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Femtosecond laser-induced periodic structures: mechanisms, techniques, and applications

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Over the past two decades, femtosecond laser-induced periodic structures (femtosecond-LIPs) have become ubiquitous in a variety of materials, including metals, semiconductors, dielectrics, and polymers. Femtosecond-LIPs have become a useful laser processing method, with broad prospects in adjusting material properties such as structural color, data storage, light absorption, and luminescence. This review discusses the formation mechanism of LIPs, specifically the LIPs formation processes based on the pump-probe imaging method. The pulse shaping of a femtosecond laser in terms of the time/frequency, polarization, and spatial distribution is an efficient method for fabricating high-quality LIPs. Various LIPs applications are also briefly introduced. The last part of this paper discusses the LIPs formation mechanism, as well as the high-efficiency and high-quality processing of LIPs using shaped ultrafast lasers and their applications.

Keywords: laser-induced periodic structures (LIPs); formation mechanisms; femtosecond pulse shaping; pump-probe imaging; structural color; birefringent effects; optical absorption; photoluminescence

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Introduction

Femtosecond lasers have ultrashort pulse durations and high peak powers¹. The interaction between femtosecond laser and matter has the advantages of minimal thermal effect and no material selectivity^{2,3}. Compared with nanosecond and other long-pulse lasers, femtosecond lasers could induce more regular and deeper periodic structures on different types of materials, such as metals, semiconductors, dielectrics, and polymers, without being overwhelmed by huge thermal effects^{4–14}.

Laser-induced periodic structures (LIPs) have been intensively studied since 1965 when Birnbaum et al. found LIPs on the surface of germanium and gallium arsenide^{15,16}. Over the past two decades, ultrafast laser technology has developed rapidly. Femtosecond LIPs (femtosecond-LIPs) have become ubiquitous in a variety of materials, including metals, semiconductors, dielectrics, and polymers^{4–9,17–31}. The periods of LIPs change significantly with the laser fluence and the number of overlapping pulses. Based on the ratio of the period

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(Λ) to the laser wavelength (λ), LIPs are divided into low-spatial-frequency LIPs (LSFLs, $\Lambda > 0.5\lambda$) and high-spatial-frequency LIPs (HSFLs, $\Lambda < 0.5\lambda$)⁴, as shown in Fig. 1. However, the formation mechanism of femtosecond-LIPs does not yet have a clear explanation.

Femtosecond-LIPs have become a useful laser processing method, with broad prospects for adjusting material properties^{9,32–38}. HSFLs on the surfaces and interiors of dielectrics demonstrate birefringence effects, which have been used in data storage and waveplates^{34,36,38}. LIPs produce a significant dispersion of incident light and have been used to prepare pure and bright structural color surface^{32,35}. Composite structures with LIPs can enhance light absorption and luminescence and adjust the wetting properties of a material surface^{33,37,39}. The anisotropic electrical properties of LIPs are an emerging research topic^{40,41}.

The main contents of this review are as follows. Section *Several models of LIPS formation* discusses the formation mechanism of LIPs. Section *Ultrafast dynamics of LIPS formation based on pump-probe imaging* presents the ultrafast dynamics studied by the pump-probe imaging method. The pulse shaping of a femtosecond laser in the temporal–frequency domain, polarization, and spatial distribution is an efficient method for fabricating regular LIPs with high efficiency, as described in Section *Efficient fabrication of high-quality LIPs with shaped ultrafast laser pulses*. Section *Femtosecond laser direct writing of <100 nm nanostructures* introduces promising methods for laser processing nanostructures with feature sizes smaller than 100 nm. In the Section *Applications of LIPs*, we briefly introduce LIP applications in the fields of data storage, structural color surfaces, light absorption and luminescence enhancement,

and the adjustment of conductive properties. The last section presents the outlook on the LIPs. storage, structural color surfaces, light absorption and luminescence enhancement, and the adjustment of conductive properties. The last section presents the outlook on the LIPs.

Several models of LIPS formation

The formation of periodic nanostructures after irradiation with a single laser beam is an interesting phenomenon. In particular, the periods of femtosecond-LIPs change significantly with the laser fluence and the number of overlapping pulses, which renders the formation mechanism of LIPs more complicated to determine. Numerous experimental, theoretical, and numerical studies have been performed, and several models have been proposed to explain the formation of LIPs^{9,16–18,20,21,25,42}, which are described below.

Scattering light model

After irradiation with continuous and long-pulse lasers, LSFLs perpendicular or parallel to the polarization direction formed on the surfaces of semiconductor materials, where the periods were nearly equal to the laser wavelength, λ . It was proposed that these LSFLs were caused by the interference between the incident light and scattered light from the ablated surface^{16,18,43}, as shown in Fig. 2. When the laser was perpendicular to the sample, period Λ was equal to laser wavelength λ . When the laser was obliquely incident at an angle θ , period $\Lambda = \lambda / (1 \pm \sin \theta)$, where + and – represent the forward and backward scattered light, respectively⁴⁴. This model was refined and developed by Sipe et al. They performed detailed theoretical studies and found that the generation of periodic structures depended significantly on the

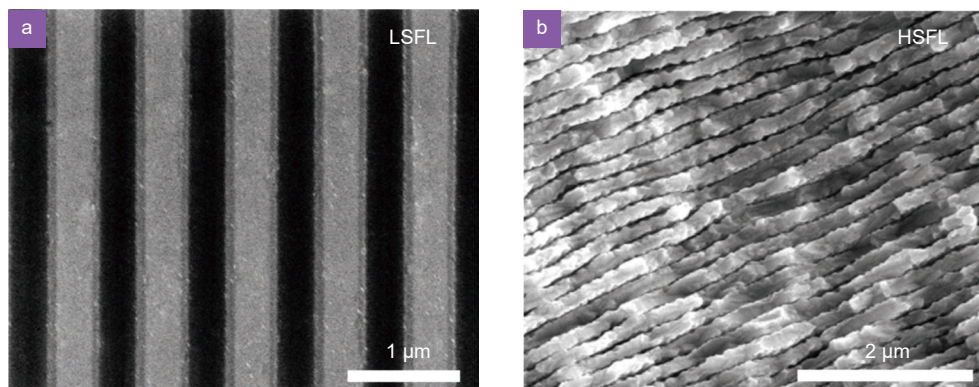


Fig. 1 | Scanning electron microscopy (SEM) images of (a) LSFLs on silicon⁸, and (b) HSFLs on ZnSe induced by 800 nm femtosecond laser. Figure reproduced with permission from: (a) ref.⁸, Optica Publishing Group, under the Optica Open Access Publishing Agreement; (b) ref.⁹, American Physical Society.

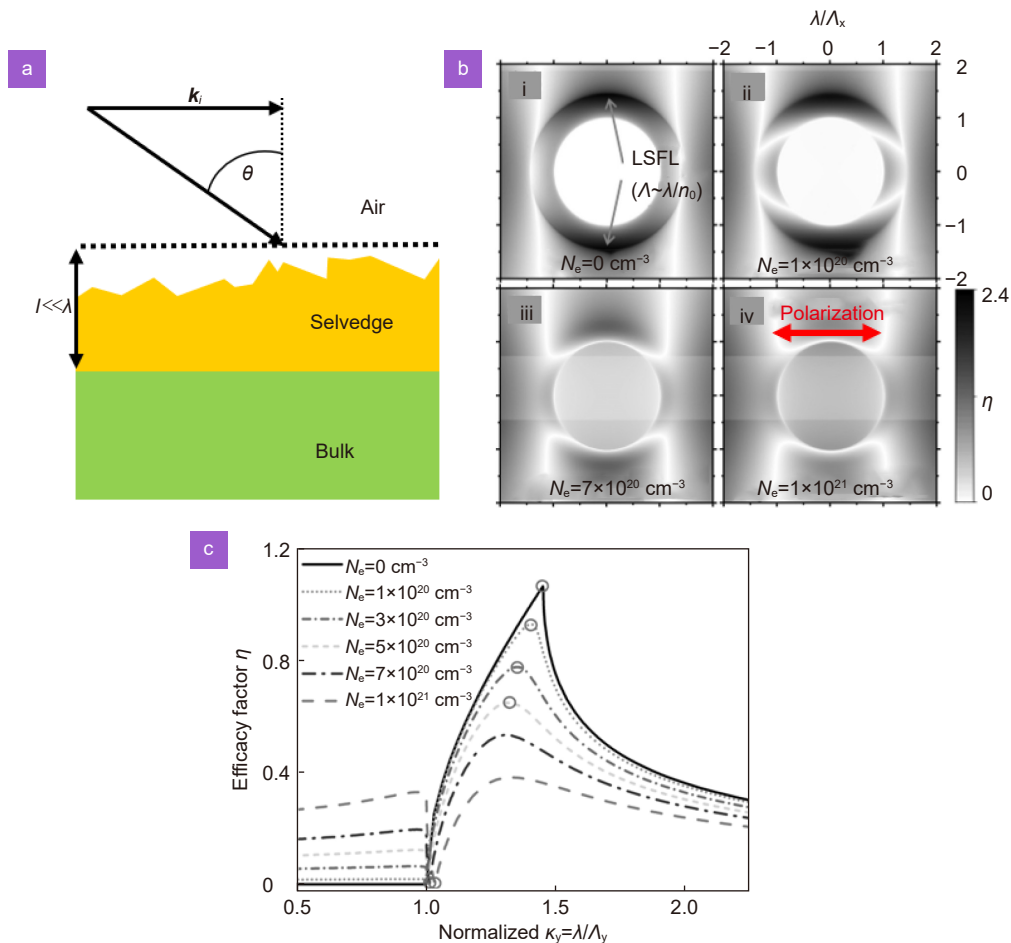


Fig. 2 | (a) The schematic of light irradiated on a rough surface at an incident angle θ , where k_i is the wave-vector component parallel to the surface, and l is the thickness of selvedge region. (b) The efficacy factor η as a function of the normalized LIPS wave vector for silica at different excited state. (c) The efficacy factor η along the positive k_y -axis for silica at different excitation levels, with circles marking k_y -positions where LIPS formation could be expected. Figure reproduced with permission from: (a) ref.¹⁶, American Physical Society; (b, c) ref.²², AIP Publishing.

roughness of the material surface¹⁶. Rough structures with a height significantly smaller than the laser wavelength resulted in a periodic distribution of the laser field itself. The incident angle and polarization direction highly influenced the period and orientation of the LSFLs. This model has often been used to explain classical LSFLs induced by nanosecond or continuous wave (CW) lasers.

Over the last two decades, scattering models have been developed and improved to explain the formation of femtosecond-LIPs^{45,46}. According to the Drude model, the dielectric constants of semiconductors and dielectrics depend on the numerical density of free electrons excited by femtosecond laser. According to the Sipe model, the periodic deposition of laser energy depends on the surface roughness and dielectric constants. The formation of femtosecond-LIPs on semiconductors and dielectrics was studied by comprehensively considering the Sipe and Drude models^{22,23,47,48}, and developed into

the Sipe-Drude model²². The numerical density of free electrons and dielectric constants varied with the laser fluence, wavelength, and pulse number, which further tuned the periods of LIPs, as shown in Fig. 2.

Surface plasmon polariton model

Under femtosecond laser irradiation, a large number of free electrons are excited, forming a plasma layer on the surface of a material. The femtosecond laser further causes the collective oscillation of the surface plasma and forms surface plasmon polaritons (SPPs), as shown in Fig. 3(a). The excitation of SPPs causes a periodic distribution of the laser field and energy deposition in the free electrons. The lattice is heated, melted, or even ablated via electron-phonon coupling, which further induces the formation of LIPs^{8,21,25,48,50-62}. However, in the case of vertical incidence (Fig. 3(b)), the wave vector of the SPPs is always larger than that of the incidence light. Therefore, grating structures⁴² or a single nanogroove^{25,52} are

fabricated on the sample surface in advance, and the scattered light can provide an additional wave vector, Δk , which satisfies the vector matching condition and induces SPP excitation, as shown in Fig. 3(c)²¹.

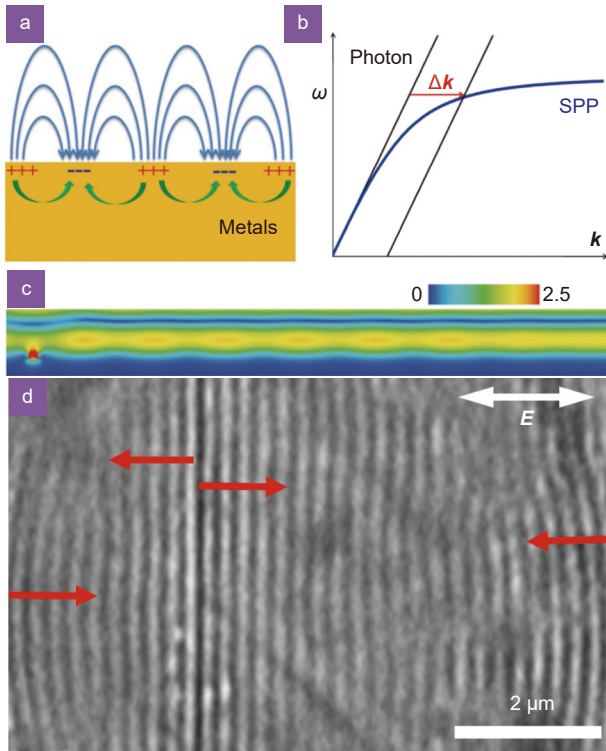


Fig. 3 | (a) Schematic illustration of SPPs. (b) Dispersion curves of SPPs and light in air. (c) SPP excitation on ZnO surface in the excited state launched by a groove. (d) The SEM image of the nanostructures on the ZnO surface irradiated by 800 nm femtosecond laser, where the LSFLs originated from the nanogroove or the ablation edge, as shown as the red arrows. Figure reproduced with permission from: (b) ref.⁴⁹, AIP Publishing; (c, d) ref.²¹, American Chemical Society.

The SPP model was used to explain the formation of LSFLs on semiconductors. The periods are expressed as follows²¹

$$\Lambda = \frac{\lambda}{\frac{\lambda}{\lambda_{\text{SPP}}} \pm \sin(\theta)}, \quad (1)$$

$$\lambda_{\text{SPP}} = \lambda \times \text{Re} \left(\sqrt{\frac{\varepsilon + \varepsilon_d}{\varepsilon \varepsilon_d}} \right),$$

where λ_{SPP} is the SPP wavelength, θ is the angle of incidence, ε is the dielectric constant of the metal, and ε_d is the dielectric constant of the dielectric. Under femtosecond laser irradiation, a large number of free electrons are excited, resulting in a significant change in the dielectric constant, according to the Drude model. The SPP wavelength and LSFL period can be calculated based on

the dielectric constant in the excited state, which agrees well with the experimental results for semiconductors^{42,51,52}. The LSFL periods induced by femtosecond laser pulses on semiconductors and metals are always smaller than the SPP wavelengths. With more laser pulse irradiation, the LSFL-assisted SPP is coupled with the laser field, forming a positive feedback effect, and leading to the formation of shorter-period LSFLs²¹.

Model of nanoplasmonic enhancement of laser field

The groove widths of HSFLs induced by femtosecond laser pulses are usually smaller than 100 nm, whereas the depth can reach hundreds of nanometers to micrometers. The widths of these periodically arranged nanogrooves are significantly smaller than the diffraction limit of the laser^{17,20,63–72}. Figure 4(a) shows 20 nm-thick nanosheet-like nanostructures with a period of $\sim \lambda/2n$ in fused silica (refractive index n) induced by linearly polarized 800 nm femtosecond laser pulses. It was proposed that these nanosheets were produced by the asymmetrically localized laser-field enhancement of the nanoplasma²⁰, as shown in Fig. 4(b).

The periodic distribution of the nanoplanes evolved from a random distribution of nanoplasma over many shots owing to the memory mechanism and mode selection. The period, $\lambda_0/2n$, is the minimum spacing required in planar metal waveguides to support this mode with the field maxima at the metal–dielectric interface²⁰.

Another mechanism that has been proposed for the formation of HSFLs in fused silica is photon–plasmon scattering¹⁷. By adjusting the pulse energy and cumulative pulse number of the 800 nm femtosecond laser, the HSFLs period varied from 140 to 320 nm. This phenomenon was interpreted in terms of the interference between the incident light field and the electric field of the bulk plasma wave, resulting in the periodic modulation of the electron concentration and structural changes in the fused silica¹⁷.

Other models

In recent years, several other models have been proposed to explain the formation of LIPs, such as self-organization^{24,26,73}, evanescent waves⁷⁴, and Coulomb explosions^{75,76}. During the femtosecond laser irradiation of SiC, ZnSe, and ZnO crystals, the generation of second harmonics induces the formation of HSFLs^{9,77,78}. The ripples of LSFLs on metal surfaces gradually split and evolve into HSFLs with an increase in femtosecond laser pulse irradiation^{79,80}.

During the femtosecond laser irradiation of semiconductor materials such as Si and ZnS, the surface plasmon layer supports two types of SPPs: those at the plasma–air interface and those at the plasma–substrate interface⁸¹. The local laser fluence and accumulated pulse number are key factors affecting the formation of LSFLs and HSFLs. Two SPP waves interfere with each other to form standing waves that induce the formation of HSFLs^{82,83}. A femtosecond laser was obliquely incident on a stainless steel surface and induced two periodic structures, LSFLs and HSFLs, which were caused by forward- and backward-propagating SPPs, respectively⁸⁴. Numerical simulation results using the finite-difference time-domain (FDTD) method indicated that the coherent superposition of the scattering far-field (propagation) of the microstructured surface and refracted fields induced LSFLs, while the scattering near-field (evanescent) and refracted fields induced HSFLs⁸⁵.

Ultrafast dynamics of LIPS formation based on pump-probe imaging

The phenomena of femtosecond-LIPSs are very rich, and the formation mechanisms are very complicated. Are LIPSs caused by the periodic deposition of laser energy or by self-organization during laser excitation, melting, and solidification? The propagating SPP model has been widely accepted to explain the formation of LSFLs on semiconductor and metal surfaces, however several key issues remain to be addressed. First, the SPPs on the surfaces of gold and silver are very strong⁸⁶, however it is difficult to form regular LSFLs on these noble metals¹⁰. Second, the periods of the LIPSs on metal surfaces are significantly smaller than the SPP wavelengths^{49,87}. The

further development of new methods is required to solve these problems.

Scanning electron microscopy (SEM) and atomic force microscopy (AFM) are typically used to observe LIPSs. The resolutions of these methods are very high, however they cannot be used to study the dynamics of LIPS formation. Pump-probe spectroscopy is an effective method to study the kinetics of femtosecond laser ablation⁸⁸. However, transient images of LIPSs cannot be observed directly^{19,79}. Therefore, we developed a collinear pump-probe imaging method to study the transient processes of LIPS formation^{25,49,52,55,56,89}. The spatial resolution is 300 nm and the temporal resolution is 0.6 ps. To show clearly the changes in the surface microstructure, the same spot was observed at three different times: before the arrival of the pump pulse, at the designed delay time, and after the ablation spot solidified.

Figure 5 shows the optical microscope (OM) images of the transient LSFLs on the Si surface at different delay times⁵². The LSFL period was 680 ± 15 nm, and the direction was parallel to the nanogroove. The annular structures were caused by the diffraction effect of the concave lens. Surprisingly, periodic ripples quickly emerged. The transient ripples were clear at a delay time of 4.0 ps, which indicated that these ripples were due to surface melting, excluding other thermodynamics such as ablation and hydrodynamics. The AFM measurements further showed that the height fluctuation in the LSFL region was less than 0.6 nm, and had no spatial periodicity. The LSFL period was predicted based on the SPP model, and it coincided with the experimental results. The formation of LSFLs on silicon was due to the periodic energy deposition caused by the SPP excitation, rather than

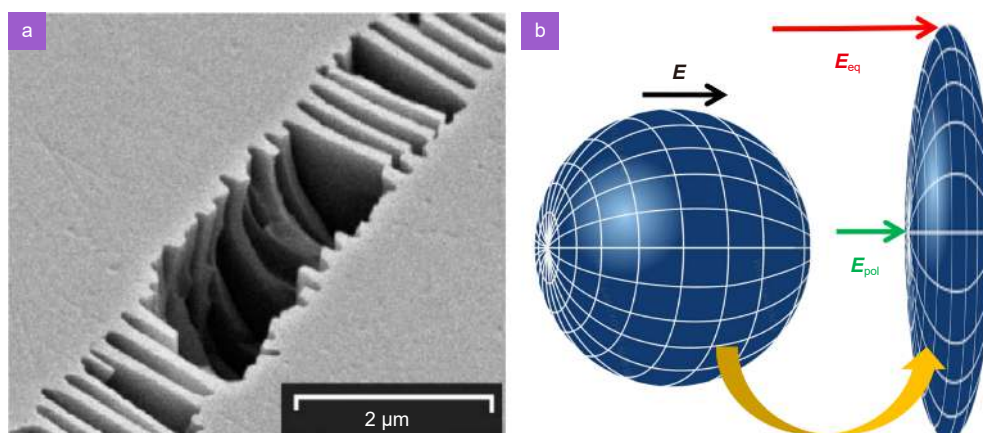


Fig. 4 | (a) SEM image of HSFLs inside fused silica induced by 800 nm femtosecond laser pulses. (b) Nanoplasma growth into plane governed by local field distribution, where E_{pol} and E_{eq} are the local fields on the polar axis and in the equatorial plane, respectively^{4,20}. Figure reproduced with permission from: (a) ref.²⁰, American Physical Society.

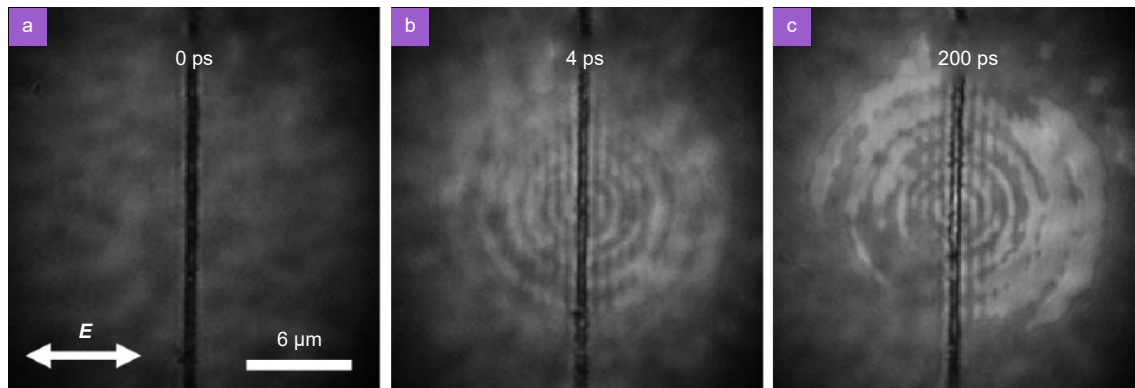


Fig. 5 | OM images of silicon surface irradiated by a single 800 nm femtosecond pulse of 0.18 J/cm². The double arrows in (a) show the laser polarization direction. Figure reproduced with permission from ref.⁵², Optica Publishing Group, under the Optica Open Access Publishing Agreement.

self-organization processes.

The pump-probe imaging experiments were further carried out to investigate the role of SPPs and thermal effects on the formation of LIPs on noble-metal surfaces⁴⁹. Under the irradiation of the second femtosecond laser pulse, transient periodic ripples were clearly observed on the gold film, but no obvious ripples remained after the surface solidified. The surface defects produced by the first laser pulse launched SPPs on the Au film, which caused further modulated energy deposition and induced transient LIPs. However, because of the small electron–phonon coupling coefficient of Au, the slow heat process and strong melting effect caused the ripples to disappear. When the Au film was immersed in water, the LIPs were retained because of the high cooling rate of the molten layers⁴⁹. These experimental results indicated that the SPPs played an important role in the formation of LIPs, while the thermal effect greatly influenced whether the transient ripples were retained after the molten surface solidified⁴⁹.

However, the period dependence of the LSFLs on metal surfaces was much different from the SPP model^{87,90,91}. The SPP wavelengths were calculated by using the dielectric constants of silver, gold, copper, and aluminum in the ground states, which were significantly larger than the periods of LSFL ripples^{10,49,92}. The d-band electrons in gold and silver could be excited to states above the Fermi surface^{93,94}. The free-electron density and plasma frequency both increased with laser fluence, resulting in longer SPP wavelengths. The LSFL periods were very different from those of the SPP model, which is a difficult problem to solve.

LSFLs are typically induced by multiple laser pulses. The material at the laser focus is partially ablated after

each laser pulse irradiation, which makes the formation mechanisms more difficult to understand because of the effects of the scattered light and grating coupling feedback. Therefore, theoretical and experimental studies on the LSFLs induced by a single femtosecond laser pulse provide fundamental information on the origin of LSFLs on metal surfaces^{52,95–97}.

Figure 6 shows the evolution of the LSFLs formed on an Au film. The transient LSFLs began to appear at 45 ± 10 ps, and become more numerous and clearer over time²⁵. Between 400 and 600 ps, the ripples appear to be the most distinct and regular. The ripples are perpendicular to the laser polarization with a period of 740 ± 10 nm.

Figure 7 shows that the LSFL period increased from 685 nm to 770 nm when the fluence increased from 0.73 to 3.42 J/cm²²⁵, which was similar to the results for Si, GaP, and ZnO crystals^{51,52,98,99}. The SPP wavelength of gold was 780 nm for 800 nm light, which was significantly larger than the experimental value. With increasing laser fluence, free-electron density became larger, which led to longer SPP wavelength and LSFL period. Obviously, there is a great deviation between the SPP theory and the experimental results.

The dielectric constant is a key factor affecting the SPP wavelength^{21,25}. The dielectric constant in the ground state was usually used to study SPP on metal surfaces, which was an important factor that caused the deviation between the SPP theory and the experimental results. Therefore, Cheng et al. proposed and studied in detail the effects of hot electron localization and d-band transitions on the dielectric constant and SPP wavelength for gold at the highly excited states during femtosecond laser irradiation²⁵. The LSFL period calculated by the developed SPP model decreased from 780 nm to 685 nm as the fluence increased to 0.75 J/cm². However, as F was

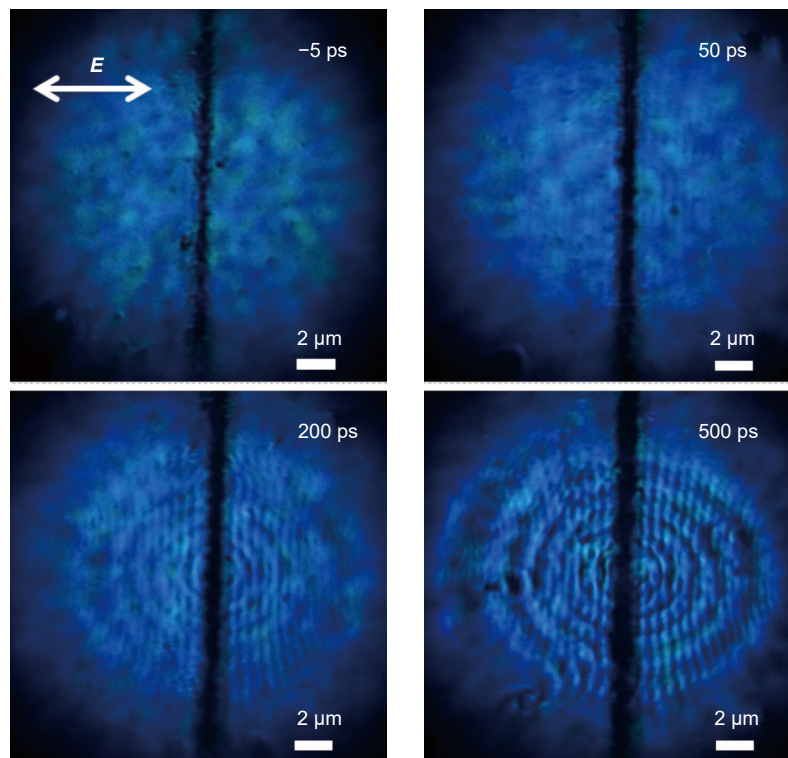


Fig. 6 | Evolution of LSFLs formed on Au film irradiated by a single 800 nm laser pulse with a fluence of 1.96 J/cm². The laser polarization, E , is perpendicular to the prefabricated nanogroove. Figure reproduced with permission from ref.²⁵, American Physical Society.

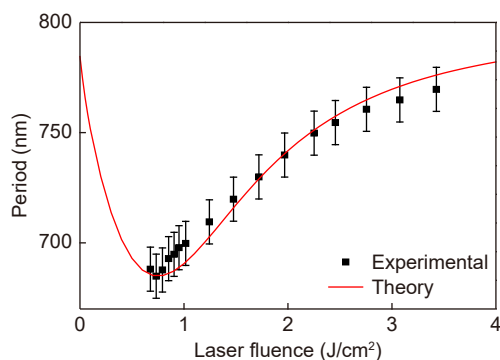


Fig. 7 | LSFL periods as a function of laser fluence F . The black squares and error bars show the experimental data, and the red curve shows the theoretical values. Figure reproduced with permission from ref.²⁵, American Physical Society.

further increased to 2.5 J/cm², the LSFL period increased to 756 nm. The developed SPP model accorded well with the experimental results, indicating that SPP excitation was the key during the formation of LSFLs on metal surfaces.

The electronic band structures and optical properties of Ag and Ni are significantly different, and can represent noble and common metals, respectively. The dependence of the transient LSFL periods of Ag and Ni films on the laser fluence was studied using the collinear pump-probe imaging method⁵⁶. The experimental results

agreed well with the theoretical values based on the developed SPP theory. Thus, the developed SPP model explains the formation mechanism of LSFLs on different types of metal surfaces.

Noncollinear pump-probe imaging techniques have also been applied to observe the formation of LSFLs. Murphy et al. reported the formation of LSFLs on silicon irradiated with multiple pulses¹⁰⁰. Using optical diffraction microscopy, Kafka et al. detected the formation of LSFLs on Cu films irradiated with a single femtosecond pulse⁵⁴. Garcia-Lechuga et al. used a moving-spot multiple-pulse irradiation approach, and observed the birth and growth of individual ripples on a silicon surface¹⁰¹. It was demonstrated that the formation of LIPs was initiated by free carrier generation, leading to non-thermal melting, liquid-phase overheating, and rapid solidification into the amorphous phase.

Efficient fabrication of high-quality LIPs with shaped ultrafast laser pulses

Temporally shaped ultrafast laser-induced high-quality LIPs

To efficiently fabricate uniform and regular LIPs, three main challenges should be addressed: enhancement of periodic deposition of laser energy, reduction of residual

heat, and avoidance of debris deposited on the ablation spots⁸. A temporally shaped ultrafast laser is an efficient tool for fabricating regular LIPs¹⁰². By changing the sub-pulse interval, pulse number, and sub-pulse energy distribution of the burst pulse train, the regularity and uniformity of LIPs were obviously improved, and the structural color was more vivid^{103,104}.

Double temporally delayed femtosecond laser beams can efficiently induce uniform LSFLs in semiconductors, metals and dielectrics^{105,106}. A femtosecond laser was further shaped into symmetrical three- and four-pulse sequences for the fabrication of double-grating structures and HSFLs on fused silica¹⁰⁷.

By controlling the voltage of each element of the spatial light modulator (SLM) via computer programming, the phase and/or amplitude of the laser spectrum can be modulated, and the 4f zero-dispersion pulse-shaping system can generate shaped laser pulses with arbitrary temporal distributions⁸. Specifically, the number of sub-pulses, interval between adjacent sub-pulses, and pulse energy of each subpulse can be flexibly adjusted.

Regular LSFLs on silicon were processed using a 4f configuration zero-dispersion pulse-shaping system⁸. A Gaussian pulse is shaped into pulse trains with an interval of 16.2 ps by using periodic π -phase step modulation. The transient LIPs started appearing on the Si surface at a delay time of 4 ps⁵², which was shorter than the interval between adjacent sub-pulses. Thus, the transient LIPs have started to appear under the illumination of the two main subpulses. When the subsequent subpulse reached the sample surface, the transient LIPs induced by the previous subpulses enhanced the excitation of the SPPs, as well as the periodic distribution of the laser field.

When the subsequent small sub-pulses reached, the surface layer remained at a very high temperature. It was further excited and partially ablated, taking away some of the remaining heat (ablative cooling effect). Moreover, the ablated plume was further excited by the subsequent subpulses, and the debris was further ionized and vaporized, resulting in fewer deposited particles. Therefore, regular and uniform LSFLs were induced on the Si surface using a shaped pulse of 16.2 ps⁸, as shown in Fig. 8.

The fabrication efficiency, depth, and regularity of the LSFLs when using shaped pulses of 16.2 ps were significantly better than those using Gaussian femtosecond laser pulses⁸. The scan velocity for fabricating regular LSFLs was 2.3 times faster, while the LSFLs depth was 2 times deeper, and the diffraction efficiency was 3 times higher

than that of LSFLs using Gaussian femtosecond laser pulses.

Spatially shaped femtosecond laser-induced LIPs with high efficiency

Arbitrary spatial intensity distribution of femtosecond laser pulses can be modulated by loading a computer-generated hologram (CGH) on a SLM, and LIPs can be processed in parallel with high efficiency¹⁰⁸. For example, Hayasaki et al. successfully realized the parallel processing of microstructures by loading a mixed-phase Fresnel lens on the SLM^{109,110}.

Maskless flexible spatial shaping based on spatiotemporal interference can modulate the interference intensity distribution into arbitrary patterns¹¹¹. It was verified using a Michelson interferometer, where the phase of one laser beam was controlled via SLM.

Lin et al. developed a correction method for the spatial distortion and light intensity, which could greatly improve the accuracy of the spatial pattern and uniformity of the light intensity simply by changing the phase map loaded on the SLM¹¹². This method was used to efficiently process colorful two-dimensional codes on silicon and shark-skin-like structures with superhydrophobic properties on stainless steel^{112,113}.

The laser focal point of a cylindrical lens is only a few to tens of micrometers wide, while its length is on the order of 1–10 mm^{114,115}. Compared with ordinary circular lenses, the efficiency of femtosecond-LIPs can be improved by a factor of 10^3 – 10^4 by using cylindrical lenses, which have been demonstrated in materials such as silicon and graphene oxide^{115,116–118}. This method is very simple, and has important implications for the industrial application of femtosecond-LIPs in the future.

Polarization shaped femtosecond-LIPs

By using a liquid crystal SLM and/or Q-plate, a femtosecond Gaussian laser beam could be modulated into vortex light, radially polarized or angularly polarized beam^{119–122}, and used to directly fabricate complex LIP patterns^{77,121–129}. Two laser beams with s and p polarizations were collinearly focused on the SiC surface, and nanostructures with different morphologies were fabricated by adjusting the energy ratio of these two laser beams⁷⁷. Radially and angularly polarized femtosecond lasers were focused on transparent materials and HSFL patterns consistent with the polarization distribution were fabricated¹²⁵.

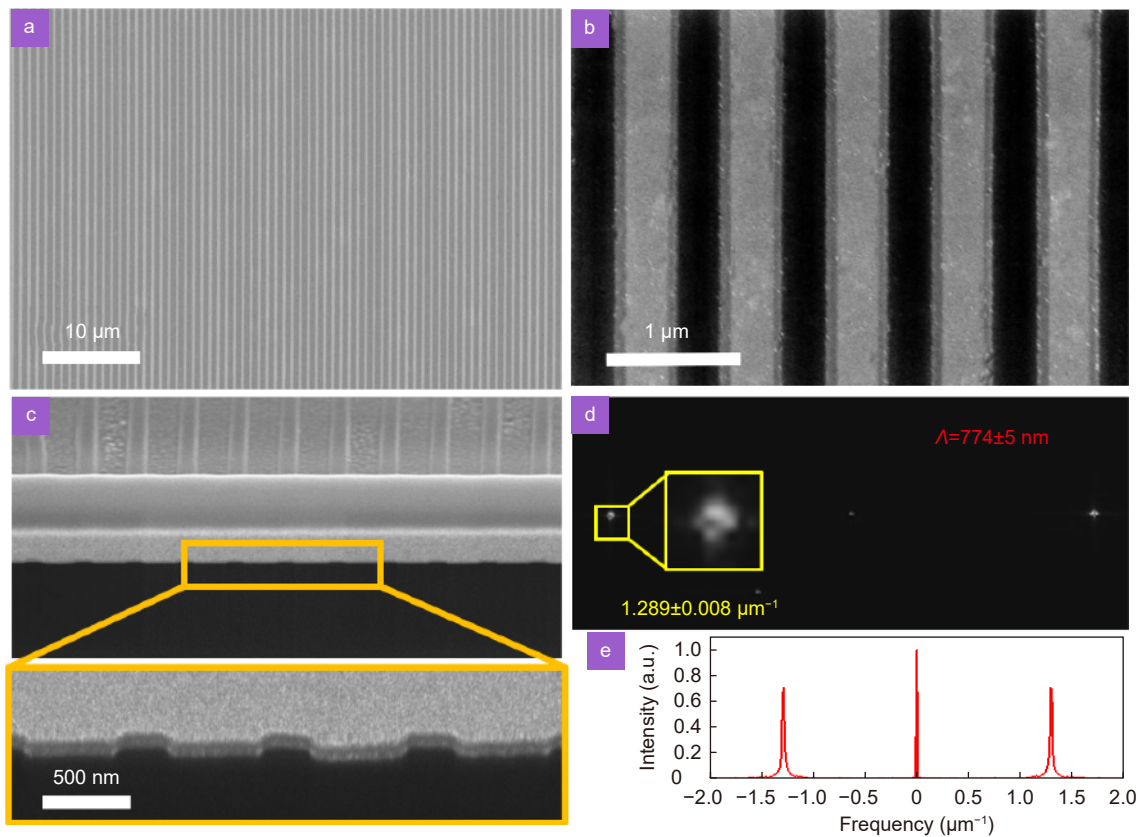


Fig. 8 | (a–b) SEM pictures of LSFLs induced by the shaped pulse trains with an interval of 16.2 ps. (c) The cross section of LSFLs. (d) The 2D fast Fourier transform of (b). (e) Spectra of the 2D Fourier transform along the x-axis. Figure reproduced with permission from ref.⁸, Optica Publishing Group, under the Optica Open Access Publishing Agreement.

Efficient fabrication of complex femtosecond-LIPS patterns by multi-beam interference

Multibeam interference is an efficient etching technology for processing regular periodic patterns, where the light intensity distribution in the interference field is usually imprinted on the materials. By combining laser interference technology with femtosecond-LIPSs, many complex LIPS patterns were fabricated with high efficiency and quality by simultaneously controlling the light intensity distribution and polarization distribution^{37,130}. Experimental and theoretical results showed that the longer-period structure was determined by the intensity distribution, whereas the LIPS pattern was determined by the polarization distribution¹³¹.

Two-beam femtosecond laser interference was used to fabricate grating structures. By changing the laser polarization direction, interesting complex periodic structures covered with LIPSs have been efficiently fabricated^{130,132}. Recently, large-area regular LSFLs were efficiently fabricated on a silicon wafer using double-beam femtosecond laser interference focused by cylindrical lens. The space of the double-beam interference

fringes was an integer multiple of the SPP wavelength, thereby effectively exciting the resonance-enhanced SPPs. The fabricated nanogratings were very regular, uniform, and smooth, and the resolution of the diffraction light was almost the same as that with commercial gold gratings⁵⁷. Using a similar method, a large area of regular and uniform LIPSs was processed on the surface of quartz glass¹¹⁸.

A wide variety of composite LIPSs such as hexagonally distributed nanogratings, hexagonal nanoflowers, and nanorings were efficiently fabricated on wide-bandgap SiC and ZnO semiconductors by changing the polarization combination of the three beams^{37,131}, as shown in Fig. 9. The four-beam interference method was further developed, and composite LIPSs such as square nanostructures, symmetric petal structures, and asymmetric helix-like structures were prepared on the surface of a ZnO crystal¹³³.

Femtosecond laser direct writing of <100 nm nanostructures

Because of the diffraction limit, the size of the laser focus is typically a few hundred nanometers. The resolution of

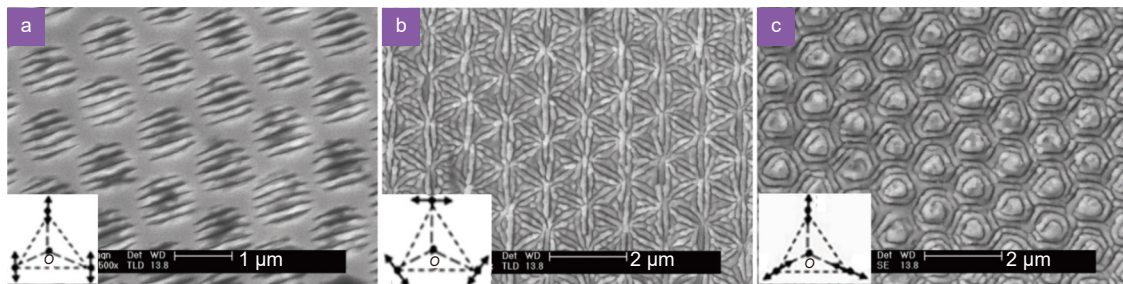


Fig. 9 | SEM images of composite LIPs prepared by three-beam interference. The insets show the polarization combinations of the three laser beams. Figure reproduced with permission from ref.³⁷, under a Creative Commons Attribution 4.0 International License.

laser processing with high numerical aperture objectives is also on the order of hundreds of nanometers. The groove widths of HSFLs on semiconductor, dielectric, and metal surfaces induced by a femtosecond laser are typically less than 50 nm and less than 1/10 of the laser wavelength, opening up new avenues for laser processing with feature sizes smaller than 100 nm^{134–138}. Liao et al. reported a single nanohollow channel with a width of 40 nm in porous glass using femtosecond laser-induced HSFL technology. It was used in nanofluidics in the field of single-molecule detection and biochip preparation. By precisely controlling the laser focus, fluences, and scanning speed, straight nanogrooves with widths smaller than 100 nm were prepared on the surfaces of fused silica and sapphire crystals¹³⁵.

A single nanogroove with a width of 30 nm and a double-groove structure with an interval of 150 nm were fabricated on a ZnO crystal surface immersed in water by precisely controlling the 800-nm femtosecond laser fluence and scanning speed¹³⁶, as shown in Fig. 10. Double-groove grating structures and single-groove grating structures with different spacings were fabricated, with groove depths ranging from 200 to 300 nm and groove widths smaller than 40 nm. Nanosquare structures with dimensions of 150 nm × 150 nm and 150 nm × 250 nm were fabricated by direct writing twice along the vertical direction.

By controlling the laser polarization always perpendicular to the scanning direction, curved nanogrooves with widths smaller than 20 nm and curvature radii smaller than 100 nm were fabricated on a silicon surface¹³⁹. Lin et al. processed curved nanogrooves with a feature size of 12 nm on silicon surfaces using 800 nm femtosecond laser direct writing with orthogonally polarized dual beams²⁷.

Applications of LIPs

Femtosecond-LIPs provide a mask-free, high-efficiency processing method for tuning material properties. This

method has been used in many fields, with applications that include structural colors^{8,32,35}, birefringent optical components^{34,36,38}, optical absorption and luminescence^{33,37,39}, and electrical properties⁴⁰.

Structural colors

Structural colors originate from the interference, diffraction, or scattering produced by micro/nanostructures on the surface of a material³². LIPs have been widely used to produce structural colors on metals, semiconductors and transparent materials^{8,35,118,140–151}.

The properties of such structural colors are determined by the period, depth, and orientation of the LIPs. LIPs with periods in the range of 400–1500 nm were prepared on the surface of stainless steel using femtosecond lasers with different wavelengths¹⁴⁰. These LIPs demonstrated completely different colors under each diffraction order, such that the structural color of the stainless steel surface could cover most of the color gamut. The same LIPs displayed different colors at different angles^{118,142,148}. Therefore, surfaces with different colors were prepared by controlling the orientation of the LIPs. Liu et al. proposed to control the orientation of LIPs in real time using a femtosecond laser double-pulse sequence with orthogonal polarization and equal pulse energy and fabricated LIPs with arbitrary orientations on silicon surfaces that were not affected by the scanning path¹⁴⁷. This is of great significance for multi-layer encryption, anti-counterfeiting, and so on.

The brightness and purity of the structural colors were affected by the regularity and depth of the LIPs. The high-efficiency preparation of high-quality LIPs has attracted increasing attention. Zhang et al.⁸ fabricated very regular and deep LSFLs on a silicon surface using temporally shaped pulses via a 4f zero-dispersion system and demonstrated very bright and pure structural colors, as shown in Fig. 11. Vivid structural colors on fused silica surfaces were prepared with high efficiency using the double-beam interference of femtosecond laser focused

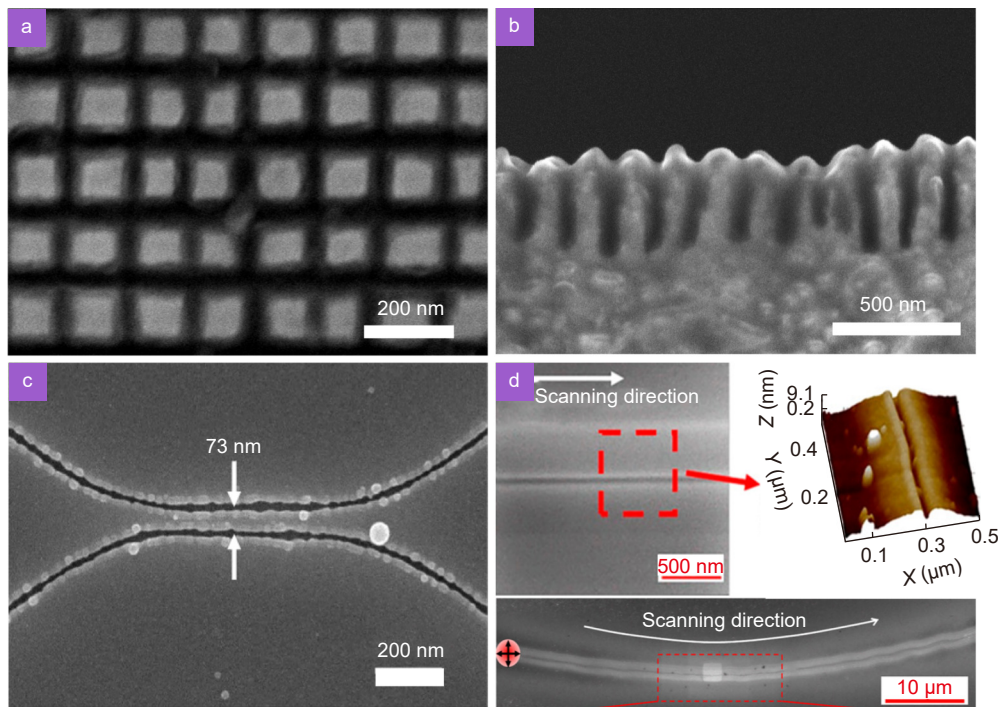


Fig. 10 | (a) SEM images of nanosquares, and (b) the cross section of nanogratings. (c) Curvature and separation control of nanogrooves fabricated by 800 nm femtosecond laser with polarization control. (d) SEM and AFM images of curved single nanogroove on silicon surface induced by 800 nm femtosecond laser with orthogonally polarized dual beams. Figure reproduced with permission from: (a, b) ref.¹³⁶, under Optica Open Access Publishing Agreement; (c) ref.¹³⁹, under a Creative Commons Attribution 4.0 International License; (d) ref.²⁷, American Chemical Society.



Fig. 11 | Structural colors of “Chinese knot” pattern made up of LSFLs fabricated by a shaped pulse of 16.2 ps on Si surface under different diffraction angles. The inset shows an SEM image of regular LSFLs. Figure reproduced with permission from ref.⁸, Optica Publishing Group, under the Optica Open Access Publishing Agreement.

by cylindrical lens¹¹⁸. Chemical etching-assisted LIPs are also an effective method for producing bright structural colors^{148,150}.

Birefringent optical components and information storage

HSFL nanostructures inside fused silica exhibit birefringence effects comparable to those of quartz crystals^{34,36,152,153} and have been used in the storage of multi-dimensional data, as shown in Fig. 12. Zhang et al.¹⁵⁴ demonstrated that HSFLs could encode the intens-

ity and polarization state of light at multiple levels and successfully retrieved digital files that were optically encrypted into five dimensions. Experiments indicated that HSFLs in silica glass have high stability and rewritability, meeting some requirements for developing high-density data storage³⁸. Recently, Wang et al. demonstrated 100-layer 5D optical data storage based on femtosecond laser-induced birefringence with a high readout accuracy and no bit error rates¹⁵⁵, as shown in Fig. 12. At present, the limitations of optical storage technology lie mainly in efficient writing and fast and accurate reading.

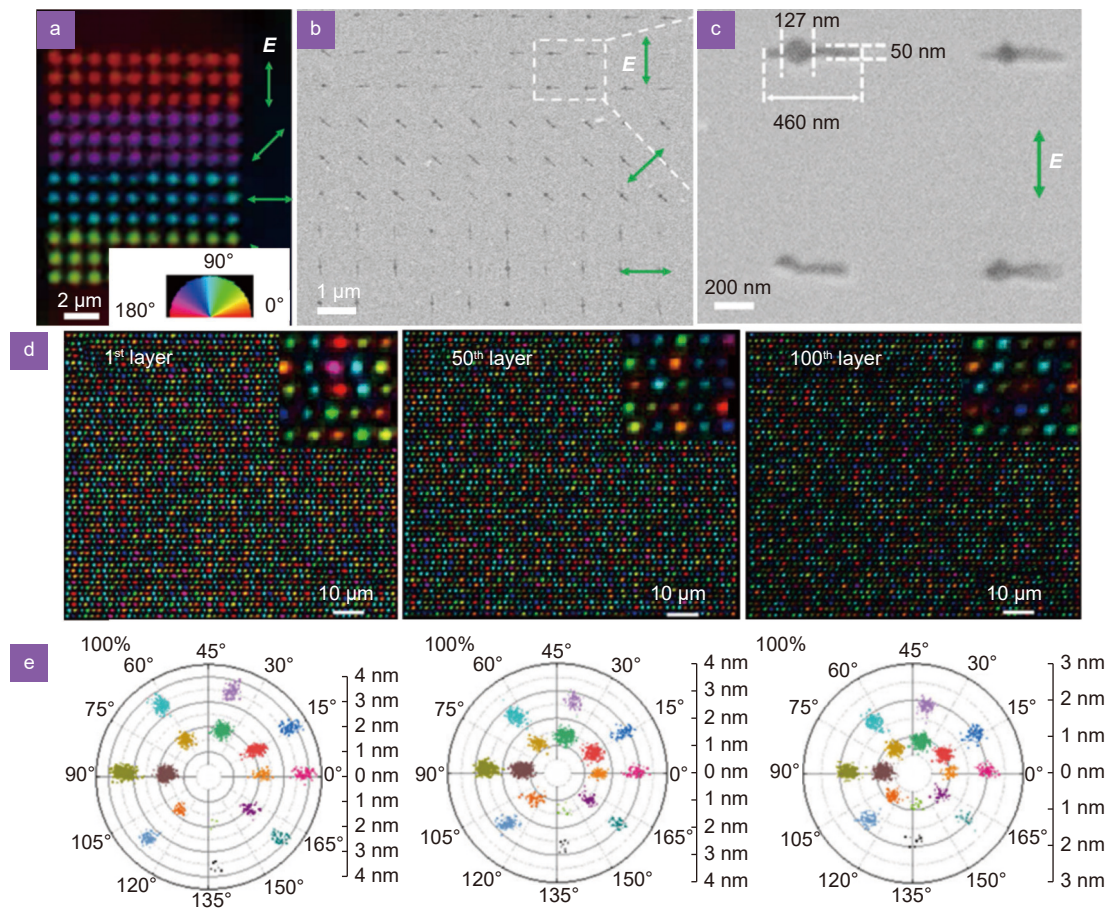


Fig. 12 | (a–c) Imaging of anisotropic nanostructures and 5D optical data storage in fused silica^{153,155}. (a) Image of the slow axis azimuth of voxels, where the pseudo-color represents the slow axis azimuth. (b) SEM image of the nanolamella-like structure after polishing and KOH etching. (c) The enlarged area in the dashed square in (b). (d) The birefringent images of data voxels of different layers in a 100-layer 5D optical data storage in fused silica after removing the background. Insets are enlargements of small region (10 μm \times 10 μm). (e) Polar diagram of the measured retardance and azimuth of all voxels in (d). Figure reproduced with permission from: (a–c) ref.¹⁵³, Optica Publishing Group, under the Optica Open Access Publishing Agreement; (d, e) ref.¹⁵⁵, under a Creative Commons Attribution License.

The writing speed has been increased to 225 kB/s^{153–155}, which is mainly a result of the higher laser power and repetition frequency with pulse energy modulation, as shown in Fig. 12.

Mechanisms and applications based on femtosecond laser-induced birefringence have been extensively studied, including various polarization-sensitive elements^{70,156–158} such as polarization beam splitters¹⁵⁹, polarization diffraction gratings¹⁶⁰, and polarization optical vortex rotation converters¹²³. HSFLs fabricated in amorphous silicon^{161,162}, silicon carbide¹⁶³ and indium tin oxide (ITO) thin films^{164,165} exhibit birefringence effects two orders of magnitude higher than those of quartz glass, demonstrating that high-refractive-index materials can be used to fabricate birefringent elements. Large-area HSFLs have been efficiently fabricated on a glass surface coated with an ITO thin film using femtosecond laser direct writing focused via a cylindrical lens¹⁶⁶. The

optical retardation reached 44 nm, which was eight times that of the HSFLs processed directly on a bare glass surface.

Enhancement of optical absorption and photoluminescence

The optical properties were significantly tuned after forming complex micro-nanostructures covered with LIPs on the surfaces of metals, semiconductors, and dielectrics^{33,35,37,39,118,167–174}. “Black” silicon has been extensively studied and was shown to exhibit ultra-low reflectivity and very high absorption in the UV–NIR region as a result of nanostructures fabricated by femtosecond lasers^{33,39,171,172}.

Various composite LIPs have been efficiently fabricated on wide-bandgap SiC and ZnO semiconductors by the multi-beam interference of femtosecond lasers^{174,176}.

These composite nanostructures exhibited greatly enhanced light absorption in the UV–NIR region, as well as an intense blue emission when excited by infrared femtosecond lasers. Compared with the smooth surface of ZnSe crystals, the blue light intensity of the composite nanostructures excited by a 1200 nm femtosecond laser was enhanced by 30–100 times¹⁷⁵, as shown in Fig. 13.

Electrical properties improved by LIPs

Recently, the effect of LIPs on the electrical properties of ITO^{40,41,177} and the superconducting material niobium^{178,179} has received increasing attention. ITO films are widely used as transparent conductive materials in solar cells, sensors, and displays. An ITO film with LIPs exhibited good anisotropic conductivity and, if fabricated under appropriate conditions, could even achieve unidirectional conductivity, as shown in Fig. 14⁴¹. Moreover, the transmittance in the near-infrared region was greatly enhanced^{40,41,177}. These results demonstrate the potentials of femtosecond LIPs for efficiently fabricating large-scale transparent nanowires¹⁸⁰.

Cubero et al.^{178,179} analyzed the effect of laser irradiation on the critical current value of superconductors by measuring hysteresis loops, and proved that LIPs could change the superconducting properties of niobium. The secondary electron emission from a copper surface with LIPs was effectively reduced¹⁸¹, which was very effective in suppressing secondary electron emission in accelerators.

Other applications

After femtosecond laser processing, the entire ablation area is covered with nanostructures, which can effect-

ively increase the surface roughness and reduce the contact area with the droplet. This is an efficient method to alter the hydrophilicity and hydrophobicity of sample surfaces, and has been successfully demonstrated on various materials including metals, semiconductors, dielectrics, and polymers^{182–192}.

Femtosecond-LIPs were optimized on steel, titanium alloy, and atomic layer deposition cemented carbide surfaces. The friction coefficient and the wear were reduced significantly, which revealed the great potential for tribological applications^{193–199}.

Outlook

In this paper, the formation mechanisms, high-quality and high-efficiency processing methods, and applications of LIPs were reviewed. We believe that the following problems that may be worth further investigation.

Formation mechanisms of LIPs

The formation mechanism of LIPs has always been a hot topic in this field. The scattered light model and propagating SPP model are continuously being developed and refined and have been used to explain the formation of LSFLs on dielectrics, semiconductors, and metals. However, the formation mechanism of LSFLs requires further experimental and theoretical studies, especially ultrafast dynamics.

HSFLs are typically formed after irradiation with multiple pulses. Their formation mechanism is more complex and difficult to determine than that of LSFLs, requiring extensive experimental and theoretical studies, including numerical simulations of the dynamics using the COMSOL and FDTD methods. In recent years,

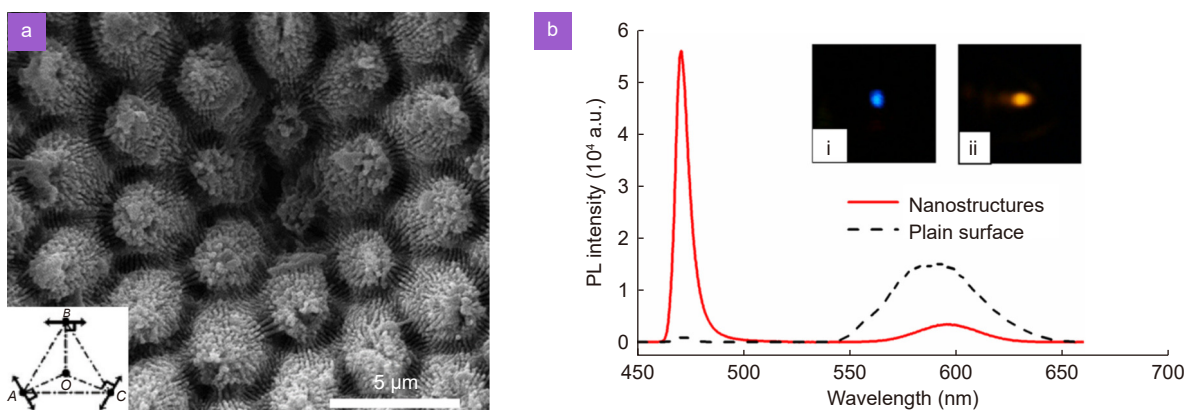


Fig. 13 | (a) SEM image of composite nanostructures on ZnSe crystal. (b) The photoluminescence (PL) spectra of the plane surface and composite nanostructures irradiated by 1200 nm femtosecond laser. Insets (i) and (ii) show PL images of the nanostructures and plane surface, respectively. Figure reproduced with permission from ref.¹⁷⁵, AIP Publishing.

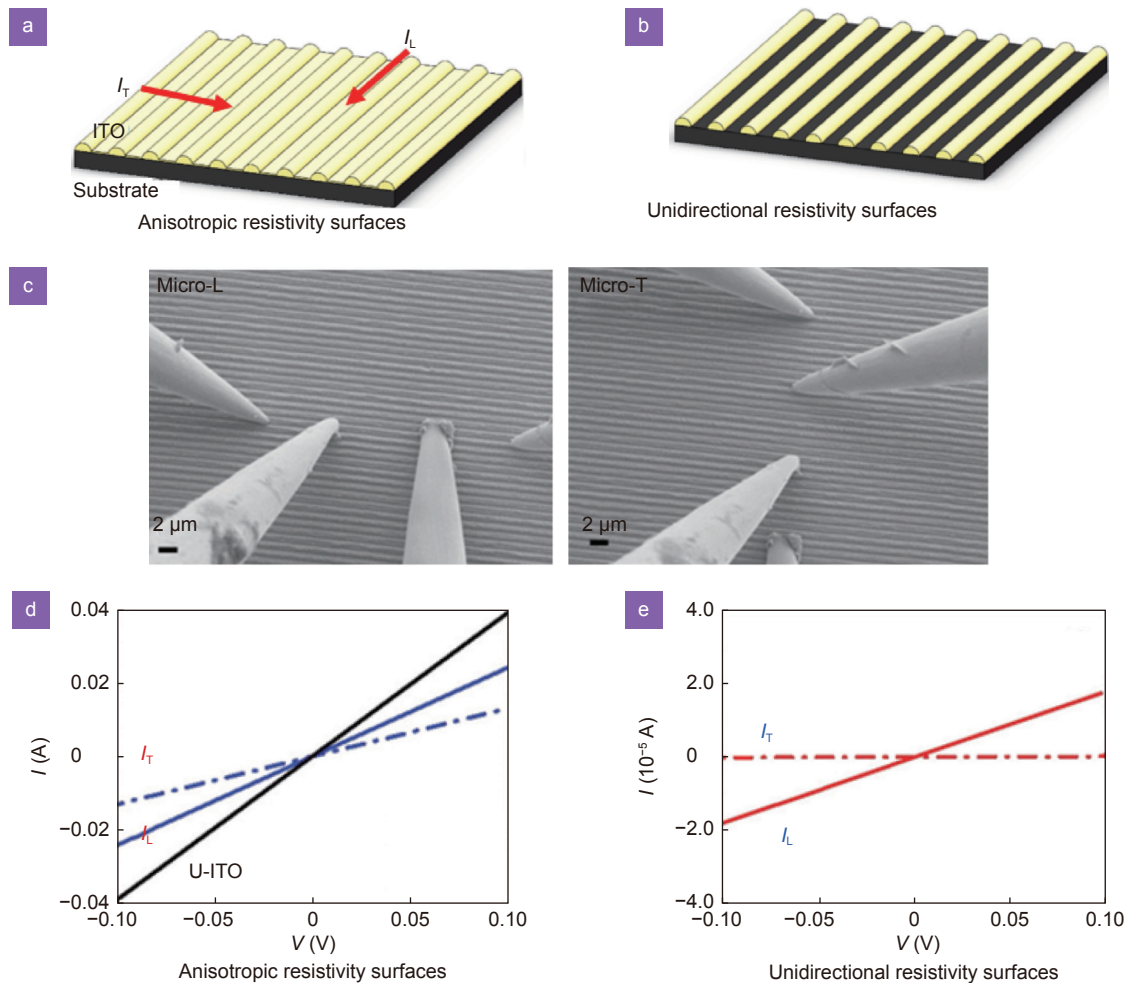


Fig. 14 | (a–b) Schematic illustrations of the laser-induced structure in the ITO thin film on the substrate, and the resistivity measured longitudinally (I_L) or transversely (I_T) along the LIPS direction. (c) Microscopic configuration of the four-point probe measurement with the probes longitudinal (Micro-L) or transverse (Micro-T) aligned with the LIPSS. (d) and (e) are the I - V curves of the anisotropic resistivity surface and the unidirectional resistivity surface measured by the Micro-L or Micro-T method, respectively. Figure reproduced with permission from ref.⁴¹, under a Creative Commons Attribution License.

various far-field super-resolution imaging methods have been reported^{200–202}. Combining the pump-probe method with far-field super-resolution imaging is very interesting and may be helpful in revealing the formation mechanism of HSFLs irradiated by a femtosecond laser.

Patterns with features of <30 nm obtained by direct writing

The groove width of HSFLs is only tens of nanometers, which provides a new method of nanofabrication using ultrafast laser direct writing. However, before this can be widely applied, there are still many problems to be solved such as the slow scanning speed and poor uniformity of the nanogrooves. In the last decade, multi-dimensional laser shaping technologies in time/frequency and polarization have been rapidly developed^{108,203}. Synchronizing

laser direct writing and multi-dimensional laser shaping in real time will ensure that the nanogrooves grow along the direction of the direct laser writing, improve the local optical field enhancement, and reduce the residual heat of ablation. This is expected to efficiently fabricate high-quality nanopatterns with features less than 30 nm.

LIPSS based on multi-dimensional laser shaping and its applications

Large-area LIPSS fabricated with high quality, efficiency, and stability can greatly promote their applications in many fields. By developing pulse-shaping methods based on the 4f configuration zero-dispersion system or Fabry-Perot interferometer²⁰⁴, the regularity and depth of LIPSS can be effectively improved, which will improve the phase retardation and writing speed and reduce the bit

error rate when reading out. Combining time/frequency shaping and spatial shaping can improve the regularity and depth of LIPSs fabricated with high efficiency, thereby improving the purity and brightness of the structural colors. By simultaneously controlling the polarization and spatial distribution of the laser field, nanostructures with designed patterns and specific nanogroove distributions can be processed with high efficiency. Combining time/frequency domain shaping and polarization shaping can improve the quality and efficiency of far-field nanofabrication and allow LIPS applications in new fields such as metasurfaces and microelectronics.

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Competing interests

The authors declare no competing financial interests.