ICP polishing of silicon for high-quality optical resonators on a chip

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Abstract

Miniature concave hollows, made by wet etching silicon through a circular mask, can be used as mirror substrates for building optical micro-cavities on a chip. In this paper, we investigate how inductively coupled plasma (ICP) polishing improves both shape and roughness of the mirror substrates. We characterize the evolution of the surfaces during the ICP polishing using white-light optical profilometry and atomic force microscopy. A surface roughness of 1 nm is reached, which reduces to 0.5 nm after coating with a high reflectivity dielectric. With such smooth mirrors, the optical cavity finesse is now limited by the shape of the underlying mirror.

(Some figures may appear in colour only in the online journal)

1. Introduction

Microfabricated atom chips [1] that manipulate ultracold atoms are becoming increasingly important in a variety of applications, such as clocks [2], Bose–Einstein condensates [3, 4], matter wave interferometers [5, 6] and quantum metrology [7]. These chips miniaturize cold atom experiments into small packages, integrating functions such as trapping [8–10], guiding [11, 12] and detecting cold atoms [13]. There is great interest now in integrating Fabry–Perot optical resonators into an atom chip. Low finesse cavities have been used to detect atoms with high fidelity and fast response time [14, 15], while higher finesse gives stronger atom–photon coupling for dispersive atom detection [16] and other applications in quantum information processing [17]. The desire for high-quality optics integrated on chips has created the need for new miniature optical components and for fabrication techniques to shape their surfaces accurately and make them smooth.

One miniature Fabry–Perot cavity consists of a concave mirror etched into a silicon substrate, together with a plane mirror at the end of a cleaved optical fibre, as illustrated in figure 1(a). Light leaves the fibre with a waist of $w_0 \sim 5 \mu\text{m}$. Because of diffraction this expands until it reaches the curved mirror when it has a spot size of $w_1$, typically between 5 and 10 $\mu\text{m}$. Both the fibre and the curved mirror are coated with multi-layer dielectric stacks to achieve high finesse. The fabrication and characterization of this cavity are described in [18] and improved in [19]. An alternative geometry [20], in which the fibre tip is laser-machined to make a concave mirror, has recently reached even higher finesse. These cavities are still improving as fabrication techniques develop and it is not yet known how good they can become or which approach will be better. It is therefore valuable to understand and refine the mirror fabrication.

It is known that inductively coupled plasma (ICP) etching with precise control over the etching parameters can polish silicon surfaces [21, 22]. In this paper, we investigate the effects of unmasked ICP polishing on cavity mirrors that have been created by wet etching in a solution of hydrofluoric, nitric and acetic acids (HNA). Detailed measurements of the shape and roughness are made at various times throughout the ICP polishing. We characterize the shape by the radius $a$ of the aperture, the depth $d$ and the radius of curvature $R$, shown in figure 1(b). We find an improvement in both the profile and the roughness of the silicon surface, making it a more suitable mirror substrate for optical micro-cavities of high finesse.

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This paper is organized as follows. Section 2 describes the process used to fabricate the hollows and compares hollows of various sizes. Section 3 describes the ICP polishing process and its effect on the shape of the hollows. In section 4, we investigate how the roughness of the silicon surface evolves during ICP polishing. We conclude with a discussion of the optical mirror quality and its application to optical micro-cavities.

2. Wet-etching the hollows

We started with 20 (100)-oriented, P-type silicon wafers of 600 μm thickness on which a 60 nm layer of silicon nitride was deposited by low-pressure chemical vapour deposition (LPCVD). Each wafer was then primed using hexamethyldisilazane vapour and coated with a 1.3 μm thick HPR-504 photore sist, which we lithographically patterned with circles of radii 10, 20, 30 and 40 μm. The silicon nitride layer was then opened by a 20 s isotropic reactive ion etch (RIE) using 515 W of RF power, with a gas flow of 5 sccm CHF₃, 25 sccm CF₄ and 60 sccm Ar at a gas pressure of 40 mTorr. The etch rates for silicon and silicon nitride are very similar, so this step requires care to avoid over-etching into the silicon wafer. Following the ideas of [21], the hollows were then fabricated by wet etching all the wafers for 2 min at room temperature under constant agitation, using an HNA solution of (HF:HNO₃:CH₃CO₂H) in the ratio (30:43:27) with concentrations of (49:70:99.5)%wt. Although the HNA etch rate and the resulting hollow profiles are highly dependent on the composition and agitation of the solution [23], this recipe gave repeatable surface profiles with approximately 6 nm rms roughness in the hollows [18].

Figure 2 shows a scanning electron microscope image showing the cross section of the hollow made using a 10 μm radius opening in the nitride mask. It has an aperture of a ≃ 30 μm and a depth d ≃ a. The surface is roughly spherical at the bottom of the hollow. In order to see how these parameters vary with the size of the mask opening, all the hollows were measured using a stylus profilometer, and their dimensions are plotted in figure 3 versus mask opening. We see that the depth does not increase as rapidly as the radius, making the larger hollows less than a hemisphere.

3. Effect of ICP polishing on the shape

The RIE, previously used to open holes in the nitride mask, was used again to remove the rest of the nitride. Some silicon is inevitably removed at the end of this step, slightly roughening the silicon surface, so care was taken to use the minimum etching time required to remove the nitride. This
was preferable to a conventional orthophosphoric acid dip, which caused visible degradation of the silicon surface. Next, the wafers were ICP polished, in an STS Advanced Silicon Etcher using a chuck temperature of 20°C. Each wafer was polished for a different length of time, spanning the range 0–38 min in steps of 2 min.

The profiles of the hollows were measured again after ICP polishing. We use these images to determine $R(t)$ as a function of ICP polishing time, as shown in figure 5, together with a straight line fit. The radius does indeed grow linearly, with the best fit to the growth rate being $\Gamma = 4.3(2) \mu m min^{-1}$, in agreement with the rate deduced above from the aperture size. Here the uncertainty is principally due to the variation in the etch.

The hollows deviate from being spherical, as plotted in figure 6. Here, we show four representative samples, which have been etched for 0, 12, 24 and 38 min. Initially, the residuals vary typically over the range ±15 nm, with a correlation length has doubled. This amounts to an order of magnitude reduction in the noise power of the residuals and almost two orders of magnitude in the angular noise power. Mirrors with this level of sphericity have been used in our laboratory to make cavities with finesse exceeding 6000.

**4. Measurements of roughness**

In order to complete our picture of the polishing, we used an atomic force microscope (AFM) to see the roughness with much higher transverse resolution over a $10 \mu m \times 10 \mu m$ square. Figures 7(a)–(c) show scans on the flat part of the wafer, zoomed into a $2 \mu m \times 2 \mu m$ region. Before ICP polishing, this part of the wafer has no appreciable roughness, the standard deviation of the height being only 0.2 nm over
Figure 6. Deviation of hollow surfaces from spherical, measured over a circular aperture of 11.6 μm diameter at the bottom of the hollows. (a)–(d) These graphs show the residual surfaces from a fit to a spherical section after ICP etching for 0, 12, 24 and 38 min. The residuals initially span a range ±15 nm and are reduced after 38 mins of etching to ±3 nm, while the correlation length increases from ∼2 μm to ∼4 μm.

Figure 7. AFM scans depicting the evolution of the surface under ICP polishing. (a)–(c) Flat part of the wafer. (a) The initial roughness is negligible (rms = 0.2 nm). (b) At 10 min, unknown contaminants are clearly seen on the surface. (c) After 38 min they are almost entirely removed. (d)–(f) Deviation of the hollows from a sphere. (d) There are large initial deviations from spherical. (e) At 18 min the shape is improved and the contaminant particles are already much reduced in size compared with (b). (f) After 38 min they are small and as infrequent as in (c). The polished surface has rms roughness ≤1 nm.

the whole wavelength band of 40 nm–10 μm, corresponding to a spatial frequency of 0.1–25 μm⁻¹. During the first 10–15 min of polishing, however, the standard deviation grows to 3.5 nm due to the appearance of particles—typically 20–40 nm in diameter with a density of order 2 per μm²—whose origin is unknown. After that, the contaminant particles are gradually removed and the standard deviation decreases again. At 38 min, these particle are still present, but their height is reduced to a few nm, giving an rms variation of 1.1 nm. Discounting them, the roughness of the underlying
surface is approximately 0.5 nm. Figure 8(a) plots the average power spectrum of height noise measured along the x direction (the y direction gives the same result). Here, the arrival of the localized contaminant particles shows up as a broadband increase in the noise power density, followed by a gradual return to a lower noise, particularly at high spatial frequency. The roughening of the underlying surface prevents the low-frequency noise power density from returning to its original value after 38 min.

Figures 7(d)–(f) show a similar set of scans, this time at the base of the hollow. In (d), the initial unpolished profile deviates from spherical over the range ±15 nm, as already seen with the Zygo profilometer. This image could only be made after cleaving the wafer as the opening is otherwise too small to accommodate the AFM cantilever. The spectral density of the noise is plotted in figure 8(b). We see that the roughness is predominantly due to Fourier components at low spatial frequencies (≤1 μm⁻¹) associated with the shape of the hollow, rather than the microscopic roughness of the surface. After 18 min of polishing, the hollow is large enough to measure without cleaving. The scan of this hollow shows that the large-scale structure is becoming smoother and, in addition, we see the same covering of contaminant particles found on the flat surface. In the Fourier spectrum this appears as a decrease in the low-frequency noise power and an increase at high frequency, i.e. above 4 μm⁻¹. After 38 min, the particle contamination has been largely cleaned away, as on the flat surface, and the deviations of the surface from spherical are reduced to the typical range ±2 nm, as also seen with the Zygo profilometer. The low frequency noise is the polished relic of the much larger shape imperfection seen initially. In the spatial frequency band above 1 μm⁻¹, the variance of the surface height is less than 1 nm².

5. Conclusions

We have found that the hollow prepared by wet etching has an undulating surface that deviates from spherical by typically ±15 nm over distances of order 2 μm. The ICP polishing reduces these low-frequency deviations dramatically. As far as an optical cavity is concerned, this part of the spectrum describes the deviation of the surface from spherical. It may be regarded as noise in the shape of the mirror substrate, producing geometrical aberrations of the cavity that were also seen using the white light interferometer. In the frequency band above 1 μm⁻¹, there is an initial increase of noise due to the deposition of contaminant particles on the surface. After 38 min, these particles are largely polished away and the variance of the surface is decreased to ≤1 nm². These higher frequency Fourier components are mostly missed by the optical profilometer because they are not resolved by the light and merely serve to reduce the reflectivity of the surface from its ideal value ρ₀. The Debye–Waller formula

\[ \rho = \rho_0 \exp\left(-\frac{4\pi\sigma}{\lambda^2}\right) \]

provides a simple estimate of the reduced reflectivity, where \( \sigma \) is the surface roughness and \( \lambda \) is the wavelength of the light. A typical application of the cavity is in cavity QED [14]. For example, using the D line of Cs atoms at 852 nm, a roughness of 1 nm permits a reflectivity of 99.98%, corresponding to a cavity finesse (\( \pi\sqrt{\rho}/(1-\rho) \)) with matched mirrors) exceeding 14 000 for \( \rho_0 = 1 \). However, the reflectivity \( \rho_0 \) is low in the visible and near-infrared because of absorption by the silicon, so the hollow has to be coated by a dielectric Bragg stack several microns thick, depending on the required reflectivity. With the AFM, we measure that the surface roughness of this coating is below 0.5 nm which can support a reflectivity of 99.995% or a finesse exceeding 60 000.

In search of the highest finesse, Biedermann et al [19] added a final silicon polishing stage, in which a thick oxide was twice grown on the surface and removed by HF. This left the silicon surface with a roughness of \( \sigma = 0.22 \) nm at spatial frequencies above 1 μm⁻¹. The roughness of the Bragg stack deposited on top of this was 0.26 nm, slightly better than the roughness of the optical coating in our laboratory. The maximum finesse they achieved was 64 000, impressively high but much less than the Debye–Waller maximum of 260 000.

We conclude that our technique for polishing silicon provides a smooth substrate, suitable for building micro-mirrors of high reflectivity corresponding to a finesse in excess of 60 000, once the mirrors are reflection coated. Even smoother surfaces can be achieved, but at this point, the limiting imperfection is the shape of the surface, rather than its roughness. This might be improved by even longer ICP etching time, but a better approach is probably to replace the initial wet etch by an ICP etch.
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