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Uniformity Control of Laser-Induced Periodic Surface Structures

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Laser-induced periodic surface structures (LIPSSs) are a universal phenomenon that can be observed on a variety of materials, including metals, semiconductors, and dielectrics, upon irradiation with ultrafast laser pulses. It has found various potential applications in the fields of optics, biologics, and mechatronics due to its efficient and flexible fabrication process and subwavelength quasi-periodic property. However, LIPSSs face the challenge of uniformity control because the formation of micro-/nanostructures induced by ultrafast laser is a complex process involving multiple interacting factors, including laser energy deposition, phase change, light scattering, and instantaneous local changes of material properties and their feedback mechanisms. Recently, there has been some significant progress regarding the control of LIPSS uniformity. In this work, we review recent experimental and methodological advances on this topic from three aspects: 1) laser-induced modified-LIPSS, 2) feedback mechanism of LIPSS formation, and 3) ultrafast laser pulse shaping. This review can stimulate further investigations into the uniformity control of LIPSSs to support and accelerate the industrial applications of uniform LIPSSs.

Keywords: laser-induced periodic surface structures, ultrafast laser, uniformity control, laser-induced modification, pulse shaping

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INTRODUCTION

Due to ultrashort irradiation periods and ultra-high intensities, ultrafast laser pulses have unique processing advantages such as high processing accuracy, high processing flexibility, and strong material adaptability compared with long laser pulses [1, 2]. Ultrafast laser pulse technology has become one of the promising methods for surface micro/nano structure fabrication [3]. Laser-induced periodic surface structures (LIPSSs) are a universal phenomenon upon irradiation with linearly polarized ultrafast laser pulses, which were first observed by Birnbaum in 1965 [4]. While LIPSS technology is an efficient and flexible method for the fabrication of subwavelength periodic surface structures, the formation of LIPSS is quite a complex process, and the mechanism has not yet been determined. Currently, the widely accepted definition arises from the interaction between an incident ultrafast laser beam and surface electromagnetic waves scattered by rough surfaces and may involve the excitation and propagation of surface plasmon polaritons (SPPs) [5–7]. Huang et al. believed that these periodic structures with periods smaller than the incident wavelength were formed by the interference of the initially excited surface plasmons (SPs) with the incident femtosecond laser and the subsequent grating-assisted SP/laser coupling [8].

After decades of research and discovery, LIPSS investigation has developed into a scientific evergreen [9]. LIPSS technology has been realized on various metals, semiconductors, dielectrics, and polymer films [10–13], and has found plenty of potential applications in the fields of optics, biologics, and mechatronics [9]. Yu et al. [14] proved the potential of LIPSS applied in the field of tribology, and Bonse et al. [15] demonstrated that the coefficient of friction of LIPSS-treated titanium surfaces was reduced by more than two times. Sugioka et al. proposed a novel fabrication of LIPSS inside 3D glass microfluidic channels and realized the application for real-time surface-enhanced Raman spectroscopy (SERS) [16]. Yin et al. utilized the ultrafast laser-induced formation of LIPSS on the surface of a stainless steel mesh to modulate the surface wettability and achieved the preparation of superhydrophilic and underwater superoleophobic structures, which have been applied to oil–water separation [17]. Jalil et al. reported the preparation of nanostructure-covered LIPSS on metal surfaces by ultrafast laser direct writing, which created broadband optical absorbers and selective solar absorbers [18].

Submicron gratings, as one of the most important diffractive optical elements, are favored by researchers because of their outstanding capability of precisely controlling the dispersion and steering characteristics of light [19–21]. LIPSS technology can flexibly fabricate periodically arranged structures with a submicron period, providing a powerful non-contact processing technology for the preparation of surface gratings on various materials [10, 22–25]. Vorobyev et al. reported the creation of various colors on a metal surface by LIPSS technology [10]. Dusser et al. systematically studied the LIPSS morphological changes and their corresponding color gamut properties induced by ultrafast laser pulses with different linear polarizations and realized complex coloring designs on stainless steel surfaces [22].

However, LIPSS technology still faces severe challenges in uniformity control [26, 27], limiting its wide application such as in the field of optics. Laser energy deposition is first absorbed by the electronic system of the irradiated materials, then the deposited energy is transferred to the lattice system, which subsequently induces various thermal effects, possibly hydrodynamic or chemical effects, etc. The effect of ultrafast pulses and the instantaneous local property changes of materials are dynamic processes of mutual feedback, which greatly increase the difficulty of controlling the formation of structures in a reliable way. In conventional LIPSS technologies, periodic structures always arise from the distributed light field caused by the large number of debris and surface defects during laser ablation, making it difficult to ensure long-range uniformity in larger-area preparation. Up to now, there have been a series of studies on the uniformity control of LIPSS. In this work, we review recent experimental advances on this issue from the perspectives of laser-induced modified-LIPSS, the feedback mechanism of LIPSS formation, and ultrafast laser pulse shaping.

LASER-INDUCED MODIFIED-LIPSS

In view of the issue about LIPSS uniformity control, a series of studies have been reported. For instance, Ardron et al. utilized linear pre-polished samples to regularize the morphology of LIPSS [28] and Harzic et al. reported an approach to improve the uniformity of LIPSS by using a nanojoule-femtosecond laser at a high repetition rate [29]. However, these methods were all performed under the conventional ablation regime, and the formation of LIPSS is always accompanied by the generation of a large number of debris and surface defects, which greatly affects the uniform excitation/propagation of subsequent SPPs, resulting in non-uniform periodic structures with numerous visible bifurcations [30]. Upon the irradiation of ultrafast laser pulses, the electron temperature and the lattice temperature of the material are in a strong non-equilibrium state, so that the kinetics of the phase transition may lead to a new phenomenon, that is, laser-induced modification. This phenomenon has been realized and applied in various scenarios, such as laser-induced single-crystal silicon [31, 32], $Ge_2Sb_2Te_5$ [33], MoS_2 [34] phase change, and laser-induced reduction of graphene oxide [35]. Based on the above revelations, Puerto et al. reported a regular grating structure preparation in silicon by taking advantage of laser-induced amorphization of crystalline silicon and LIPSS technology [36]. As demonstrated in **Figure 1A**, the fabricated structures consist of alternating amorphous-crystalline silicon stripes without any visible inhomogeneous cross-linked structure. When the incident laser fluence is higher than the melting threshold of crystalline silicon but lower than its ablation threshold, localized melting occurs under ultrafast laser pulse irradiation. Then liquid phase silicon overheats, and rapidly solidifies into an amorphous phase thin layer [37, 38]. Raman spectroscopic analysis confirmed the existence of laser-induced amorphous silicon, and the maximum thickness of the amorphous phase layer was around 60 nm [39]. Similarly, Zou et al. exploited ultrafast laser pulse for direct writing on graphene oxide (GO) films and successfully produced uniform subwavelength rGO-LIPSS (**Figure 1B**) [40]. The authors believe that the ultrafast laser-induced nonthermal or thermal photoreduction effects play a crucial role in the uniform formation of LIPSS [40, 41].

In addition, on the basis of modified-LIPSS technology, Huang et al. innovatively proposed a chemical etching–assisted ultrafast laser modification method to further promote the uniformity control of LIPSS [30]. As illustrated in **Figure 1C**, this method combines the advantages of modified-LIPSS with the specialty of great difference in chemical activity between amorphous and crystalline phase of silicon [42]. First, by precisely manipulating the laser-material interaction process, alternating amorphous-crystalline stripes are created by ultrafast laser scanning over silicon substrates; second, assisted by further chemical etching, the amorphous-stripes act as fine etch stops to prepare the uniform structures. The fabricated structures by this chemical etching–assisted ultrafast laser modification method are periodically and regularly distributed, and there is no visible crosslinking (**Figures 1Ciii,iv**). Compared with the conventional ablation LIPSS (**Figures 1Cv,vi**), the uniformity of the grating structures is significantly improved [30]. Meanwhile, researchers from the same group extended the laser focusing mode from point to line, further expanding the applicability of the chemical etching–assisted ultrafast laser modification method, and long-range uniformity in larger-area preparation of periodic surface structures is achieved (**Figures 1Di–iv**) [43]. The incomplete

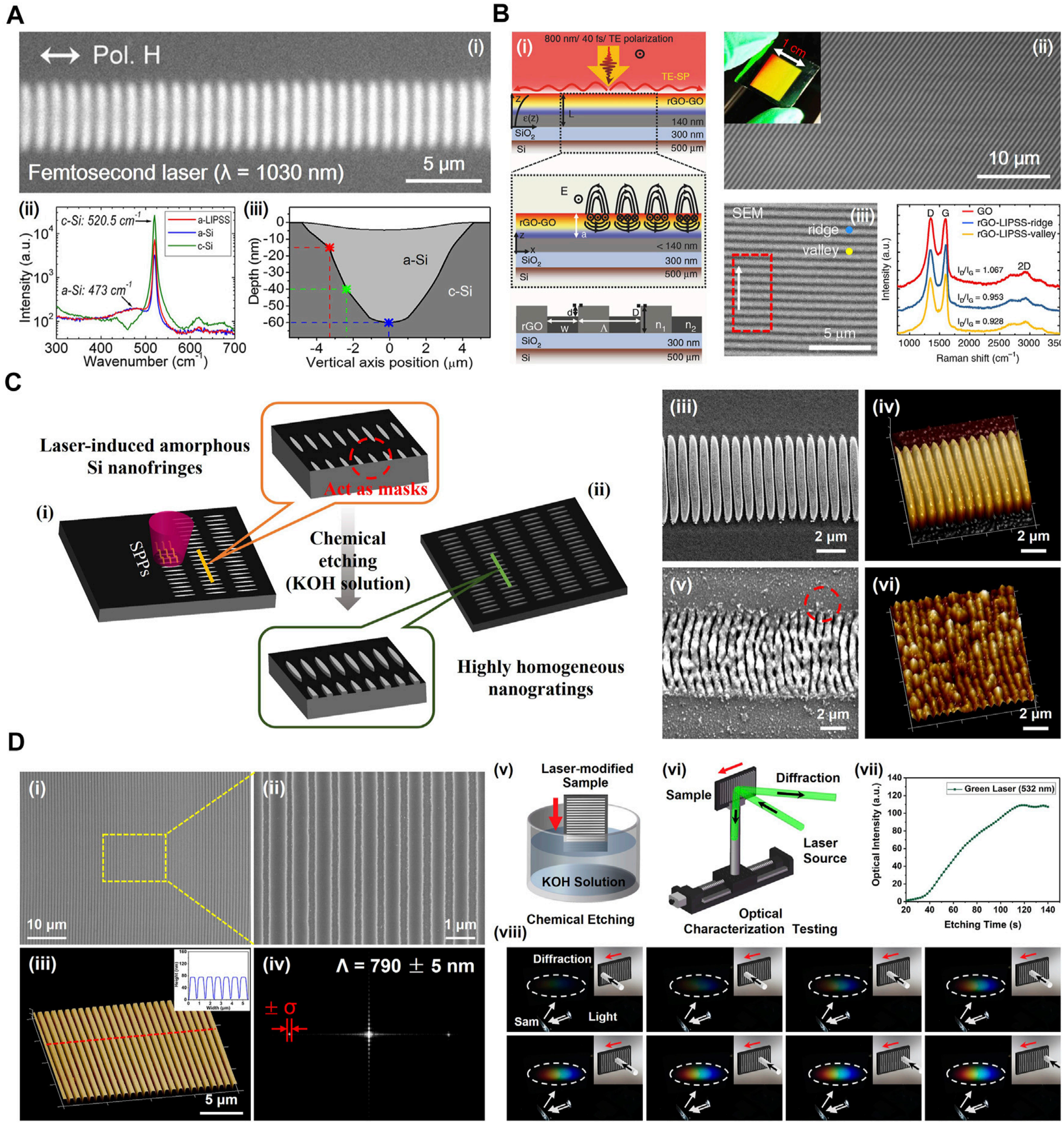

FIGURE 1 | Laser-induced modified-LIPSS. **(A)** Femtosecond laser-induced high-precision amorphous-crystalline nanogratings in silicon [36, 39]. **(B)** Uniform fabrication of subwavelength grating structures on GO thin films by femtosecond laser-induced reduction of GO [40]. **(C)** Highly homogeneous LIPSS on silicon *via* chemical etching–assisted femtosecond laser modification [30]. **(D)** Long-range uniform periodic surface structures realized by a modified regime cylindrically focused on femtosecond laser processing [43].

statistics show that the structures fabricated by this technology achieve optimal long-range uniformity compared to the reported LIPSS, which gains a minimum divergence of structure-orientation angles (<5°) [44]. Additionally, the etching time at different positions of the sample can be continuously and accurately controlled by a carefully designed etching process in which the laser-treated samples are gradually and uniformly immersed in the KOH etching solution. Then, periodic surface structures with continuous gradient changes in morphology are prepared in crystalline silicon. Benefiting from the uniformity and topographic features of the grating, the diffraction efficiency of the designed structure exhibits a continuous near-linear variation with the irradiation position, which will be widely used in optical sensing (**Figures 1Dv-viii**).

FEEDBACK MECHANISM OF LIPSS FORMATION

As mentioned above, ultrafast laser-induced LIPSS formation is a complex process involving mutual feedback mechanisms. Wang et al. have proved the relationship between the laser-

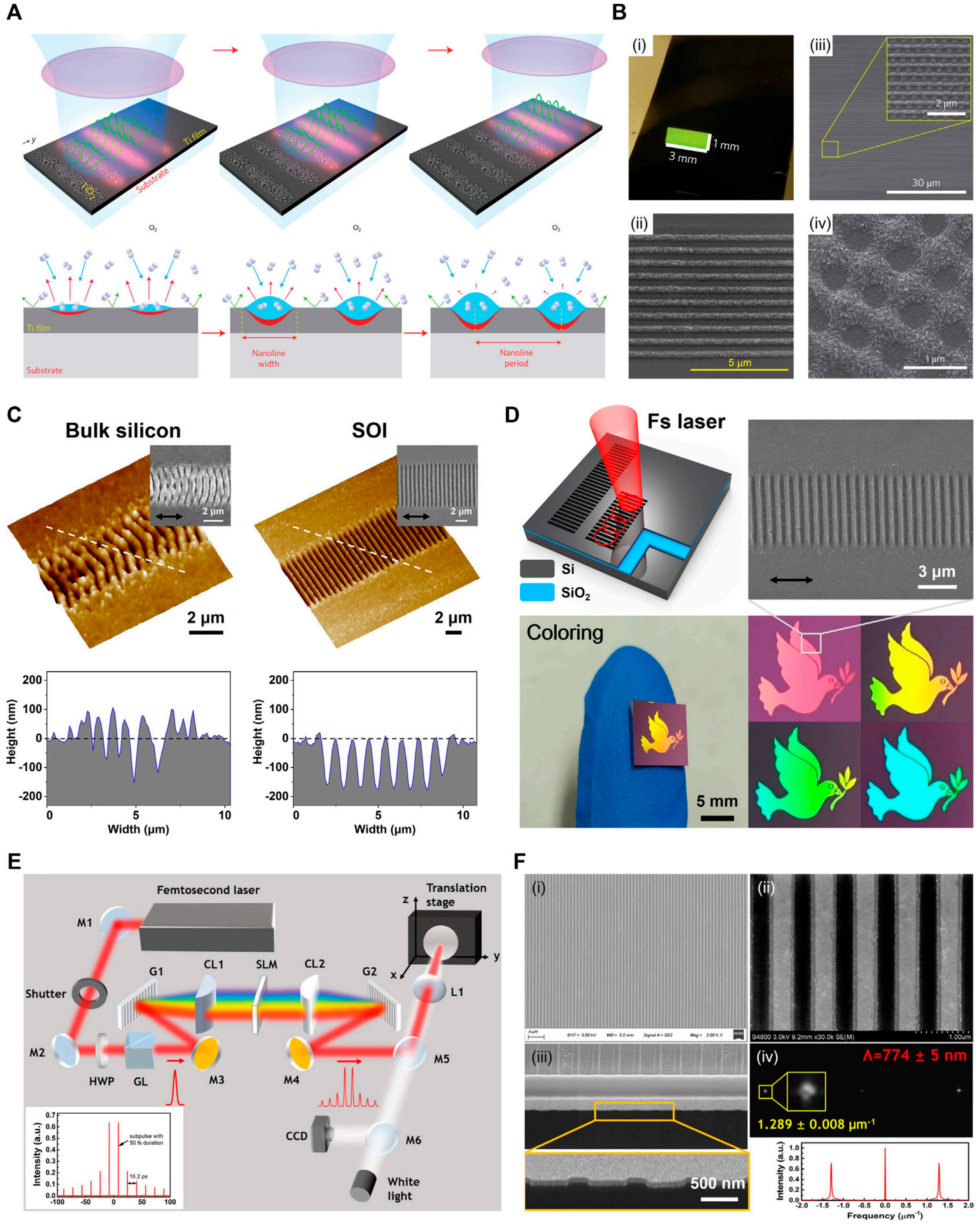

FIGURE 2 | The feedback mechanism of LIPSS formation and ultrafast laser pulse shaping. **(A,B)** Growth of metal-oxide periodic nanostructures with long-range uniformity by exploiting positive non-local feedback to initiate and negative local feedback to regulate [46]. **(C,D)** Regularizing the generation of subwavelength gratings on SOI by the feedback mechanisms of sample structural properties [50]. **(E,F)** A highly uniform LIPSS was fabricated on silicon based on temporally shaped femtosecond laser pulses [67].

induced plasma layer and the formation of ultrafast laser-induced subwavelength and deep-subwavelength structures on silicon [45]. The study by Han et al. found that the grating structure formed by the pre-pulse would facilitate the SPPs' coupling and directional scattering effects, thereby enhancing the anisotropic characteristics of the subsequent structure [11]. Recently, Öktem et al. proposed a new mentality based on nonlinear feedback mechanisms and realized the preparation of LIPSS with long-range uniformity on titanium surfaces [46]. The method tightly regulates the formation of nanostructures induced by ultrashort pulses by carefully exploiting feedback mechanisms, which can be summarized in the following steps: 1) An ultrafast laser beam with a peak intensity close to the ablation threshold of titanium is focused on the titanium surface, which is scattered by the existing nanostructures or any surface defects. The interference of the scattered field with the incident field results in the variation of the laser intensity near the scattered points. 2) At points where the laser energy exceeds the ablation threshold, titanium reacts rapidly with O_2 in the air and forms titanium dioxide (TiO_2). As shown in **Figure 2A**, the first two steps constitute a positive feedback loop, and as the nanostructure grows, so does its scattering power. 3) The growth mechanism of TiO_2 also possesses an imbedded negative feedback loop. As TiO_2 grows on top of the titanium, the penetration of O_2 through the oxide layer decreases exponentially. Based on this new mentality, various periodic nanostructures with long-range uniformity have been successfully fabricated on titanium. **Figure 2Bi** presents a photograph of nanostructures covering a 3-mm^2 area, consisting of the highly uniform TiO_2 nanogratings shown in **Figure 2Bii**. As demonstrated in **Figures 2Biii,iv**, a mesh structure is generated on titanium by two scans of orthogonally polarized laser beams, and a regular array of nanocircles is obtained using circularly polarized light. Moreover, Dostovalov et al. successfully fabricated regular periodic structures on metal thin films (such as titanium films and chromium films) by using a similar method [47, 48].

On the other hand, researchers regulated laser-processed structures by carefully exploiting feedback mechanisms of sample material/structural properties. Feng et al. utilized the feedback mechanism of the thin gold film coat on silicon surfaces for the laser processing, which enhances interfacial electron–phonon coupling to form a high and uniform electron density, and suppresses the impact of defects on the silicon, thereby achieving a uniform and stable LIPSS fabrication [49]. Recently, Huang et al. deeply analyzed the structural characteristics of silicon-on-insulator (SOI) and exploited the feedback mechanism of the intermediate buried oxide layer (SiO_2 layer) of the SOI on the laser-induced phase change process to regulate the generation of subwavelength gratings [50]. For a bulk Si wafer, the multi-pulse–induced incubation heat [51, 52] quickly transfers inside the substrate because of the high thermal conductivity of Si, balancing the local surface temperature of the initial structure between melting and vaporization. As demonstrated in **Figure 2C**, the generated LIPSS structures on the bulk Si were subject to the mechanism of melt flow, in accordance with the results from Tsibidis et al. [53], where the bendings and uneven cross-linkings obviously result from the long-term melt disturbance.

However, for an SOI wafer, the SiO_2 layer of SOI plays a critical role in the formation of surface structures. Since the intermediate SiO_2 layer possesses high thermal insulation, a feedback mechanism is constituted for the laser-induced incubation heat. The non-local nature of the feedback seriously prevents the incubation heat from spreading into the underneath silicon substrate, resulting in the accumulation of a large amount of heat in the top thin device layer of the SOI, reaching the vaporization temperature threshold. Then, uniform structures are generated on the top Si device layer of SOI as a result of direct local vaporization of the material instead of long-term melt flow. As shown in **Figure 2D**, highly uniform subwavelength gratings are flexibly prepared on SOI based on exploiting the feedback mechanisms to regulate the formation of structures. Furthermore, periodic surface structures with high uniformity achieve superior structure coloring, and a large-area cross-scale "peace dove" pattern was flexibly prepared on an SOI wafer, which exhibits a vivid and distinguishable structural color under indoor lighting [50].

ULTRAFAST LASER PULSE SHAPING

Under single femtosecond laser beam irradiation conditions, researchers have made efforts to manipulate the characteristics of LIPSS by varying the incident laser parameters (such as wavelength, energy fluence, pulse number, and polarization state) [54–57]. However, the adjusting ability appears to be limited because the formation of LIPSS is essentially determined by the transient properties of the irradiated material [58]. Temporal pulse shaping enables the generation of sub-pulses with ultrashort pulse delays (typically tens of femtoseconds to tens of picoseconds) so that the localized transient material properties can be under control. Ultrafast imaging results show that a transient metallic state can be generated on the material surface within a few tens of ps after femtosecond pulse irradiation and that a transient LIPSS structure has begun to form [59]. Therefore, the formation process of LIPSS can be further regulated by the temporal pulse-shaping method [8]. Several studies have investigated LIPSS formation by using delayed femtosecond double pulses with crossed or parallel polarizations and obtained some interesting results [60, 61]; for example, by controlling the pulse delay to tune the grating period [62], enabling the processing of ring-shaped LIPSS structures [63] and realizing the preparation of large-area 2D metal photonic crystal structures on tungsten surfaces [64].

Recently, ultrafast laser pulse shaping technologies have been used to control the uniformity of LIPSS. Jalil et al. employed a double temporally delayed femtosecond laser beam to adjust the propagation length of excited SPPs to improve LIPSS uniformity [65]. Lei et al. reported the fabrication of uniform subwavelength grating structures on metallic glass by a double-pulse femtosecond laser with nondegenerate directions of the linear polarizations [66]. Additionally, Zhang et al. fabricated a highly regular LIPSS on a silicon wafer by femtosecond laser pulse trains (**Figure 2E**) with a pulse delay of 16.2 ps and half in a symmetric energy distribution ratio of 0.09:0.13:0.21:0.69 [67]. **Figure 2F** shows the top-view SEM images (i–ii), cross-sectional SEM images (iii), and the corresponding 2D-FFT image (iv) of the fabricated LIPSS structure, clearly demonstrating that each ripple of the structure is completely straight and uniform, with a small fluctuation of period. The authors suggest that the underlying mechanism for the formation of highly uniform LIPSS is that, first, temporally shaped femtosecond laser pulse can enhance the excitation of the SPPs and periodic energy deposition [8, 59]. Second, the residual thermal effect on the ablation spot is greatly reduced due to the "ablation cooling" effect [68]. Besides, the ejected plume and debris from the previous sub-pulse are further excited by the subsequent sub-pulses, and the debris will be further ionized and vaporized into aerosol, avoiding the deposition of the ejected debris [69]. This ultrafast laser pulse–shaping technology will potentially be extended to other materials as a general process for the fabrication of large-area uniform LIPSS.

CONCLUSION

In summary, LIPSS shows immense potential for applications in various fields such as optics, biologics, and mechatronics by virtue of its efficient and flexible fabrication process and subwavelength periodic property. However, the formation of LIPSS is determined by the parameters of the incident femtosecond laser, the material properties, and the feedback mechanism of laser-material interaction, which makes the fabrication of LIPSS with high uniformity difficult and greatly limits its application. In this article, recent experimental and methodological advances in LIPSS uniformity control are reviewed and summarized into three aspects: laser-induced modified-LIPSS, feedback mechanism of LIPSS formation, and ultrafast laser pulse shaping, which are discussed separately. The review can provide a reference guide for the research on the preparation of highly uniform LIPSS and will promote the industrial application prospects of LIPSS. In future work, a general process for the preparation of LIPSS with high uniformity on various materials should be further developed.

AUTHOR CONTRIBUTIONS

JH and KW initiated the project. JH wrote the manuscript. YL, SJ, ZW, YQ, JZ, and RQ made intellectual contributions and edited the manuscript.

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