

Cleaning Silicon Nitride Gratings with Liquid Immersion

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We demonstrate a method for cleaning sodium atom deposits off of nano-fabricated silicon nitride gratings using liquid immersion, without damaging the grating. The possibility of cleaning a grating successfully depends on its physical parameters and the type of liquid to be used. We present calculations on surface tension interactions between a liquid and the bars the grating, and derive the maximum grating bar span that will survive immersion.

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1. Introduction

Free-standing silicon nitride gratings with periods down to 100 nm are currently used as transmission diffraction gratings for atom interferometry¹. In the interferometer operating at the University of Arizona, the gratings are subject to a beam of sodium atoms and after prolonged exposure to the beam, the gratings become clogged with sodium deposits. They become progressively less effective at transmitting and diffracting atoms.

Since a set of high-quality homogeneous gratings is assembled only after much testing, it is of interest to develop a cleaning method to remove sodium from the gratings, while leaving the grating structure intact and undamaged. This allows for repeated use of the same gratings.

Previously, free-standing 100 nm period gratings which were exposed to liquid have been damaged, as the surface tension of the liquid can be strong enough to pull the grating bars together. However, the ability to withstand the forces created by the surface tension of the liquid depends on the physical dimensions of the support structure and the grating bars.

2. Grating Cleaning

The gratings are made by T. Savas and H. I. Smith at the MIT NanoStructures Laboratory^{2,3}. The gratings consist of a support structure with a period of a few microns, between which smaller bars with a period of a few hundred nanometers are spanned. The gratings are physically open, to allow passage of particles. The open fraction of the bars, and the periods of the support structure and grating bars vary depending on the manufacturing run.

A 200 nm period grating used in the interferometer that was covered in sodium was cleaned using liquid immersion. Figure 1 shows atom force microscope (AFM) images of the grating with sodium deposits on the surface, and the same grating cleaned after immersion in liquid. To clean the grating, it was first placed in acetone. The acetone was then flushed with water, which is a good solvent for sodium and sodium oxide. The grating is then flushed with acetone again, and then allowed to dry. In this way the grating is exposed to water, but never exposed to a water-air boundary. Acetone is used to buffer the grating from direct water immersion, since acetone has less surface tension than water.

This procedure has been repeated with 100 nm gratings, also without damage to the grating. Though the AFM images show that there is no visible damage to the bars, there is the possibility that there would be a thin film left behind after drying the grating. To check that the transmission properties are still intact, a 100 nm period grating that was cleaned with the same procedure was used in an SEM to observe diffraction, thus proving the grating still transmits 5 keV electron waves coherently. The details of this method of observing diffraction are available in Cronin et al.⁴ Figure 2 shows the diffracted image of a wire mesh as viewed through a cleaned grating.

Although these trials were all successful, there are also examples of gratings that have been damaged by water immersion. As seen in Figure 3, the left image is an SEM frame of a cleaned and undamaged grating, while the right image is of a grating that was unsuccessfully immersed in water. The grating bars have clumped together, presumably as a result of the surface tension of the drying liquid pulling them together in clumps of 2 to 5 bars. In order to determine which gratings are likely to survive an immersion in liquid, we present calculations on the mechanics of the grating cleaning process.

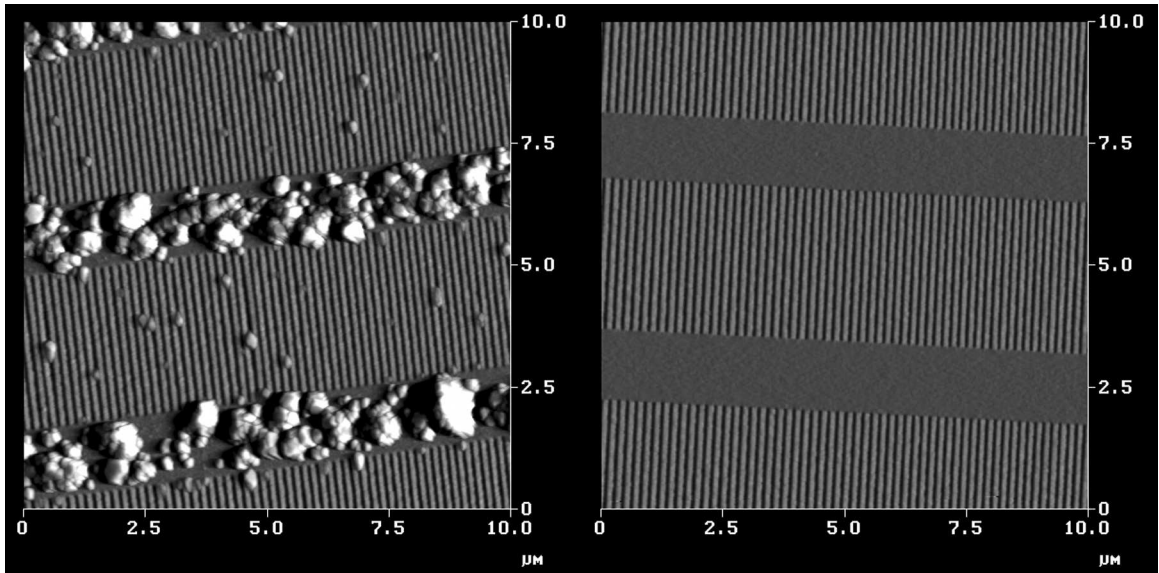


Fig. 1. A 200 nm period grating that shows sodium deposits, and the same grating after cleaning in water.

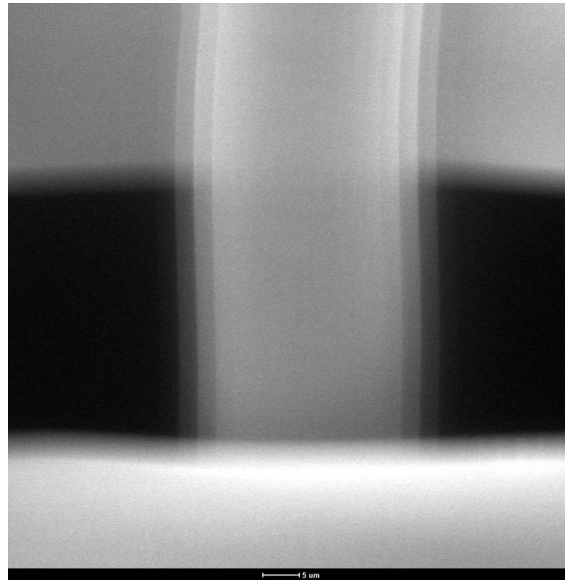


Fig. 2. Electron diffraction of a wire mesh through a 100 nm period grating.

3. Bar Bending

Consider a bar fixed at both ends, as depicted in Figures 4a,b. The bar has a length L , width a , and depth b . Under a uniform load F , the bar will bend in the y direction. The beam's curvature, $y(x)$, can be expressed mathematically as⁵

$$y(x) = \frac{F}{24LEI}(x^4 - 2Lx^3 + L^2x^2), \quad (1)$$

where F is the external force, L is the beam length, E is the Young's modulus of the bar material, and I is the bar's moment of inertia.

At the midpoint of the bar, the displacement, $y(L/2)$, which we define as δ , will be a maximum. Substituting $x = L/2$ into Eqn (1), we have

$$\frac{FL^3}{32ba^3E} = y(L/2) \equiv \delta, \quad (2)$$

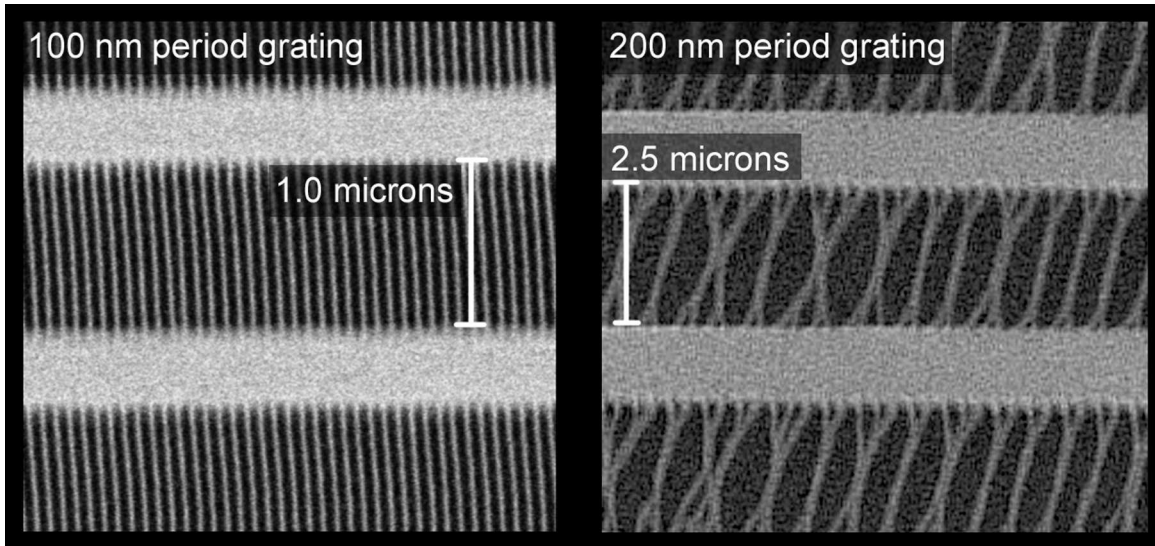


Fig. 3. Left: a 100 nm period grating that survived cleaning in liquid. Right: a 200 nm period grating that was damaged by liquid immersion.

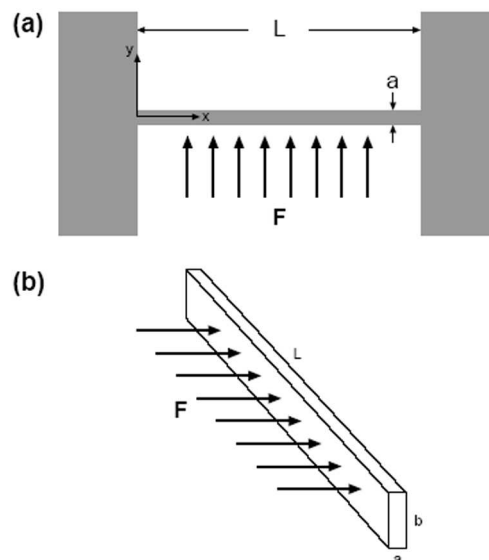


Fig. 4. Depiction of a bar fixed at both ends under a uniform load F . (a) Top view. (b) Side view.

where we used $I = ba^3/12$. Eqn (2) may be rewritten in terms of pressure rather than force, so that

$$\delta = \frac{PL^4}{32a^3E}, \quad (3)$$

where $P = F/bL$. Viewed alternatively, Eqn (2) gives the internal restoring force, F_I , that balances the external force bending a bar along $y(x)$. Rearranging Eqn (2), the restoring force is

$$F_I = \frac{32\delta ba^3E}{L^3}. \quad (4)$$

4. Surface Tension Calculations

Figure 5 depicts four grating bars, in cross section, after wetting. Since the liquid does not dry uniformly, occasionally two bars will have liquid between them while the spaces to either side of these bars will be dry. We consider this worst case in which the net force on two bars is maximum.

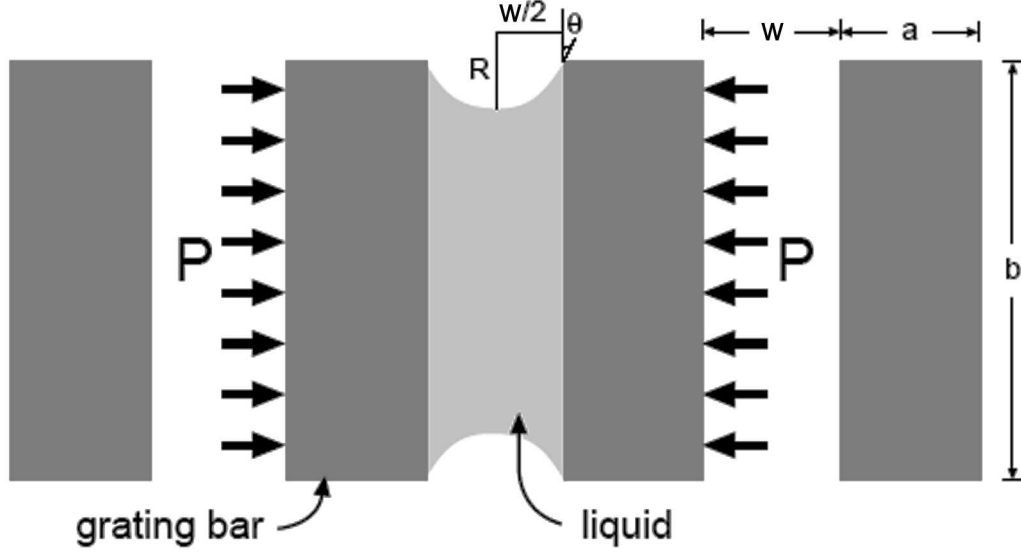


Fig. 5. Schematic showing wet grating bars in cross section.

As shown in the figure, the pressure difference between the inside and outside of the liquid will produce a force on the grating bars. In this case, the liquid pulls the bars together (if the liquid surfaces curved outward, the surface-tension force would push the bars apart). One can show that the pressure difference, P , between the inside and outside of the liquid is

$$P = \frac{2\gamma}{R}, \quad (5)$$

where γ is the surface tension of the liquid and R is the surface's radius of curvature. As shown in Figure 5, the radius of curvature is given by

$$R = \frac{\frac{w}{2} - \delta}{\cos \theta}, \quad (6)$$

where w is the distance between walls of the two bars. The term $w/2 - \delta$ accounts for bars bent closer than $w/2$. Substituting Eqn (6) in Eqn (5), we have

$$P = \frac{2\gamma \cos \theta}{\frac{w}{2} - \delta}. \quad (7)$$

Therefore, the force, F_γ , exerted by the surface tension is

$$F_\gamma = \frac{2\gamma b L \cos \theta}{\frac{w}{2} - \delta}. \quad (8)$$

For static equilibrium, $F_\gamma = F_I$, where F_I is given by Eqn (4). Rather than setting the two forces equal to one another, the same result may be obtained by substituting the result for P given by Eqn (7) into Eqn (3). We then have

$$\delta = \frac{L^4 \gamma \cos \theta}{16a^3 E} \cdot \frac{1}{\frac{w}{2} - \delta} \equiv \frac{\beta}{\frac{w}{2} - \delta}, \quad (9)$$

where β is defined. Solving for delta, we get

$$\delta = \frac{w}{4} \left(1 - \sqrt{1 - \frac{16\beta}{w^2}} \right). \quad (10)$$

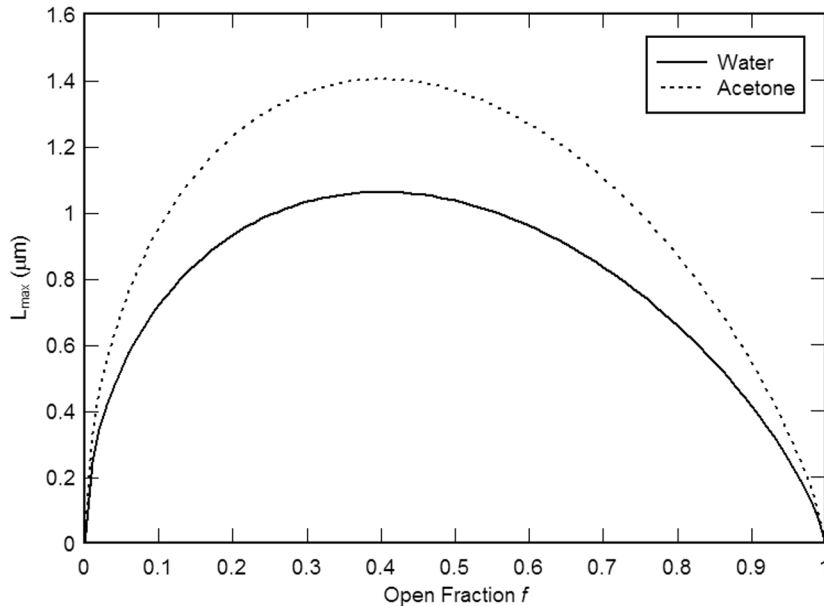


Fig. 6. A plot of L_{max} versus open fraction f for 100 nm period gratings, in water and acetone.

The surface tension force balances the bars' internal restoring forces up to a distance $w/4$ beyond which the surface tension overcomes the restoring force and the bars pull together. Examining the square root in Eqn (10), we find

$$\beta \leq \frac{w^2}{16} \quad (11)$$

which leads to an upper limit for L of

$$L \leq \left(\frac{w^2 a^3 E}{\gamma \cos \theta} \right)^{\frac{1}{4}}. \quad (12)$$

That is, the grating-bar span, L , must not exceed the amount given by Eqn (12) or else the bars after wetting will contact one another. We may set $\cos^{\frac{1}{4}} \theta \approx 1$ for a few reasons. First, the contact angles are small for most liquids on silicon nitride ($\theta = 3.2$ degrees⁶ for water on silicon nitride). Even for angles as large as 50 degrees, this is still a good approximation. Finally, L is smallest when $\cos \theta = 1$, so setting $\cos \theta = 1$, we obtain a value for the maximum span. To obtain a more general relationship, we recast Eqn (12) in terms of the period, d , and the open fraction, f , of the grating, by making the substitutions $d = a + w$ and $f = w/d$. Eqn (12) then becomes

$$L \leq \left(\frac{d^5 E}{\gamma} \right)^{\frac{1}{4}} (f^2 (1-f)^3)^{\frac{1}{4}} \equiv L_{max}. \quad (13)$$

Using $d = 100$ nm, $E = 270$ GPa = 2.7×10^{12} dynes/cm², $\gamma_{(H_2O)} = 73$ dyne/cm, and $\gamma_{(acetone)} = 24$ dyne/cm, Figure 6 shows a graph of L_{max} versus open fraction f , for both acetone and water. The curves have maxima at $f = 0.4$, but are relatively constant for open fractions between 20 and 60 percent, which is a typical open fraction for manufactured gratings. If we use acetone as our first and last liquid of immersion, we can expect a 100 nm period grating with bar length of 1.4 microns or less to survive. If we use 200 nm period gratings, the maximum of the curves peak at 3.2 microns for acetone and 2.4 microns for water. The grating in figure 1 is a 200 nm period grating, and the bars span about 3 microns, just below the theoretical limit of L_{max} derived for acetone. In figure 3, the 100 nm period grating on the left has a bar span of about 1.3 microns, also just below the limit for acetone. The grating on the right, damaged by water, is a 200 nm period grating, and its bar span is 2.5 microns, slightly above the limit of L_{max} for water.

In the future, it would be of interest to see if other atoms can be cleaned from the gratings. For example, aluminum is removed by a solution of water and lye, so gratings that have been exposed to aluminum atoms could possibly be cleaned in a similar manner. Since lye dries with a crystalline residue, liquid replacement techniques for the cleaning would be crucial. Of additional concern would be what effect bubbles caused by the aluminum-lye reaction would have on the cleaning process. At the moment the technique is viable for sodium and other elements or compounds that would be cleaned away by water or acetone.

5. Conclusions

Within certain guidelines, free-standing silicon nitride gratings can be cleaned of sodium by immersion in water. The geometric properties of the grating and the surface tension of the liquid affect the possibility of success. Gratings with larger periods for the support structure, and those with very high or low open fraction between nanometer-period bars have more likelihood of being damaged by surface tension. By using a technique to replace fluids with progressively larger surface tensions and then reversing the procedure, one can avoid having the grating dry with high surface tension liquids between the bars. This technique increases the maximum allowable span of the support structure bars.

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