Structures fabrication on the sapphire surface by femtosecond laser pulses

Yoshiharu Namba, Litao Qi
Motohiro Yasui, Kazuhiro Nishii, Hikoharu Aoki (Brother Industries, LTD.)

Experiments on structures fabricated on the sapphire surface by femtosecond laser pulses are carried out. The sapphire is first irradiated under static irradiation, and then the fabrication of microgrooves on sapphire by femtosecond laser pulses is investigated through line-scanning experiments. Experiments on femtosecond laser ablation of sapphire of different crystal-plane are carried out. High-spatial-frequency laser-induced periodic surface structures with spatial period of 340 nm and the craters free of the cracks are obtained. The mechanism of femtosecond laser ablation of sapphire is studied through the ablation depth of the craters varied with the pulse energies and number of laser pulses. Two ablation phases are observed, which is in agreement with previous works. The transition regime between the two ablation phases is found. The craters ablated in the transition regime shows good surface profile and surface quality. On the microgroove fabrication, the surface quality of the fabricated microgrooves could be improved through increasing the number of laser scans.

1. Introduction

Sapphire is widely used in the field of optical components and micromechanical devices owing to its useful mechanical, optical, and electrical properties. However, sapphire is mechanically and chemically difficult to machine because of its high hardness and chemical stability. The difficulty of machining sapphire has restricted the development of advanced device structures. As sapphire is inert to most types of wet chemical and dry etching, laser ablation has been proposed as a potential machining method. The interaction of laser pulses with sapphire materials has been investigated for many years [1-7]. However, a thermal effect is unavoidable, even in ultraviolet laser processing. Recently, the femtosecond laser ablation of sapphire has been attracting much attention because femtosecond laser ablation can produce precise, well-defined micrometer-sized structures in materials that cannot be processed with nanosecond pulsed lasers. A number of studies on the femtosecond laser ablation of sapphire have been carried out [5, 7-12], and a mechanism for the femtosecond laser ablation of sapphire has been proposed [5, 9-11].

The advanced development of III-V nitride devices such as those of GaN and InGaN [11, 12] has prompted research into a suitable method of patterning sapphire. However, further experiments need to be carried out to obtain fine structures on sapphire. In this study, experiments on the femtosecond laser ablation of sapphire are carried out focusing on several aspects including mechanism of femtosecond laser ablation of sapphire through the ablation depth, femtosecond laser ablation sapphire on different crystal-plane, and then a method of improving the surface quality of microgrooves on sapphire. As a result of our investigations, a sample of sapphire with parallel microgrooves on the surface is fabricated.

2. Experiment

In the experiments, we used a commercially available amplified Ti:sapphire laser system that generated 164 fs laser pulses with a maximum pulse energy (E_p) of 1 mJ at a 1 kHz repetition rate and with a central wavelength of λ = 780 nm. The laser beam had a Gaussian profile with a diameter of 6 mm. The incident laser beam was irradiated onto a sapphire sample through a 4 mm aperture positioned normal to the sample and a 10× microscope objective lens with the numerical aperture of 0.30. The laser spot size on the sample was estimated to 8~10 μm. The sample was placed on a scanning stage and translated at various scanning speeds (ν) under computer control during irradiation. The scanning direction was perpendicular or parallel to the laser polarization direction. All experiments were carried out in air at atmospheric pressure and room temperature. After irradiation, the sample was rinsed for 30 minutes with acetone in an ultrasonic cleaner to remove any debris from the ablation process. The morphology of the structures was examined by scanning electron microscopy (SEM). The sample was coated with a gold layer of approximately 20 nm thickness to ensure conduction in the SEM observation. The surface profile was measured by atomic force microscopy (AFM).

3. Results and discussion

The sapphire is first irradiated by different pulse energies and numbers of laser pulses under static irradiation, and then the fabrication of microgrooves on sapphire by femtosecond laser pulses is investigated through line-scanning experiments.

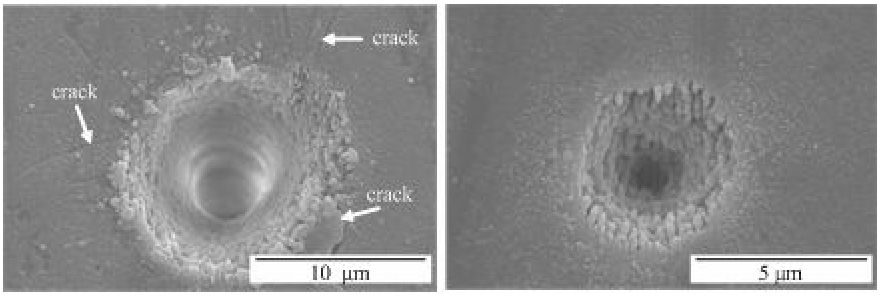
The surface damage threshold is determined by ex situ optic microscope. The damage thresholds obtain from an analysis of ex situ light scattering is identical for more than 5 shots, within the experimental error bars. The damage threshold of femtosecond laser ablation of sapphire is around 4.0 J/cm^2.

3.1 Characteristics of femtosecond laser ablation of sapphire

Cracks have a negative effect on the applicability of sapphire. The formation of cracks is unavoidable, even in ultraviolet laser processing with long pulses, because of the thermal effect [6]. The sapphire ablated by femtosecond laser pulses is free of cracks under appropriate irradiating conditions. Figure 1 shows SEM images of the craters produced by the irradiation of femtosecond laser pulses with 1000 laser shots and different pulse energies. When the pulse energy is high, cracks are observed around the ablated crater, as shown in Figs. 1(a). When the pulse energy decreases, no cracks are observed around the ablated crater, as shown in Figs. 1(b). Figure 2 shows an image of laser-induced periodic surface structures (LIPSS) formed on the sapphire surface when it is irradiated with pulse energy close to the ablation threshold and 50 laser shots. The spatial period of LIPSS on the sapphire surface is approximately 340 nm. The orientation of the LIPSS is perpendicular to the polarization direction of the laser beam. The formation of LIPSS on sapphire is caused by interference between the laser-induced plasma and the incident laser beam [14]. Further investigations on the fine nanostructures formed on sapphire by femtosecond laser pulses similar to those that Shinoda *et al.* obtained on diamond [14] are being carried out because of their potential applications to nanotechnology.

3.2 Femtosecond laser ablation of sapphire near the surface damage threshold

Upon irradiation with ultraviolet and ultrashort laser pulses, two ablation phases can be observed with increasing number of laser pulses [3, 5]. One is a ‘gentle’ ablation phase characterized by a low rate of material removal, which has been attributed to particle vaporization or Coulomb explosion [9, 15]. The second phase is characterized by a high rate of material removal and the production of molten droplets, which has been attributed to a phase explosion [3, 5]. In our experiment, the same results are obtained, in agreement with previous works by Tam *et al.* [3] and Ashkenasi *et al.* [5]. Figure 3 shows the AFM images of the 3D profile of the crater produced by the irradiation of femtosecond laser pulses with different pulse energies and number of laser pulses. Figure 4 shows the ablation depth of the crater varied with the pulse energy. From the Fig. 3 and Fig.4, the surface quality and the ablation depth of the crater varies with the pulse energy. When the pulse energy is high, the molten droplets are observed around the crater (see in Figs. 3(a)) and the ablation rate is high, as show in Figs. 4(a). When the pulse energy is below the single-shot ablation threshold, no particles are found around the crater (see in Figs. 3(c)) and the ablation rate is low, as shown in Figs. 4(b).



(a) E_p = 16.49 μJ (b) E_p = 1.33 μJ

Fig.1. SEM images of the craters produced by irradiation of femtosecond laser pulses with 1000 laser shots and different pulse energies.

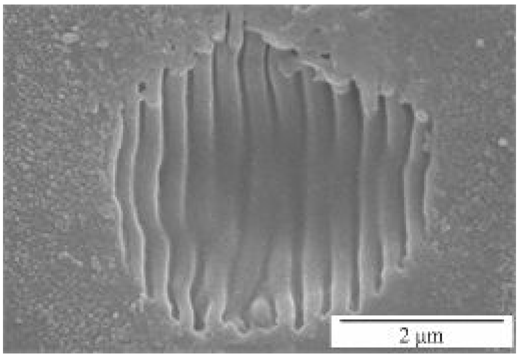
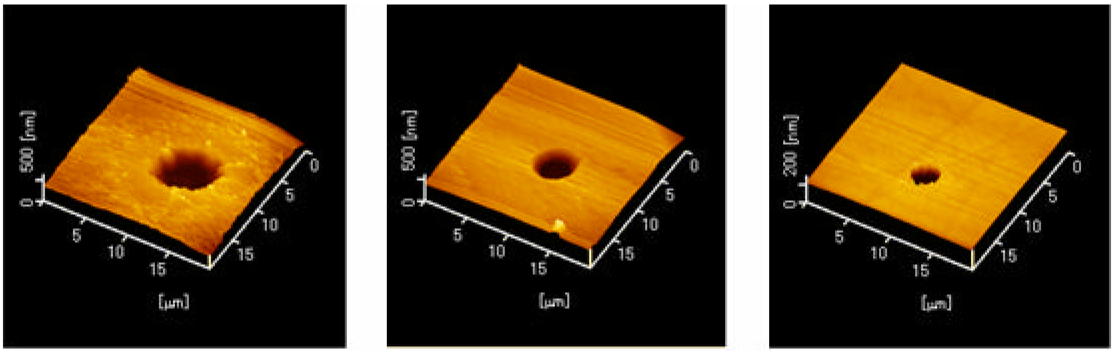


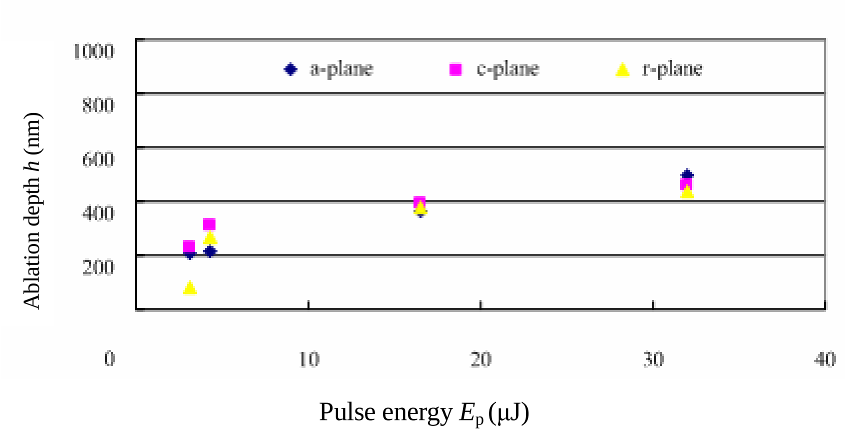
Fig.2. SEM image of LIPSS formed on sapphire by femtosecond laser pulses with E_p = 1.95 μJ and 50 laser shots.

A ‘transition’ phase between the ‘gentle’ phase and the strong’ phase for the pulse energy between 2.27 μJ and 4.35 μJ is observed from the Fig.4. The ‘transition’ phase depends on the number of laser pulses. Table 1 shows the ablation depth varies the pulse energy with a parameter of the number of laser pulse. When the number of laser pulse is 1, the pulse energy for the ‘transition’ phase is from 3.15 μJ to 4.35 μJ. As the number of laser pulse increases to 5 or 10, the pulse energy for the ‘transition’ phase is from 2.27 μJ to 3.15 μJ. The pulse energy for the ‘transition’ phase affects by the number of laser pulses because of the incubation effect. The threshold decrease with an increase in the number of laser pulses until it reaches saturation value [7]. As the number of laser pulse increases, the ablation threshold decreases and correspondingly, the pulse energy for the ‘transition’ phase decreases. The surface quality of the crater irradiated by femtosecond laser pulse with the pulse energy for the ‘transition’ phase is better than that obtains on the ‘gentle’ and ‘strong’ phases, as shown in Fig. 3(b).

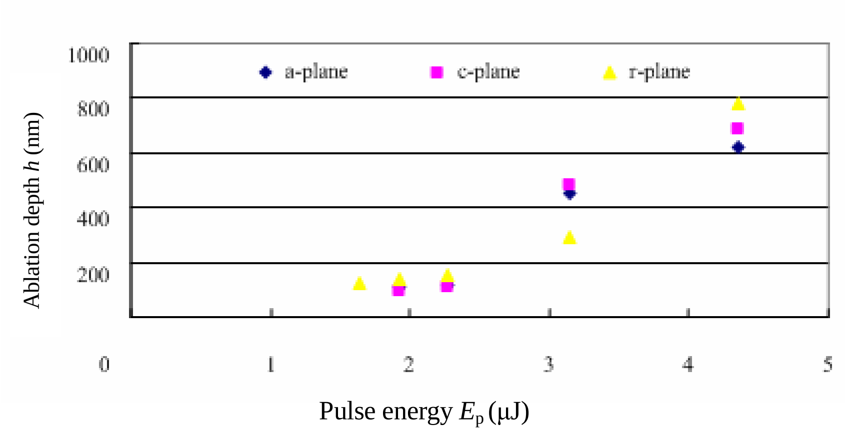


(a) E_p = 16.49 μJ, N = 1 (b) E_p = 3.15 μJ, N = 5 (c) E_p = 1.93μJ, N = 10

Fig. 3. AFM images of the 3D profile of the crater produced by the irradiation of femtosecond laser pulses.



(a) Ablation depth of the craters irradiated by single laser pulse at high pulse energy



(b) Ablation depth of the craters irradiated by 5 laser pulses at low pulse energy

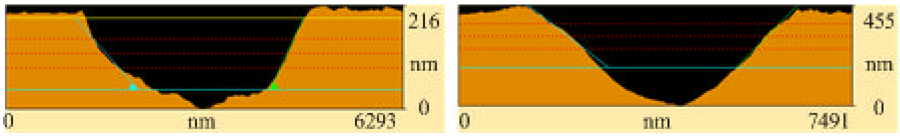
Fig. 4. Relationship between the ablation depth of the crater and pulse energy on different crystal-planes.

Upon irradiation with ultraviolet and ultrashort laser pulses, two ablation phases can be observed with increasing number of laser pulses Figure 5 shows AFM images of the cross-sectional profile of the craters produced by the irradiation of femtosecond laser pulses with pulse energy of 3.64 μJ and different number of laser pulses. During the first 10 pulses, the laser-ablated area is generally smooth, as shown in Figs. 5(a), 5(b), and 5(c). After 10 laser shots, rough regions start to form in the ablated area, as shown in Figs. 5(d).

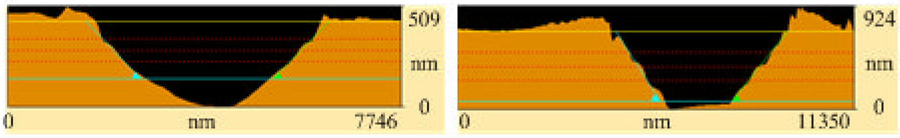
Table 1 Ablation depth of the craters varies with the pulse energy

Pulse energy (μJ) / Ablation depth (nm)		1.64	1.93	2.27	3.15	4.35	16.49	32.01
1	c-plane	x	x	x	228.37	312.35	390.73	456.21
5		x	96.51	112.29	480.85	686.17	0	0
10		226.48	231.27	264.18	819.97	0	0	0

x - no ablation; 0 - no results (out of the scope of AFM measurement); 1, 5, 10 - number of laser pulses; c-plane - femtosecond laser irradiation normal to the c-plane surface of the sapphire.



(a) $N = 1$ (b) $N = 5$



(c) $N = 10$ (d) $N = 20$

Fig. 5. AFM images of cross-section profile of the crater produced by irradiation of femtosecond laser pulses with Ep = 3.64 μJ and different number of laser pulses.

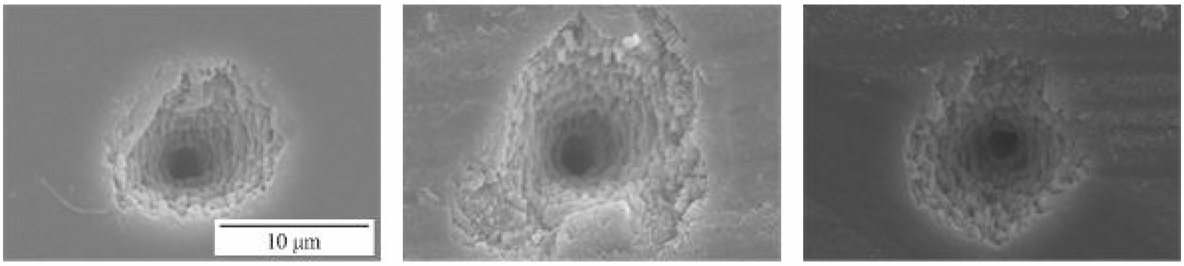
3.3 Femtosecond laser ablation of sapphire on different crystal-plane

Sapphire shows different optical and mechanical properties on different crystal-planes [16]. Experiments to determine the dependence of the surface quality of the ablated crater on the crystal plane of the sapphire are carried out. Three types of crystal plane, c-plane, a-plane, and r-plane, are considered. The experiments are carried out with different pulse energies and number of laser pulses. The results show that the surface quality of the ablated crater is higher when femtosecond laser pulses are irradiated on the c-plane. The SEM images of comparative results of femtosecond laser ablation sapphire on different crystal-plane are shown in Fig.6.

3.4 Microgroove fabrication on sapphire by femtosecond laser pulses

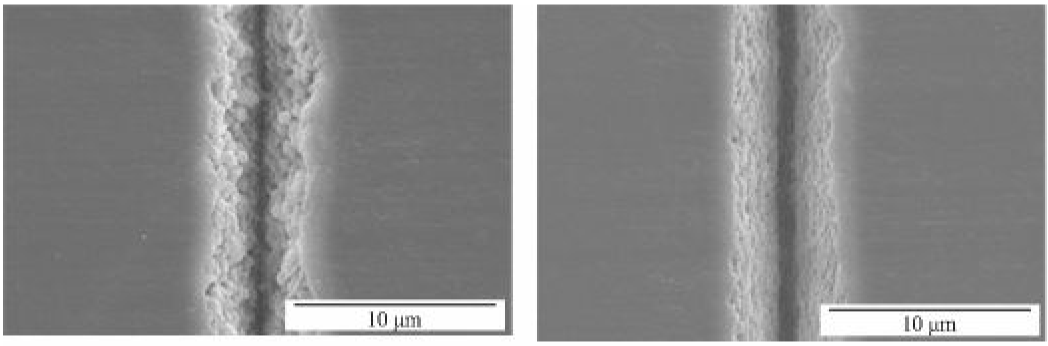
A microgroove fabrication on c-plane are carried out resulting from the surface quality of the ablated crater is higher when femtosecond laser pulses are irradiated on the c-plane. Experiments on the fabrication of microgrooves on sapphire by femtosecond laser pulses are carried out under various conditions of pulse energy, scanning speed, and number of laser scans. The surface quality of the microgrooves is improved when the number of laser scans increases, as shown in Fig. 7. Although as the number of laser scans increases, the ablation depth of the microgrooves remains almost the same. The finding that the ablation depth of the microgrooves is independent of the number of laser scans is consistent with the results of the static irradiation experiments [17]. From the Fig. 7, one can see that after the first pass, the microgrooves fabricated by femtosecond laser pulses leases a fairly rough surface. During the subsequent laser passes, the most intense central part of the laser pulses propagates through the bulk of sapphire without absorption because the laser intensity out of the Rayleigh length is not enough to lead to nonlinear effects on sapphire. The interaction with the cut walls occurs only at the pulse edge, where the local laser intensity is low. In this low fluence regime, only a small amount of materials can be removed from the walls, which is equivalent to a 'polishing' effects that remarkably improves the surface quality.

A sample of parallel microgrooves with high surface quality is fabricated on sapphire by femtosecond laser pulses, as shown in Fig. 8.



(a) c-plane (b) a-plane (c) r-plane

Fig. 6. SEM images of comparative results of femtosecond laser ablation of sapphire along different crystal planes with E_p = 2.27 μJ and number of laser pulses is 250.



(a) Number of laser scans = 2 (b) Number of laser scans = 10

Fig. 7. SEM images of microgrooves fabricated with different numbers of laser scans by femtosecond laser pulses with E_p = 1.64 μJ and ν = 100 μm/s.

4. Conclusion

Experiments on the femtosecond laser ablation of sapphire are carried out. The main conclusions are as follows.

1) Nano- and microstructures are obtained on sapphire.

2) There are two different ablation phase when femtosecond laser ablation of sapphire. One is the 'gentle' ablation and the other is the 'strong' ablation. The transition between

the two phases is observed. The mechanism of two phases is attributed to the Coulomb explosion and phase explosion, respectively.

3) The craters formed by laser ablation are free of cracks under appropriate irradiating conditions.

4) The surface quality of the ablated crater is higher when femtosecond laser pulses are irradiated on c-plane sapphire.

5) In the line-scanning experiments, the surface quality of the ablated microgrooves fabricated by femtosecond laser pulses is improved by increasing the number of laser scans.

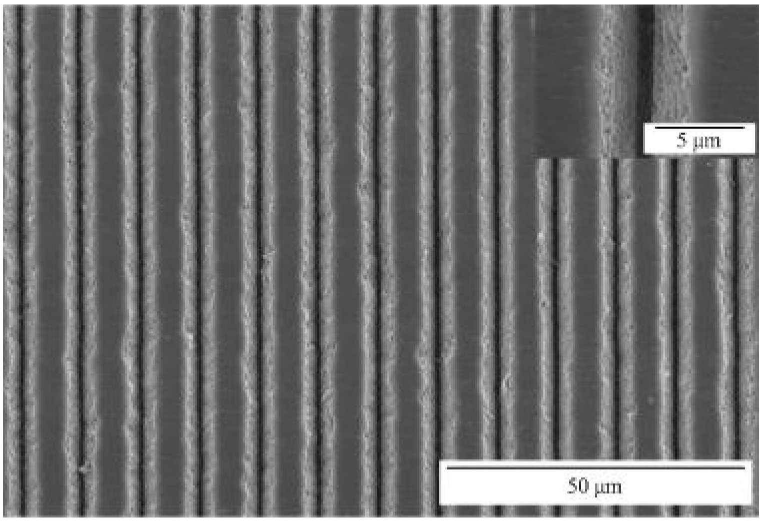


Fig.8. SEM image of parallel microgrooves on sapphire fabricated by femtosecond laser pulses with E_p = 1.64 μJ, ν = 100 μm/s, and 10 laser scans.

References

1) R. W. Dreyfus, F. A. McDonald, and R. J. von Gutfeld: Laser Energy Deposition at Sapphire Surfaces Studied by Pulsed Photothermal Deformation, Appl. Phys. Lett., **50**, (1987) p.1491-1493.

2) J. E. Rothenberg and G. Koren: Laser Produced Plasma in Crystalline α-Al_2O_3 and Aluminum Metal, Appl. Phys. Lett., **44**, (1984)p.664-666.

3) A. Tam, J. Brand, D. Cheng, and W. Zapka: Picosecond Laser Sputtering of Sapphire at 266 nm, Appl. Phys. Lett., **55**, (1989)p.2045-2047.

4) J. Brand and A. Tam: Mechanism of Picosecond Ultraviolet Laser Sputtering of Sapphire at 266 nm, Appl. Phys. Lett., **56**, (1990)p.883-885.

5) D. Ashkenasi, A. Rosenfeld, H. Varel, M. Wähmer, and E. E. B. Campbell: Laser Processing of Sapphire with Picosecond and Sub-Picosecond Pulses, Appl. Surf. Sci., **120**, (1997)p.65-80.

6) H. Horisawa, H. Emura, and N. Yasunaga: Surface Machining Characteristics of Sapphire with Fifth Harmonic YAG Laser Pulses, Vaccum, **19**, (2004)p.661-664.

7) X. Wang, G. Lim, W. Liu, S. Chua, H. Zheng, and F. Ng: Femtosecond Pulse Laser Ablation of Sapphire in Ambient Air, Appl. Surf. Sci., **228**, (2004)p.221-226.

8) S. Matsuo, K. Tokumi, T. Tomita, and S. Hashimoto: Three-Dimensional Residue-Free Volume Removal inside Sapphire by High-Temperature Etching after Irradiation of Femtosecond Laser Pulses, Laser Chem., **2008** (2008)p.892721.

9) R. Stoian, A. Rosenfeld, D. Ashkenasi, I. Hertel, N. Bulgakova, and E. E. B. Campbell: Surface Charging and Impulsive Ion Ejection during Ultrashort Pulsed Laser Ablation, Phys.

Rev. Lett., **88**, (2002) p. 097603.

10) M. Perry, B. Stuart, P. Banks, M. Feit, V. Yanovsky, and A. Rubenchik: Ultrashort-Pulse Laser Machining of Dielectric Materials. J. Appl. Phys., **85**, (1999) p. 6803-6810.

11) E. Glezer and E. Mazur: Ultrafast-Laser Driven Micro-Explosions in Transparent Materials, Appl. Phys. Lett., **71**, (1997) p. 882-884.

12) W. Wong, T. Sands, and N. Cheung: Damage-Free Separation of GaN Thin Films from Sapphire Substrates, Appl. Phys. Lett., **72**, (1998) p. 599-601.

13) W. Wong, T. Sands, N. Cheung, M. Kneissl, D. Bour, P. Mei, T. Romano, and N. Johnson: Fabrication of Thin-Film InGaN Light-Emitting Diode Membranes by Laser Lift-off, Appl. Phys. Lett., **75**, (1999) p. 1360-1362.

14) M. Shinoda, R. Gattass, and E. Mazur: Femtosecond Laser-Induced Formation of Nanometer-Width Grooves on Synthetic Single-Crystal Diamond Surfaces, J. Appl. Phys., **105**, (2009) p. 053102.

15) R. Stoian, D. Ashkenasi, A. Rosenfeld, and E. E. B. Cambell: Coulomb Explosion in Ultrashort Pulsed Laser Ablation of Al_2O_3, Phys. Rev. B, **62**, (2000) p. 13167.

16) L.M. Belyaev: Ruby and Sapphire, First Ed., Amerind, New Delhi, 1980.

17) L. Qi, K. Nishii, M. Yasui, H. Aoki, and Y. Namba: Femtosecond Laser Ablation of Sapphire, Proc. of ASPE 2009, Monterey, America (2009) p. 231-234.