Scanning tunneling microscopy applied to optical surfaces

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The technique of scanning tunneling microscopy has been applied to topographic mapping of two optical surfaces: a ruled grating replica and a diamond-turned gold mirror. We have demonstrated the ability of the scanning tunneling microscope to measure surface topography of a ruled-grating replica over an area of 2 μm × 2 μm. Furthermore, surface structure on a diamond-turned gold mirror was observed that could not be detected by any other type of surface-sensitive microscope. These measurements yield information necessary for gaining a complete understanding of the diamond-turning process.

Recently, the scanning tunneling microscope (STM) has been shown to be capable of producing topographic maps of single-crystal metal[1] and semiconductor[2] surfaces with resolution sufficient to permit the observation of individual atoms. The surfaces were prepared and measured in an ultrahigh-vacuum environment and were atomically flat with occasional, single-atom high steps. We report the application of the same technique to the characterization of polycrystalline gold optical surfaces. Operating our STM in high vacuum and with moderate resolution (~0.1 nm vertical, ~1 nm lateral), we have examined two optical surfaces: a ruled grating replica and a diamond-turned gold mirror. These measurements represent the first step in developing the STM to provide topographs of mechanically generated optical surfaces, similar to those currently obtained with stylus instruments but with near-atomic vertical and lateral resolution and the advantage of a noncontacting probe.

The measurement technique involves scanning a conducting tip across a conducting surface while maintaining a constant tip-to-surface distance. Three-dimensional positioning of the tip is achieved by using a piezoelectric tripod. The tip-to-surface distance is fixed at a value necessary to obtain an electron tunneling current of ~1 nA for a typical bias voltage between the two electrodes of 0.1 V. Topographic information is obtained by monitoring the servo voltage applied to the z piezo as the tip is scanned in x and y across the surface. A description of this mapping technique is included in some early work by Young.[3] The STM used is nearly identical to the third-generation instrument developed at IBM/Zurich, which is described in detail in the work of Binnig and Rohrer.[4] While atomic lateral resolution has been obtained only in an ultrahigh-vacuum environment, operation of the instrument in air at somewhat decreased resolution has also been demonstrated.[5]

The atomic resolution that has been obtained when profiles of near atomically flat surfaces were made is thought to be due to the reduction of the tip size to an effective area of a few, or perhaps one, atoms because of the exponential dependence of tunneling current on gap distance. In these cases a blunt, jagged tip may work quite well.[4] However, for surfaces with a greater degree of roughness, the site of this effective tip area may move around the tip within the tip diameter. In the current work, we have used an etching technique to produce rigid, yet sharp tips (Fig. 1) with diameters characterized to <1 μm with the possibility of sharp (~20-nm-diameter) protrusions. Clearly much can be gained by developing more-carefully controlled tip preparation and characterization procedures to take full advantage of the wear-free noncontacting nature of the STM tip.

The etching technique consists of using a 1-N KOH solution in a glass vessel with a carbon rod anode. A tungsten rod (1-mm diameter) is masked with plastic sleeves exposing a 1-mm narrow band to the etchant. A pulsed ac current of 50 mA at 3.5 V is used to etch the W rod at the exposed band with electrical contact

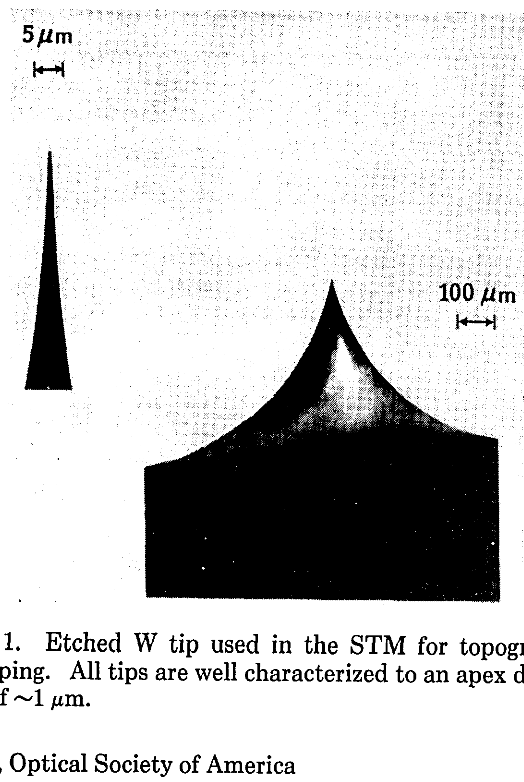

Fig. 1. Etched W tip used in the STM for topographic mapping. All tips are well characterized to an apex diameter of ~1 μm.

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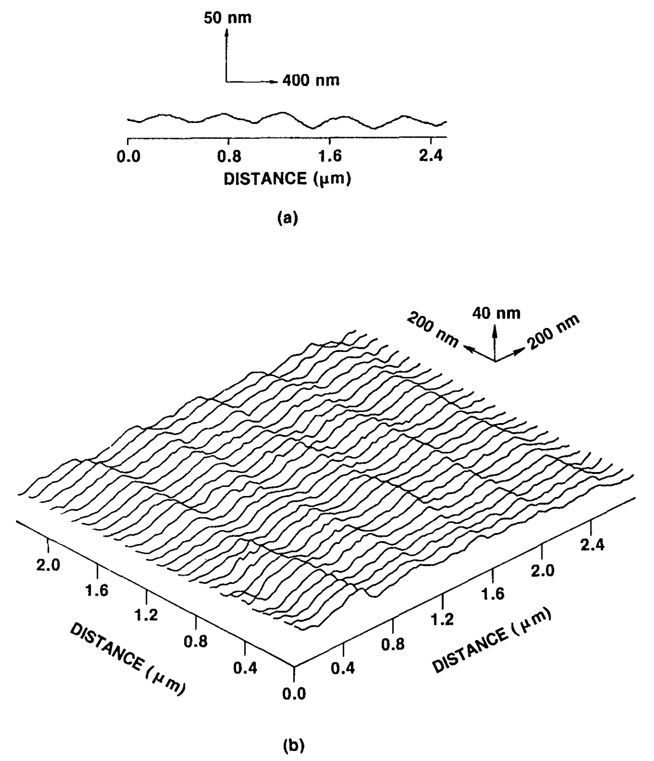

Fig. 2. (a) Single profile of a grating replica measured by a conventional stylus instrument. The measured line spacing is 460 nm, and the measured roughness is ~4.3 nm. (b) STM topographic map of a grating replica. The measured surface rms roughness is ~5.4 nm.

being made at the top of the W rod. The etching process of the bottom piece of W immediately stops as the W rod is etched through, thus preventing tip blunting. The short, stubby quality of these tips ensures a high resonant frequency, whereas the sharp tip permits examination of surface features with substantial slopes such as are found on a ruled grating.

The grating replica examined was produced by coating a glass master grating (463-nm line spacing) with a gold film, electrodepositing a heavy nickel base (2 mm thick) to the Au film, and then separating the Ni/Au replica from the master grating. The shape of the grating replica approximates raised lines separated by flat areas. The line spacing in this type of grating is used for dimensional calibration of electron microscopes, and no information concerning amplitude was available. Whereas the central portion of the replica is cross ruled, measurements were recorded in a 4 mm × 6 mm area cut from a single-ruled portion of the replica. Profiles of the grating were measured using a conventional stylus instrument.[6,7] Measurements were recorded using a previously unused, pyramid-shaped diamond stylus with a 0.1-μm nominal radius, a loading force of 2 mg, a sampling interval of 3.3 nm, and no filtering. However, owing to the possiblity of wear of the diamond stylus, the effective radius may have been somewhat higher. By measuring profiles of the grating replica, an average surface rms roughness of 4.3 nm was determined relative to a best-fit straight line for each profile [Fig. 2(a)].

The piezoelectric elements used in the STM have good range but exhibit substantial nonlinearity when scanned large distances. A measure of the nonlinearity was obtained using a calibrated eddy-current sensing probe. The nonlinearity must be corrected in the lateral dimensions over the 2.7 μm × 2.3 μm scan area shown in Fig. 2(b). The ruling spacing determined from the STM results was found to be ~470 nm with an average surface rms roughness of 5.4 nm relative to a best-fit straight line for each scan line. The improvement in resolution and consequent increase in measured surface rms roughness over that of the stylus determination is thought to be due to a sharp protrusion on the STM tip.

Diamond turning, a machining process that can produce mirror finishes on contoured surfaces, has been applied to the manufacture of mirrors such as those used in laser fusion research and in optical scanners.[8] Topographic measurements of such surfaces, in particular with the sensitivity to fine detail afforded by the STM, are crucial to a better understanding of the manufacturing process and, ultimately, to the controlled production of optical surfaces of the highest quality.

The polycrystalline diamond-turned Au sample (5.7-mm diameter × 0.8 mm) was produced by flycutting at 1000 rpm with a feed rate of 0.1 mm/min to yield cuts of 100-nm width. There was no discernible structure in single profiles measured by the stylus instrument. The spacing between the cuts as measured by the STM is seen to be irregular (Fig. 3), even after the large scan area (1.6 μm by 1.6 μm) is corrected for piezo nonlinearity. This irregularity could be produced by nonuniformity of the feed mechanism for the diamond-cutting tool or could be caused by variation in the depth (≈2.5 μm) of the cuts. A heavy cut can remove the adjacent peaks left by a previous lighter cut, creating the appearance of an irregular feed rate or missing cuts. Both types of structure appear to be occurring on this surface—large gaps between obvious cuts (i.e., missing cuts) and uneven spacing between cuts. The average height of the tool markings is ~3 nm.

In addition to the irregularity of the cuts on the Au

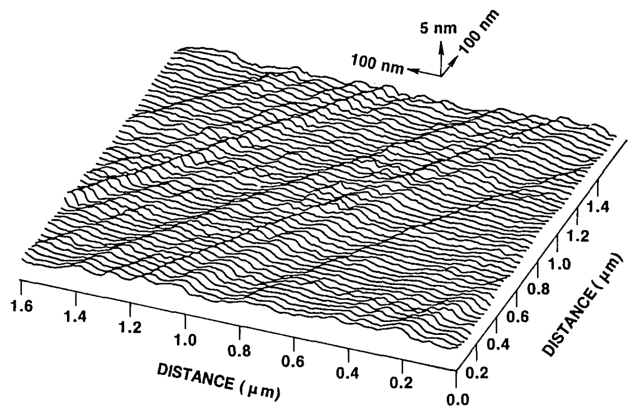

Fig. 3. Large-area topographic map of a diamond-turned Au mirror. The surface rms roughness is ~0.6 nm.

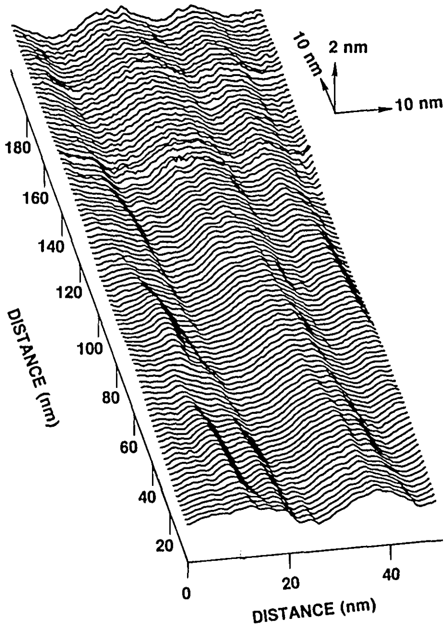

Fig. 4. Surface structure of a diamond-turned Au mirror within one pass of the cutting tool. The surface rms roughness is ~0.4 nm.

diamond-turned sample, there is evidence of tool signature variation between cuts that could be caused by tool chatter or grain relief.[7,9] As shown in Fig. 4, there is structure within a single pass of the tool that changes as the tool progresses across the surface. The 50 nm × 200 nm area scanned has a peak-to-valley spacing of ~1.3 nm. The z displacement has been magnified to display the surface structure. No correction for nonlinearity was necessary owing to the relatively small scan range. The scanning angle has been adjusted so that the individual scans are approximately perpendicular to the tool path, which permits following the tool signature over a relatively long range. The structure noticeably changes in the lower left portion of the figure. Furthermore, this tool signature (furrows spaced ~20 nm apart) changes dramatically between tool cuts over the surface of the diamond-turned sample.

We have shown that STM measurements of optical surfaces reveal much finer detail than observable with a conventional stylus instrument. Such topographic information should be useful in understanding the remnant roughness of diamond-turned samples, which as yet has not been explained by taking into account such machining conditions as tool radius, feed rate, and vibration.[10] Further work is needed, and is planned, to assess fully the effects of tip geometry and surface work-function variation on the observed topography.

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