vibration frequencies of 150 Hz, lead to 1.6 µm and 660 nm uniform period gratings, respectively. Insets reveal a) deep and b) shallow pattern profiles controlled by the tool-substrate gap during VIP. Note the patterns shown in the insets to (a,b) have different periods.

Here the pattern profile (e.g., trench depth) can be controlled by adjusting the indenting force which depends on the contacting point of the oscillation cycle. When indentation occurs at the midpoint of the oscillation cycle where the force is the maximum, the deepest trenches can be achieved (inset to Figure 2a). On the other hand, if the tool just touches the substrate at the lowest point of oscillation cycle, very shallow trenches are created (Figure 2b).

By combining higher vibration frequency and slower substrate feeding, the resulting pattern period can become essentially as small as desired. The practical limit, however, emerges from several issues. The sharpness of the indenting tool needs to be guaranteed. It is more challenging for the vibration at very high motor rotation rate (i.e., faster than ≈25 000 rpm in our setup) to stay at steady and uniform stroke because of the motor resolution. The motorized linear stage used for substrate feeding also has the minimum incremental speed (i.e., ≈25 µm s^{-1} in our setup). Aside from these instrumental limitations, the principle of VIP process also predicts that the vertical response becomes larger as the vibration frequency increases (see Equation (2)); this is counterproductive to the fact that smaller pattern typically requires smaller indentation amplitude. Considering all these aspects, the smallest feature size that VIP can reliably achieve with our current experimental system is ≈100 nm.

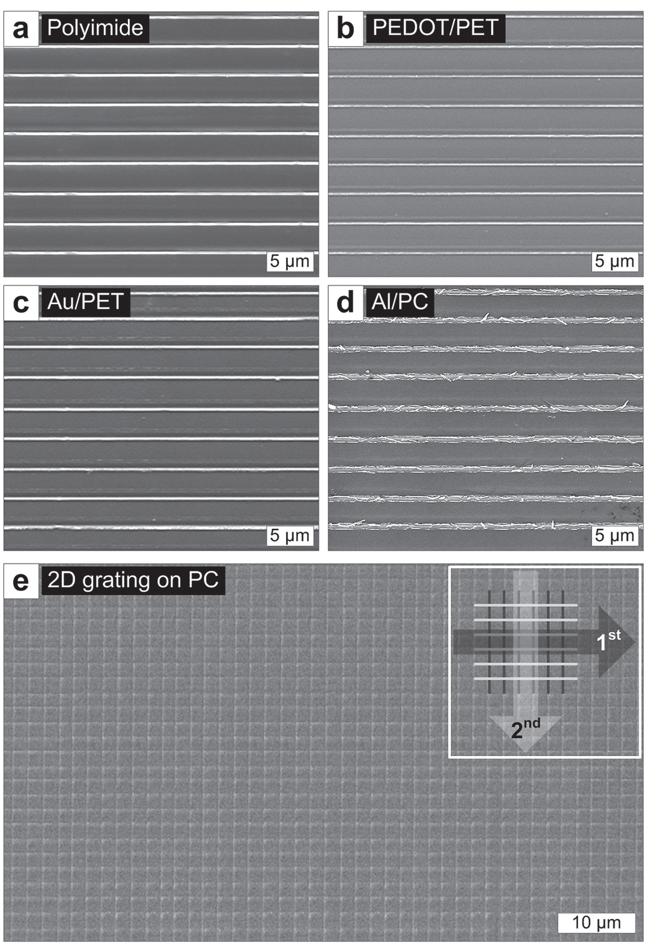

Figure 3. a–d) Grating patterns with 3 µm periods fabricated by VIP on various substrates: a) polyimide, b) PEDOT-coated PET, c) 50 nm Au-coated PET, and d) 50 nm Al-coated PC. e) 2D grating structures of 2 µm period fabricated on PC by two sequential VIP processes along the perpendicular axes as illustrated on the upper-right corner.

3.2. Applicability to Various Substrate Materials and Multidimensional Patterns

Based on pure mechanical indentation, VIP can create grating patterns on any substrate material softer than the tool material. Essentially the substrate does not require any pretreatment (e.g., surface chemical modification or coating of a resist material), which is a great benefit over conventional lithographical technologies. Furthermore, since VIP does not involve any heating or chemical agent, it can be used to pattern thermosensitive organic materials at ambient environment.

Figure 3 demonstrates ≈3 µm period grating patterns fabricated by VIP on various substrates. A polyimide film, widely useful for its excellent chemical/mechanical stability, is typically uneasy to pattern due to very high glass-transition temperature (T_g > 350 °C) and low toughness, but VIP can readily make clear patterns as shown in Figure 3a. Poly(3,4-ethylenedioxythiophene) (PEDOT) is a key conductive polymer material popularly used in organic light-emitting and photovoltaic applications,[9] yet is vulnerable to the exposure to heat and chemicals. Although we previously demonstrated that localized heated-DNI (LH-DNI) could create nanogratings on PEDOT and poly(3-hexylthiophene) (P3HT),[4] the applied heat might affect the properties of those organic materials over times. Here, VIP provides a safer solution to fabricate grating pattern on the heat-sensitive organic layers at room temperature, as exemplified by PEDOT patterns on PET as shown in Figure 3b.

The thin metal film deposited on a polymer film can also be ‘cut’ into the metal grating structures as VIP makes sufficiently deep indentations into the polymer substrate. For example, a 50 nm-thick Au film coated on a PET substrate is cleanly cut to form discrete lines (Figure 3c). Similar is for an Al layer on a polycarbonate (PC) substrate (Figure 3d); the debris generated

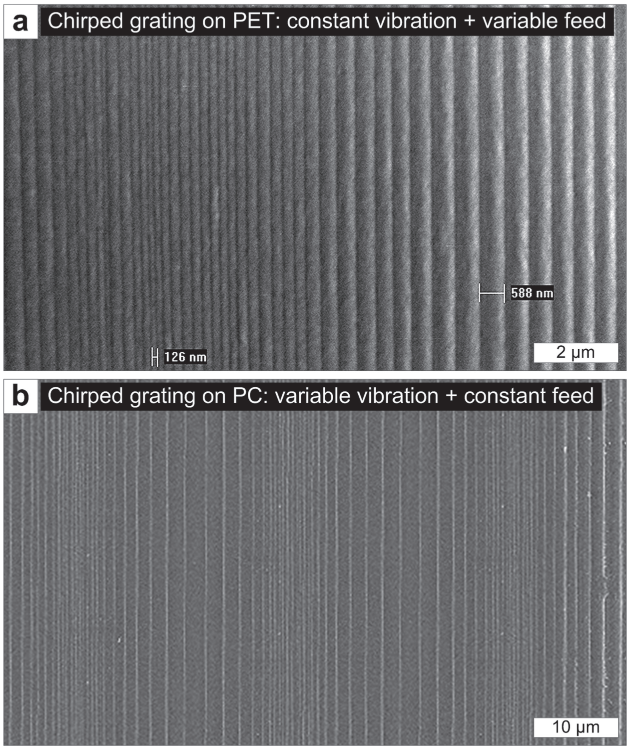

Figure 4. Grating patterns having variable periods (chirped gratings) fabricated by VIP in a single process a) on a PET substrate by modulating the substrate feeding speed between 25 and 120 µm s^{-1} under constant 200 Hz vibrational indentations and b) on a PC substrate by modulating the vibration frequency between 40 and 200 Hz under constant 100 µm s^{-1} feeding.

by the fracture of Al due to its lower toughness can be removed by subsequent cleaning such as ultrasonic agitation.

Since the tool does not touch the substrate surface between indentations in well-controlled VIP, the grating structure first formed by VIP along one direction is not disturbed by the subsequent VIP process along another direction. This allows sequential VIP processes on the same substrate to create multidimensional pattern structures. Figure 3e demonstrates the 2D grating structure faithfully fabricated by two VIP processes sequentially performed in orthogonal directions. Note that such flexibility is difficult to obtain in conventional NIL as well as the inscription-based methods such as DNI and NCL. Hence, VIP can provide a practical alternative to easily and rapidly create 2D patterns which are useful for certain optical and electronic applications. For example, such 2D micro/nanopatterns can be adapted to the light-trapping surfaces in photovoltaic cells to increase the energy conversion efficiency.[10]

3.3. Real-Time Fabrication of Period-Variable Chirped Gratings

The period-variable, namely, "chirped" gratings are useful for a variety of applications ranging from light dispersing devices to high-power ultrafast lasers,[11] but the fabrication of chirped gratings has mostly relied on e-beam lithography of very low processing speed. Recent effort to produce chirped gratings in faster and more straightforward way has been made based on gradually increased surface strain from the stretched PDMS with geometric gradient,[12] but the precise pitch control is still challenging because it needs a delicate pre-fabrication step for the PDMS stamps that cannot be modified during the process.

Providing a breakthrough solution, VIP enables a single-step fabrication of chirped grating structures by real-time modulating the indenting frequency and/or substrate feeding speed during a single process. **Figure 4** demonstrates chirped gratings fabricated in two ways; periods are real-time varied by either modulating the substrate feeding speed between 25 and 120 µm s^{-1} under constant 200 Hz vibration (Figure 4a) or modulating the vibration frequency between 40 and 200 Hz under constant 100 µm s^{-1} feeding (Figure 4b). This process is especially practical as it can be performed in a very simple, single stroke without the need of stamps. Such grating patterns having steeply varying periods can further be applied to optical color filters[13] and plasmonic antennas.[14]

3.4. Fabrication of Angle-Tunable Blazed Gratings and Application to IR Polarizers

VIP trenches are intrinsically of V-shaped profiles (i.e., blazed grating) as originating from the indentation of a right-angle cleaved Si edge. Blazed grating structures have served as essential components for optical diffractions[15] and derivative applications such as optical demultiplexers,[16] beam deflectors,[17] and field-of-view enhancement in imaging systems.[18] However, fabrication of blazed gratings has typically relied on diamond ruling or complex procedures involving reactive ion-etching and/or wet-etching of masked substrates in tilted angle.[19]

Here VIP provides a facile, mask-free route to creating an angle-tunable blazed grating in a single step by controlling the indenting angle θ, as depicted in **Figure 5a**. Figure 5b shows the cross-sections of blazed gratings fabricated on perfluoroalkoxy (PFA) films by VIP at different tool-tilting angles, demonstrating successful and easy control of the blaze angles. As the process is purely based on mechanical deformation free from chemical or wet process, a very clean and smooth sawtooth profile can be obtained, as confirmed by the AFM profiling over the ≈5 µm-period 20°-blazed grating fabricated on PC (Figure 5c). Smooth surface is essential for high performance optical blazed gratings.

The 45°-blazed grating structure formed on a transparent film can function as micro-prism arrays that can be used in light guide plates[20] or diffusion films[21] in liquid crystal display (LCD) panels. Furthermore, when one side of prism planes is covered with metal, the structure conducts directional transmission of incident light that realizes autostereoscopic three-dimensional displays.[22] Here we show another application by demonstrating a metal wire-grid polarizer, which is fabricated by shadow-evaporating a 50 nm-thick Al layer on one side of the 45°-blazed grating on a PFA film. The period of a blazed grating used is ≈1 µm, which works for mid-IR range. Figure 5e shows the optical transmission of transverse-magnetic (TM) and transverse-electric (TE) modes measured on Fourier transform infrared spectroscopy (FT-IR) by using the polarized incident IR light. The structure achieved ≈80% transmission in

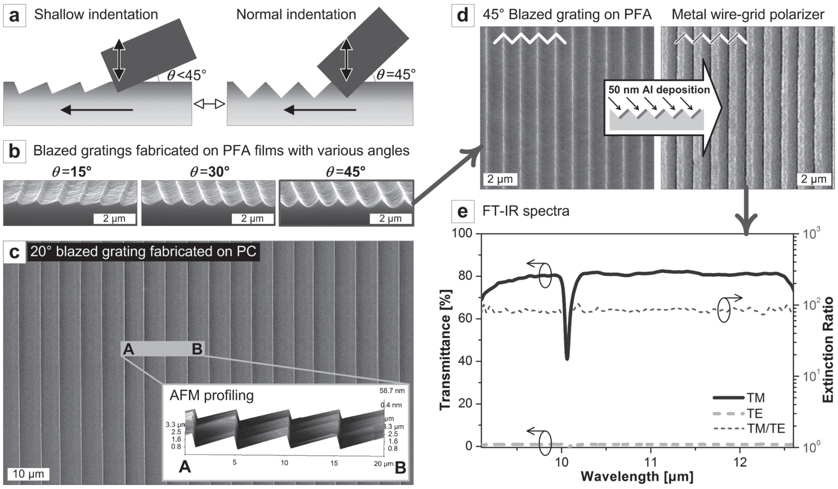

Figure 5. a) Schematic illustrations of blaze angle control by adjusting the tool tilting angle in VIP. b) Cross-sectional SEM images of blazed gratings fabricated on PFA by VIP with different tool tilting angles. c) 20° blazed grating structure with 5 μm period fabricated on PC. Inset to (c) shows the enlarged topology characterized by AFM across the four pitches, indicating clear blazed grating profile. d) SEM images of 45° blazed gratings formed on PFA, before (left) and after (right) 50 nm Al shadow evaporation. e) Measured TM and TE transmission from the Al-deposited 45° PFA blazed grating using polarized incident IR light, along with calculated extinction ratio (TM/TE).

TM mode with well-suppressed (<1%) TE transmission. The extinction ratio (i.e., ratio of TM to TE transmission) is quite uniform at ≈85–90 in the measurement range. The transmission dip emerging in ≈10 μm wavelength originates from the specific absorption of the PFA substrate, which can be removed by using different substrate materials.

4. Conclusions

In conclusion, we have demonstrated VIP, a template-free, high-throughput patterning technique, that realizes continuous, period-tunable fabrication of micro/nano-scale gratings by vertical indentations of a vibrating flat tool edge on a moving substrate. As VIP is based on mechanical deformation, any materials softer than the tool can be patterned in one or multiple directions with full control of grating periods. Real-time modulation of vibration and substrate feeding enables a single-step fabrication of chirped gratings. The blazed gratings with various blaze angles can also be readily created by adjusting the tool tilting. With its simple control and great versatility, VIP may be applicable to continuous and scalable fabrication of functional gratings and find further applications in optics, electronics, and energy conversion devices.

5. Experimental Section

VIP Processing: A high-speed DC motor (up to 24 000 rpm) with an eccentric mass (≈10 g) was fixed to an aluminum tool holder. The motor was connected to an adjustable resistance circuit to allow real-time speed control. A well-cleaved Si piece was mounted to this motor-assembled tool holder, which was inclined typically at an angle of 15–45° with respect to the substrate plane. This tool assembly unit was designed to be as light as possible to maximize the vibration amplitude and also to avoid resonance. A substrate was placed on a thin polydimethylsiloxane (PDMS; Sylgard 184, Dow Corning Corp.) pad attached to a homemade 5-DOF stage (components from Newport Corp.). The PDMS pad provided the attached substrate surface with an ideal flatness by conformal contact to maintain the uniform distance from the vibrating tool edge, thereby ensuring stable and consistent periodic indentations throughout the process. By adjusting the 5-DOF stage, the tool edge and the substrate surface were positioned to be parallel with a small gap controlled to be within the oscillation amplitude of the tool edge. With the motor turned on, the substrate was transferred by a motorized linear motor (CMA-12CC, Newport Corp.) at a controlled speed (≈25–250 μm s^{-1}), to create grating patterns on a substrate by periodical vertical indentations of a tool edge.

Substrate Preparations: All polymer substrates (PET; Melinex 454, Tekra Corp., PC; Lexan 8010, Tekra Corp., and polyimide; Kapton HN, DuPont) were used as purchased or after IPA cleaning followed by nitrogen drying. Electron beam evaporation (Enerjet Evaporator, Denton Vacuum, Inc.) was performed in case for the deposition of metals (e.g. Au, Al). For the coating of PEDOT, the as-purchased PEDOT (Clevios PH 500, Heraeus Holding) was spin-casted on a cleaned PET substrate (2000 rpm, 30 s) and then baked (110 °C, 15 min) to remove residual solvent.

Characterizations: SEM (Philips XL30-FEG) imaging was carried out typically at the operating voltage of 10–25 kV, after sputtering a thin Au film (≈3–5 nm) to avoid electron charging, if needed. A Veeco Dimension Icon AFM was used for the AFM profiling under the soft-tapping mode (sweeping rate of 2 Hz, 1024 lines per sample). The FT-IR measurement was performed using a Spectrum GX FT-IR spectroscopy (Perkin-Elmer) with the incident IR wavenumbers swept from 1100 cm^{-1} to 800 cm^{-1}) through a high-quality IR polarizer (KRS-5, Perkin-Elmer).

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