Fabrication of natural diamond microlenses by plasma etching

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Advantageous properties including optical transparency, high thermal conductivity, and high carrier mobility make natural diamond an attractive choice for a range of optical and electrical devices. However, its hardness and chemical inertness provide a significant challenge for device processing. We demonstrate the ability to etch natural type IIa diamond using inductively coupled plasma etching with a significant etch rate of 228 nm/min. The etched surfaces were characterized by atomic force microscopy and found to have a root-mean-square roughness of below 3 nm. Using the photoresist reflow technique, refractive microlens arrays, with diameters ranging from 10 to 100 μm, were fabricated on the same diamond substrates. The lenses were characterized by confocal microscopy, which showed that their focal lengths, ranging from 5 to 500 μm, were in excellent agreement with the predicted values, demonstrating the high fidelity of the fabrication process.
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Wide band gap inorganic materials, including GaN, SiC, and ZnO, have attracted considerable attention in recent years, due to their attractive optical and electronic properties. However, it is anticipated that these materials will be superseded for many applications by diamond. The properties of diamond are unique: it is the hardest material known, and it has a very wide optical transparency window (from ~220 nm to ~12 μm) allied with a high thermal conductivity (2000 $Wm^{-1} K^{-1}$), all attractive features for optical and optoelectronic structures. Additionally, it is anticipated that diamond-based electronic devices will deliver outstanding performance due to the material's high carrier mobility and high breakdown field.

The hardness and chemical inertness of diamond, however, means that the controlled processing of diamond presents a considerable technical challenge. Patterning of diamond by focused-ion beam milling,[1] excimer laser ablation,[2] reactive ion etching,[3] electron-cyclotron resonance etching,[4] and inductively coupled plasma (ICP) etching[5] have been reported previously, however using synthetic diamond. Although synthetic diamonds, typically grown using high-pressure high temperature technology and more recently by chemical vapor deposition (CVD), are less costly than natural diamonds, they have until very recently—as reflected in the aforementioned work—been predominantly polycrystalline in nature, and the presence of grain boundaries impedes both the optical and electrical performance of the material.

In this article, we report on the patterning of microoptics on natural diamond substrates by ICP etching. Despite the higher cost of natural diamond compared to CVD diamond, its usage is justified by the extremely high performance it offers, with specific applications in various niche markets such as space environments. We note, furthermore, that our results should be directly transferable to the new generation of single-crystal CVD diamond material.[6]

The experiments were conducted on commercial 5 mm ×5 mm natural-type IIa diamond with a platelet thickness of 250 μm. Spherical microlenses were fabricated by the photoresist-reflow method.[7] A 7 μm layer of Shipley SPR220 photoresist was spin coated onto the substrates and cylindrical pillars were patterned onto the photoresist by standard photolithography, as shown in Fig. 1(a). The diameters of the pillars range from 10 to 100 μm. The substrates were then placed on a hot plate (115 °C) for approximately 15 min to melt the photoresist, forming almost perfect spherical caps due to surface tension,[7] as illustrated in Fig. 1(b). The pattern was subsequently transferred onto the diamond substrate by ICP etching in a Surface Technology Systems multiplex etching system. The substrates were etched with a 10 sccm Ar/30 sccm O_2 plasma at 5 mTorr with the chuck cooled to 0 °C to avoid resist burning. The ICP power was varied be-

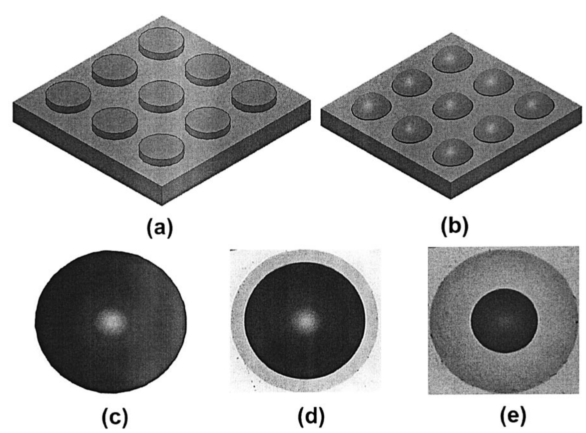

FIG. 1. Schematic diagram of the patterned photoresist pillars (a) before and (b) after resist reflow. Microphotographs showing evolution of the etch process, (c) before etching, (d) after 20 min, and (d) after 40 min.

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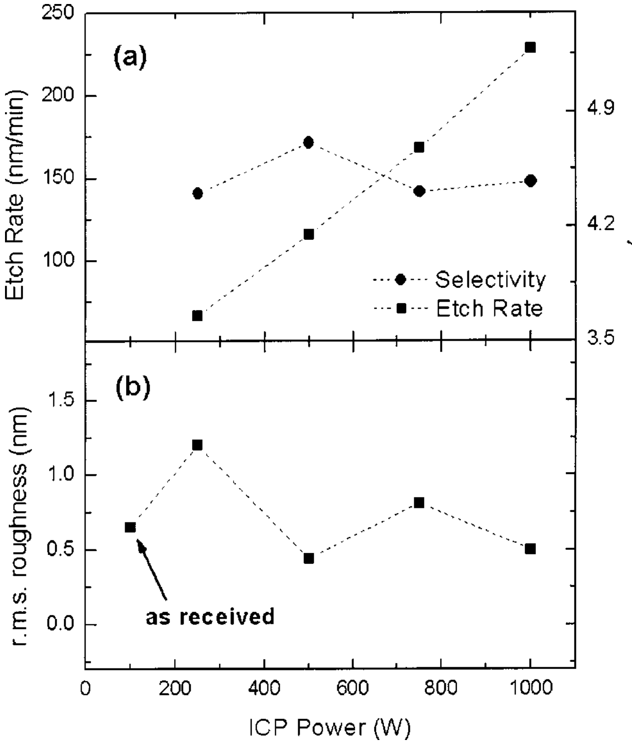

FIG. 2. (a) Etch rate and selectivity of diamond as a function of incident ICP power, and (b) corresponding rms surface roughness.

tween 250 and 1000 W. The resulting profiles of the lenses were evaluated by atomic force microscopy (AFM), while their focal lengths were measured by confocal microscopy.[8]

The etch rates and etch selectivites of diamond etched in this manner, as a function of ICP power, are plotted in Fig. 2(a). The etch selectivity is defined as the ratio of the photoresist etch rate to the diamond etch rate, and is determined by etching photoresist-patterned mesa structures alongside the microlens samples. The mesa heights before and after etching were evaluated using a stylus profile. The etch rate increased linearly with ICP power, reaching a maximum of 228 nm/min, which is comparable to the best reported results using CVD diamond.[4,9] The ICP etch process proceeds by a combination of ion bombardments by Ar ions and the formation of volatile compounds between carbon and oxygen radicals. As a result, the application of a larger ICP power increases the ion densities and provides a greater supply of heavy ions and oxygen radicals. On the other hand, the etch selectivity [Fig. 2(a)] remains in the range of 4.4–4.8 over

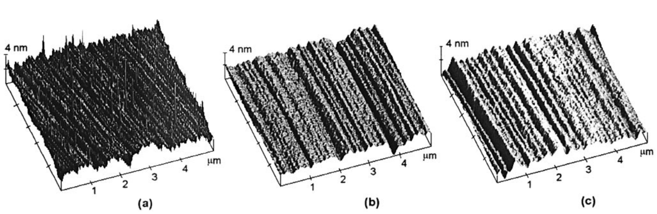

FIG. 3. AFM 3D images of the (a) as-received diamond surface, and diamond surfaces etched for 5 min at an ICP power of (b) 500 W and (c) 1000 W.

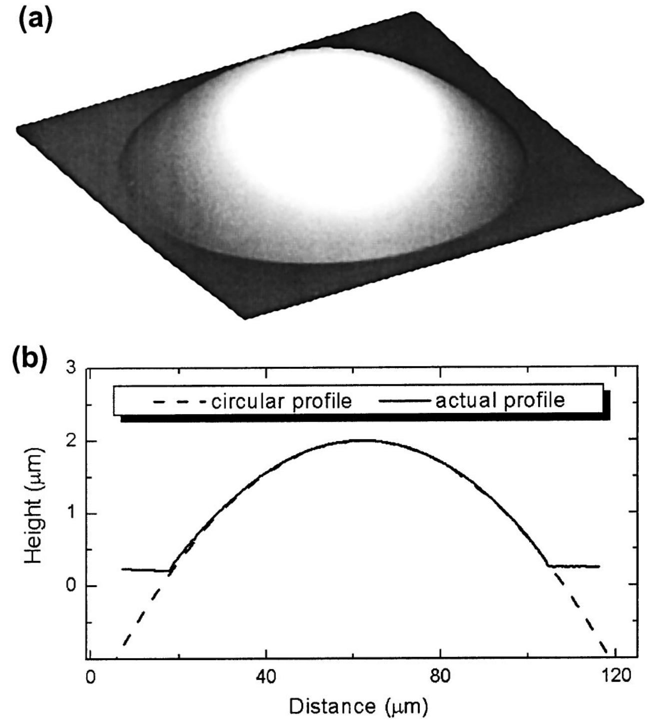

FIG. 4. (a) 5×5 μm AFM 3D image of the microlens with a diameter of 90 µm. (b) Curve fitting of the cross-sectional profile of the lens against the theoretical curvature of a circle.

the range of ICP powers used. The substrates were supplied polished to a root-mean-square (rms) roughness of less than 3 nm. Figure 3 shows the AFM images of the as-received surface, together with planar surface images of diamond samples etched in the Ar/O_2 plasma at ICP powers of 500 and 1000 W, respectively, for 5 min. The nanoscale stripes are a feature of the diamond related to its (110) crystalline orientation. The rms roughnesses of all the etched samples, extracted from the AFM data, are plotted in Fig. 2(b). It can

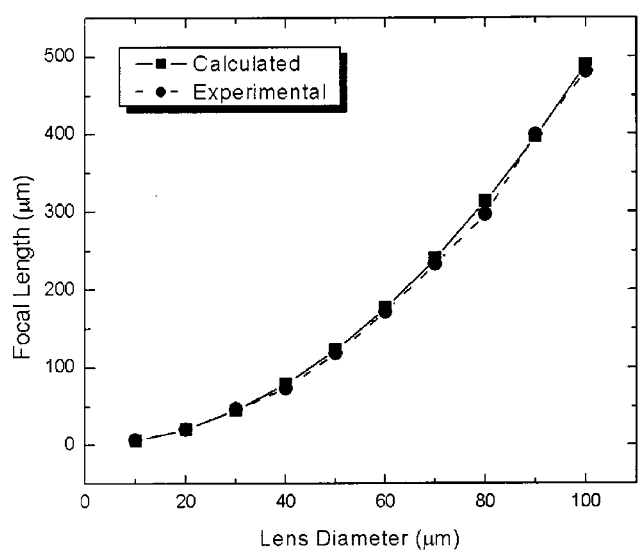

FIG. 5. Plot of calculated and experimental focal lengths vs lens diameter.

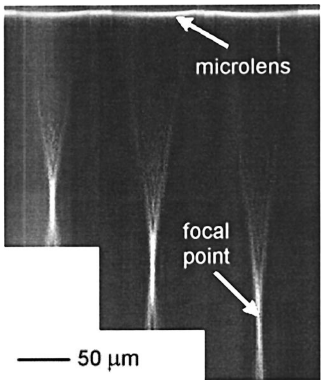

FIG. 6. Cross-sectional confocal images, illustrating the focusing capabilities of the microlenses with diameters of 50, 60, and 70 μm with 1 μm dome height.

be seen that high-density plasma processing of the substrates did not alter its surface structure nor roughness, confirming the suitably of this process for the fabrication of optical elements, where high quality surfaces are desired.

The microlens arrays were fabricated onto the diamond substrates using an ICP power of 500 W. Since the etch selectivity was unchanged under varying ICP power, the choice of parameters is a compromise between the etch rate and the amount of heat generated. Excessive heating of the substrate surface, caused by high-density ion bombardments, could modify the photoresist profile and hardness, and affect the final shape of the fabricated lenses. The etch pattern consisted of microlens arrays with individual lens diameters ranging from 10 to 100 μm, in diameter steps of 5 μm. An AFM three-dimensional (3D) scan of a representative fabricated microlens with a diameter of 90 μm is illustrated in Fig. 4(a). Since the etch selectivity between diamond and photoresist is nonunity, a spherical photoresist pattern will not translate into an identical profile on the diamond. Evaluation of the profiles of the lenses was undertaken by examining multiple cross-sectional profiles extracted from the 3D AFM images. These experimental profiles were then compared to the theoretical curvature of a circle using a chi-squared fitting algorithm, the result of which is shown in Fig. 4(b). Application of this procedure reveals that the maximum deviation from a perfect spherical surface was less than 10 nm over 80% of the lens area for lenses over the full range (10–100 μm) of diameters. These results were confirmed using a Fizeau optical interferometer (Zygo Corp.) to examine the optical performance of the lenses. On lenses down to 50 μm in diameter (the smallest that could be measured) the rms deviation from a sphere was found to be around 15 nm for the entire lens surface. These results indicate that the lenses produced are very close to spherical in shape.

As the height of the lenses, in this case, are approximately equal (since the same photoresist was employed across the lens array), the variation in focal length is determined by their base diameters. The calculated focal lengths, using the procedure outlined in Ref. 10, range from ~5 to 500 μm, and are plotted in Fig. 5. The actual focal lengths were determined using the confocal microscopy technique.[8] Cross-sectional images taken in this manner, illustrating the different focusing abilities of the 50-, 60-, and 70-μm-diam microlenses, respectively, are shown in Fig. 6. The focal lengths of the lenses were determined directly from the images, and are plotted alongside the calculated data in Fig. 5. It can be seen that the experimental data are very close to their predicted values, confirming the high fidelity of the fabrication process.

In summary, high-rate ICP etching of natural-type IIa diamond substrates has been demonstrated. The etched surfaces maintain high optical quality with rms roughness of better than 3 nm. Micro-optical elements have been fabricated onto the diamond substrates using the photoresist-reflow technique and ICP etching. The microlenses with diameters ranging from 10 to 100 μm, all have near-perfect spherical profiles and the focal lengths, measured using confocal microscopy, range from 5 to 500 μm, in very close agreement to their predicted values. We anticipate these lenses to have an important role in areas including high-resolution photolithography and *in vitro* imaging. We note, furthermore, that very similar results should be achievable under the same conditions using the new generation of single-crystal CVD diamond.

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