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Fabrication of surface relief gratings on transparent dielectric materials by two-beam holographic method using infrared femtosecond laser pulses

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Abstract. Fabrication of surface relief-type gratings in transparent dielectrics, which are hard to machine, has been achieved by a holographic technique using two infrared femtosecond (fs) pulses from a mode-locked Ti:sapphire laser. The present method can be applied for a variety of transparent dielectrics, Al_2O_3 (sapphire), TiO_2, ZrO_2, $LiNbO_3$, SiC, ZnO, CdF_2, MgO, CaF_2 crystals, and SiO_2 glass. It is found that the grating formation is due primarily to laser ablation processes. Planar surface relief gratings can be fabricated by colliding two fs laser pulses on the surface of substrates which move at a constant speed, synchronized with the laser repetition rate.

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A mode-locked (ML) Ti:sapphire laser has two unique features among pulsed lasers. One is, of course, its ultrashort pulses of femtosecond (fs) to subpicosecond duration. Such ultrashort pulses opened up new possibilities for the laser micromachining of wide band gap materials [1–4] as well as the observation of transient phenomena such as carrier dynamics in semiconductors [5]. There are a number of advantages in using lasers for micromachining. However, one requires very high laser intensity with nanosecond (ns) pulses in order to obtain sufficient energy absorption in the optically transparent dielectrics to observe macroscopic material removal. The thermal stresses built up in the materials during irradiation and the extensive cracking and exfoliation are induced by intense ns laser pulse irradiation. These faults may be effectively avoided by using ultrashort laser pulses instead of ns laser pulses. Dots with differing refractive indexes and well-defined channels are successfully formed in these materials by near IR light pulses from Ti:sapphire lasers [6–8] . Another feature of ML Ti:sapphire lasers is that the pulse is close to Fourier-limited, i.e., $\Delta\nu\Delta\tau < \alpha$, where $\Delta\nu$ and $\Delta\tau$ denote the spectral width and the time width of the pulse, respectively, and α is a constant depending on the intensity profile [9]. This means that when two pulses collide with each other, high optical interference occurs in a temporary overlapped region. We may anticipate a new development of laser micromachining for transparent dielectric materials utilizing this feature. Here we report the first demonstration that surface relief gratings in transparent dielectric materials, which are hard to machine, can be fabricated by holographic methods using two crossed pulses of a single shot from a fs ML Ti:sapphire laser.

1 Experimental

The substrates used are single crystal plates of sapphire, SiC, $LiNbO_3$, ZrO_2 (cubic), ZnO, CaF_2, CdF_2, MgO, TiO_2, and SiO_2 glass plates. Figure 1 shows an experimental setup. Near infrared light (800 nm) pulses from a ML Ti:sapphire laser were amplified by a regenerative amplifying system using an Nd:YAG laser operated at 10 Hz, separated into 2 paths (intensity ratio $\sim 3:7$), and finally these two focused pulses were crossed on the top surface of the substrate. The beam diameter on the sample surface is $\sim 100\,\mu m$. The angle between these two beams was varied in the range of 10 to 30° and the time delay between the two pulses was adjusted in the range of 0.2 to 2 ps by changing the optical path of one of the pulses. The pulse duration was ~ 100 fs and the laser power at the substrate position was varied in the range of 0.1 to 3 mJ/pulse. Writing of gratings was monitored in situ by observing diffracted light source: He-Ne laser light) from the spot. For the fabrication of planar gratings, the substrate is moved using a pulsed stage at a constant speed, synchronized with the laser pulse repetition rate.

The formation of gratings was confirmed ex situ by observation of the irradiated surface area with a differential interference microscope (DIC), a scanning electron microscope (SEM), and an atomic force microscope (AFM). Micro-Raman scattering spectra (spot size, $10\,\mu m$ in diameter) were measured on the encoded gratings.

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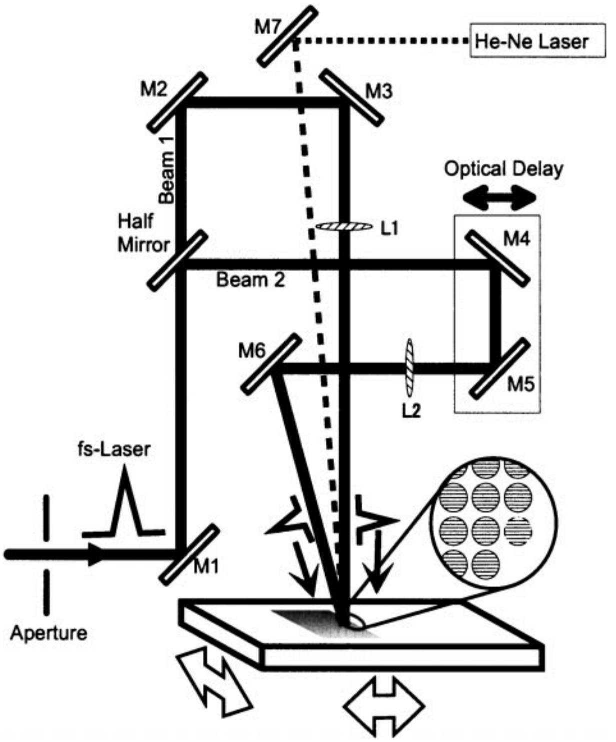

Fig. 1. Experimental setup for holographic encoding of permanent gratings by holographic technique using fs pulses from a ML Ti:sapphire laser. For the fabrication of planar gratings, a substrate is moved at a constant speed, synchronized with laser pulse repetition rate, i.e., 0.1 mm/s

2 Results and discussion

Figure 2 shows an example of an AFM photo showing the formation of surface gratings encoded on a sapphire surface by the present method. A periodic line valley with a constant spacing of $\sim 2.3\,\mu$m is clearly visible and the depth of the valley is 120–140 nm. No such periodic pattern formation was observed for cases in which the relative time delay of the two pulses was 0.2–10 ps, indicating that temporal and spatial overlapping of the two pulses is indispensable for the grating formation. A threshold power density for the recording was noted to exist. The order of recording threshold power density for various transparent dielectrics is SiO_2 glass $>$ Al_2O_3, CaF_2 $>$ ZrO_2 $>$ MgO $>$ $LiNbO_3$ $>$ CdF_2 $>$

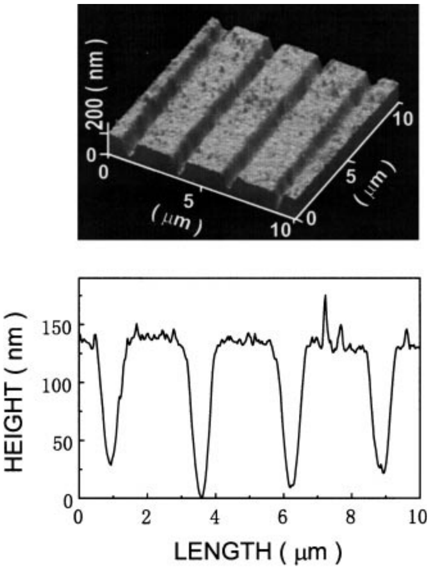

Fig. 2. Atomic force microscopic photo showing a surface relief-type grating on sapphire

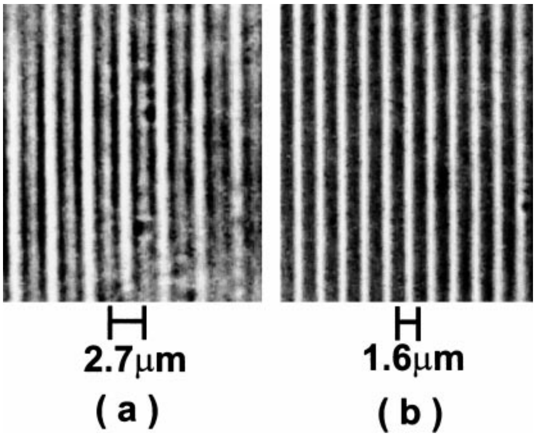

Fig. 3a,b. Optical micrographic photos of surface relief-type gratings encoded on sapphire by holographic irradiation with fs laser pulses. Cross-beam angle: 17° (**a**) and 30° (**b**)

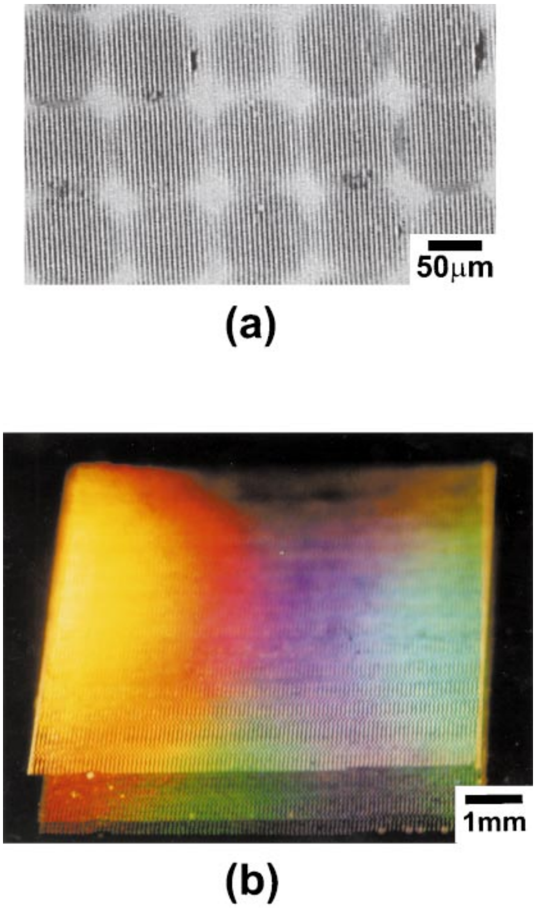

Fig. 4a,b. Optical microscopic photo of planar gratings encoded on SiO_2 glass. **a** Top view of the gratings. **b** Bird's-eye view of the gratings. The reflection image from the bottom surface is also seen

$TiO_2 > SiC > ZnO$. Although the threshold value changes for each material, surface relief gratings could be formed for all the transparent dielectric materials we examined. The encoded periodic spacing (d) was changed by varying the angle (θ) between the two crossed beams. Figure 3 shows examples of surface relief gratings encoded on sapphire by irradiation with crossed fs laser pulses with differing θ, 17° and 30°. The d varies with the θ, i.e., 1.6 μm for 17° and 2.7 μm for 30°. These correspondences meet the well-known relation for two beam interference at the surface: $d = \lambda/[2\sin(\theta/2)]$, where λ denotes the wavelength (∼ 800 nm) of light for recording. The above results demonstrate that surface relief gratings are formed by the interference of two fs pulses from a single shot of a ML Ti:sapphire laser.

No significant changes were noted in micro-Raman spectra between the grating-encoded areas and pristine areas of various materials, indicating that no significant structural alteration remains on the surface and in the near-surface regions of the irradiated areas. These findings lead to the conclusion that the encoded surface relief gratings are primarily formed by laser ablation processes.

Figure 4 shows photos of planar gratings encoded on a SiO_2 glass (5 mm × 5 mm) fabricated by moving a substrate at a constant speed, synchronized with the laser pulse repetition rate (10 Hz). Each grating spot of ∼ 100 μm diameter is encoded by a single shot.

Gratings are the key optical elements for diffractive optics [10]. The holographic encoding of gratings on photosensitive glass such as SiO_2:GeO_2 glass has been extensively studied, aiming at diffractive devices for wavelength division multiplexity (WDM) communications [11–14]. However, encoding surface gratings on pure SiO_2 glass by laser light has not succeeded to date as far as we know. Furthermore, the present technique can be applied to various transparent dielectric materials. We anticipate that a new frontier of sophisticated micromachining of transparent dielectrics will be open with the present holographic technique using fs light pulses from a ML Ti:sapphire laser.

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