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Femtosecond laser-induced subwavelength ripples formed by asymmetrical grating splitting

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ABSTRACT

The formation process and mechanism of subwavelength ripples were studied upon irradiation of ZnO by a femtosecond laser (800 nm, 50 fs, 1 kHz). An abnormally asymmetrical grating-splitting phenomenon was discovered. At relatively high laser fluences (F = 0.51–0.63 J/cm^2), near-wavelength ripples were split asymmetrically to create subwavelength laser-induced periodic surface structures (LIPSS) with dual gaps (~230 nm and ~430 nm) on the primary grooves. At relatively low laser fluences (F = 0.4–0.45 J/cm^2), near-wavelength ripples were split symmetrically, leading to the formation of uniform subwavelength structures with a period of ~340 nm. The splitting phenomena are related to the varying laser beam dose induced by the overlapping during line scanning. The two grating-splitting types further imply that the dominated mechanism for LIPSS formation may be changed under different processing conditions.

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1. Introduction

Laser-induced periodic surface structures (LIPSS) have been widely studied due to their potential industrial applications, especially in absorption enhancement and the generation of functional surfaces [1,2]. The LIPSS with a period close to the laser wavelength are referred as low spatial frequency LIPSS (LSFL). However, in very recent studies, the LIPSS with periods significantly smaller than a wavelength can be obtained on various materials, which are defined as high spatial frequency LIPSS (HSFL) [3–6]. Furthermore, single-crystalline ZnO, a promising wide bandgap optoelectronic material, is always selected for study by researchers as it shows high nonlinear optical properties and is transparent at the laser irradiation wavelengths in the near-infrared (NIR) spectral region [7].

Generally speaking, the spatial characteristics of LIPSS may depend strongly on processing parameters, such as laser fluence and pulse number [8–10]. The formation mechanism of LIPSS is quite a controversial topic, which has been frequently discussed in recent years.

The explanation for the formation of LSFL has been widely accepted to be a result of the interference between the incident laser and surface plasmons (SPs) [11]. The period-decreasing phenomenon may originate in the effect of the field distribution caused by the grating-assisted SP-laser coupling effect [12]. However, several mechanisms, such as excitation of surface plasmon polaritons (SPP) [13], the effect of grating-assisted SP-laser coupling [14], or second harmonic generation (SHG) [15,16] have been proposed to explain the formation of HSFL. For example, Das et al. has applied an extended Drude-Sipe formalism to define conditions for a continuous transfer between LSFL and HSFL [13]. The SPP wavelength of ZnO can be theoretically predicted as a function of the carrier density. The results of simulations and experiments indicate that the generation of SPP plays a major role in the mechanism of HSFL formation. From another point of view, Dufft et al. has presented a complementary study on the formation of LIPSS in single-crystalline ZnO after irradiation with near-infrared femtosecond laser pulses [17]. The spectroscopic experiments reveal the generation of frequency-doubled radiation SHG in the regime of HSFL formation. However, the origin of the LIPSS formation and evolution on a ZnO surface is still an open question. Many researchers have reported the symmetrical grating-splitting phenomenon on various materials; however, we report the asymmetrical grating splitting here, for the first time.

We present a systematic study on the formation and evolution of LIPSS (HSFL/LSFL) in single-crystalline ZnO after irradiation with a linearly polarized femtosecond laser with a wavelength of 800 nm and a pulse duration of 50 fs in air. Here, an interesting phenomenon is reported, manifested as asymmetrical grating-splitting from LSFL to HSFL to form bi-periodic structures when the applied laser fluence is between 0.51 J/cm^2 and 0.63 J/cm^2. For lower laser fluence (0.4 J/cm^2 ~ 0.45 J/cm^2), LSFL split symmetrically, which may be caused by the admixture of SPP and the redistribution of the field intensity localized from the grooves to the ridges. The observation of a special transition (LSFL/HSFL) provides morphological evidence that would be helpful for us in further understanding basic physical mechanisms.

2. Experiments

A Ti: sapphire laser system consisting of a mode-locked oscillator and a regenerative amplifier was used in the experiment to generate linearly polarized laser pulses (λ = 800 nm, τ = 35 fs, ν = 1 kHz), fitting well with a Gaussian profile. The single-crystalline <0001> ZnO sample (5 mm × 5 mm × 0.5 mm) was mechanically

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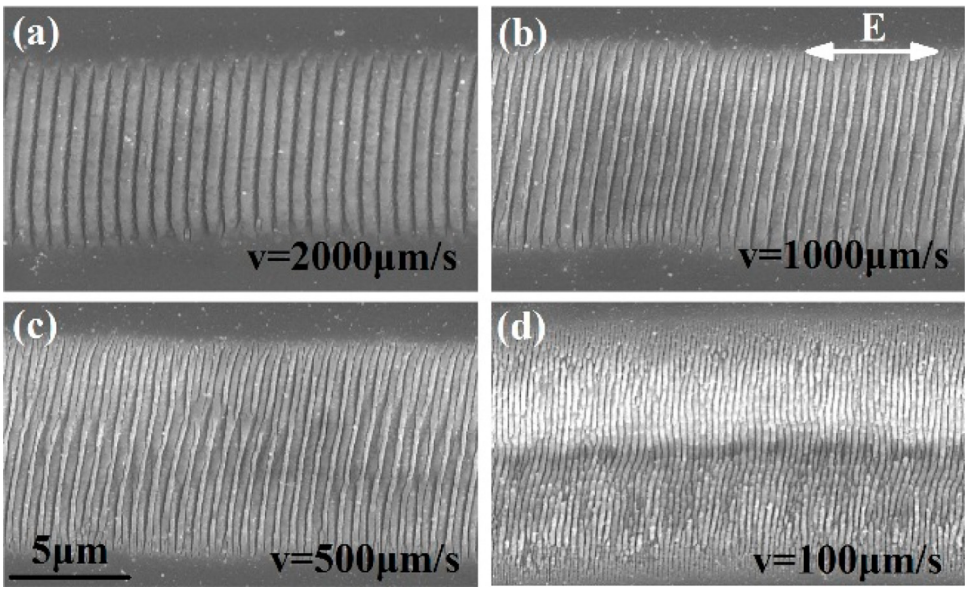

Fig. 1. SEM images of the scanned lines obtained on the surface of single-crystalline ZnO at different scanning speeds. The polarization direction is indicated by the arrow. The laser fluence was fixed at 0.51 J/cm^2.

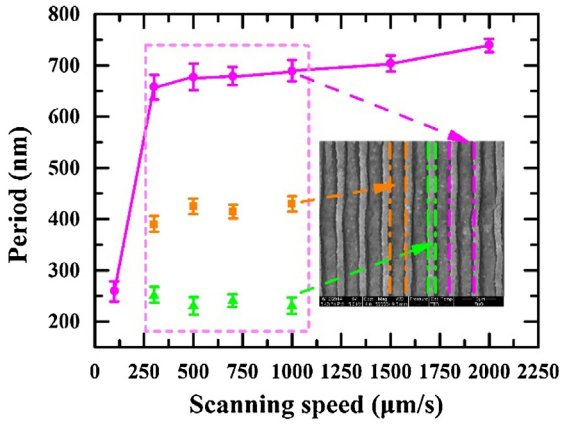

Fig. 2. Dependence of the LIPSS periods as a function of the scanning speed at 0.51 J/cm^2.

polished to a surface roughness of 0.5 nm Ra. The sample was mounted on a computer-controlled, six-axis translation stage (M-840.5DG, PI, Inc.) with a positioning accuracy of 1 μm. The linearly polarized laser beam was incident normal to the sample surface focused by a quartz convex lens with a focal length of 100 mm, corresponding to a spot size diameter of ~20 μm determined using a method by Liu [18]. The laser fluence was continuously adjusted by using a combination of a half-wave plate and a polarizer. All experiments were carried out in air at ambient pressure and temperature. The surface and spatial morphologies after laser processing were imaged by scanning electron microscopy (SEM) and atomic force microscopy (AFM) in tapping mode, respectively.

3. Results and discussion

It is known that the formation and evolution of LIPSS (LSFL/HSFL) depends strongly on the laser fluence and average number of irradiated pulses. Fig. 1 shows the SEM images for the scanning lines obtained at different scanning speeds. The laser fluence was fixed at 0.51 J/cm^2. The number of femtosecond laser pulses ranged from 10 to 200 adjusted by varying the scanning speed (v) of the laser beam from 2000 to 100 μm/s. It can be concluded that the periods of LIPSS monotonically decrease with a decrease in scanning speed (the increase of the average number of irradiated pulses), which is illustrated in Fig. 2. When the scanning speed was 2000 μm/s, uniform LSFL with a period of ~700 nm close to the laser wavelength (800 nm) were found, as illustrated in Fig. 1(a). The orientation of the LIPSS was perpendicular to the laser polarization. The nanostructures obtained were not only well-patterned but also clearly long-range ordered, which may show novel properties and great interest for manipulating them to the level of industrial applications. By reducing the scanning speed to 1000 μm/s, we clearly found that the LSFL split asymmetrically so that bi-periodic structures with dual gaps of ~230 nm and ~430 nm were generated on the primary grooves simultaneously (Fig. 1(b)). At the same time, on repeating this experiment, similar results were obtained when we fixed the laser fluence at 0.58 J/cm^2 and 0.63 J/cm^2. Although the LIPSS on ZnO have been extensively studied in recent years, the interesting phenomenon of asymmetrical grating-splitting forming bi-periodic structures is reported here for the first time. Additionally, when the scanning speed of the laser beam was decreased to 100 μm/s, it was found that the HSFL induced completely substituted for the LSFL, exhibiting spatial periods between 230 nm and 280 nm (Fig. 1(d)), which was consistent with previous reports.

When the applied laser fluence was 0.45 J/cm^2, another ablation topography appeared with changes in the scanning speed, as shown in Fig. 3. Similarly, when the scanning speeds were varied, the periods of the LIPSS would transform from ~710 nm to ~230 nm; and the orientations were also perpendicular to the laser polarization. However, the evolution process deserves our attention.

Different from previously mentioned in this paper, LSFL split symmetrically led to the formation of regular, uniform HSFL with a period of ~340 nm, that is, the intermediate values of the period for asymmetrical grating splitting. Moreover, AFM was used in order to gain a deeper insight into the information about the spatial characteristics of HSFL. Therefore, it was clearly shown that laser fluence, as well as pulse number, significantly influenced the evolution process. Similar results were obtained on metals by using femtosecond laser pulses, and it was suggested that redistribution of the electric field intensity due to the formation of LSFL plays a crucial role in the creation of HSFL [14].

In addition, our study systematically investigated the relevant parameter window (laser fluence, scanning speed) for which differ-

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