An overview of surface-enhanced Raman scattering substrates by pulsed laser deposition technique: fundamentals and applications

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Abstract
Metallic nanoparticles (NPs), as an efficient substrate for surface-enhanced Raman scattering (SERS), attract much interests because of their various shapes and sizes. The appropriate size and morphology of metallic NPs are critical to serve as the substrate for achieving an efficient SERS. Pulsed laser deposition (PLD) is one of the feasible physical methods employed to synthesize metallic NPs with controllable sizes and surface characteristics. It has been recognized to be a successful tool for the deposition of SERS substrates due to its good controllability and high reproducibility in the manufacture of metallic NPs. This review provides an overview about the recent advances for the preparation of SERS substrates by PLD technique. The influences of parameters on the sizes and morphologies of metallic NPs during the deposition processes in PLD technique including laser output parameters, gas medium, liquid medium, substrate temperature, and properties of 3D substrate are presented. The applications of SERS substrates produced by PLD in the environmental monitoring and biomedical analysis are summarized. This knowledge could serve as a guideline for the researchers in exploring further applications of PLD technique in the production of SERS substrate.

Keywords Pulsed laser deposition · Surface-enhanced Raman scattering · Metallic nanoparticles · Substrate morphology

1 Introduction

Raman spectroscopy, based on the inelastic scattering that has been firstly discovered by Raman [1], plays a major role in providing structure information, surface, and interfacial process at the molecular level. However, the signals of Raman scattering are too weak to be detected since $10^7$ of incident phonons could excite only one Raman phonon. Later on, a phenomenon of an enhanced Raman signal in the pyridine adsorbed on the surface of a roughened silver electrode has been firstly observed by Fleischman et al. in 1974 [2], which was explained by the adsorbed more molecules on the surface of rough electrode arising from its larger specific surface area than that of smooth electrode. However, in 1977, Van Duyne [3] and Creighton [4] found that the increased number of adsorbed molecules caused by the high specific surface area of rough electrode was not consistent with the enhancement of Raman scattering intensity. Subsequently, this new phenomenon was received tremendous interests and named as surface-enhanced Raman scattering (SERS). After development over more than 40 years, SERS is widely used in the fields of physics, chemistry, biology, medicine, etc. [5–8] resulting from its extremely high sensitivity, good selectivity, and rapid response. It is possible to characterize molecules adsorbed on the surface of SERS substrate, even to realize the detection of a single molecule [9].

Normally, it is well accepted that the electromagnetic mechanism and chemical mechanism are two major enhancement mechanisms in the SERS theory. Electromagnetic mechanism is regarded as the dominant model in the understanding of SERS theory because the intensity of SERS is proportional to the fourth power of the incident electromagnetic field amplitude [3, 4]. The origin of electromagnetic enhancement on the nanoscale is from the collective oscillation of electrons excited by the incident light on the metal surface. This collective oscillation is called surface plasmon resonance (SPR) that is influenced

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by the size and morphology of SERS substrate [10, 11]. By controlling the shape, size, and interparticle spacing of SERS substrate, the SPR frequency could be altered, further attaining an optimized SERS enhancement [12].

It is evident that the selection of proper methods for preparing substrates is significant because the efficiency of SERS technique distinctly depends on the characteristics of SERS substrates. Metallic nanoparticles (NPs) are recognized as the most studied substrates after the discovery of SERS effect. In general, the synthesis methods of metallic NPs could be divided into chemical methods and physical methods [13–17]. The reduction of metal salts in aqueous or nonaqueous solution [18–21] is the main chemical method for the fabrication of metallic NPs. Both experimental and theoretical results have indicated that a junction or close interaction of two or more metallic NPs (that has been called hot spot) would produce strong SERS enhancement owing to the coupling of electromagnetic field [9, 22, 23]. However, the SERS substrate produced by chemical reduction method has a poor reproducibility because of the randomness of interparticle distance and arrangement. Therefore, the enhancement effect is not easy to be predicted. Self-assembly method is a commonly used chemical method to improve the reproducibility of SERS signal. This is the reason that in the self-assembly process [24–26], the metallic NPs could be fixed on the glass slide surface via the presence of functional groups like amine or thiol groups by immersing the glass slide surface into metallic NPs suspension, leading to a stable SERS effect. Then, the adjustable distance between two metallic NPs by changing the immersion time is contributed to the SERS enhancement [27]. Unfortunately, various reducing agents, surfactant, and adhesion agent have been added in the chemical process for fabricating the metallic NPs to prevent their agglomeration/oxidation and adjust their growth rate [28–30]. Consequently, a large number of reagents used in the chemical methods result in the contamination of fabricated metallic NPs.

The manufacture of metallic NPs is an easy task, but keeping the uniform dispersion of uncontaminated metallic NPs is a challenge. By contrast, the physical methods could be broadly exploited in the synthesis of metallic NPs thanks to the good controllability and high reproducibility. In addition, physical methods could also yield the uniformly monodisperse metallic NPs without solvent contamination. Thermal deposition [31, 32], sputtering deposition [33–36], electron beam evaporation [37–39], and pulsed laser deposition (PLD) [40–43] are some of the most commonly used physical methods to produce the metallic NPs. Mechanical pressure, high-energy radiation, thermal energy, and electrical energy have been applied to generate the metallic NPs by triggering the melting, evaporation, condensation, and ablation [28].

Among these physical methods, PLD is a relatively flexible and effective technique for the fabrication of metallic NPs with controllable sizes. Metallic NPs produced in ultrahigh vacuum feature are different from that in the conventional deposition techniques such as evaporation or sputtering [44]. In PLD technique, the pulsed laser beam like excimer laser or Nd:YAG laser is utilized for the ablation of any type of target material. It is easy to modify the morphology of fabricated metallic NPs by adjusting the laser wavelength, pulse duration, or laser fluence. Inert gas environment could also provide an additional condition to systematically vary the properties of metallic NPs [45]. For example, a good SERS performance of Ag NP film deposited by PLD was proved by Smyth et al. [46]. They investigated the SERS sensitivity of three different SERS substrates including pulsed laser–deposited Ag NP film (which was done in a high vacuum environment at room temperature), conventional silver colloids, and a commercial film (Klarite® substrate from Renishaw Diagnostics). The results showed that the SERS signals of both pulsed laser–deposited Ag NP film and conventional silver colloids were superior to that of commercial film. Moreover, pulsed laser–deposited Ag NP film offered much better reproducibility than conventional silver colloids.

Up to now, several review articles [23, 47–52] have displayed the various top-down, bottom-up, and combination technologies on the fabrication of SERS substrates. Nevertheless, PLD technique has been reported curtly in a few reviews. In this review, we have comprehensively summarized the detailed description of various SERS substrates fabricated by PLD technique. Firstly, we present the fundamental processes occurred in the ablation and deposition of metallic NPs in PLD technique. Secondly, many deposition process conditions of substrates using metallic NPs are considered. Lastly, the applications of SERS substrates fabricated by PLD in the environmental and biomedical analysis are described and the potential future trend in this area is outlined with the aim of stimulating the broader interest in developing PLD technique in the SERS substrates.

2 Fundamentals of pulsed laser deposition

Although the first experiment of PLD was carried out in the 1960s [53, 54], the extensive application in growing oxide film rapidly and efficiently was arisen in the late 1980s [55]. PLD technique has gradually developed over the past few decades. Today, it has been considered as one of the most popular techniques for depositing semiconductors [56, 57], metals [40–42], alloys [58, 59], and compounds [60, 61]. In addition, it could be applied to most of materials due to the high-power density near the focus of a pulsed laser (> 10⁶ W/cm²).
PLD technique presents many advantages in fabricating nanostructures. For example, benefiting from the intense energy of laser, almost all the materials could be deposited with the stoichiometry retained. The high instantaneous fluxes would lead to the high nucleation densities [62], which is beneficial for the formation of hot spots. Besides, since it could be operated at low temperature, the temperature sensitive materials could also be manufactured with this technique. Remarkably, the composite films with complex composition could be deposited by using several targets as well, and their thickness and morphology could be easily controlled.

PLD is a physical vapor deposition process, which requires a vacuum system and a pulsed laser. As shown in Fig. 1, the construction of PLD experiment is relatively simple. A pulsed laser with high laser energy density concentrates onto a rotating target in a vacuum. The extreme energy of the focused beam vaporizes or ablates a small surface area of target material to create a plasma plume for film growth. The ejected plasma plume travels at an initial velocity $\geq 10^6$ cm/s that is formed with ions and atoms [63] through the vacuum chamber, causing a highly directional expansion perpendicular to the target. The ejected flux impinges on the surface of substrate, and the film deposition is fulfilled.

In contrast to its simplicity in construction, the mechanisms of PLD are rather convoluted. As shown in Fig. 2a, the PLD process is normally classified into four substages: (I) laser ablation and target vaporization, (II) plasma formation and interaction with the incident laser, (III) plasma expansion in vacuum, (IV) deposition at the substrate [64]. When
the laser is concentrated on the metal surface, part of the laser penetrates the metal target and gets absorbed. During the process of laser ablation, metal target starts to melt and evaporate since the vaporization temperature is acquired. Next, the plasma plume formed at the metal target surface further interacts with the incident laser. The effect of plasma plume on absorbing the incident laser is closely related to the distance between plasma and target surface. Strong absorption happens when the distance is close enough to allow high density of plasma plume to be built on the target. With the expansion of plasma plume, it retards the absorption of incoming radiation and the deposition rate of substrate turns to rely on the rate of plasma plume expansion. Therefore, it is usually deposited in vacuum or inert gas atmosphere to avoid the interaction between plasma plume and ambient molecules. Finally, the plasma plume reaches the substrate followed by the nucleation and growth of island. In the most simplified model, an atom meets the nearest neighbor atom to nucleate an island. The number of islands grows rapidly within the deposition of a few percent of a monolayer [65]. Then, the probability of atoms encountering another diffusion atom is reduced, whereas the probability of atoms jumps into the island is raised, causing the enlarged size of islands [66]. This leads to descending the number density of atoms while increasing the number density of step edge and islands [65]. For PLD, the number of nucleation sites hinges on the first laser pulse and the additional atoms coalesce the existing islands. The steps involved in the formation and growth of islands are shown in Fig. 2c. The first laser pulse results in the nucleation of islands at a characteristic distance that is greatly affected by the diffusion rates. Ideally, the size of island remains lower than this distance until the coalescence begins. New islands nucleate on the top of existing islands as soon as its size is comparable to or larger than the nucleation site spacing. Therefore, the ideal case of layer-by-layer growth is not accomplished [67].

Previous works [9, 68–71] have showed the excellent SERS effect in the gap between neighboring Au or Ag NPs, that is, hot spot. However, the SERS effect is weakened when the islands are cross-linked with each other resulting from the decreased numbers of hot spots. As a result, it is necessary to control the parameters in PLD technique and prepare the suitable SERS substrate in order to obtain a better SERS effect. The quality of SERS substrate deposited by PLD generally depends on the experimental conditions, including laser output parameters, ambient medium, substrate temperature, properties of PLD substrate, etc. With the appropriate laser wavelength and fluence, high-energy densities are absorbed by a small volume of material, leading to the stoichiometric transfer of ablated material from the target. In PLD process, some introduced background gas (e.g., oxygen) interacts with the ejected plume, causing the formation of complex compounds. The background gas (e.g., N₂ and Ar) could also be used to slow the plume expansion. Moreover, the substrate temperature influences the kinetic energy of metallic NPs and results in the formation of a highly ordered substrate structure [72]. Additionally, the morphology of PLD substrate could strongly affect the surface morphology of metallic NPs. Summarily, the convenient manipulation of sizes and morphologies of metallic NPs allow PLD technique to be an effective method for preparing SERS substrates.

3 Parameters effect in PLD technique for preparation of SERS substrates

It is well known that the Raman intensity is proportional to the square of the induced dipole moment that is a product of the incident electromagnetic field magnitude and the molecular polarizability, so it is reasonable to deduce two major enhancement mechanisms (i.e., electromagnetic mechanism and chemical mechanism). The latter involves in the charge transfer process [73]. The former is due to the interaction between incident or scattered light and SERS substrate and served as the most important mechanism in the understanding of SERS. When SPR is excited on the surface of metallic NPs, the electromagnetic fields would be greatly enhanced at the specific position around the metallic NPs.

Like the harmonic oscillator model, which is a classical physical model, the electrons in the metal could be coherently excited to oscillate by incident laser. Under the irradiation of incident light, electrons move toward the surface of metallic NPs, resulting in the separation of charges and generation of electric dipoles (Fig. 3). The electric field opposite to that of the light induced by the dipole makes the charges return to the equilibrium position and causes the resonance collectively [74–76]. The type that electrons oscillate around the surface of metallic NPs or nanoscale crevices is termed as localized SPR [10]. The oscillating
amplitude could be determined by the extinction spectrum indirectly. According to Mie theory, the extinction spectrum, \( E(\lambda) \), is given by Eq. (1) [74]:

\[
E(\lambda) = \frac{24\pi^2 N a^3/2}{\lambda \ln(10)} \left[ \frac{\varepsilon_r(\lambda)}{(\varepsilon_r(\lambda) + 2\varepsilon_{\text{out}})^2 + \varepsilon_i(\lambda)^2} \right]
\]

where \( \lambda \) is the wavelength of incident light, \( N \) is the molecule surface density, \( a \) is the radius of a spherical nanoparticle, \( \varepsilon_{\text{out}} \) is the dielectric function of the surrounding medium, and \( \varepsilon_r \) and \( \varepsilon_i \) are the real and imaginary components of the metallic dielectric function, respectively.

Therefore, the wavelength and strength of the SPR are not only responsive to the dielectric function of both metallic NPs and surrounding medium, but also tunable by the surface morphology of substrate. Specific adjustment of the variables to change the surface morphology of metallic NPs assists SERS optimization because the strong amplification of local electromagnetic field is obtained by SPR, leading to the increase in SERS signal of analytes [77, 78]. PLD is a relatively simple and effective nano-fabrication technique to immobilize metallic NPs upon a given substrate. Structure and optical property of the grown metallic NP film are strictly dependent on the deposition conditions. In this process, laser output parameters, gas medium, liquid medium, substrate temperature, and 3D substrate morphology significantly affect the sizes and morphologies of metallic NPs.

### 3.1 Effect of laser output parameters

Several laser output parameters such as laser pulse number, pulse duration, and mass per pulse must be taken into consideration to control the size and distribution of metallic NPs by PLD. A series of studies have been done to understand the effect of laser pulse number on the morphology of SERS substrates. Alonso et al. [79] initially realized the formation of Ag NPs by PLD without any ambient gas. They found that the condensation in the gas phase during the plume expansion was unnecessary for the formation of Ag NPs, and the surface morphology of Ag NPs arrays could be optimized by tuning the laser pulse number. The TEM images (Fig. 4a-d) showed that the size of Ag NPs was positively correlated to the laser pulse numbers. Because of the laser pulse number deposited was from 500 to 10,000, the mean Feret’s diameters of isolated Ag NPs were in the range of 5 to 32 nm. Ag NPs turned to ellipsoidal and bean-like shapes. Then, the number of bean-like shapes was increased, and the islands started to be cross-linked with 15,000 laser shots. Additionally, AFM images suggested that the nanostructures had a columnar-like growth arising from the increasing height. The correlation SPR peak was red shifted from 425 to 737 nm, and the resonance band was widened as the pulse number was increased from 1000 to 15,000.

Furthermore, an important parameter of PLD process, namely the ablated mass per laser pulse (\( M_p \)), could obviously affect the structural properties of metallic NP films. Although the \( M_p \) depends on the fluence, the fluence is not
At a fixed pulse energy density, the $M_p$ could be affected by the irradiated area where the laser beam focuses on. Spadaro et al. [80] showed that the effect of $M_p$ was controlled by the laser spot area on the structure of Ag NP films when the laser fluence was fixed. The morphologies were apparently distinguishable between two samples with different $M_p$. Isolated, nearly spherical Ag NPs were observed at a lower $M_p$, but the coalescent Ag islands were displayed at the higher $M_p$, resulting from the clustering of spherical Ag NPs. The SPR position and width of samples deposited at the same experimental conditions were determined by $M_p$. When the $M_p$ was lower, the SPR peak showed an evident maximum at about 590 nm, while the broad maximum was detected around 820 nm at the higher $M_p$. The red shift of SPR and the increase of linewidths at the higher $M_p$ pointed out the detuned optical transitions due to the dipole–dipole interactions.

The previous studies [81] suggested that a longer pulse laser ablation, like ps or ns, would suppress the formation of metallic NPs due to the effects of laser absorption, heating, and evaporation of the metal target. Unlike the ps or ns PLD, an fs laser pulse duration is shorter than the full ablation process and the ablated species do not interact with the laser pulse itself, thereby avoiding the complicated secondary laser interactions. Bonis et al. [82] investigated the characteristics of Ag film obtained by ns and fs ablation in PLD process. The atomic velocity distribution, excitation temperature, and electron density were evaluated to show the effect of plasma dynamics on the Ag film properties. Surprisingly, the features of Ag films like morphology, peak position, and widening of the SPR deposited by fs pulsed laser were similar to those of the films deposited by ns pulsed laser, which was different from the previous reports [79, 83]. Generally, the gas phase condensation occurred in the ns ablation could result in the formation of droplets. It is clearly different from the situation in the fs ablation, in which a new plume with a lower velocity than the primary plume is expanded [82]. They supposed that this similarity between ns and fs laser-induced plasmas was related to the Ag bulk properties.

Because of easily controllable tuning of SPR band, Ag–Au alloy films were considered by Verma et al. [84]. Unlike chemical methods, PLD technique is able to synthesize the uniformly mixed Ag–Au alloy film with various sizes within a single step without stabilizing agents or other extraneous chemical species [85]. The relationship between SPR response and Ag–Au alloy film grown under different process parameters (including the Ag–Au pulse ratio and total number of pulses) was studied further. The result depicted that the red shift of SPR band was accompanied with the decreasing Ag–Au pulse ratio and the increasing mass thickness which was under the control of pulse number. Generally, the increase of mass thickness means the enlargement of metallic NP size [85]. In the present case, 20–50 nm size of Ag–Au alloy films boasted strong SERS response based on the size dependent near-fields [86, 87], in which the near-field enhancement was initially increased with increasing the size of metallic NPs and started to reduce after reaching a maximum, arising from the competition between nonradiative damping and radiative damping of the excited near-field. Moreover, SPR wavelength of the Ag rich alloy film and the pure Ag film closely corresponded to the laser excitation wavelength, resulting in the highest SERS response. Therefore, the fabrication of Ag–Au alloy film allowed the easy tuning of SPR band without changing the particles size, and the higher SERS intensity under different laser excitation wavelengths was obtained as a result of the alloy films with tunable SPR band.

### 3.2 Effect of gas medium

In the PLD process, a background gas is often introduced to provide a reactive species or reduce the kinetic energies of ablated species [44]. The expansion of laser ablation plume is strongly affected by the ambient gas pressure. To promote the formation of metallic NPs, an inert gas is adopted to confine the expansion of plume, which allows the sufficient time for nucleation.

For example, the performance of Ag NPs deposited in the Ar gas is not identical to that in the vacuum. Össi et al. [83, 88, 89] reported a series of pulsed laser–deposited Ag NPs in the Ar. As shown in Fig. 4e-h, the mean size of Ag NPs is approximately the same under the different laser pulse numbers, while the number density of Ag NPs increases as a function of the number of laser pulses at a fixed Ar pressure of 70 Pa. This is supposed to be a typical model for the growth of metallic NPs at the high laser shot number and low buffer gas pressure.

Moreover, they [83, 88, 89] not only changed the pulse laser numbers, but also optimized the Ar pressure to figure out the highest SERS activity. Qualitative differences in the surface morphologies of Ag NPs deposited at the different Ar pressures were clearly visual. SEM images proved that the Ar pressure made a notable contribution to the morphology of Ag NPs. The Ag NPs grown at the high Ar pressure showed the isolated nearly spherical with no evidence of a coalescence phenomenon. At the low Ar pressure, the presence of Ag islands with smooth rounded edges indicated that they were caused by the coalescence of small nearly spherical particles. The SERS substrate formation mechanism was summarized into two steps: (I) the aggregation of Ag atoms induced by the Ar atmosphere caused the formation of Ag NPs; (II) the coalescence of Ag NPs led to the appearance of interconnected network of Ag islands [90]. In this process, the dimensions of Ag NPs were determined by the Ar
pressure, that is, the higher Ar pressure, the larger Ag NPs were formed [91]. In addition, at a constant Ar pressure, the number density of Ag NPs was increased with increasing the laser pulse number, resulting in the beginning of aggregation process. It might be the result of high resistance from the Ar environment to the plume motion [92].

The different morphologies of Ag films lead to the different optical properties. Bulk Ag showed an absorption peak in near-UV [93], while the SPR peak of isolated Ag NPs shifted to the visible region at about 390 nm [94]. It was clear that the plasmon position of deposited Ag films was red-shifted from 440 to 632 nm with decreasing Ar pressure and the full width at half maximum (FWHM) was broadened from 150 to 630 nm, indicating the non-visual aggregation of isolated spherical Ag NPs. The absorption spectra of Ag films deposited in the Ar gas at different pulse numbers were investigated as well. The SPR peaks were red-shifted and broadened when the number of laser shots was increased. This implied an effective aggregation process among Ag NPs with the collective optical behavior [89]. In this research, the highest SERS activity was attained at 70 Pa with 30,000 laser shots. The corresponding SEM and TEM images illustrated that the Ag NPs with appropriate sizes were closely arranged to form a dense network, arousing the red shift of SPR peak position and the largest FWHM. The enormous enhancement of SERS signal was attributed to the red shift of the coupled plasmon resonance and the increase of electromagnetic field at the junction between the two closely adjacent Ag NPs [95, 96].

In order to reduce the effect of gas on the plume, PLD is usually carried out in the vacuum or low-pressure gas. The formation of metallic NPs by PLD in the air is a complex and low efficient process as a result of re-deposition of the ablated material [97, 98]. It is reported that the growth of metallic NPs in the air was realized in the ablation plume due to the intensive interparticle collisions, while the metallic NPs deposited in the vacuum were created on the surface of PLD substrate [99]. Nedyalkov et al. [100] deposited Au NP film in the atmospheric pressure and air condition to understand this process. The red shift of SPR wavelength revealed that no significant oxidation or other chemicals generated in the air. Moreover, SERS spectra demonstrated that this Au NP film could be a cheap alternative for other SERS substrate deposited in the high vacuum conditions. Khan et al. [101] were also interested in using PLD at atmospheric pressure (which was named as APLD), generating strong collisional coupling of the ablated material with the ambient gas. Highly different morphological characteristics of Ag NP films made by a high repetition rate (HRR) vacuum PLD and HRR supersonic APLD were obtained. A substantial fraction of elongated Ag NPs was found in the film that was produced by vacuum PLD. By contrast, the Ag NP films made by HRR supersonic APLD were more densely packed with more uniform size distribution. Then they found that the enhancement factor of HRR supersonic APLD film was about 30 times more sensitive than that of the PLD film prepared in the vacuum, which demonstrated the utility of APLD.

### 3.3 Influence of liquid medium

In principle, if the laser energy is not extremely attenuated and its density is still enough to perturb the material away from equilibrium state, the laser ablation could also occur in liquid [102]. The pulsed laser ablation in liquid (PLAL) attracts increasing attention since 1980s [103, 104]. Contemporarily, this technique is widely utilized for the nanostructure generation [105–108] because of its reliability and easy applicability without any contaminations. Desired metallic NPs could be obtained in one step due to the high energetic state of ablated species [109, 110]. Unlike conventional PLD technique, PLAL process does not need costly vacuum chambers and the products are easy to be collected from solutions.

Typical experimental setup for PLAL is laid out in Fig. 5a, which is composed of a pulsed laser, a set of focusing optics, and a vessel containing the metal target. The metal target might rotate to avoid a deep ablation trace, and the magnetic stirred liquid solution is adopted to achieve a better ablation and a higher reproducibility [111, 112].

Figure 5b–e schematically depict the evolution of the laser ablated plasma in liquid phase. Similar to the conventional PLD, the plasma plume is produced when the incident laser pulse irradiates the solid target through the liquid. Compared with ablation in vacuum or in gas, the expansion of plasma is strongly confined by liquid, and the thermodynamic and kinetic properties of plasma are able to be changed significantly. Different kinds of chemical reactions are yielded in the plasma and the interface between liquid and plasma provides the opportunities for the formation of new materials by the combination of target with liquid. Finally, the plasma cools down and condenses in the confining liquid and the metallic NPs start to nucleate, grow, and coalesce.

Up to now, a large number of literature [105–108, 113–115] about metallic NPs formation by PLAL and the effect of solvent types, solutes, stabilizers, and laser output parameters have been studied. Size and aggregation state of metallic NPs are controlled by the addition of charge-changing chemicals such as surfactants. Kadhim et al. [116] prepared Au NPs by PLAL in an aqueous solution of sodium dodecyl sulfate (SDS). They found that the size of Au NPs signified a negative correlation with the concentration of SDS. The concentration-dependent size relationship was explained by a dynamic formation mechanism [117, 118]. The rapid nucleation of Au NPs and further growth with supplying Au atoms were terminated when the surfaces were
covered with the surfactant molecules. Alluhyabi et al. [119] demonstrated that the size of Au NPs could be altered by changing the laser energy and laser pulse repetition rate. The mean size of Ag NPs was decreased by increasing laser energy originated from the disintegration of large Au NPs at high effective transient temperature. Similarly, high repetition rates of the laser pulses with high effective transient temperature could also fracture large Au NPs into small ones.

Giorgetti et al. [105] investigated the Ag NPs acquired with different laser pulse durations by PLAL. They found that the Ag NPs attained with ps pulses in the pure water were more dispersible and spherical with a relatively small size than that achieved by ns pulses. Compared with theoretical spectra calculated with the Mie theory, both the SPR spectra of Ag NPs with ps and ns pulses were red shifted and the SPR band of ps pulses was wider than that of ns pulses. The formation of Ag$_2$O layer surrounding was proved to make a great contribution to the red shift of SPR peak position for Ag NPs [120]. Owing to the presence of Ag$_2$O, the ablation-prepared Ag NPs could keep stable for months but displayed a weakened SERS activity. However, the addition of chloride ions into Ag NPs was able to promote an efficient SERS signal [121].

Vinod et al. [106] synthesized Ag@Au core–shell NPs by ablating Au target in the Ag colloidal suspension using low energy laser pulses. Au target absorbed the energy of pulse laser and Au atoms jetted on the spherical surface of the fragmented core Ag NPs [122]. The thickness of Au shell was controlled by changing the ablating duration of Au target. Two distinct SPR bands appeared when the Au shell was thin or no dense, while peculiar SPR band was observed due to the dense and thick Au shell. The interparticle distance between Ag@Au core–shell structures was decreased with increasing the number of Ag@Au core–shell NPs per unit volume, leading to the formation of “hot spots.” Therefore, SERS spectra strongly depended on the shape and interparticle distance of Ag@Au core-shells by controlling the ablation duration and colloid concentration. In addition, the pure metal colloidal solutions without stabilizer or polymer are preferred as an optimal SERS substrate for biological applications.

Besides, AgCl NPs were confirmed to be directly generated by pulsed laser ablation of Ag target in the sodium chloride (NaCl) solution [123]. The AgCl NPs were cubes with the sizes in several hundred nanometers. Cl$^-$ ions could not only promote the generation of cubic morphology [124], but also prevent the aggregation of Ag nanoclusters [121]. Furthermore, the AgCl NPs possessed a high decontamination capability of gram-negative bacteria and obvious SERS activity as well.

### 3.4 Effect of substrate temperature

The ambient medium can control the morphology of grown substrates without changing their stoichiometry of the target material. The change of substrate temperature also affects the properties of formed metallic NPs; particularly, the size and number density of metallic NPs can be adjusted. Budner et al. [72] reported the effect of parameters in the PLD process on the morphology and SERS enhancement property of Ag NP films. The effective SERS enhancement was reached by altering the laser fluence, number of laser pulses, and substrate temperature. Especially, the substrate temperature played a key role in the formation of Ag NP films. The thermal energy increased the kinetic energy of Ag atoms, causing a highly ordered structure. The best SERS performance was noted at the coalesced Ag NPs with decrease in the dimensions of gaps.

Kamakshi et al. [125] showed the effect of substrate temperature on the morphology of Ag NPs, which were grown by means of the Volmer-Weber growth mode [126] at the
substrate temperature ranging from 100 to 500 °C and the maximum size of Ag NPs appeared at substrate temperature of 300 °C. They noticed that the Ag NPs size and substrate temperature followed a non-linear relationship, meaning that there was more than one competing growth mechanism. On the one hand, the surface mobility of Ag NPs was raised with the increase of substrate temperature, resulting in the increase in the Ag NPs size. On the other hand, the increase of substrate temperature aroused the decrease in the density of ambient gas and collisional rate inside the plasma, declining the size of Ag NPs [127]. Besides, at high substrate temperature of 600 °C, the average size and interparticle distance of Ag NPs were larger than that deposited in the substrate temperature range of 100–500 °C, which suggested that the growth mechanism of Ag NPs was changed. It was reported that appropriate size and interparticle spacing of Ag NPs played key roles on the SPR and SERS effect [128]. As expected, the highest SERS activity was reached at the deposition temperature of 300 °C.

### 3.5 Effect of 3D substrates

Recently, three-dimensional (3D) nanostructures with high specific surface area are regarded as one of the solutions to promote giant enhancement of SERS [68, 69, 129, 130]. Furthermore, the local electromagnetic field could be facilitated by additional charge transfer between the metallic NPs and the adjacent 3D nanostructures [68, 69]. These evidences have prompted the study of composite substrates formed by 3D nanostructures and metallic NPs.

D’Andrea et al. [131] fabricated the 3D Si nanowires with Ag NPs coating and investigated the effect of size and density for Ag NPs and length of 3D Si nanowires on the SERS performance. Their experimental results are depicted in Fig. 6A. It is observed that the Si nanowire array was still intact after PLD deposition, illustrating that the deposition process had no effect on the 3D Si nanowires. It was seen that the Ag NPs size was increased from the bottom to the top section of the 3D Si nanowires, whereas the distance between Ag NPs was decreased. At the bottom and central sections of the 3D Si nanowires with 1.7 μm long, the mean radiuses of the separated Ag NPs were estimated to be 5.5 ± 1.1 and 7.5 ± 2.7 nm, respectively. On the top of 3D Si nanowires, the mean radius was 8.3 ± 2.7 nm and the Ag NPs started to agglomerate. The coalesced Ag NPs with larger size and smaller interparticle spacing were formed on the top section relative to the bottom and central section due to the greater possibility for the top of 3D Si nanowires to catch the incident Ag NPs. In addition, the mean radius was decreased to 7.3 ± 4.7 nm on the top of 3D Si nanowires with 3.4 μm long. Accordingly, it was easy to change the size distribution and the interparticle distance by changing the length of nanowires and the laser pulse number for increasing the SERS efficiency.

As the growing interest in the fabrication of flexible plasmonic substrates, Rebollar et al. [132] fabricated and characterized a range of Au-coated 3D nanostructured polymer films to explore the relationship between the Au-coated 3D nanostructured polymer films and the SERS signal (Fig. 6B). The polymer films were irradiated under different laser systems to generate the periodic surface of 3D structures with different periods, depths, and roughness. After coated with a thin layer of Au NPs by PLD, the 3D nanostructured polymer films exhibited no significant change in the period and height. Owing to the same morphology of Au layer on both flat and 3D nanostructured polymer films, they supposed that the SERS signal was more related to the period, depth, and roughness of polymer nanostructures. Particularly, the greater roughness, longer period, and deeper depth of 3D nanostructures, the higher SERS enhancement was obtained, attributing to the larger effective area of interaction between substrate and analyte.

Wang’s group originally fabricated Ag NPs/Ni(OH)2 nanosheet arrays to study the effect of 3D Ni(OH)2 nanosheet arrays on the SERS intensity (Fig. 6C) [68]. These 3D Ni(OH)2 nanosheets demonstrated a highly ordered and porous nanoarray structure, providing a large specific surface area for the deposition of Ag NPs. As the deposition time was varied from 10 to 60 min, the diameter of Ag NPs went up from 55 to 212 nm. When depositing for 50 min, SERS enhancement reached the maximum because of the optimal interparticle gap and particle size. Moreover, as shown in Fig. 6C, the red shift of SPR band for Ag NPs/Ni(OH)2 nanosheet arrays verified the decrease in the electron density of Ag NPs. The charge transfer from Ag NPs to 3D Ni(OH)2 was facilitated by the stacking of positively charged Ni(OH)2 layers [133]. Finally, the positive charges were left on the Ag NPs, while 3D Ni(OH)2 nanosheets were negatively charged and the charge density became higher at the adjacent surface of Ag NPs and Ni(OH)2 nanosheets. The surface plasmon between 3D Ni(OH)2 and Ag NPs would be strongly excited, devoting to the increase of SERS enhancement.

This group was further investigated the SERS effect of Ag–coated 3D nanostructure arrays (Fig. 6D) [69]. The naked 3D Ti(OH)4 and TiO2 nanobelt arrays with similar morphology feature were studied. The distribution of Ag NPs on the 3D Ti(OH)4 nanobelt arrays was more uniform than that of Ag–decorated 3D TiO2 nanobelt arrays. According to the XPS spectra, Ag2O might be formed arising from the reaction between Ti(OH)4 and Ag on the surface of 3D Ti(OH)4 nanobelt arrays with the high kinetic energy supplied by PLD [63]. The Ag2O could serve as a nucleation center to grow the ordered Ag NPs. The red shift of SPR band of both Ag decorated 3D Ti(OH)4 and TiO2...
nanobelt arrays in the UV–Vis spectra proved the charge transfer from Ag NPs to 3D nanobelt arrays. The more red shifts for Ag decorated 3D Ti(OH)$_4$ nanobelt arrays than that for Ag decorated 3D TiO$_2$ nanobelt arrays implied a higher charge density and a stronger local electromagnetic field, leading to an increased SERS effect. Additionally, the SPR of Ag decorated 3D Ti(OH)$_4$ nanobelt arrays was closer to the wavelength of the incident light than that of Ag-coated 3D TiO$_2$ nanobelt arrays, which also made contribution to the enhancement of SERS signals [12].

In summary, among these parameters, the laser output parameters are the simplest one to adjust the size and morphology of SERS substrates. Under the high-energy laser ablation, the metallic NPs jet with a very high initial velocity, which is easy to form the irregular nanoislands. The introduction of gas medium effectively alleviates the formation of nanoislands, especially in the deposition process of Au NPs. Moreover, the SERS substrate fabricated in the liquid medium exists in the form of metallic NP colloidal solution. Hence, its SERS sensitivity is high since the molecules to be detected could easily combine with metallic NPs in the colloidal solution. The substrate temperature has a certain effect on the morphology of metallic NPs. In addition, the morphology of 3D substrate is changeable and its large specific surface area could increase the density of hot spots. More and more research reports on the 3D substrate suggest a great potential of 3D substrate in the SERS applications.

4 Applications

SERS has the wide applications in the field of food safety [18, 134, 135], environmental analysis [136–138], drug and explosive monitoring [139–142], biomedical analysis [143–146], etc. However, the application of SERS substrates
prepared by PLD technique mostly focuses on the environmental and biomedical analysis fields.

4.1 Environmental analysis

As a result of irrational human activities, the potential hazards in environments have been the center of gravity in the environmental detection field and analytical chemistry. Thus, it is very important to develop the corresponding analytical techniques for monitoring the trace pollutants. A number of analytical techniques, including spectrophotometry [147–149], fluorescence [150–152], chromatography [153, 154], and electrochemical methods [155, 156] have been reported. However, the complicated instruments, complex sampling processes, and insufficient recognition capability restrict their applications. SERS technique has attracted wide attention for its simple, and fast process, as well as simultaneously efficient detection of multiple contaminant classes.

There are several review papers that have already mentioned the application of SERS technique in the environmental analysis [157–161]. The analysis target of SERS mostly included organic pollutant such as dyes [68, 69], pesticides [136–138], and polycyclic aromatic hydrocarbon (PAHs) [162–166]. Some of the analytes with strong affinity toward a substrate could easily realize the SERS detection. Dye molecules, like rhodamine 6G (R6G) and crystal violet (CV), are representative SERS analytes that could be efficiently adsorbed on the Au and Ag surfaces. Unsurprisingly, most of literature referring to Sect. 3 used R6G as a probe molecule to verify the SERS effect of metallic NP substrates. Moreover, fungicide Dithane DG [167], neonicotinoid insecticide thiacloprid [168], and benzenethiol [132] with special functional groups containing sulfur or nitrogen that usually applied in the agriculture or industry could be easily detected.

However, the analytes with low affinity to the substrate are still challenges to be directly detected by SERS. For instance, PAHs are highly carcinogenic and bioaccumulative [169, 170], and they could accumulate to the harmful concentrations from very low levels. Unfortunately, the apolar nature of aromatic rings causes a low affinity to the metal surface, which limits the SERS application in the PAH detection. To overcome this limitation, SERS substrates were often modified by the thiol [163, 171], humic acid [162], calixarene [172, 173], cyclodextrin [164], and other molecules like carbon nanotubes (CNTs) [174]. The basic principles of above modification methods are similar, that is, they employed the interaction between PAH molecules and functional groups/molecules to adsorb PAHs on the surface of SERS substrate for SERS detection. However, thiol, calixarenes, and cyclodextrin have strong absorption peaks in the range of characteristic peaks of PAHs, which is easy to interfere with the detection of PAHs.

Wang’s group used a simple and rapid method to prepare Al2O3 nanosheet array structure [175] and utilized PLD technique to composite Ag NPs with Al2O3, as shown in Fig. 7A. Figure 7B shows that the Ag NPs were uniformly distributed on the surface of Al2O3 nanosheet array with a mean diameter of 20 nm. Because of the porous structure of Al2O3 nanosheet array, the number density of hot spots was increased, which was beneficial for the enhancement of SERS effect. The SERS performance of Ag/Al2O3 nanosheet array and Ag/Al foil are investigated in Fig. 7C by using the Al foil without Ag NPs as a comparison. Raman spectrum of R6G affirmed the excellent SERS performance of Ag/Al2O3 nanosheet array. Pyrene was chosen as a molecular model because it was recognized as a key indicator of PAHs and occurred in all mixtures of PAHs. As shown in Fig. 7D, the limit for the detection concentration of pyrene whose absorption peaks were located at the blue region achieved based on the SERS results is evaluated to be ≤1 mM. Compared with other modification methods as mentioned above [164, 171–173], the Ag/Al2O3 nanosheet array substrate depicted no obvious characteristic peak with no interference of PAH detection. The promising experimental results were acquired and demonstrated a relatively cheap and easy fabrication strategy of SERS active substrates by PLD for trace analysis of harmful pollutants.

CNTs were utilized to enrich and extract the pollutants because of the high specific surface area and π-π stack of non-covalent bonds [176–178]. Wang’s group decorated CNTs that were prepared by the chemical vapor deposition (CVD) [179] with Ag NPs (Ag/CNTs) by the PLD technique. The fabrication process of Ag/CNTs is listed in Fig. 8A.

SEM images of CNTs and Ag/CNTs obtained from Ni-Ag catalyst are shown in Fig. 8B. The generated CNTs grew perpendicular to the substrate and formed an intersecting network structure, providing a larger specific surface area. Ag NPs with similar size were uniformly deposited on CNTs. All the characteristic peaks of pyrene were detected from the SERS spectrum (Fig. 8C). However, the differences of peak position between SERS spectra and Raman spectra for pyrene were speculated due to the different orientations of the PAH molecules relative to the substrate surface [163]. Although the SERS effect could be further optimized, the results suggested that the Ag/CNTs were a promising substrate in the optical sensor devices for detecting the PAHs.

4.2 Biomedical analysis

To comprehensively understand the biological process and early diagnose the complicated disease, the sensitive and specific methods are sought for the biomedical analysis.
The changes of biomolecules like proteins, nucleic acids, and metabolites suggest the presence and course of disease [180–183]. Therefore, to monitor these changes is conducive to further understand the life process and disease mechanisms. Various techniques, including nuclear magnetic resonance, mass spectroscopy, electrochemistry, fluorescence microscopy, electron microscope, etc., are exploited for biomedical research [184–188]. With the discovery of SERS phenomena, SERS-based investigation in biomedical application is fascinated originating from its excellent molecular selectivity, high sensitivity, intense signal, and great precision [189]. Considerable investigations have been taken to extend the multi-functional SERS technology to realize the detection of tissues, cells (apoptosis, secretion, cellular stress, cellular cycle) and biomolecules (proteins, enzymes, nucleic acid), tumor makers, and metabolic products in vitro and in vivo, especially for the detection of problems in the complex biological compositions [51, 190–192].

A feasible SERS application of in vivo analysis is the blood glucose quantification. The inconstant monitoring of blood glucose is potentially dangerous for patients. To overcome this obstacle, a direct sensing of glucose at physiologically relevant concentrations with SERS on the Au film-over-nanosphere (AuFON) substrates functionalized with bisboronic acid receptors was developed to resolve the glucose in the high background of fructose and accurately detect the glucose in the 1–10 mM range, which was promising in the SERS-based in vivo glucose monitoring sensors [193]. Recent progress by applying SERS substrate in lateral flow immunoassay (LFIA) strip instead of colloidal gold as signal reporters is valuable. SERS-based LFIA strips were introduced to detect trace amount of influenza H1N1 virus and human adenovirus (HAdV) with a value of 50 and 10

Fig. 7 A Fabrication processes of Ag/Al₂O₃ nanosheet arrays. B SEM image of Ag/Al₂O₃ nanosheet arrays. C SERS spectra of 10⁻⁶ M R6G adsorbed on (a) Ag/Al₂O₃ nanosheet arrays and (b) silver-decorated Al foil. Raman spectrum of 10⁻² M R6G adsorbed on (c) Al foil. D SERS spectra of pyrene molecules (a–d) in different concentrations on Ag/Al₂O₃ nanosheet arrays, (e) Raman spectra of Ag/Al₂O₃ nanosheet arrays, and (f) solid pyrene.
pfu mL$^{-1}$, respectively, which was 2000 times sensitive than the standard colloidal gold strip method [194]. Based on this, a pioneering study magnified that the trace spike protein (S protein) of SARS-CoV-2 in human saliva was detected by SERS biosensors with an extremely low concentrations of $10 \times 10^{-9}$ g mL$^{-1}$, exhibiting the significant differences from Raman spectra. These results represented a facile, rapid, and generalizable strategy for the future investigation of SARS-CoV-2 by SERS-based detection [195].

The detection of complex molecular systems such as proteins is usually time-consuming and expensive. Common detection of proteins in clinical applications is not practical enough in medical emergency. Agarwal et al. [196] showed the fast response and low-cost SERS method obtained with Au NP substrates deposited by PLD technique (Fig. 9A). Colloids systems were more popular for SERS detection, which was possible to probe the protein from all directions [197]. However, the interaction was easily uncontrollable between protein and metallic NPs, resulting in the poor molecular-structure sensitivity and scarcely reproducible. The SERS substrates obtained by PLD technique were more controllable, which made it possible to reveal the fingerprint of protein structure. The demonstration of binding for specific amino acid residues to the Au NPs indicated that the SERS enabled label-free detection of a protein.

Breast cancer has a high morbidity rates in female malignant tumor and is the major cause of cancer mortality in Chinese women. HER2, a kind of the human epidermal growth factor receptor, could be detected in the different biosensors for breast cancer diagnosis [199]. In order to simply
and directly detect the breast cancer, Téllez-Plancarte et al. [198] immobilized recombinant humanized anti-HER2 monoclonal antibody (trastuzumab) on an Ag nanoplate made by PLD technique and investigated its SERS activity (Fig. 9B).

The number of laser pulse was adjusted to find the best experimental conditions that provided the highest SERS effect. A clean Au (111) substrate and the specially prepared Ag nanoplates with high affinity for HER2 were analyzed. Compared with the Au nanoplates, the SERS spectra of Ag nanoplates provided higher peak intensity, better peak definition, and higher S/N ratio. The remarkable peaks at 1002 and 1030 cm⁻¹ belonged to the phenylalanine symmetric ring breathing mode and in-plane C–H bend mode, respectively [200]. They proposed that the phenylalanine could be used as a label to identify the breast cancer cells with HER2 expression and monitored by SERS.

SERS has received extensive attention in the environmental and biomedical analysis thanks to its capability of high sensitivity and rapidness during the detection process. However, many of the experimental results remain at the stage of laboratory research, and it still takes a long way to apply these results to the real life and production. For example, PLD technique is a small-scale production technique in the laboratory which may not fulfill the industrial demands for large amounts. In addition, even though it is possible to detect multiple pollutants simultaneously, the molecules with similar structure are not easy to be identified. Furthermore, the design of human-friendly SERS substrates is still the focus of future research to realize the in vivo detection.

5 Summary and outlook

The design and fabrication of SERS substrates with suitable morphology is the primary issue for SERS applications. In this review, we have outlined a variety of SERS-active substrates manufactured by PLD technique. Generally, PLD is utilized to synthesize conventional films, especially oxide-based compounds. However, recent
studies provide the strong support for broadening its application scope from traditional film to a new platform for SERS-active substrates. The PLD deposited SERS substrates exhibit the high SERS activity, high controllability, good adhesion, and great reproducibility resulting from the controllable growth conditions. However, there also exist some important challenges. For example, the morphology of SERS substrates prepared by PLD technique is relatively simple and limited to metallic NPs or films; the distribution of metallic NPs is random, which is easy to form the metallic islands, causing a weakened SERS effect. Thus, further research is necessary for solving or improving these problems. The manufacture of composites formed by 3D nanostructures and metallic NPs might be a feasible solution. In fact, metal oxides, carbon, and silicon materials with different morphologies are preliminarily used in the SERS substrates because of their intrinsic properties like magnetism, porosity, and photosensitivity and controllable preparation process. Furthermore, the electrochemical methods might be used to assist the growth of metallic NPs deposited by PLD. The growth process of NPs could be finely regulated to reduce the production of metallic islands. Moreover, the quantitative study on charge transfer between 3D nanostructures and noble NPs should be further carried out for the enhancement of SERS effect. In addition, it is still a challenge for PLD to satisfy the industrial demands with large amounts. The large-area PLD technique is a requisite prerequisite for SERS substrate to move from laboratorial fundamental research to practical application. However, conventional PLD grown sample with good uniformity is limited to a small area of 10 × 10 mm. Laser energy, repetition rate, deposition time, and gas pressure would be adjusted to realize the large-area deposition. High-temperature superconductor layer with length more than 600 m has been deposited by multi-beam pulsed laser deposition technique [201]. This method could be extended to the deposition of SERS substrates in the future. As a conclusion, this review paves the way to an extensive use of PLD technique as a reliable and efficient manufacture technology for SERS substrate. It is believed that the study and development of SERS substrates produced by PLD would make SERS technology a facile and indispensable tool in the future.

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Declarations

Conflict of interest  The authors declare no competing interests.

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