

SEMI C87-0515

TEST METHOD FOR DETERMINING ROUGHNESS OF POLYMER SURFACES USED IN ULTRAPURE WATER AND LIQUID CHEMICAL DISTRIBUTION SYSTEMS BY CONTACT PROFILOMETRY

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1 Purpose

1.1 This Document provides a test method for determining the roughness of polymer surfaces using contact profilometry (CP).

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 CP for measuring the roughness of polymer surfaces is outlined in this Test Method. CP 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 profile the sample topography.

2.2 Values of the scan obtained using this method are reported in Ra and Rq. Refer to ISO 4287 for information about these terms.

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 atomic force microscopy (AFM) in that CP generally measures a single line profile. At the same time, CP exerts a higher pressure on the surface than AFM and the minimum detectable patterns are not as fine as for AFM.

3.2 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.

3.3 A CP method is already approved for metallic surfaces, known as ANSI/ASME B46-1. For polymer surfaces the method referred to in this document shall be used. Particularly soft polymers, for which no experimental validation of the method was made, shall be excluded.

3.4 Depending on the type of instrument used (high end/low cost) the smallest vertical difference and height to be measured is <1 nm to 10 nm. Differences in noise will also be a factor. Refer to the supplier's manual for details. Additionally, the analyst shall be aware of some limitations in the maximum vertical range of the CP. For example, if the sample's curvature radius is particularly small, then the maximum vertical range may be exceeded during the scan.

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

3.6 If the surface roughness is expected to fall outside of a range of 0.02 to 2.0 μm then use other parameters that are called out in ISO 4288 and ISO 3274.

4 Referenced Standards and Documents

4.1 ISO Standards¹

ISO 3274 — Geometrical Product Specifications (GPS) – Surface Texture: Profile Method – Nominal Characteristics of Contact (Stylus) Instruments

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

ISO 4288 — Geometrical Product Specifications (GPS) – Surface Texture: Profile Method – Rules and Procedures for the Assessment of Surface Texture

4.2 ASME Standard²

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

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

5 Terminology

5.1 Definitions

5.1.1 *axial direction* — obtaining a scan along the length of a component, such as the extrusion direction of a piece of pipe. See Figure 1.

5.1.2 *contact profilometry (CP)* — an instrument used to measure a surface's profile in order to quantify its roughness. A diamond (or sapphire) stylus moves vertically while in contact with a sample as either the stylus or the sample are moved laterally for a specified distance and with a specified contact force.

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

5.1.4 *filter cutoff wavelength or cutoff wavelength (λ_S)* — refer to ISO 3274 for information about this term.

5.1.5 *first order flattening* — a technique to eliminate the effect of sample tilt on surface roughness calculations. The technique consists of fitting the data using a first order polynomial and subtracting the best fit line from the data. A first order polynomial is an equation of the type $A + B \times x$ in which A and B are constants and x is the position on the line along which data are collected during a measurement. The best fit line is established through least square fitting (see Appendix 2).

5.1.6 *maximal vertical range* — CP supplier specified maximum vertical travel distance of the stylus in nanometers or micrometers

5.1.7 *roughness cutoff wavelength (λ_C)* — refer to ISO 3274; also known as Roughness Sampling Length (L_r) as referred to in ISO 4288. This is the length in the direction of the X-axis used for identifying the irregularities characterizing the profile under evaluation. See Figure 2.

5.1.8 *roughness evaluation length (L_n)* — refer to ISO 4288 for information about this term. This is the length in the direction of the X-axis used for as the profile under evaluation.

5.1.9 *radial direction* — obtaining a scan perpendicular to the length of a component, such as 90° to the extrusion direction of a piece of pipe. See Figure 1.

5.1.10 *static measuring force* — this is the normal force applied to the stylus tip.

5.2 The following terminology is defined in ISO 4287 for line profiles.

5.2.1 *Ra* — the arithmetical mean deviation of all scan lines (profiles). Refer to ISO 4287 for information about this term.

¹ International Organization for Standardization, ISO Central Secretariat, 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland; Telephone: 41.22.749.01.11, Fax: 41.22.733.34.30, <http://www.iso.org>

² American Society of Mechanical Engineers, Three Park Avenue, New York, NY 10016-5990, USA; Telephone: 973.882.1170, Fax: 973.882.1717, <https://www.asme.org>

5.2.2 R_q or RMS — the root mean square deviation of all scan lines (profiles). Refer to ISO 4287 for information about this term.

5.2.3 Z_p — the distance between the mean roughness baseline (see Figure 2) and the highest point of the profile peak.

5.2.4 Z_v — the distance between the mean roughness baseline (see Figure 2) and the lowest point of the profile valley.

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.

6.3 With CP 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 1: CP 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.

6.4 Upon obtaining a representative piece, choose a random area and view the sample surface with an optical microscope at 10× magnification. If the area shows any particulate contamination then choose a new area. If it is not possible to obtain a measurement due to particulate contamination then re-clean the sample.

6.5 If particles can be seen at 10× 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 10× 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 sample. 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.

7 Instrument Set-Up

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

7.2 Verify that the CP supplier recommended recalibration procedures have been followed to ensure accurate height and lateral distance readings. In any case it is good practice, but not required by this Standard, to verify accuracy of height measurements using reference samples from industry recognized sources.

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

NOTE 2: See Limitations, ¶ 3.6.

7.4 Methodology

7.4.1 Flat Samples

7.4.1.1 Mount the sample onto the CP sample holder such that all regions of interest of the sample can be measured.

7.4.1.2 Acquire a minimum of three scans from the same sample at randomly chosen, non-overlapping areas, within the surface area, which was identified in ¶ 6.3.

7.4.1.3 If it is not possible to perform the measurement on an area due to vertical limitation of the instrument, it shall be noted on the report form located in Appendix 1.

7.5 Curved Samples — Pipe, Fittings, Valves.

7.5.1 Mount the sample onto the CP sample holder such that sample tilt along lines perpendicular to the natural curvature of the conduit sample is minimized (see Figure 1) and that its identifying markings are up (zero degrees).