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Standard Test Method for Measurement of Percent Crystallinity of Polyetheretherketone (PEEK) Polymers by Means of Specular Reflectance Fourier Transform Infrared Spectroscopy (R-FTIR)[1]

This standard is issued under the fixed designation F2778; 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.

1. Scope

1.1 This test method describes the collection of absorption spectra of polyetheretherketone (PEEK) polymer in filled and unfilled grades, as supplied by a vendor, and the subsequent calculation of the percent crystallinity. The material is evaluated by infrared spectroscopy. The intensity (height) of the absorbance peaks is related to the amount of crystalline regions present in the material.

1.2 This test method can be used for PEEK consolidated forms, such as injection molded parts, as long as the samples are optically flat and smooth.

1.3 The applicability of the infrared method to industrial and medical grade PEEK materials has been demonstrated by scientific studies.[2,3] Percentage of crystallinity is related to R-FTIR measurement by calibration through wide-angle x-ray scattering (WAXS) crystallinity measurements.[2,3] It is anticipated that this test method, involving the peak heights near 1305 cm^{-1} and 1280 cm^{-1}, will be evaluated in an Interlaboratory Study (ILS) conducted according to Test Method E691.

1.4 This test method does not suggest a desired range of crystallinity for specific applications.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 This standard may involve hazardous materials, operations, and equipment. *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:*[4]

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *crystallinity index (CI),, n*—the ratio of the height between the absorption peaks 1305 cm^{-1} and 1280 cm^{-1}.

4. Significance and Use

4.1 Mechanical properties of PEEK, such as stiffness or yield strength, are influenced by the level of crystallinity.[5] The reported crystallinity index determined by this test method has been correlated with percent crystallinity in PEEK by wide-angle X-ray scattering (WAXS) experiments.[2,3]

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[1] This test method is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

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[2] Chalmers J. M., Everall, N. J., Hewitson, K., Chesters, M. A., Pearson, M., Grady, A., Kuzicka, B., "Fourier Transform Infrared Microscopy: Some Advances in Techniques for Characterisation and Structure-Property Elucidations of Industrial Material," *The Analyst*, Vol 23, 1998, pp. 579–586.

[3] Jaekel, D. J., Medel, F. J., Kurtz, S. M., "Validation of Crystallinity Measurements of Medical Grade PEEK Using Specular Reflectance FTIR-microscopy," *Society of Plastics Engineers Annual Technical Conference 2009*, Chicago 2009, Manuscript ID ANTEC-0248-2009.

[4] 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.

[5] Kurtz, S. M., Devine, J. N., "PEEK biomaterials in trauma, orthopedic, and spinal implants," *Biomaterials* , Vol 28, No. 32, 2007, pp. 4845–4869.

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4.2 This test method may be useful for both process development, process control, product development, and research.

5. Interferences

5.1 Samples must be smooth and optically flat over the area of investigation, typical of injection-molded specimens. They must be sufficiently thick (for example, 1 to 2 mm) such that there is no detectable back surface reflected radiation.

5.2 Samples must be sized appropriately to be accommodated in the FTIR apparatus.

6. Apparatus

6.1 *Infrared Spectrometer:*
6.1.1 An infrared spectrometer capable of recording a reflection absorption spectrum over the range of 500 cm^{-1} to 1500 cm^{-1} is necessary.
6.1.1.1 A minimum scan resolution of 4 cm^{-1} shall be used.
6.1.1.2 A minimum aperture of 360 × 360 µm shall be used.
6.1.1.3 A minimum of 100 scans shall be collected per spectrum.
6.1.2 Software capable of using the Kramers-Kronig transform algorithm to derive the absorbance spectra.

6.2 *Specimen Holder*—Equipment capable of accurately positioning the sample under the orifice and allowing the sample to be in focus.

6.3 *Samples Preparation Equipment*—Equipment capable of producing optically flat and optically thick samples.

7. Preparation of Apparatus

7.1 Prior to testing, the FTIR dewer chamber should be filled with liquid nitrogen until peak-to-peak signal in transmission mode is over 10.

8. Calibration and Standardization

8.1 Validation and calibration should be conducted weekly by running a validation check of the infrared spectrometer based on manufacturer's instructions.

8.2 A background scan should be conducted at the start of testing and every 30 min thereafter for the duration of testing while using the same scanning settings as the test parameters dictate. The background scan should be taken from a completely reflective surface.

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9. Procedure

9.1 Check settings to ensure you are in reflectance mode, collecting at 100 scans per spectrum with a resolution of 4 cm^{-1}, an aperture of 360 × 360 µm, and the spectrometer is set to use a Kramers-Kronig transform algorithm.

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9.2 Place and secure the sample in the holder so that it appears level and flat.

9.3 Through the microscope, focus the view on the surface of the sample.

9.4 Samples can continue to be tested as long as a new background is collected every 30 min.

10. Calculation or Interpretation of Results

10.1 *Crystallinity Index (CI):*
10.1.1 Open a spectrum data file, and restrict the absorption band range to 900 cm^{-1} and 1400 cm^{-1}.
10.1.2 Perform an automatic baseline correct of this spectrum range.
10.1.2.1 Determine a reference baseline for the height measurements from the zero value absorbance points on the spectrum as shown in Fig. 1. The first point for the baseline is the lowest absorbance value point between the spectra bands, 1340 to 1375 cm^{-1}, and the second point is lowest absorbance value point between 1000 cm^{-1} and 1080 cm^{-1}.
10.1.3 Calculate the heights of the peaks near 1305 cm^{-1} (HA) and 1280 cm^{-1} (HB) as shown in Fig. 1.
10.1.3.1 HA is the peak height corresponding to carbonyl linkages, while HB is primarily influenced by the diphenyl ether groups of the PEEK molecular chain. The bands influenced by the diphenyl ether groups (HB) are invariant to the level of crystallinity, whereas the absorption peak corresponding to carbonyl linkages (HA) increases with the level of crystallinity.[6]
10.1.4 CI shall be calculated by dividing the calculated height HA (peak height at 1305 cm^{-1}) by the calculated height HB (peak height at 1280 cm^{-1}) (Fig. 1):

$$CI = HA/HB \tag{1}$$

10.2 *Percent Crystallinity:*

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[6] Nguyen, H. X., and Ishida, H., "Molecular Analysis of the Melting Behavior of Poly(Aryl-Ether-Ether-Ketone)," *Polymer*, Vol 27, No. 9, 1986, pp. 1400–1405.