Large-area, free-standing gratings for atom interferometry produced using holographic lithography

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Interferometers based on matter waves promise orders-of-magnitude improvements in metrology over laser-based systems by virtue of the fact that the de Broglie wavelengths of atoms are about 10^4 times shorter. To date, the required matched set of four aligned gratings for such atom interferometers has been made using electron beam lithography and, as a result, such gratings suffer from a lack of spatial-phase coherence. We report on processes we have developed for fabricating free-standing gratings over large areas using conventional holographic lithography and achromatic holographic lithography to achieve spatial periods of 200 and 100 nm, respectively (i.e., nominal linewidths of 100 and 50 nm, respectively).

Atomic or "matter-wave" interferometry has potential for a variety of fundamental studies and practical applications.[1,2] Some have been reviewed in the semitechnical literature.[3] In metrology, for example, an interferometer based on matter waves should provide orders-of-magnitude improvement over laser-based systems because the de Broglie wavelength of supersonic atoms is about 10^4 times shorter.

A key component of an atom-beam interferometer is the matched set of four aligned diffraction gratings. These gratings must be free-standing (that is, they consist of lines of material separated by open spaces); they must have fine periods (e.g., 200 nm and below); and they must be phase coherent over large areas (e.g., 100 μm$\times$1 mm or more). Even larger areas are desirable, and may be required in the future. The grating lines can be supported by crossing struts, provided they do not significantly obstruct the beam.

The requirement of spatial-phase coherence can be understood as follows: if we imagine an abstract, perfect grating of period p, the lines of the real grating must superimpose on, or be congruent with, the ideal grating lines to within a small fraction of p. A small percentage of missing line segments, random errors, and line edge raggedness are permitted. Systematic placement errors and distortion beyond a fraction of p are not acceptable.

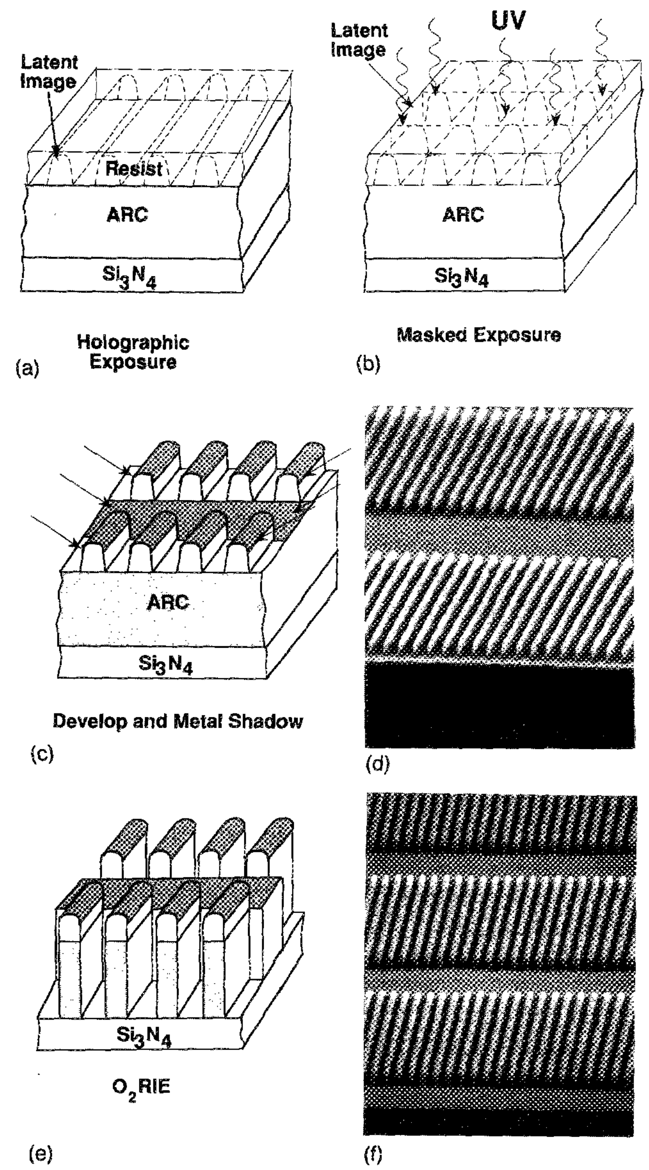
The initial experiments in atom beam diffraction with free-standing gratings utilized gratings designed for x-ray spectroscopy.[2] They were 200 nm period, fabricated using holographic lithography and electroplating of gold, 0.5 μm thick. They were suitable for demonstrating diffraction of matter waves, but due to distortion induced by stress in the thick plated gold, they did not meet the stricter requirements of spatial-phase coherence for interferometry.

In atomic-beam interferometry there is no need to make the grating lines thick, as there is in diffraction gratings for x rays. (In fact, the lines need be only one atomic layer thick!) Considering the materials available, the optimal choice appears to be Si_3N_4, because high-quality membranes, with controlled internal stress, can be produced, and free-standing microstructures[4] and gratings[5] have been fabricated from such membranes. To produce a membrane, the Si_3N_4 is first deposited on a clean Si wafer, polished on both sides. By patterning the back side with a rectangular opening in the Si_3N_4, a KOH solution can etch the exposed Si in a highly anisotropic manner, leaving a free-standing Si_3N_4 membrane supported on a "window frame" of rigid Si, in the form of a truncated pyramid with (111) facets.

Given that Si_3N_4 is the material of choice, the problems in fabricating free-standing gratings for atomic interferometry are (1) how to generate the pattern of grating lines and crossing support struts with the spatial-phase coherence required for interferometry; (2) how to etch the free-standing grating and struts in the Si_3N_4 without breaking.

The first successful fabrication of free-standing Si_3N_4 gratings with support struts for atomic-beam interferometry utilized scanning electron beam lithography (SEBL) in PMMA, and reactive ion etching (RIE) in CF_4: H_2 mixtures.[5] Although successful in demonstrating split-beam atomic interferometry, concerns were expressed about the spatial-phase errors that are intrinsic to conventional SEBL. These phase errors (i.e., placement errors) arise from a number of sources: distortion within a single writing field of the SEBL; digital-to-analog converter (DAC) errors (e.g., missing steps, nonlinearities, etc.); stitching errors at field boundaries (i.e., spatial-phase discontinuities); drift due to charging in the sample and the column; drift due to thermal expansion; height variation on the sample, etc. Anderson *et al.* have shown that DAC errors and distortion within a SEBL scan field can be eliminated, at least for small fields ($\sim 100\times100$ μm), by calibrating the field using a holographically generated grid.[6] The other placement errors of SEBL should likewise be correctable by reference to a holographically generated global-fiducial grid on the sample surface, as has been proposed,[7] but not yet implemented.

In order to fabricate gratings for atom beam interferometry the preferred solution is to bypass SEBL and use holographic lithography directly.[8] The benefits include not only the elimination of concerns about spatial-phase errors in the pattern generation process, but also the opportunity to use very thick resist, and exposure times that are orders of magnitude shorter than with SEBL. Moreover, holographic methods cover large areas, are ideally matched to the system problem of interferometry, and can probably be extended to 50 nm periods.[9]



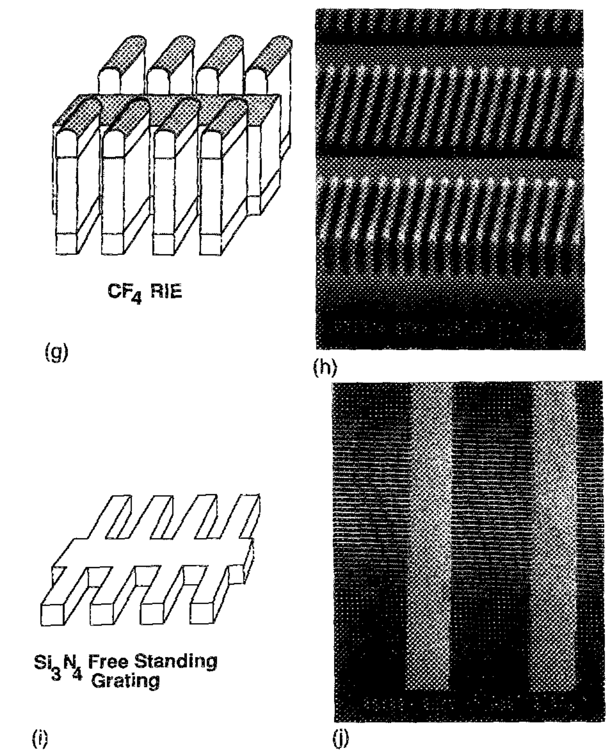


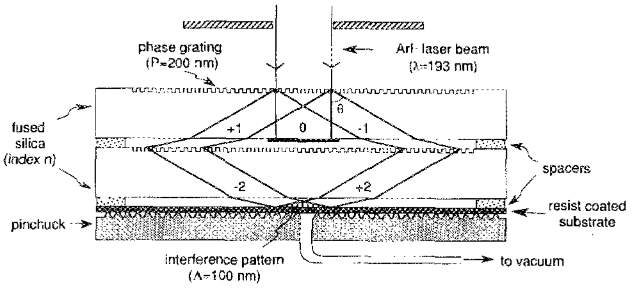
FIG. 1. Steps of process for fabricating free-standing gratings with support struts: (a) Holographic lithography is used to expose a fine-period grating in resist on an ARC. The latent image is not developed. (b) The crossing support struts are exposed by ordinary photolithography producing a second, superimposed latent image. The resist is then developed. (c) The developed relief image is shadowed with a thin metal. (d) Corresponding electron micrograph. (e) An oxygen RIE steps etches through the ARC except in areas covered with metal. (f) Corresponding electron micrograph. (g) RIE in CF_4 to etch through the Si_3N_4 membrane. (h) Corresponding electron micrograph, but on a Si substrate. (i) Schematic of free-standing Si_3N_4 grating with support struts. (j) Corresponding electron micrograph at normal incidence. Grating period is 200 nm.

One problem with holographic lithography is that it produces only the fine period grating; the support struts must be produced by another means. Figure 1 illustrates the steps of the lithographic process we have developed, in which the pattern of support struts is exposed directly on top of the latent image of the holographic exposure. (With care the support grid can be made perpendicular to the fine-period grating, which is helpful in aligning an atom interferometer.) After a single development step, the entire surface-relief pattern is shadowed with e-beam evaporated metal (in this case Ni) which protects the pattern from being undermined during the oxygen RIE down through the antireflection coating (ARC).[8] The component of the shadowing vector in the plane of the substrate is parallel to the grating "*k* vector" (i.e., it is perpendicular to the grating lines). Shadowing is done from two sides, as depicted, and the angle chosen determines the line-to-space ratio of the final grating. The support struts are metallized at the same time as the grating lines. We have a specially designed fixture in our e-beam evaporator to accomplish the precision shadowing.

By far, the major problem in fabricating the free-standing gratings is the step of etching through the Si_3N_4, and removal of the resist thereafter. In many respects it would be easier to etch the grating and support struts through the Si_3N_4 while still supported on the Si substrate, and then etch the Si beneath the grating as the last step. We have actually used this process successfully, but were concerned that the etching of the Si may cause the grating lines to move laterally, thereby loosing spatial-phase coherence. We therefore chose to do patterning and etching directly on Si_3N_4 membranes by an entirely dry process.

We experienced less breakage if low stress, Si-rich Si_3N_4 membranes were used.[10] The Si_3N_4 membrane window-frame structures, which measured 0.1×1.8 mm, are fabricated using well-known techniques discussed above. A Si wafer contains many such windows. ARC and photoresist are readily spun on the membranes. Exposure in the holographic system is straightforward when our "conventional" holographic lithography (CHL) is used, which produces a uniform 200 nm-period grating over an entire 7.5 cm-diam Si wafer.[8] The support struts are exposed by contact photolithography, as depicted in Fig. 1(b).

To achieve gratings finer than 200 nm we have to use



$$\sin\theta = \lambda/(nP),$$
$$\Lambda = \lambda/(2n\sin\theta)$$
$$= P/2$$

FIG. 2. Schematic of AHL configuration used to expose 100 nm-period gratings.

achromatic holographic lithography (AHL),[11] depicted in Fig. 2. This technique produces 100 nm-period gratings in PMMA, as shown in Fig. 3. The area coverage of the AHL gratings is not limited by the source temporal coherence; it depends only on geometry, i.e., the spacing of the two phase gratings. Source spatial coherence, which can be made arbitrarily high by means of apertures, affects only the depth-of-focus.[11] We have fabricated gratings with areas $\sim 5\times 20$ mm, much smaller than that of the CHL gratings (75 mm diam), but much larger than required for atom-beam interferometry. The process of Fig. 1 need be modified only slightly for the 100 nm periods, i.e., the

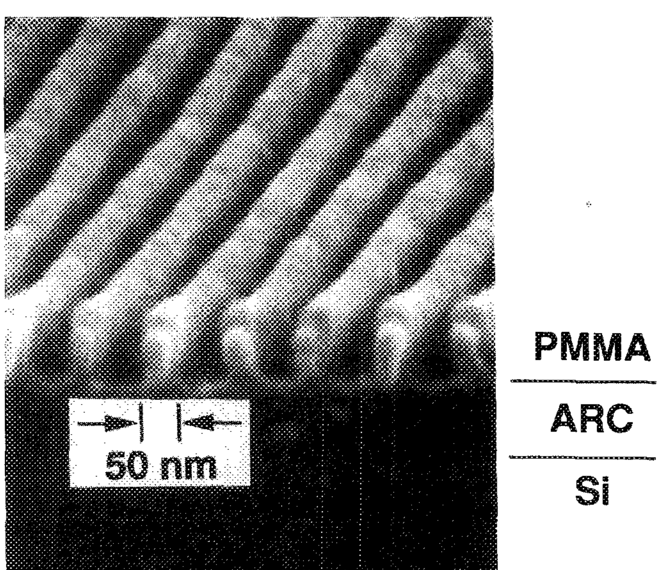


FIG. 3. Scanning electron micrograph of 100 nm-period grating in PMMA, exposed by AHL on top of a specially designed ARC (Ref. 12).

exposure of support struts must be done with a deep ultraviolet source and quartz masks.

The RIE is done in a parallel plate system at a bias voltage of 400 V, a power density of 0.14 W/cm^2, a pressure of 10 mTorr, and with the Si wafers on a water-cooled baseplate. To avoid overheating of the membranes, etching is done in short bursts of 10 s separated by cool down cycles of 90–120 s. The gasses are O_2 for etching down through the photoresist and ARC, and CF_4 for etching through the Si_3N_4. The remaining ARC and resist need not be removed, but if this is desirable (e.g., to avoid any possibility of stress-induced distortion due to changes in the polymers with time), it can be done in liquid resist strippers (which has the problem that the grating lines sometimes pull together and stick to one another) or in an O_2 plasma.

An alternative approach would be to use holographic lithography to fabricate x-ray masks containing the fine-period gratings and support struts, and to use x ray to expose on the Si_3N_4 membranes. This would avoid the shadowing and O_2 RIE steps. The CF_4 RIE step would remain substantially the same.

In summary, we have described processes for fabricating free-standing gratings in Si_3N_4 for atomic beam interferometry that take advantage of the ideal spatial-phase coherence of holographic lithography. Large numbers of identical gratings can be produced at low cost. The technique should be extendable to 50-nm periods using a proposed new form of AHL in which a 13 nm undulator beam is the radiation source.

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