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Standard Test Method for Determination of Surface Roughness by Scanning Tunneling Microscopy for Gas Distribution System Components[1]

This standard is issued under the fixed designation F1438; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Semiconductor clean rooms are serviced by high-purity gas distribution systems. This test method presents a procedure that may be applied for the evaluation of one or more components considered for use in such systems.

1. Scope

1.1 The purpose of this test method is to define a method for analyzing the surface texture of the above-mentioned components using a scanning tunneling microscope (STM). STM is a noncontact method of surface profiling that can measure three-dimensional surface features in the nanometer size range, which can then be used to represent the surface texture or to provide figures of merit. Application of this test method, where surface texture is used as a selection criterion, is expected to yield comparable data among different components tested.

1.2 *Limitations:*

1.2.1 This test method is limited to characterization of stainless steel surfaces that are smoother than $R_a = 0.25$ µm, as determined by a contact-stylus profilometer and defined by ANSI B46.1. The magnifications and height scales used in this test method were chosen with this smoothness in mind.

1.2.2 Intentional etching or conductive coating of the surface are considered modifications of the gas-wetted surface and are not covered by this test method.

1.2.3 This test method does not cover steels that have an oxide layer too thick to permit tunneling under the test conditions outlined in 11.3.

1.3 This technique is written with the assumption that the STM operator understands the use of the instrument, its governing principles, and any artifacts that can arise. Discussion of these points is beyond the scope of this test method.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[2]

2.2 *ANSI Standard:*

ANSI B.46.1-85, "Surface Texture (Surface Roughness, Waviness, and Lay)," ANSI/ASME, 1985[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *artifact*—any contribution to an image from other than true surface morphology. This could include such examples as vibration, electronic noise, thermal drift, or tip imperfections.

3.1.2 *center line (graphical center line)*—line parallel to the direction of profile measurement, such that the sum of the areas contained between it and the profile contained on either side are equal (see Calculation Section).

3.1.3 *cutoff length (l_c)—for profiles in this context*, the sampling length, that is, the length of a single scan, in nanometers (see Calculation Section).

3.1.4 *current*— in this context, the tunneling current (expressed in nanoamperes) that flows in either direction between the tip and surface, under the conditions specified.

[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronicsand is the direct responsibility of Subcommittee F01.10 on Contamination Control.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute, 13th Floor, 11 W. 42nd St., New York, NY 10036.

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3.1.5 *feature height*—datum (height in the z-direction) of any point in the scan area, relative to the lowest point in the scan area, as derived from tunneling current during tip rastering.

3.1.6 *filter*—process of modification of surface data for purposes of numerical analysis or data presentation. Examples include high or low pass filters and plane-fitting.

3.1.7 *gold ruled grating*—gold surface having uniformly spaced grooves of known depth and separation; used for micrometer scale *x-y* calibration.

3.1.8 *illuminated surface*—three-dimensional image representation that simulates a reflective surface illuminated obliquely or from overhead.

3.1.9 *image*—surface topography represented by plotting feature height as a function of tip position. The feature height data is derived from the amount of tunneling current flowing between the tip and surface during rastering.

3.1.10 *line plot*—three-dimensional image given as side-by-side surface profiles.

3.1.11 *mean roughness (R_a)*—average deviation from the mean of all profile heights (see algebraic definition in the Calculation Section).

3.1.12 *peak*—highest point between two crossing points of a profile and its center line.

3.1.13 *profile*—the cross-sectional data that has been high pass filtered with a two-pole filter having a gain of 75 % at the cutoff length l_c (in nanometers).

3.1.14 *raster*—repetitive scanning in the x-direction while moving stepwise in the *y*-direction; also the area defined by such action.

3.1.15 *scan*—a single, continuous movement in one direction (defined as the *x*-direction) of the tip relative to sample surface.

3.1.16 *scan area*—area covered by successive, side by side scans.

3.1.17 *scan length*—distance from start to end of a single scan, without moving in the y-direction (see cutoff length).

3.1.18 *scan rate*—the speed at which the tip moves relative to the surface.

3.1.19 *shaded height plot*—image representing feature height as dark or light shades (any color) over a two-dimensional area. Higher features are shaded lighter and lower features are shaded darker.

3.1.20 *thermal drift*—movement of the surface with respect to the tip due to a lack of thermal equilibrium.

3.1.21 *tilted surface*—three-dimensional image showing surface tilted away from viewer, as opposed to a topview.

3.1.22 *tip crash*—touching of tip to surface, during rastering or attempts to initiate tunneling, usually resulting in damage to one or both.

3.1.23 *top view*—image represented as a surface viewed from overhead.

3.1.24 *tunneling*—in this context, the flow of current between the tip and surface (see **current**); more discussion can be found in additional references.[4]

3.1.25 *valley*— lowest point between two crossing points of a profile and its center line.

3.1.26 *voltage*—bias voltage, expressed in volts (V) or millivolts (mV), applied between the tip and the surface.

3.2 *Abbreviations and Symbols:*

3.2.1 *HOPG*—highly ordered pyrolytic graphite; used for atomic scale *x-y* calibration of the scanning tunneling microscopy.

3.2.2 *STM*—scanning tunneling microscopy (or microscope).

3.2.3 *nA*—nanoamperes (1×10^{-9} amperes).

3.2.4 *Pt/Ir*—platinum and iridium alloy wire used to make tunneling tips.

3.2.5 R_a—see mean roughness.

3.2.6 R_{max}—maximum height difference between the highest and the lowest points on the profile over the length of the profile (see Calculation Section).

3.2.7 *root mean square (RMS)*—see algebraic definition in Calculation Section.

3.2.8 R_z—the 10-point mean roughness; that is, the average difference in height between the five highest peaks and the five lowest valleys over the length of the profile (see Calculation Section).

3.2.9 *x-direction*—see *scan*.

3.2.10 *y-direction*—the direction, in the sample plane, over which successive scans are taken, orthogonal to the scan direction.

3.2.11 *z-direction*—the direction perpendicular to the sample plane. Also referred to as the feature height direction.

3.2.12 Z_i—same as feature height (see Calculation Section).

3.2.13 Z_{max}—maximum height difference over entire surface (see Calculation Section).

3.2.14 Z_{rms}—root-mean-square of all surface heights (see Calculation Section).

4. Summary of Test Method

4.1 In this test method a sharp, conductive tip is scanned over very closely but not in contact with a conductive surface; that is, they are separated by a gap of several angstroms. A bias voltage present between them causes a flow of electrons through, rather than over, the energy barrier represented by this tip-surface gap. This flow is referred to as the tunneling current. The manner in which the current fluctuates during the scanning process is used to indicate the surface's topography. Though the tip or sample can be scanned, the method described here considers only the tip to be in motion. A more extensive discussion of the operating principles can be found in other literature.

[4] Binning, G., et al., "Surface Studies by Scanning Tunneling Microscopy," *Physical Review Letters,* Vol 49, No. 1, July 1982, pp. 57–61.

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4.2 In this test method, stainless steel tubing is used as an example of a component surface. An area of the surface is first scanned at a width of 500 nA, then 2000 nA. Even though larger areas can be scanned by most instruments, these magnifications are chosen to show surface texture in a size range beyond that measured by contact stylus type surface profiling instruments, but not at an atomic scale. The surface scans are then compared for damage, artifacts, etc. Numerical analysis can then proceed using these data for roughness or surface area or both, following the model of other standards such as ANSI B46.1.

5. Significance and Use

5.1 The use of STM images and data is for purposes of textural quality assessment and calculation of figures of merit, and for high purity gas system clean room components.

5.2 This test method defines a standard data presentation format and suggests figures of merit that utilize STM's ability to analyze three-dimensional surface features.

6. Interferences

6.1 Some (stainless steel) component surfaces have an oxide layer that prevents tunneling from occurring under any conditions without affecting tip or surface morphology. This results in ambiguous surface data. Such surfaces require the use of other techniques for topographic measurement.

6.2 This test method assumes that the images obtained are unperturbed by very thin, non-solid layers (for example, hydrocarbons, moisture) on the surface.

6.3 Operation with the surface in air, vacuum, or under inert liquids is permissible. (The liquids must be suitably inert and fluid, so as to not modify the apparent surface topography or introduce artifacts into the image.) Water is not recommended.

6.4 The tip shall be made from platinum/iridium or tungsten.

NOTE 1—**Caution:** The tip must not have previously touched any surface (see 11.7).

7. Apparatus

7.1 *Scanning Tunneling Microscope* , capable of the following may be used:

7.1.1 Scanning lengths up to at least 50 µm,

7.1.2 Substaining ± 3 V between the tip and sample,

7.1.3 Monitoring tunneling current as low as 0.05 nA,

7.1.4 Traversing feature height variations as great as 2 µm without touching the tip to the surface, and

7.1.5 Providing the surface topography as a shaded height plot or line plot.

7.2 Inert atmospheres, temperature controls, acoustic isolation, and vibration isolation are to be provided as necessary to obtain artifact-free images.

8. Sampling

8.1 Many components are too large or irregularly shaped to permit STM analysis without cutting a sample from the component. Low speed cutting, preferably without lubricants, using a diamond blade saw is recommended over high speed abrasive cutting or hacksaws.

8.2 This sampling must not modify the surface topography, such as effects due to stress, heat, corrosion, or combination thereof, from its condition as found in the component.

8.3 Cleaning the surface using an inert fluid to remove cutting contamination is permitted.

9. Calibration

9.1 Calibration frequency may vary with different instrument manufacturers. It should be performed, at least initially, then yearly, and after any repair or addition to the instrument's hardware and software.

9.2 Following the manufacturer's recommendations, the STM will be calibrated using the HOPG, gold ruled grating, or some other suitable dimensional standard, depending upon the size range to be used and the accuracy of the standard.

10. Conditioning

10.1 A conductive path for the sample shall be provided for voltage biasing of the sample with respect to the tip.

10.2 Mount the sample so the tip will scan an arbitrarily chosen representative area.

10.3 Bring the sample and microscope to thermal equilibrium.

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11. Procedure

11.1 As stated in 1.2.3, this test method does not cover steels that have an oxide layer too thick to permit tunneling under the test conditions outlined in this test method.

11.2 Make sure that a minimum of 200 data points is collected in the x-direction, and at least 200 scans per raster is in the y-direction ($200 \times 200 = 40\,000$ data points).

11.3 Initiate tunneling between a Pt/Ir tip and the sample in accordance with the manufacturer's instructions under conditions to provide artifact free images. Suggested starting values: voltage bias of 1800 mV, and current levels monitored at 0.1 nA (*Standard Test Conditions*—Room temperature and ambient pressure (101.3 kPa, 25° ± 2°C)).

11.4 Scan an area 500-nm across at a rate of approximately 2 s/µm (or slow enough to prevent the tip from touching the surface during rastering), collecting at least 200 data points with each *x*-direction scan.

11.5 Collect area scans so that comparisons can be made between the first two successive rasters. Ensure that the area has not drifted more than 10 % of the scan width in any direction. If it has, discontinue scanning for sufficient time to allow thermal equilibrium to be obtained. Determine this by repeating 11.3 through 11.5.

11.6 Scan the selected 500-nm area for at least five full, successive rasters and then store at least the last (fifth) image of that scan area. Fig. 1, Fig. 2, and Fig. 3 show an example of such an area after the fifth successive raster (see data presentation section for explanation of format shown).

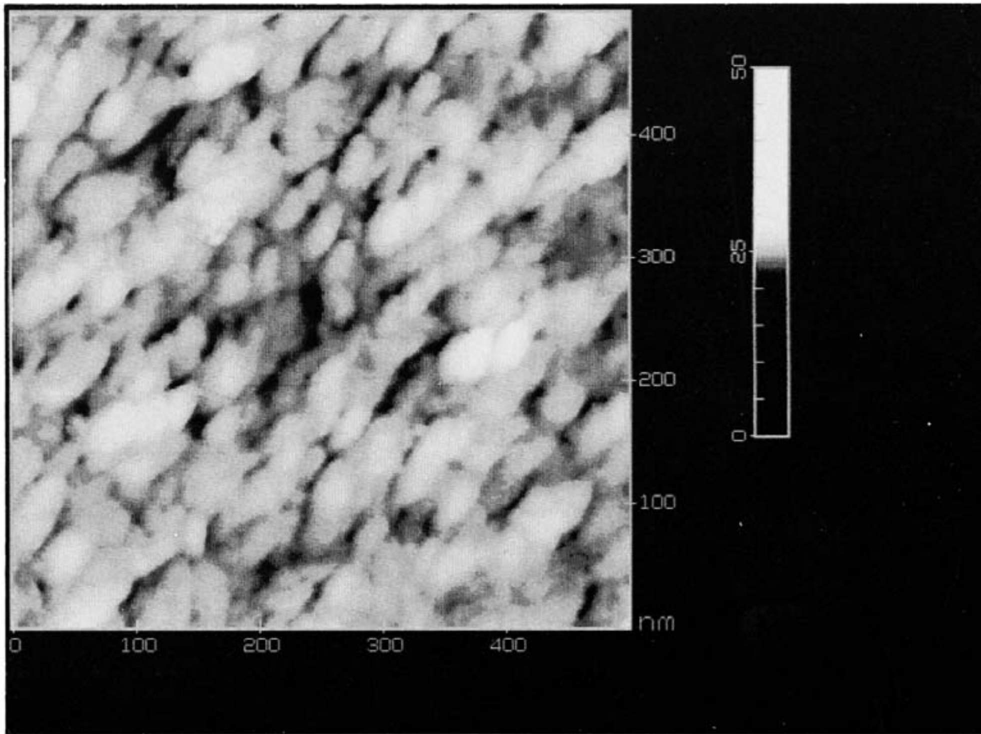

FIG. 1 Shaded Height Topview of Electropolished 316L Stainless Steel, 500-nm Across

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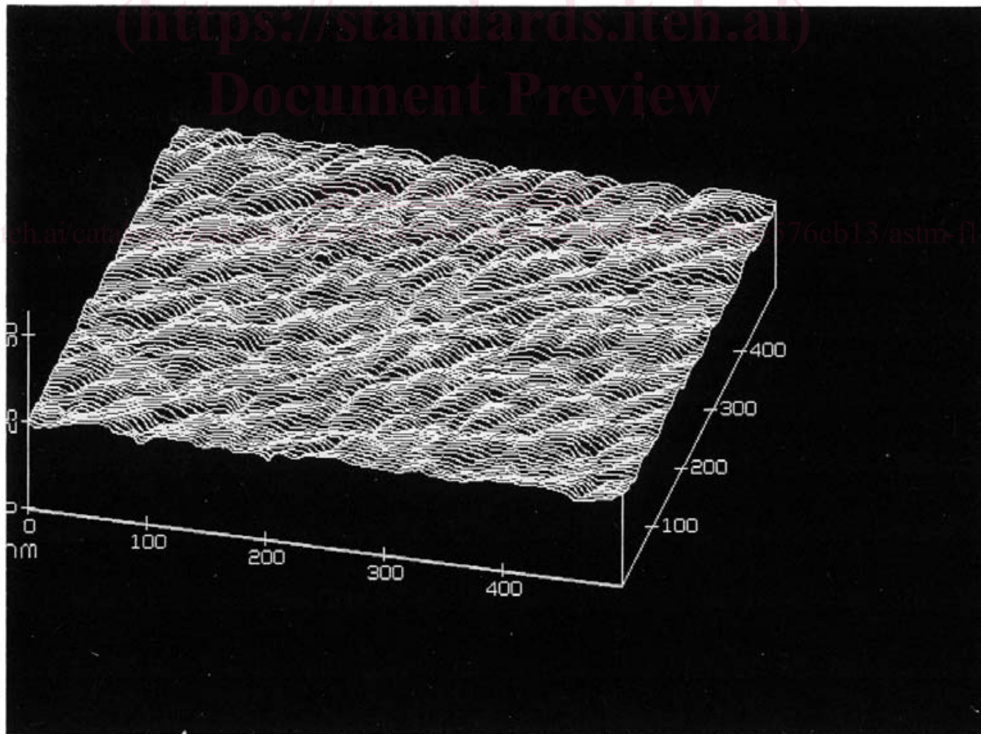

FIG. 2 Line Plot of Same Scan as Shown in Fig. 1

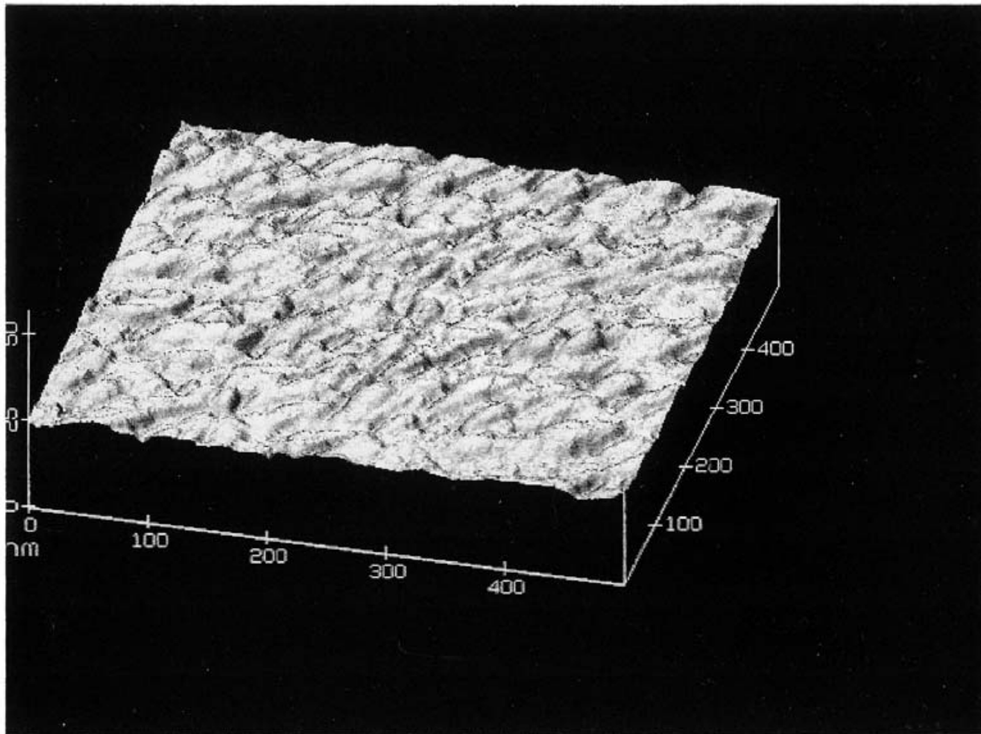

FIG. 3 Top-Illuminated (Tilted Surface) View of Same Scan as Shown in Fig. 1

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11.7 Inconsistent topography from one scan to the next is a sign of tip crashing. Fig. 4 shows an example of where the tip touched the surface during rastering.

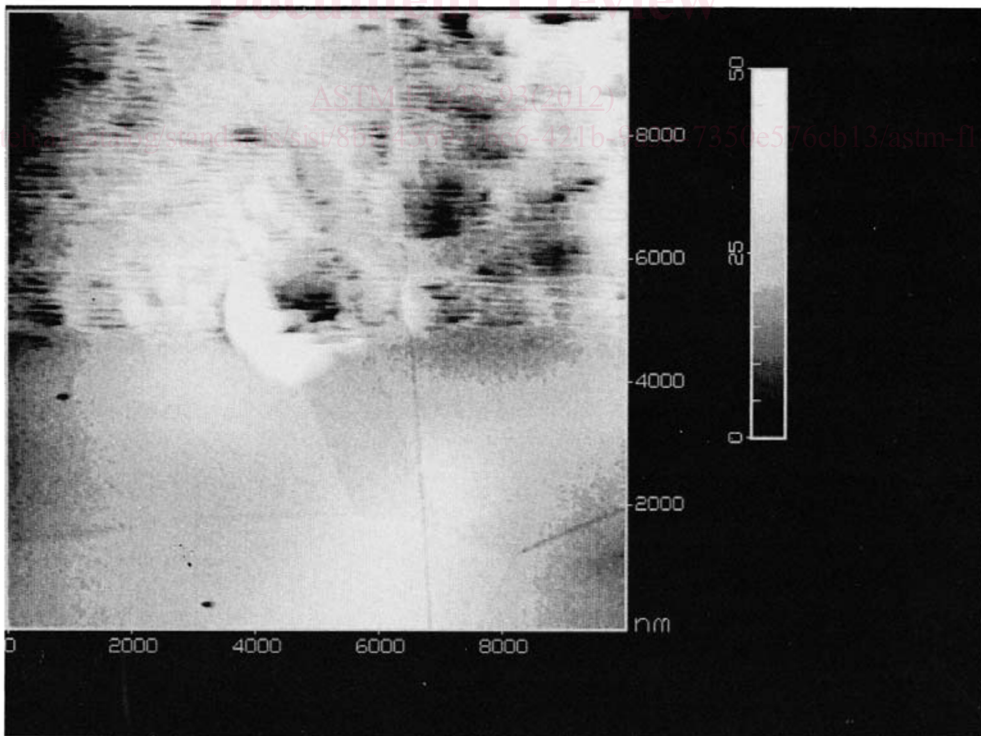

FIG. 4 Shaded Height of Topview of Stainless Steel Surface Showing Example of Tip "Crashing" Artifact (Upper Portion is Distorted Due to Damaged Tip.)

11.8 Change the scanned width to 2000 nm across, centered around the same region, and immediately obtain an image of that area. Fig. 5, Fig. 6, and Fig. 7 show an example of a 2000-nm wide area.

11.9 Observe whether the 2000-nm image shows evidence of damage from scanning the previous 500-nm (smaller) area. This generally appears as a 500-nm square of very different topography or height near the center of the 2000-nm image. If there is such evidence, the surface may be too oxidized to analyze (see 6.1). Fig. 8 shows an example of damage apparent at 2000 nm, which occurred during the 500 nm-wide rasters.

11.10 If no scanning damage is apparent, store the data for the 2000-nm area.

11.11 Disengage tunneling, relocate the tip or sample to a different surface area, and repeat 11.4 through 11.10 using the best imaging conditions. Make sure that the distance moved is at least far enough to prevent reanalyzing a portion of the previously scanned areas.

11.12 If there is any uncertainty about tip artifacts or tip-crashing, replace the tip and repeat 11.3 through 11.10.

11.13 Calculations of figures of merit (see Calculation Section) should utilize data from at least three different areas of the same scan width. If such figures are required, analyze additional areas in accordance with 11.3 through 11.10.

12. Calculations

12.1 Figures of merit can be calculated from the surface area or surface profiles data.

12.2 Surface data consists of successive scans across an area. Profiles extracted from the surface data that cross these scans may contain artifacts due to slight discontinuities from scan to scan, which may appear as small peaks in the profile. If these artifacts are present, then use only profiles taken in the scan direction.

12.3 The results of numerical analysis of the data may include the following, as defined in 12.10.1 and 12.10.2, in tabular form:

$$R_a, R_{max}, \text{ and } R_z \text{ (from profiles).}$$

$$Z_{max}, \text{ and } Z_{rms} \text{(from surface areas).}$$

12.4 As stated in the profile definition, the surface profile (cross-sectional data) must be cutoff filtered before R_a or R_z can be determined.

12.5 Calculate profile figures of merit (R_a and R_z) from data for five profiles each on at least three different areas on the same sample surface (for a total of 15 profiles of a single scan length). Also report a 95 % confidence limit of the mean in the table, along with the profile length in nanometers.

12.6 Calculate area figures of merit (Z_{max} and Z_{rms}) from data for at least three different areas on the same sample surface. The average of at least three such areas will be the reported values in the table.

12.7 Table 1 shows results for the surfaces shown in Fig. 1, Fig. 5, and Fig. 9.

12.8 The units for each will be reported in nanometers.

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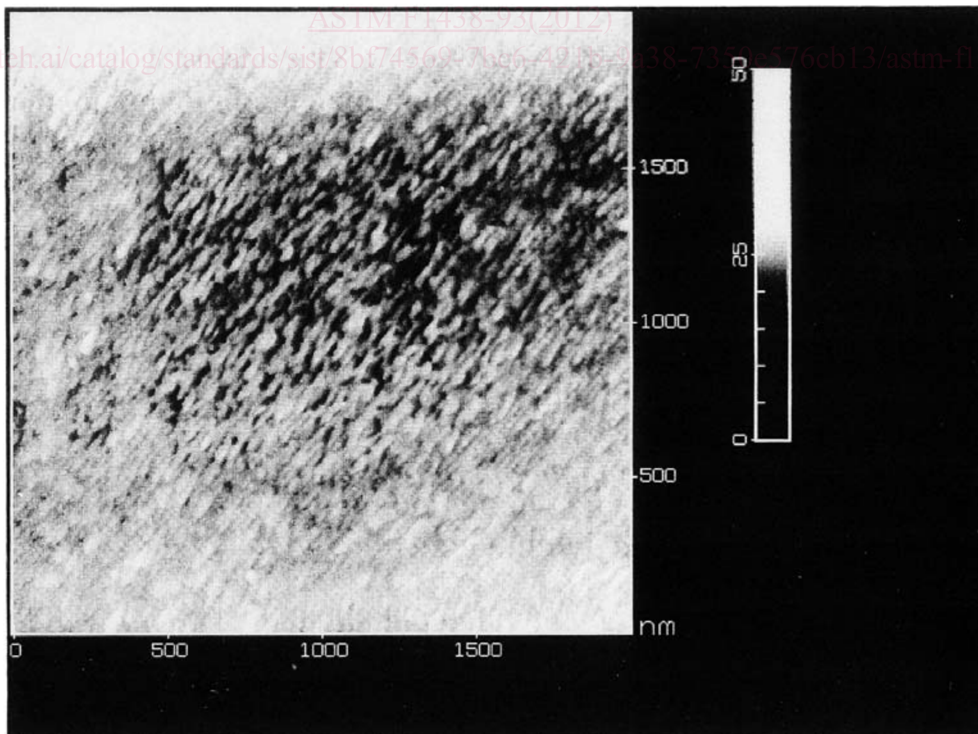

FIG. 5 Shaded Height Topview of Electropolished Stainless Steel, 2000-nm Across