euspen's 16th International Conference & Exhibition, Nottingham, UK, May 2016
www.euspen.eu

Removal of nanoscale damage induced by FIB nanofabrication on single-crystal diamond using femtosecond laser treatment

Zongwei Xu[1*], Fengzhou Fang[1*], Wei Wu[1], Wanli Li[1], Shutong He[2], Minglie Hu[2]

[1] *State Key Laboratory of Precision Measuring Technology & Instruments, Centre of MicroNano Manufacturing Technology, Tianjin University, 300072, China*
[2] *Ultrafast Laser Laboratory, Key Laboratory of Opto-electronic Information Technical Science of Ministry of Education, Tianjin University, Tianjin 300072, China*

Email: zongweixu@163.com, fzfang@tju.edu.cn

Abstract
A post-treatment process using a femtosecond laser was developed to effectively remove nanoscale damage layer covering on a single crystal diamond (SCD) substrate induced during focused ion beam (FIB) nanofabrication. The laser power density threshold for the SCD and the FIB induced damage layer was determined by investigating the morphology, element composition and structure of the irradiated surface using different characterization methods. By exploiting the differences in the ablation thresholds between the FIB modified layer and the SCD, the femtosecond laser process was optimized to fully remove the surface modified layer without affecting the underneath SCD substrate. A circularly polarized laser with a power density of 6.60 × 10^{10} W/cm^2 (4.16 × 10^{-3} J/cm^2) can effectively ablate and eliminate the 50 nm FIB induced modification layer. This study proved that ultrafast laser process is feasible to optimize the single crystal diamond devices developed by FIB nanofabrication.

Keywords: Femtosecond laser, Laser ablation, Single crystal diamond, Focused Ion Beam, Ion implantation, Amorphous layer

1. Introduction

Single crystal diamond (SCD) has excellent physical, optical and electrical properties and has been an attractive material for use in mechanical, optical and electronic devices, such as micro-tools [1], micro-cantilevers [2], electronic devices [3], etc. High-precision material removal and geometrical accuracy can be obtained using focused ion beam (FIB) processing, which has been widely used to fabricate diamond devices [1-3] because of their physical properties and precision requirements.

However, ion beam modification on the machining device's surface and subsurface is inevitable during FIB nano-machining [4]. Ion implantation results in the doping of Ga ions, which changes the nature of diamond surface layer and causes the formation of an amorphous layer [5]. For example, the micro cutting tools machined by FIB have amorphous layers at the rake and flank faces, leading to rapid increasing of edge radius in cutting process, which would deteriorate the quality of cutting surface and decrease the lifetime of the tools [6]. Therefore, developing a suitable means of removing this modified layer has become an important research topic.

Recently, several research groups have implemented different methods to address this FIB damage layer. Kupfer et al. thermally treated diamond devices at 773 - 1273 K in a flowing N_2 atmosphere after device fabrication [2]. Using an incident ion beam at low energy in sputtering processing could reduce SCD damage. However, diamond is prone to amorphization at high temperatures, while lowering the incident beam energy cannot completely remove the amorphous layer.

In this paper, the fs laser ablation thresholds was studied and exploited to effectively remove the FIB-induced amorphous layer without damaging the SCD substrate. Various microscopy techniques have been used to characterize the femtosecond laser treatment. The laser power density has been shown to be the most significant factor in the ablation process and the removal of the amorphous layer.

2. Experimental setups

A natural SCD sample was used here. A FEI Nova 200 NanoLab FIB/SEM Dualbeam system was used to perform both the ion implantation and in-situ SEM observation. Several rectangular trenches with areas of 10 × 10 μm^2 were milled by FIB on SCD with 30 kV energy and 1.0 × 10^{18} ions/cm^2 ion doses.

A Yb-doped large-mode-area photonic crystal fiber (PCF) femtosecond laser amplifier, which outputted a pulse with a center wavelength of 1040 nm, a repetition rate of 50 MHz and a pulse duration of 74 fs, was used to treat the FIB modified layer. The laser power was measured by a power detector located at the exit of the objective lens.

An AFM (Nanoscope IIIa) was used to probe the morphology of the fs laser irradiated area on diamond in tapping mode. EDX was performed to determine the relative gallium and carbon surface concentrations using a Bruker X-ray 5010 Silicon Drift Detector under a FEI Nanosem 430 SEM. A laser micro-Raman spectrometer (RENISHAW inVia Raman Microscope) was used to rapidly detect lattice defects with a wavelength of 633 nm for an integration time of 10 s.

3. Results and discussion

A circularly polarized laser with different power densities was used to remove the FIB-induced amorphous layer on SCD substrate, as shown in Figure 1. Results showed that the covering FIB implanted layer can be effectively removed by the fs laser treatments. Under the laser power densities between 4.06 × 10^{10} W/cm^2 and 6.60 × 10^{10} W/cm^2, the FIB implanted layer was removed at the center of the fs laser irradiated areas resulting in the appearance of fairly flat regions surrounded by debris, where the area of the flat regions increased with the irradiated laser power density. No nano-grooves appeared in the areas irradiated by the laser at power density below 6.60 × 10^{10} W/cm^2.

When the power density was increased above 1.02 × 10^{11} W/cm^2, much deeper swirl-shaped pits formed at the center area, which showed that the power density was larger than SCD ablation threshold, as shown in Figure 1(c).

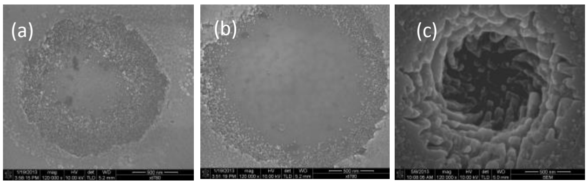

Figure 1. FIB implanted areas of SCD irradiated by a circularly polarized fs laser at power density of (a) 4.06 × 10^{10} W/cm^2, (b) 6.60 × 10^{10} W/cm^2 and (c) 1.52 × 10^{11} W/cm^2.

The morphology of the irradiated areas of Figure 1(b) was measured by AFM, as shown in Figure 2. The bottom of the pit was approximately planar with a Ra of 1.03 nm, and the pit was approximately 50 nm deep. The planar shape of the pit bottom showed that the amorphous layer had been ablated. EDX was used to detect the gallium and carbon concentrations before/after laser irradiation, as shown in Table 1. The gallium concentration dropped drastically to a very low value after fs laser processing, proving that the FIB modified layer was substantially removed by fs laser.

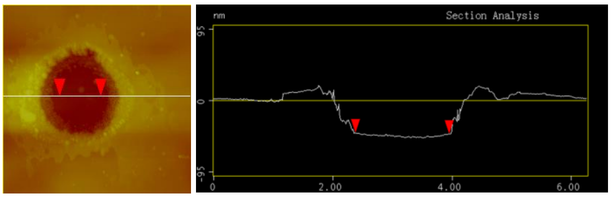

Figure 2. AFM pit profile for Figure 1(b). The material covering surrounding the pit results from the ablation of FIB modified layer during fs laser irradiation treatment. The unit of x-axis is μm.

Table 1. Gallium and carbon concentrations for an ion implanted region and a laser irradiated region

Concentration	ion implanted region		laser irradiated area	
Element	Wt %	At %	Wt %	At %
C	92.16	98.56	99.29	99.88
Ga	7.84	1.44	0.71	0.12

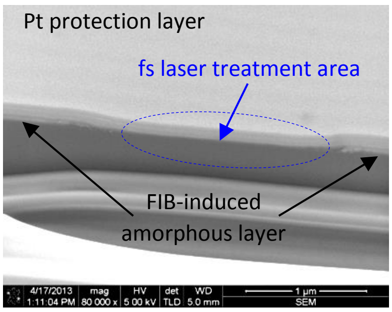

Figure 3. High resolution SEM morphology of the cross-section profile after fs laser treatment.

In order to characterize whether there was further new damage inducing by the fs laser treatment for the irradiated SCD area in Figure 1(b), a high resolution SEM image was captured using the FIB cross-section, as shown in Figure 3. Results showed that the fs laser treatment completely removed the FIB modified layer without any further modification for the subsurface underneath SCD area.

The irradiated area was further characterized by performing Raman measurements at three different points for the area in Figure 1(b): the untreated SCD region, the ion implanted region and the center of the fs laser irradiated area, as shown in Figure 4. The Raman spectrum for diamond consists of a single peak at 1332 cm^{-1}, whereas a peak is observed at 1580 cm^{-1} for graphite. The Raman results showed that adjusting the power density of the fs laser treatment can effective remove the FIB modified layer and prevent further SCD ablation.

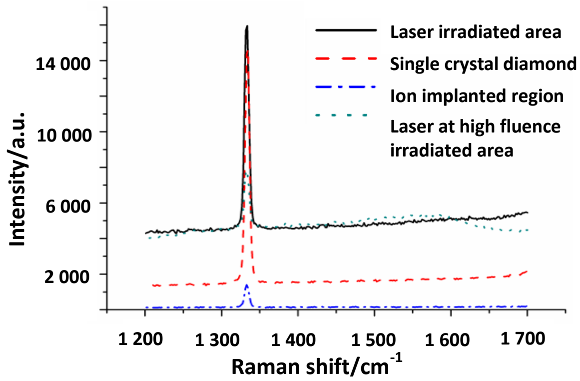

Figure 4. Raman spectrum for SCD, ion implanted area and laser irradiated areas.

Firstly, the implantation Ga ions in the modified layer are helpful for the fs laser energy absorption. More importantly, focusing a high intensity fs laser on a sample surface will produce multi-photon ionization with a large amount of free electrons within the laser spot [7]. The interactions between this large numbers of free electrons would generate a surface plasma (SP) wave, which would result in a periodic distribution of the deposed energy. Once the laser power density reached the SCD ablation threshold, a swirl-shaped pit hundreds nanometers deep would form as shown in Figure 1(c). The FIB modified layer can be effectively removed by choosing an fs laser power density which is higher than the ablation threshold of the amorphous layer and lower than that of SCD.

4. Conclusions

The FIB modified layer of single crystal diamond (SCD) can be effectively removed by choosing an femtosecond laser power density which is higher than the ablation threshold of the amorphous layer and lower than that of SCD. This study proves that fs laser process is feasible to optimize the SCD devices developed by FIB nanofabrication. In the future, how to increase the fs laser treatment efficiency should be focused on.

References

[1] Xu Z W, Fang F Z, Zhang S J, Zhang X D, Hu X T, Fu Y Q and Li L 2010 *Optics express* **18** 8025-8032
[2] Kupfer B Z, Ahmad R K, Zainal A, Jackman R B 2010 *Diamond and Related Materials* **19** 742-747
[3] Hamada M, Teraji T, Ito T 2003 *Applied surface science* **216** 65-71
[4] Xiao Y J, Fang F Z, Xu Z W and Hu X T 2015 *Applied Surface Science*, **343** 56-69
[5] Tong Z, Xu Z W, Wu W and Luo, X 2015 *Nuclear Instruments and Methods in Physics Research Section B* **358** 38-44
[6] Wu W, Xu Z W, Fang F Z 2014 *Nuclear Instruments and Methods in Physics Research B* **330** 91-98
[7] Sun H, Song J, Li C, Xu J and Wang X 2007 *Applied Physics A* **88** 285-288