中国激光

基于激光复合技术的金刚石抛光研究

赵智炎[1,2]，冯昱森[2]，罗子艺[1*]，蔡得涛[1]，薛亚飞[1]，王忠强[3]，于颜豪[2**]

[1]广东省科学院中乌焊接研究所，广东省现代焊接技术重点实验室，广东 广州 510650；
[2]吉林大学电子科学与工程学院集成光电子学国家重点实验室，吉林 长春 130012；
[3]北京大学东莞光电研究院，广东 东莞 523808

摘要 金刚石作为一种具有独特优异性能的半导体材料，在光学和电子学领域具有重要的应用价值。目前生长金刚石最常用的方法是化学气相沉积（CVD）法，采用该方法制备的金刚石薄膜通常为多晶结构，表面粗糙度高、颗粒大的缺点制约了金刚石薄膜的应用。笔者提出了飞秒-纳秒-离子束刻蚀的复合抛光方法并采用该方法对CVD金刚石薄膜进行抛光。结果表明：经飞秒-纳秒激光刻蚀后，金刚石表面粗糙度降低得十分明显，由未刻蚀时的4 μm降至0.5 μm左右，但表面出现了明显的石墨化现象；进一步采用离子束刻蚀去除表面的石墨层，最低可将表面粗糙度降至0.47 μm。所提方法实现了金刚石表面的无改性平滑抛光，为金刚石表面微纳器件的发展奠定了基础。

关键词 超快激光；金刚石薄膜；激光抛光；离子束刻蚀；粗糙度

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1 引 言

金刚石作为一种宽带隙半导体材料，具有许多优异的物理化学性质[1]：1）高达5.5 eV的超宽禁带宽度，远高于GaN、SiC等宽禁带半导体材料的禁带宽度；2）低的介电常数和摩擦因数；3）高的载流子迁移率、电子漂移速度和热导率[2-6]。这些独特的性质使得金刚石在光学和微电子学领域具有重要的应用价值[7-8]。化学气相沉积（CVD）法是制备金刚石薄膜的常见方法[9]。CVD金刚石薄膜通常为多晶膜，其表面呈现为杂乱无序的晶粒堆积，晶粒尺寸大小不一，致使薄膜表面相当粗糙[10]。金刚石薄膜较低的表面平整度限制了其在光学、电子学等领域的应用，因此必须对金刚石表面进行抛光，以达到应用要求[11]。金刚石薄膜抛光是对金刚石表面凸起的棱角进行大面积去除，目前常用的方法主要有机械抛光[12]、化学辅助机械抛光[13]、离子束抛光[14-15]、热化学抛光[16]、激光抛光[17-21]等。Shi等[22]分析了机械摩擦在金刚石抛光过程的重要作用，阐述了金刚石材料的磨损机理。由于金刚石的硬度较高，传统的机械抛光法的抛光速率较低，而且不能达到精密加工所需的粗糙度要求。Mi等[23]提出了一种用离子束刻蚀法对脆性衬底进行非接触式表面抛光的方法，该方法具有很高的效率，但离子束刻蚀成本较高，不适合工业化生产。Komlenok等[24]首次实现了飞秒和纳秒激光对粗糙度为5 μm的金刚石板材的激光抛光。激光加工技术属于非接触式加工，能够对曲面进行处理[25]，不仅加工效率高，还能够实现对各种硬质材料的高质量加工[26]。因此，激光抛光可以用于对金刚石薄膜的抛光。利用激光的高能量烧蚀金刚石颗粒的棱角，可以降低表面粗糙度，使表面平整化[27-29]，但通常会在薄膜表面诱导出表面微纳结构并引入石墨层[30-31]。

由此可见，虽然金刚石薄膜的抛光方法众多，但都存在一定的局限性，难以满足越来越高的应用需求。为了解决以上问题，笔者提出了激光抛光结合离子束刻蚀的复合抛光方法：先采用飞秒激光对金刚石薄膜进行粗抛，将其表面粗糙度从4 μm降低至2 μm左右，然后再用纳秒激光进一步将表面粗糙度降低至0.5 μm左右；最后用离子束刻蚀掉沉积在金刚石表面的石墨层（纳秒激光抛光时的热效应会使薄膜表面产生石墨化现象），从而在降低粗糙度的同时获得无改性层的金刚石表面。笔者分析了不同抛光方式对粗糙度的影响，进一步优化了抛光工艺参数。本研究结果为金刚石微加工及相关微器件的制备提供了技术支撑。

2 实验装置及方法

本文提出的复合抛光方法的示意图如图1所示。首先采用飞秒激光对CVD金刚石薄膜进行预抛光，改

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通信作者： *luozy@gwi.gd.cn；**yanhao_yu@jlu.edu.cn

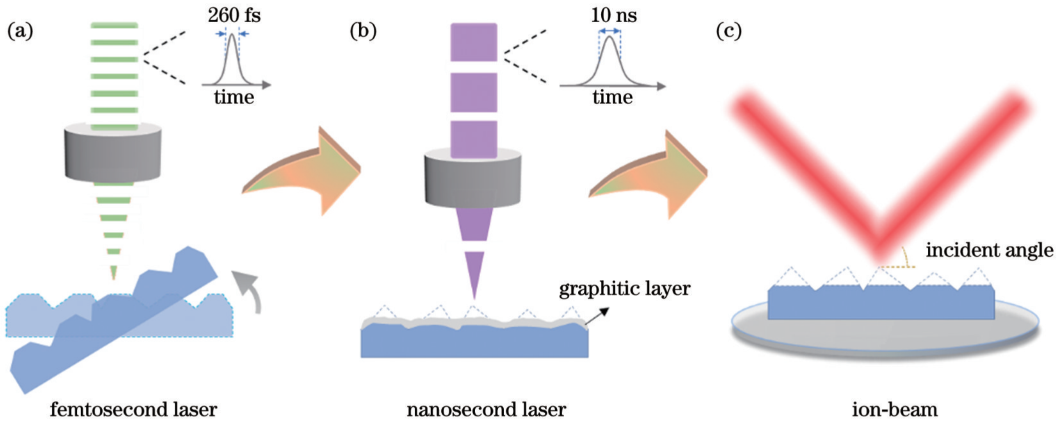

图 1 复合抛光加工示意图。(a)飞秒激光结合变角度加工;(b)纳秒激光加工;(c)离子束抛光

Fig. 1 Schematic diagram of composite polishing. (a) Femtosecond laser combined with variable angle machining; (b) nanosecond laser processing; (c) ion-beam polishing

变激光入射角度,通过控制飞秒激光输出功率及曝光时间,在金刚石表面进行粗抛光;然后控制纳秒激光的功率,在飞秒激光预抛光的基础上进行精细抛光;激光加工完成后,再进行离子束刻蚀,将CVD金刚石薄膜的表面粗糙度降低到0.5 μm左右。实验中采用的飞秒激光器(LIGHT CONVERSION)的波长为515 nm,脉宽为260 fs,重复频率为100 kHz;采用的纳秒激光器(武汉华日精密激光股份有限公司)的波长为355 nm,脉宽为10 ns,扫描速度为100 mm/s。Fischione生产的Model 1060 SEM Mill通过电离氩产生两束聚焦在样品表面的离子束。在离子束刻蚀过程中,样品架匀速旋转,以保证刻蚀的均匀性。使用奥林巴斯生产的3D测量激光显微镜OLS4100对金刚石薄膜的三维形貌和粗糙度进行表征;使用日本电子株式会社(JEOL)生产的JSM-7500F冷场发射扫描电子显微镜对金刚石薄膜的微观形貌进行观察;采用RENISHAW INVIA拉曼光谱仪采集不同样品的拉曼散射光谱(使用波长为532 nm的激发光)。

3 实验结果与讨论

3.1 飞秒激光对金刚石粗糙度的影响

激光能量与曝光时间对金刚石表面形貌有重要影响,采用控制变量法来探究激光功率对金刚石形貌的影响。选取飞秒激光器的输出功率分别为900、1000、1100、1200、1300、1400、1500 mW,选取激光曝光时间分别为1000、2500、5000、10000、25000 μs。图2为不同功率、不同曝光时间下部分飞秒激光加工样品的SEM形貌图。从图2中可以看出,输出功率和曝光时间与金刚石表面形貌、峰谷深度有很大关系。当曝光时间为1000 μs时,不同功率激光对金刚石薄膜表面的加工效果不明显。这是因为单个脉冲的作用时间太短,光子与金刚石晶格的作用时间不够长,导致对金刚石表面凸起棱角的烧蚀不够充分,因而烧蚀后的薄膜表面无明显变化。当曝光时间为2500 μs、功率为1300 mW时,金刚石薄膜表面出现了比较明显的加工效果,较大的金刚石颗粒顶部被削平,表面的起伏程度降低。当功率达到1400 mW时,虽然表面起伏程度降低,但粗糙度较大,样品表面有烧蚀之后的碎屑残渣。随着曝光时间延长,金刚石薄膜表面的起伏减小,但是表面颗粒堆积为块状,表面质量变差。

图3展示了不同功率和曝光时间下金刚石薄膜表面粗糙度的变化。如图3(a)所示,在同一输出功率下,随着曝光时间延长,表面粗糙度呈现先下降后上升最后趋于稳定的趋势。金刚石表面的原始粗糙度为4 μm左右,当曝光时间为1000 μs时,粗糙度比原始粗糙度有一定程度的降低;当曝光时间为5000 μs时,粗糙度比2500 μs时有所增加。这可能是因为能量较小时,金刚石表面的烧蚀不够充分,而能量较大时虽然波峰形貌被明显调控,但激光对波谷的加工效果同样明显,导致粗糙度反而比曝光时间为2500 μs时增大。曝光时间为2500 μs时,粗糙度下降得特别明显,在适当的功率下,粗糙度能够降到2 μm左右。从图3(b)可以看出,当曝光时间为1000、2500、5000 μs时,随着输出功率由小到大变化,粗糙度整体呈现出先逐渐下降然后趋于稳定的趋势。曝光时间为10000 μs和25000 μs时,随着功率增大,表面粗糙度也呈现出先下降后上升的趋势,这可能是因为激光作用处积累了过多的光子能量,反而对材料表面造成了过度损伤。当曝光时间为2500 μs且输出功率为1300 mW时,飞秒激光加工金刚石表面粗糙度达到最小值,为1.97 μm。

改变飞秒激光入射角对金刚石薄膜进行烧蚀实验,粗糙度随入射角度的变化如图4(a)所示。入射角分别选取为0°、5°、10°、15°、20°、30°,在上述入射角下加工了相同的三组样品(sample 1、sample 2和sample 3)。三组样品的粗糙度呈现出相同的变化趋势,即:随着入射角度增大,粗糙度先减小后增大;当入射角为15°时,粗糙度达到最小值。当入射光线沿法线方向时,激光聚焦于样品表面,落在波峰顶部的光斑能量密度较高,

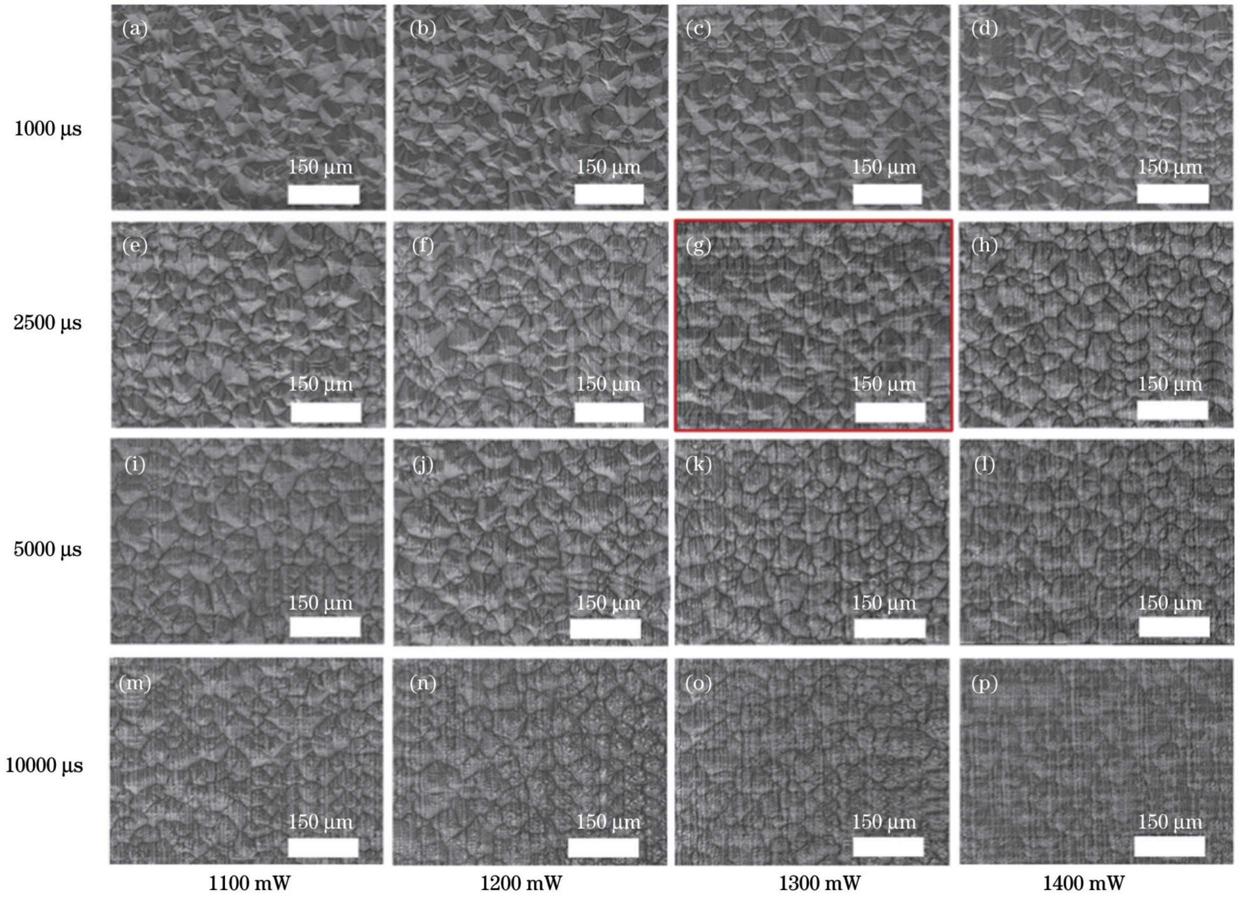

图 2　不同功率和曝光时间下飞秒激光加工金刚石表面的SEM形貌

Fig. 2　SEM morphologies of diamond surface processed by femtosecond laser under different powers and exposure time

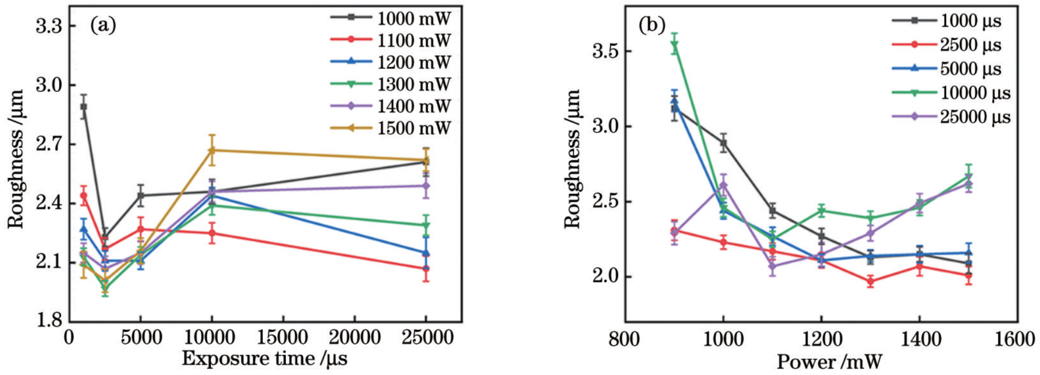

图 3　不同功率和曝光时间下飞秒激光加工金刚石表面粗糙度的变化趋势。(a)表面粗糙度随曝光时间的变化；(b)表面粗糙度随功率的变化

Fig. 3　Variation trend of surface roughness of femtosecond laser processed diamond under different powers and exposure time. (a) Variation of surface roughness with exposure time; (b) variation of surface roughness with power

峰尖首先被烧蚀削平，波谷处同样受到激光烧蚀作用的影响，凹陷区域亦被加深，可能加剧了峰谷的起伏，最终导致粗糙度较大。随着入射角增大，激光能量更多地落在了薄膜表面的凸起部分，波峰的削平效果更加明显，而波谷获得的能量较少，激光烧蚀的影响较小，去除量较小，因此粗糙度明显下降。由于采用的飞秒激光功率较小，烧蚀作用有限，当入射角过大时，金刚石颗粒凸起不能被完全烧蚀，粗糙度增大。

3.2　纳秒激光与离子束对金刚石粗糙度的影响

采用低功率的飞秒激光对金刚石薄膜进行粗抛光后，为了达到精密抛光的要求，在飞秒激光加工的基础上再用纳秒激光进行处理。图 4(b)是用不同功率的纳秒激光加工后，薄膜表面粗糙度的变化曲线，fs-ns 表示先对样品进行飞秒激光加工而后再进行纳秒激光加工。飞秒激光加工(fs)采用的曝光时间为 2500 μs，输出功率为 1300 mW，入射角为 15°。进行飞秒-纳秒

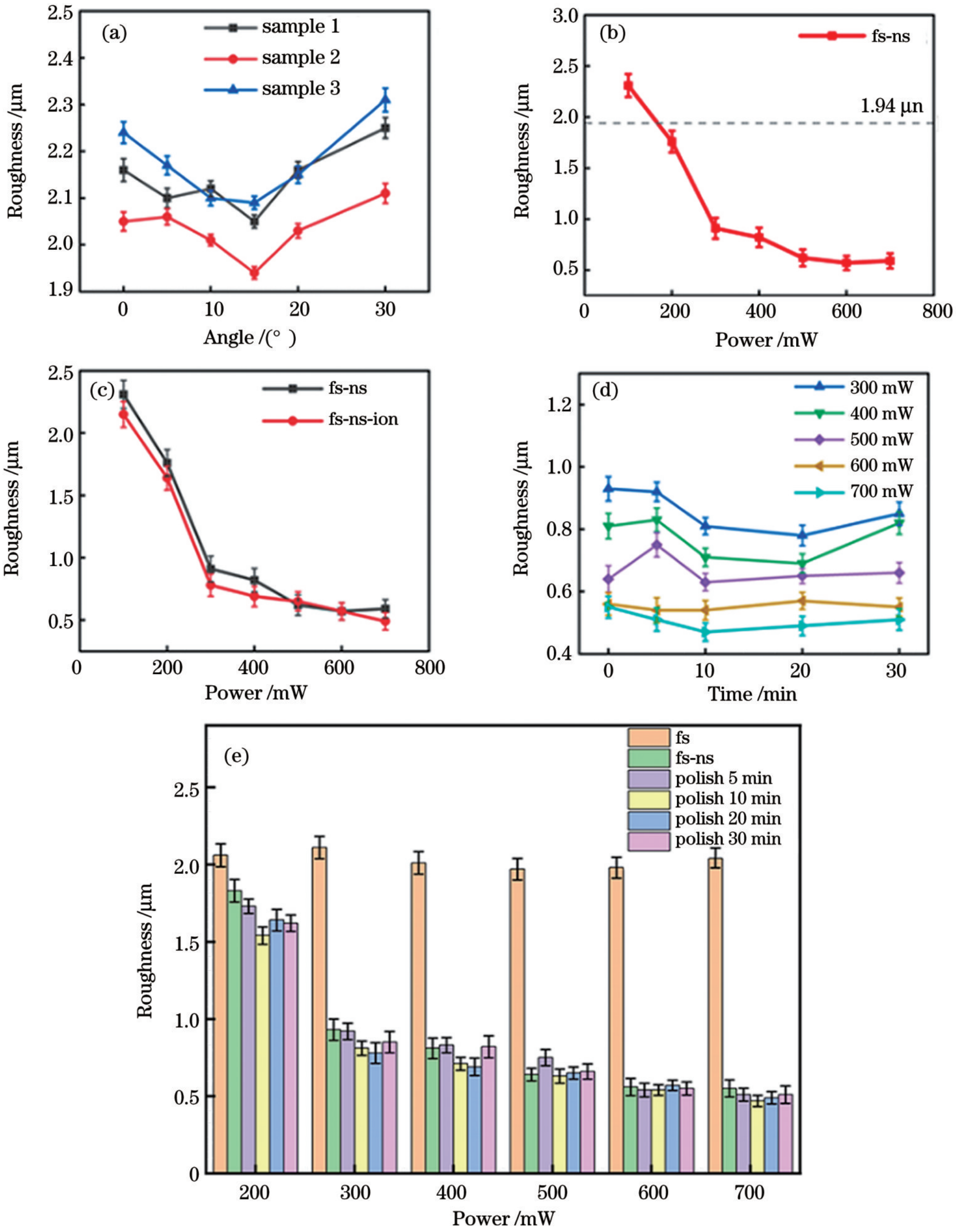

图 4　不同加工方式下粗糙度的变化趋势。(a)粗糙度随飞秒激光入射角的变化;(b)粗糙度随纳秒激光功率的变化;(c)离子束抛光前后的粗糙度;(d)粗糙度随离子束抛光时间的变化;(e)不同加工表面的粗糙度

Fig. 4　Variation trend of roughness under different machining methods. (a) Variation of roughness with incident angle of femtosecond laser; (b) variation of roughness with nanosecond laser power; (c) roughness before and after ion-beam polishing; (d) variation of roughness with ion-beam polishing time; (e) roughness of different processed surfaces

激光加工(fs-ns)时,纳秒激光加工的曝光时间为 100 μs,功率分别选取 200、300、400、500、600、700 mW。纳秒激光加工后,粗糙度变化明显,并且随着纳秒激光输出功率增加,表面粗糙度不断降低,最后趋于稳定。当纳秒激光功率为 700 mW 时,表面粗糙度接近 0.5 μm。纳秒激光的脉冲持续时间相对于飞秒激光更长,沉积的能量增加,进而传递给金刚石晶格的能量增加,能量在晶格声子中的传播距离延长;在能量传播过程中,高于石墨化温度而未达到石墨升华温度的加工区域变大,样品表面将会出现明显的石墨层。在激光抛光过程中,石墨层需要去除,同时考虑到需要进一步降低粗糙度,笔者对激光加工后的表面进行了氩离子束刻蚀抛光。不同刻蚀角度下的抛光效率不同。用两束氩离子束以 10°角度入射,刻蚀过程中样品匀速旋转,以保证刻蚀的均匀性。图 4(c)中的“fs-ns-ion”表示先对样品进行飞秒-纳秒激光加工而后再进行离子束抛光。可以看出:抛光之后的粗糙度略低于飞秒-纳秒激光加工的粗糙度,约降低了 0.1 μm。这说明氩离子束刻蚀抛光对于降低薄膜粗糙度有一定的效果。笔者统计了不同抛光时间和不同纳秒激光器功率(分别为 300、400、500、600、700 mW)下金刚石薄膜的表面粗糙度,如图 4(d)所示。当抛光时间在 10 min 以内

时，粗糙度整体上随着抛光时间延长而减小；当抛光时间超过10 min后，粗糙度整体呈逐渐上升的趋势。

从图4(e)中可以更直观地看出不同加工过程中粗糙度的变化趋势。在6组不同的功率下，比较了飞秒激光加工(fs)、飞秒-纳秒激光加工(fs-ns)和飞秒-纳秒-离子束抛光不同时间后的粗糙度变化趋势。可以看出：飞秒-纳秒激光加工后，粗糙度下降得十分明显；离子束抛光后，粗糙度呈现出略微下降的趋势。当功率为200、500、600、700 mW时，飞秒-纳秒-离子束抛光10 min后的粗糙度最小，说明通过控制离子束抛光时间来降低粗糙度是有效的。当纳秒激光加工功率为700 mW、离子束抛光时间为10 min时，薄膜表面粗糙度达到最小值0.47 μm。不同加工过程中金刚石薄膜表面的电镜形貌和共聚焦显微镜形貌如图5所示。可

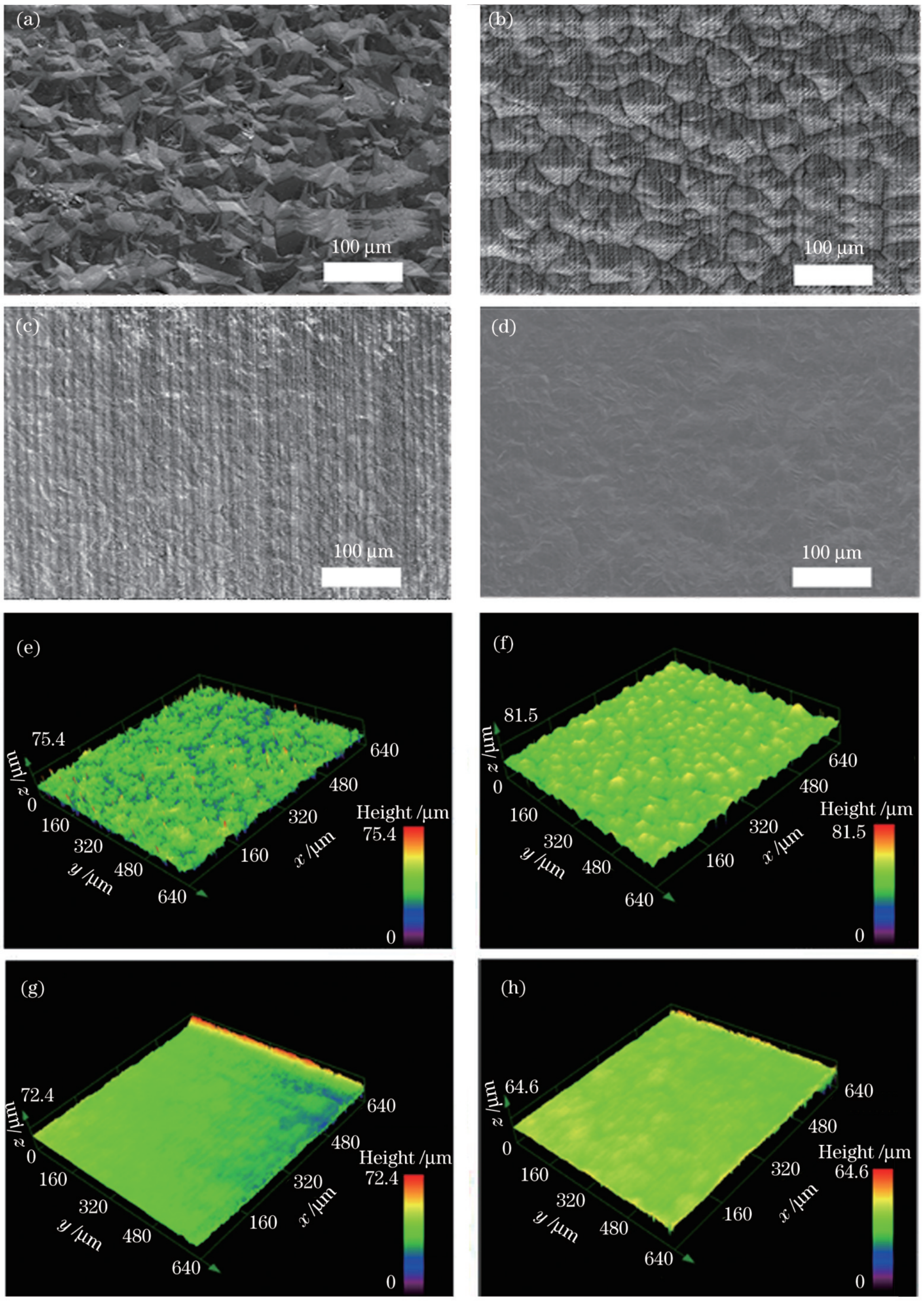

图5 不同加工方式下金刚石表面的电镜形貌和三维景深形貌。(a)(e)金刚石原始表面；(b)(f)飞秒激光加工表面；(c)(g)飞秒-纳秒激光加工表面；(d)(h)飞秒-纳秒-离子束抛光表面

Fig. 5 Scanning electron microscopy and three-dimensional depth-of-field morphologies of diamond surface under different processing methods. (a)(e) Original diamond surface; (b)(f) femtosecond laser processed surface; (c)(g) femtosecond+nanosecond laser processed surface; (d)(h) femtosecond+nanosecond+ion-beam polished surface

以看出，经过飞秒激光预抛光、纳秒激光抛光、离子束去石墨化处理后，薄膜表面烧蚀区域逐渐趋于平坦，薄膜表面凸起基本被削平，表面形貌明显改善。

3.3 复合抛光对金刚石成分的影响

使用纳秒脉冲激光对金刚石薄膜进行烧蚀时，总会在薄膜表面形成石墨层，故而采用离子束抛光进行去石墨化处理。图 6 展示了烧蚀前后金刚石表面的拉曼光谱。刻蚀前，金刚石薄膜的拉曼光谱在 1332 cm^{-1}附近出现了明显的金刚石相特征峰。飞秒激光加工后，金刚石钻石峰强度略有下降，由于材料内部存在一定的拉应力，石墨的特征峰偏移至 1570 cm^{-1}附近，说明激光扫描过程中出现了石墨化。飞秒-纳秒激光加工金刚石表面的拉曼光谱出现了十分明显的石墨化现象。这是由于纳秒激光具有很强的热效应，材料表面积累了大量的能量，金刚石晶格结构缺陷吸收激光辐射能量后引发材料改性，导致金刚石转化为石墨。同时，由于激光的烧蚀作用，石墨氧化和升华产生气相的碳材料，气相碳沉积后使石墨层更加明显，因此在 1570 cm^{-1}处出现了典型的石墨带。飞秒-纳秒-离子束抛光 10 min 后，石墨带的拉曼峰被去除，金刚石相含量增加，金刚石的特征峰变得更加尖锐。石墨化成分受到离子轰击容易被去除，进行适当时间的氩离子束抛光处理不仅能减小表面粗糙度，更有助于去除金刚石表面的石墨层。

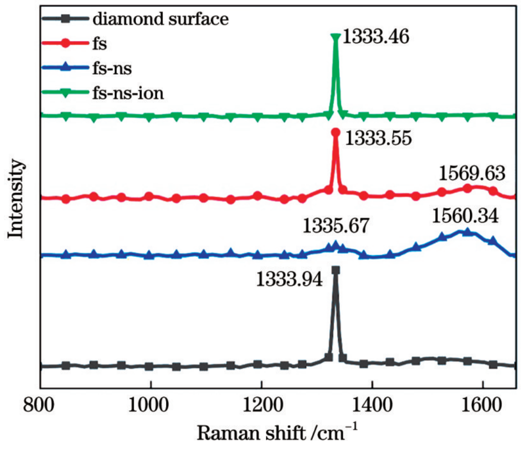

图 6 不同加工表面的拉曼光谱

Fig. 6 Raman spectra of different processed surfaces

4 结 论

笔者探究了飞秒-纳秒-离子束抛光对 CVD 金刚石薄膜粗糙度的影响，通过选择合适的激光加工与离子束抛光参数可以获得理想的表面粗糙度。采用激光抛光与离子束刻蚀相结合的复合抛光技术能够有效地对 CVD 金刚石薄膜进行抛光。通过控制飞秒激光的输出功率和曝光时间以及改变激光入射角度在金刚石表面进行粗抛光，能够在降低粗糙度的同时减少石墨层的形成。纳秒激光加工能够在飞秒激光预处理的基础上进行精细加工，但热效应会导致加工过程中形成石墨层。采用离子束刻蚀能够有效去除石墨层，实现无改性层的高质量抛光。与抛光前 4 μm 左右的粗糙度相比，飞秒-纳秒-离子束抛光金刚石薄膜的表面粗糙度明显降低，最低可以达到 0.47 μm。该方法基本上实现了对金刚石表面的抛光，为在金刚石表面进行微加工和制造微光学元件提供了参考。

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Diamond Polishing Based on Laser Composite Technology

Zhao Zhiyan[1,2], Feng Yusen[2], Luo Ziyi[1*], Cai Detao[1], Xue Yafei[1], Wang Zhongqiang[3], Yu Yanhao[2**]

[1]*China-Ukraine Institute of Welding, Guangdong Academy of Sciences, Guangdong Provincial Key Laboratory of Advanced Welding Technology, Guangzhou* 510650, *Guangdong, China*;
[2]*State Key Laboratory of Integrated Optoelectronics, College of Electronic Science and Engineering, Jilin University, Changchun* 130012, *Jilin, China*;
[3]*Dongguan Institute of Opto-Electronics Peking University, Dongguan* 523808, *Guangdong, China*

Abstract

Objective Diamond is a wide-bandgap semiconductor material with several excellent physical and chemical properties. It has an ultrawide bandgap of 5.5 eV, which is significantly higher than those of GaN, SiC, and other wide-bandgap semiconductor materials. In addition, it has a low dielectric constant, low friction coefficient, high carrier mobility, high electron drift speed, and high thermal conductivity. These unique properties make diamonds have an important application value in optics and microelectronics. Because of the high hardness of diamonds, the traditional mechanical polishing method, which yields low polishing speeds and has high costs, cannot achieve an ideal effect. Ion-beam etching is a highly efficient noncontact surface-polishing method for super-hard and brittle substrates. However, it is unsuitable for industrial production because of its high cost. Laser processing is a noncontact processing technique that can handle curved surfaces. It has high processing efficiency and can achieve high-quality processing of various hard materials. Therefore, laser polishing can be used to polish the diamond film using the high energy of the laser to ablate the edges of the diamond particles. It can reduce the surface roughness and flatten the film, but typically induces a surface microstructure or nanostructure on the film surface and introduces a graphite layer. Although several polishing methods for diamond films have been developed, they have limitations, and it is difficult to satisfy the increasing application requirements. To solve these problems, we propose a composite polishing method that uses laser polishing combined with ion-beam etching. By further optimizing the polishing process parameters, a diamond surface without a modified layer is obtained, and the roughness is reduced. The results of this study provide technical support for diamond micromachining and related microdevice preparation.

Methods The research object of this study is a diamond film prepared via chemical vapor deposition (CVD). The CVD diamond film was first prepolished using a femtosecond laser. The incidence angle of the laser was varied, and the diamond surface was initially polished by controlling the femtosecond laser output power and exposure time. The three-dimensional (3D) surface morphology and roughness of the diamond films were characterized and analyzed using 3D laser microscopy. Next, the power parameters of the nanosecond laser were controlled, and fine polishing was performed. The effect of nanosecond laser machining on the surface roughness of the films was assessed. Subsequently, the effect of the ion-beam etching time on the roughness of the CVD diamond was analyzed. The morphology of the polished diamond films was observed using cold-field emission scanning electron microscopy. The Raman scattering spectra of the samples were measured using Raman spectrometry to analyze the changes in the graphite layers during different polishing processes.

Results and Discussions After femtosecond+nanosecond machining and ion-beam etching, the roughness of diamond surface decreases significantly, from 4 μm without etching to 0.47 μm after etching. In addition, the graphite layer formed by the thermal effect during laser processing can be effectively removed, and the diamond surface can be polished without modification and with high smoothness.

1. Using the femtosecond+nanosecond polishing method to polish the surface of diamond film can effectively reduce the surface roughness and produce a smooth surface.

2. Laser-polished diamond is typically converted into graphite because of the thermal effect that accumulates on the diamond film surface, which ablates the film surface and forms a graphite layer on the surface. By bombarding the laser-polished surface structure with an ion-beam, graphitization can be effectively eliminated, and an unmodified layer can be formed.

3. The use of field mirrors to polish diamond films can result in efficient large-area processing. With shortened scanning and ion-beam etching time, rapid preparation can be achieved, creating conditions for the industrial application of diamond films.

Conclusions In this study, the effect of femtosecond+nanosecond+ion-beam polishing on the roughness of CVD diamond films was investigated. Ideal surface roughness can be achieved by selecting suitable laser processing and ion-beam polishing parameters. The composite polishing technology of laser polishing and ion-beam etching can effectively polish CVD diamond films. By controlling the femtosecond laser output power and exposure time and varying the laser incident angle, rough polishing of the diamond surface can reduce the roughness and formation of the graphite layer. Nanosecond processing can be fine-processed after applying femtosecond rough processing, however, owing to the thermal effect, a graphite layer is formed during processing. Finally, the graphite layer is effectively removed via ion-beam etching. High-quality polishing is achieved without modifying the layers. Compared with the roughness of approximately 4 μm before polishing, the surface roughness of the composite polished diamond film decreases significantly, with the minimum value reaching 0.47 μm. The proposed method polishes diamond surfaces and provides support for the micromachining and fabrication of micro-optical components on diamond surfaces.

Key words ultrafast lasers; diamond film; laser polishing; ion-beam etching; roughness