

SEMI C78-0113 (Reapproved 1218)

TEST METHOD FOR DETERMINING ROUGHNESS OF POLYMER SURFACES USED IN ULTRAPURE WATER AND LIQUID CHEMICAL DISTRIBUTION SYSTEMS BY ATOMIC FORCE MICROSCOPY

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NOTICE: This Document was reapproved with minor editorial changes.

1 Purpose

1.1 This Document provides a test method for determining the roughness of polymer surfaces using atomic force microscopy (AFM) in intermittent contact mode.

1.2 In microelectronics, the need to accurately determine roughness of polymer surfaces is important when evaluating fluid handling system components from various sources. Rough surfaces have been known to contribute to the shedding of particles, encourage the harboring of micro contamination and promote the proliferation of bacteria. This method can also be used for other applications outside of microelectronics.

2 Scope

2.1 The use of AFM for measuring the roughness of polymer surfaces is outlined in this test method. AFM uses a stylus or probe tip that interacts with the surface of interest and moves at a known speed across the surface while recording the up and down movement of the tip relative to the surface, to image the sample topography.

2.2 Values of the image obtained using this method are reported in Ra, arithmetic mean deviation of the assessed profile and Rq, root mean square deviation of the assessed profiles in nanometers. Additionally, the sum of the peak height Zp and the valley depth Zv are reported.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 This method differs from contact profilometry in that AFM measures numerous single profiles to create an image of the surface.

3.2 This method uses intermittent contact as a means of measurement, which is also called amplitude modulation or TappingMode™.¹ In contact mode, large lateral forces may cause sample deformation; particularly in polymer samples. Contrary, intermittent contact mode practically eliminates these lateral forces.

3.3 Strict adherence to the conditions specified within this method must be followed to obtain comparable results with other laboratories. For example, changes to instrument set-up conditions may accidentally omit subtle features in the surface.

3.4 Surface roughness measurements of extruded materials of perfluoroalkoxy (PFA), polyvinylidene fluoride (PVDF), ethylene chlorotrifluoroethylene (ECTFE) and polypropylene (PP) have all been validated with this method between 5 different laboratories. However, this method has not been validated for sintered materials like polytetrafluoroethylene (PTFE). It is up to the user of this document to determine suitability in the testing of alternative materials.

3.5 This method is not intended for use with determining surface roughness on metallic surfaces. For such measurements refer to ASME B46-1.

3.6 This method is limited to surface roughness larger than 5 nm, when considering the arithmetical mean deviations of the assessed profiles, otherwise commonly known as Ra. Additionally, the analyst shall be aware of some

¹ TappingMode is a trademark of Bruker Corporation.

limitations in the maximum vertical range of the AFM. For example, if the sample's curvature radius is particularly small, then the maximum vertical range may be exceeded during the scan. More information concerning the maximum vertical range can be found in ¶ 8.5.

3.7 Surface roughness is a function of material processing conditions. Therefore, different results might be obtained for the same material.

4 Referenced Standards and Documents

4.1 ASME Standard²

ASME B46.1 — Surface Texture (Surface Roughness, Waviness, and Lay)

4.2 ISO Standard³

ISO 4287 — Geometrical Product Specifications (GPS) – Surface Texture: Profile Method – Terms, Definitions and Surface Texture Parameters

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *AFM* — atomic force microscopy

5.2 Definitions

5.3 *A* — oscillation amplitude while imaging the sample, in nanometers (nm) measured at the same frequency as the free oscillation amplitude.

5.4 *atomic force microscopy (AFM)* — a sensitive laboratory instrument typically requiring a high skill level that can reconstruct a three-dimensional image of a surface based on the mechanical interaction of a nanometer sized probe tip, using very small forces and small scan areas. Typical scan areas range from 500 nm to 100 µm. Contact forces routinely range from 1 n Newton to 5 µ Newtons.

5.5 *A0* — free oscillation amplitude in air (e.g., when the tip is not in contact with the sample), in nanometers (nm) measured at or up to a maximum of 5% less than the resonance frequency.

5.6 *defect* — a pattern that is not repeated, such as a scratch, dimple, void, pit, crack, etc.

5.7 *relative setpoint* — this is a value, which equals the ratio of the setpoint amplitude (*A*) during scanning and the free oscillation amplitude, or the ratio of *A/A0* measured at the same frequency as described in ¶ 5.3.

5.8 *scan rate* — user selectable parameter, which determines how many scan lines in the fast scan direction are completed by the scanner per second.

5.9 *scanner* — piezoelectric ceramic device that either drives the movement where the sample stage moves, and the tip is stationary or the converse.

5.10 *maximal vertical range* — AFM supplier specified maximum vertical travel distance of the scanner in nanometers or micrometers.

5.11 *fast scan direction* — direction of the scanner motion, which progresses at the speed of the scan rate. The tip is moved by a distance equal or very close to the scan size along the fast scan direction to complete a scan line.

5.12 *slow scan direction* — direction of the scanner motion perpendicular (ortho scanning) or nearly perpendicular (raster scanning) to the fast scan direction.

5.13 The following terminology is defined in ISO 4287 for line profiles. The same definitions apply to each scan line (which can be thought of as a profile line captured with a contact profilometer) of an AFM image.

² American Society of Mechanical Engineers, Three Park Avenue, New York, NY 10016-5900, USA; Telephone: +1.973.882.1170, Fax: +1.973.882.1717, <https://www.asme.org>

³ International Organization for Standardization, ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland; Telephone: +41.22.749.01.11, <http://www.iso.org>

- 5.13.1 Ra — the arithmetical mean deviation of all scan lines (profiles).
- 5.13.2 Rq — the root mean square deviation of all scan lines (profiles).
- 5.13.3 *mean plane* — arithmetic average of all points of an image.
- 5.13.4 Zp — absolute value of the distance between the highest measured point of an AFM image and the mean plane.
- 5.13.5 Zv — absolute value of the distance between the lowest measured point of an AFM image and the mean plane.

6 Sample Preparation

6.1 Remove the component from its packaging and randomly choose a location in the internal flow path area to be measured for surface roughness.

6.2 Prepare the sample in a way that neither damages the surface of interest nor leaves excess particles in the sample area. An effective method for sample preparation is to immerse the piece into liquid nitrogen for approximately 10 minutes and freeze fracture it using an impact technique. This creates smaller shards of plastic that can be easier to handle.

NOTE 1: With AFM there are limitations to sample orientation and probe agility. Therefore, samples must be obtained in a manner that neither damages the surface of interest nor allows excessive particles to remain in the sample area. Cutting a plastic sample to size with a saw, for example, may result in particle buildup on the surface, which could interfere with the roughness measurement reading.

NOTE 2: AFM stages may have a thickness limitation. Since larger diameter conduits will have thicker walls, the backside wall thickness may need to be reduced by machine milling or other appropriate means before obtaining the final sample. Care should be taken that the sample surface is neither deformed nor damaged during sample prep. For example, fingerprints can leave residues that can interfere with the measurement.

6.3 Upon obtaining a representative piece, view the sample surface with an optical microscope at 50× magnification and identify an area that does not show any surface damage or particulate contamination or either.

6.4 If particles can be seen at 50× then use filtered clean dry air or nitrogen to remove them. In the case of stubborn particles, it may be necessary to wash them away with reagent grade solvent that is compatible with the material being measured. In either case, reexamine the sample at 50× magnification. If particles are still visible, correct the reasons for the contamination, such as using a solvent that will not alter the surface and proceed until a clean sample surface is obtained for analysis. This may require obtaining another shard. If it is impossible to obtain a clean sample, then stop the preparation and note the condition on the report and return the sample to the requester.

NOTE 3: Smaller particles or other types of contamination may not be visible until the initial scan lines of AFM are collected. For example, polypropylene and other materials with additives may be subject to streaking that could interfere with the roughness result. In these cases, sonication can be used if it is known not to alter the surface of the sample.

7 Instrument Set-Up (AFM)

7.1 The instrument shall be sufficiently isolated from vibration, which will artificially increase roughness measurements.

7.2 Verify that the AFM supplier recommended scanner recalibration procedures have been followed to ensure accurate height and lateral distance readings. Modern AFM instruments may not require scanner recalibration at all. In any case it is good practice, but not required by this Standard, to verify accuracy of height and lateral measurements using reference samples from industry recognized sources. Since scanner nonlinearities scale with distance it is recommended, if reference samples are used, to determine accuracy at length scales (both lateral and vertical) as close as possible to those encountered for the actual test samples.

7.3 Set up the AFM using the parameters summarized in Table 1.

7.4 The amplitude may be estimated from the cantilever deflection and the known displacement of the piezo at a fixed lateral position using a very stiff and flat sample such as silicon or diamond. Alternatively, the AFM supplier recommended amplitude and setpoint settings in volts or nanometers for imaging polymers may be used.