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Abstract: Surface-enhanced Raman spectroscopy (SERS) is an optical technique for molecule identification. However, fabrication of flexible and structured SERS substrates for performance improvement in a facile and cost-effective manner is challenging. In this work, we reported a polytetrafluoroethylene (PTFE)-based flexible SERS substrate by femtosecond laser direct writing (FsLDW) technology. The femtosecond laser-treated PTFE surface is 3D hierarchical micro-/nano-structures, and the structured PTFE-based SERS chip shows excellent performance enhancement. As a result, $10^{-7}$ M can be detected, which shows excellent potentials in developing flexible SERS for wearable electronics.

Index Terms: Raman spectroscopy, ultrafast lasers, ultrafast nonlinear processes.

1. Introduction
Surface-enhanced Raman spectroscopy (SERS) is an optical technique for molecule identification [1]–[5] and shows distinguished advantages in chemical and biological analytics [6]–[10]. Particularly, SERS can obtain a molecular fingerprint with high selectivity and sensitivity in a non-destructive manner [11]–[15]. Typically, SERS substrates show strong Raman scattering because electromagnetic (EM) field is significantly enhanced on the tip of plasmonic materials (e.g., Au, Ag) [16]–[20]. Recently, to improve SERS performance, various SERS substrates with micro-/nano-structures have been developed based on EM field enhancement mechanism. The improvement of SERS performance is attributed to creating interparticle nanogaps, localized surface plasmon resonance (LSPR) effect, incident light standing wave effect, the increasing interaction time between exciting light and matter [21]–[25]. As a result, there have been developed various SERS substrates with micro-/nano-structures for performance improvement [26]–[31].
to now, there are successful approaches (e.g., metal colloid self-assembly, soft lithography, uniaxial stretching) and materials (e.g., biocompatible poly(ε-caprolactone) film, PMMA) for the fabrication of flexible SERS substrates [32]–[37]. For example, Sharma et al. proposed “nano-fingers” on silicon nanowire arrays as SERS substrate, in which the silicon nanowire arrays are fabricated by using deep-ultraviolet lithography [38]. Li et al. prepared superhydrophobic-superhydrophilic silicon plate for SERS trace detection by metal-assist chemical etching and selectively electrochemical deposition [39]. Wang et al. fabricated porous silicon photonic crystals for SERS by electrochemical anodization [40]. Tian et al. demonstrated 3D SiO$_2$ nano-grids for SERS substrates with maximizing multiple coupling effects by using electron-beam lithography [41]. Choi et al. developed periodic MgF$_2$ nanopillar arrays on a silicon wafer for highly reliable SERS substrates by combining nanoimprint lithography and electron beam evaporation [42]. Nevertheless, it is still urgent to develop new enhancing substrates with hierarchical micro-/nano-structures in a facile and cost-effective manner despite the big success.

Laser processing has become an emerging cutting-edge technology for the preparation of hierarchical micro-/nano-structures in simple, high-speed, large area manners [43]–[48]. Recently, few SERS substrates have been fabricated by laser technologies [49]–[54]. For example, silica, sapphire, and polymethylmethacrylate polymer-based SERS substrates have been successfully demonstrated by femtosecond laser ablations [51], [52]. Bai et al. prepared periodic surface structures for SERS chip by femtosecond laser (Fs) assisted wet etching [53]. Lao et al. successfully developed nanogap plasmonic structures for localized SERS sensing by laser printing and capillary force-driven self-assembly [55]. Botta et al. demonstrated 3D structured aluminum sheets based on SERS substrate by laser engraving technology[56]. However, the fabrication of flexible and large SERS substrates by a femtosecond laser is rarely reported, especially for future wearable devices.

Herein, we proposed a polytetrafluoroethylene (PTFE) based flexible SERS substrates with hierarchical micro-/nano-structures by Fs laser direct writing (FsLDW) technology. In addition, PTFE shows excellent heat, acid, and alkali resistance, having abroad applications in power, automotive, and aerospace industries [57]–[59]. Flexible SERS chips were prepared by silver nanoparticles thermal evaporation on the structured PTFE substrates. Due to the flexibility of the PTFE membrane, the SERS chip can twine on a metal wire. In addition, because of the hybrid micro-/nano-structures on PTFE membranes, the SERS chip shows high performance by enhancing the internal light reflection and the LSPR.

2. Experimental Details

2.1 Fabrication of SERS Substrates

The flexible and structured PTFE SERS substrate was fabricated by Fs laser technology. Firstly, PTFE membranes (thickness, $\sim$100 $\mu$m) were cleaned with ethanol and deionized water. A Roban Nano System (Jicheng Ultrafast Equipment, Ltd) was employed for laser structuring PTFE membranes (L-PTFE). As for the relationship between laser processing parameters and PTFE structure, the wider the pulse width and the higher the Fs laser’s repetition frequency, the wider the resulted linewidth of the L-PTFE surface [60]–[62]. As for the period, when the period is too small, the laser scanning will repeatedly destroy the structure before fabricated. When the period is too large, the micro-/nano-structure is not enough. Therefore, an appropriate period is needed [63]–[65]. As a result, the detailed Fs laser processing parameters are as follows: $\lambda = 1030$ nm, the pulse duration $= 290$ fs, the repetition rate $= 200$ kHz, the laser power $\approx 1500$ mW, respectively. SD400B-Multisource Organic Molecular Vapor Deposition System conducts the thermal evaporation.

2.2 Characterization and Measurement

The confocal laser scanning microscopy (CLSM) images were obtained by LEXT 3D measuring laser microscope (OLS4100). The scanning electron microscopy (SEM) images were collected by
Fig. 1. Schematic Illustration of Fs laser micro-/nano-fabrication technology for flexible and structured SERS substrates based on PTFE membranes. The inset is a photograph of a flexible L-PTFE-Ag based SERS substrate twining on a metal wire.

a JEOL field-emission scanning electron microscope (JSM-7500). The Raman signals were taken by LabRAM HR Evolution (HOARIBA).

3. Results

After laser treatment, flatten PTFE membranes become rough. Silver was coated on the rough PTFE membranes by thermal evaporation (L-PTFE-Ag); flexible L-PTFE-Ag SERS substrates were designed and fabricated. As shown in the inset of Fig. 1, the flexible L-PTFE-Ag SERS substrates are capable of twining on a metal wire (diameter, \( \sim 1.17 \) mm), which demonstrated outstanding flexibility. Additionally, instead of the outstanding flexibility, the L-PTFE-Ag membrane can be used to develop high-performance SERS substrates because of the LSPR effect and 3D micro-/nano-structures. The 3D micro-/nano-structures are helpful to enhance the internal light reflection, leading to the improvement of SERS performance [66]–[71].

In order to characterize microstructures of samples, CLSM was used to examine the surface morphology of PTFE, L-PTFE, and L-PTFE-Ag (Fig. 2). The pristine PTFE surface is flat, and there are some wrinkles on the surface (Fig. 2(a)). The wrinkles may be introduced in the PTFE preparation process by mold pressing. The wrinkles are inapparent, which may limit the PTFE membrane in the fabrication of high-performance SERS substrates. After the laser treatment, gratings (\( T \approx 25 \) \( \mu \)m) could be observed (Fig. 2(b)). Simultaneously, there are micro-/nano-structures on the edge of the grating and the untreated PTFE surface. The micro-/nano-structures on the grating edge may be attributed to the instantaneous high energy interaction between Fs laser and PTFE. The micro-/nano-structures on the untreated PTFE surface maybe originate from debris that splashed on untreated areas during the interaction between Fs laser and PTFE. When it comes to the L-PTFE-Ag surface, the L-PTFE-Ag surface shows distinct bright gray due to Ag film's color (Fig. 2(c)). Ag film thickness is \( \sim 18 \) nm, which is precisely controlled by the thermal evaporation equipment. Compared with the micro-/nano-structures of L-PTFE, Ag film's nanoscale thickness has nearly no effect on the microscale surface morphology between L-PTFE and L-PTFE-Ag. As shown in Fig. 2(d), each grating’s width and height can be extracted from L-PTFE-Ag's 3D perspective. The average width is \( \sim 4 \) \( \mu \)m, and the average height is \( \sim 7.6 \) \( \mu \)m.

To investigate the micro-/nano-structures of samples, SEM has been adopted to reveal the surface morphology of PTFE, L-PTFE, and L-PTFE-Ag (Fig. 3). Not surprisingly, the PTFE surface
Fig. 2. Top-view CLSM images of (a) PTFE, (b) L-PTFE, and (c) L-PTFE-Ag surfaces. (d) 3D view and corresponding sectional profile of the L-PTFE-Ag surface.

Fig. 3. SEM images of (a) PTFE and (b) L-PTFE. The enlarged SEM images of (c), (d) L-PTFE and (e), (f) L-PTFE-Ag surfaces.
is flat. Besides, the L-PTFE surface is rough and has grating micro-structures (Fig. 3(a), (b)). It is worth mentioning that there are many nanoporous structures on the surface L-PTFE (Fig. 3(c), (d)). The focused Fs laser shows a high temperature at the focus point. Then PTFE decomposes in the form of gas when the Fs laser interacts with PTFE. As a result, the gas escaped from PTFE materials, leading to nanoporous structures. After Ag-coating, compared with the L-PTFE surface, there are many silver nanoislands on the L-PTFE surface (Fig. 3(e), f). After the Ag-coating, there are two advantages: (1) The Ag nanoislands help further enhance the internal light reflection. (2) The Ag nanoislands provide hot pots by the LSPR. The combination of these two advantages mentioned above plays a vital role in developing PTFE-based SERS substrates. Appropriate diameter and spacing of silver nanoparticles (AgNPs) will help to improve the SERS performance [1], [69], [72]. As a pioneer, Kumar et al. reported flexible SERS substrates with AgNPs on grating PDMS structures that combine LSP and surface plasmon polariton structures [73], [74]. Compared with this successful work, the laser-treated area shows much more hybrid micro-/nano-structures formation in a simple, high-speed, large-area way [75]–[80]. The porous PTFE structures enhance the interaction time between exciting light and matter, leading to the incident light reflection enhancement using the PTFE structure's porosity, which is significant for performance improvement. Besides, our methods' fabrication repeatability has been tested 100 times, which shows good repeatability.

COMSOL simulations of local electrical field distribution were employed to qualitatively analyze the enhancement effect by combining the LSPR and 3D micro-/nano-structures (Fig. 4(a)). Ag nanoislands on a structured substrate show two orders of magnitude electrical field distribution improvement. The electrical field distribution improvement between the adjacent silver nanoislands maybe because the structured substrate shortens the distance between two adjacent nanoislands. As shown in Fig. 4b, R6G probe molecule was chosen to evaluate SERS performances. The wavelength of the excitation source is 633 nm. The bands at 1360 and 1509 cm$^{-1}$ are observed. Therefore, $10^{-4}$ $\sim 10^{-7}$ M R6G were used to evaluate the L-PTFE-Ag SERS performance. The Raman signal intensity decreases with the concentration. Due to the advantages of the flexibility, flexible SERS substrates have been demonstrated for conformal rapid and non-invasive SERS detection [33], [34], [81]. Besides, we measured the sensing performance under repeated 100 cycling between different bending degrees, which shows stability without noticeable deterioration.

4. Conclusion
In conclusion, PTFE-based SERS substrates have been fabricated for developing a flexible SERS chip by FsLDW technology. After Fs laser treatment, the PTFE surface becomes rough with 3D hierarchical micro-/nano-structures. Then, The SERS chip was prepared by silver thermal evaporation
on the structured PTFE membrane. It worth mentioning that the structured PTFE-based SERS chip shows excellent performance enhancement by the LSPR effect and the increasing interaction time between exciting light and matter. The mentioned method shows excellent potentials in developing flexible SERS for wearable electronics.

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