National Institute of Standards & Technology

Report of Investigation

Reference Material 8457

Ultra High Molecular Weight Polyethylene

0.5 cm Cubes

This Reference Material (RM) is an orthopedic grade ultra high molecular weight polyethylene (UHMWPE) that consists of 0.5 cm cubes, machined from the central portion of the 7.62 cm (3.0 inch) rod of RM 8456. The 0.5 cm cubes are intended for use as controls for evaluations of cross-linking wrought by exposure to ionizing radiation, in particular, to improve wear characteristics. Cubes of this size are useful for determination of cross-link density by swelling measurements. Similar cubes, also fabricated from RM 8456 bar stock, were used in an interlaboratory comparison of cross-link density measured by a new ASTM method.

This Reference Material (RM), fabricated from the cylindrical bar stock of NIST RM 8456, is intended for evaluations of cross-linking induced by radiation exposure, such as used to improve wear characteristics. Each unit of RM 8457 consists of 10 cubes of ultra high molecular weight polyethylene (UHMWPE) of nominal dimension 0.5 cm. The cubes are sized for measurement of cross-link density by swelling. The material as supplied has not been irradiated, however.

Reference Values: Reference values and uncertainties for dimensions of cubes and surface roughness are shown in Table 1. Reference values are noncertified values that are the best estimate of the true value; however, the values **DO NOT** meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty.

Information Values: Information values for degree of swelling in xylene at 130 ˚C obtained in a round robin test on gamma-irradiated cubes are given in Table 2. Values and uncertainties for mechanical properties of the bar stock, RM 8456, from which RM 8457 was machined are shown in Table 3. The reported mechanical properties are Young's modulus, yield strength, ultimate strength, and elongation. The composition trace elements and material properties of the bar stock from which the cubes were machined are given in Table 4. The values in Table 4 are based on manufacturer-supplied information on the composition and are considered to be information values. These are noncertified values with no reported uncertainties as there is insufficient information to assess uncertainties. The information values are given to provide additional characterization of the material and should not be used for calibration or quality control.

Expiration of Value Assignment: RM 8457 is valid, within the measurement uncertainty specified, until **01 January 2017**, provided the RM is handled and stored in accordance with instructions given in this Report of Investigation (see "Instructions for Handling, Storage, and Use"). This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of this reference material were under J.A. Tesk and B.M. Fanconi of the Polymers Division. This RM was fabricated by the NIST Manufacturing Engineering Laboratory.

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See Report Revision History on Last Page

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Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

The information regarding the source of the material and the information value reported in Table 4 were provided by R.K. Wilhelm of Poly Hi Solidur, Inc., Fort Wayne, IN.

Support aspects involved in the issuance of this RM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE[1]

Source of Material: RM 8457 cubes were fabricated from one of the cylindrical bars from which test specimens were prepared for mechanical measurements on RM 8456 [1]. Only the central 5.62 cm (2.21 in) of the bar's diameter was used to fabricate the cubes because mechanical properties outside this area were found to differ statistically [1]. The source material for RM 8456 was Premium Grade UHMWPE bars (Production Code PG9981) provided by Poly Hi Solidur, Inc., MediTECH Division Fort Wayne, IN (This material was produced from Virgin UHMWPE Raw Material (Lot No. 332945), TICONA GUR 1050).

Storage: Until required for use, RM 8457 should be stored at room temperature in its original container and not exposed to intense, direct light, or ultraviolet radiation.

Use: The reference properties, cube dimensions and surface roughness, represent mean values and expanded uncertainties.

Methods of Analysis Used in Value Assignment

The cubes of RM 8457 were fabricated from one of the cylindrical bars from which test specimens were prepared for mechanical measurements on RM 8456 [1]. Only the central 5.62 cm (2.21 in) of the bar's diameter was used to fabricate the cubes because mechanical properties outside this area were found to differ statistically [1]. Preliminary measurements had shown that the swelling of gamma-irradiated cubes is not truly isotropic, hence, to reduce measurement uncertainties it was necessary to maintain consistency in measuring swelling along the same direction for all cubes. The anisotropic behavior derives from molecular orientation imparted by the extrusion process used to produce the bar stock of RM 8456. Cubes were machined with four of the faces aligned along the axial direction; the machining produced linear striations in the axial direction on those four cube faces. Circular swirls appeared on the two faces of the cube that were perpendicular to the axial direction. These distinguishing surface features are evident under low magnification optical microscopy.

Cube Dimensions

The NIST Manufacturing Engineering Laboratory fabricated the cubes. After machining, the cubes were stored together in a plastic bag. Each cube was examined under an optical microscope for imperfections that resulted from the machining. Those that possessed burrs were discarded as well as those whose edges were not beveled. The three edge dimensions were measured with a micrometer on 40 cubes randomly selected from the remainder. Owing to the different machining processes used for faces, normal and perpendicular to the bar direction, the dimensions in the axial direction were separated from those perpendicular. The mean and expanded uncertainty of the dimension in the extrusion direction were (0.4919 ± 0.005) cm and (0.5017 ± 0.006) cm in the dimension perpendicular to the extrusion direction.

[1]Certain commercial equipment, instruments, or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Surface Roughness

Measurements of the surface roughness of the cubes were made with a Federal Products Surfanalyzer 5000™, using a probe having a range of ± 50 µm. The root mean square surface roughness (R_q) of the faces normal to the bar direction was found to be no more than 5.5 µm ± 0.6 µm (0.6 µm represents the 95 % confidence interval for 4 cubes, chosen at random, from which the measurements were made). The standard deviation of the measurements is 0.3 µm.

Table 1. Reference Values for Cube Dimensions and Surface Roughness

Cube Dimension	Dimension (cm)	Surface Roughness (µm)
Faces normal to bar direction	0.4919 ± 0.005	5.5 ± 0.6
Faces perpendicular to bar direction	0.5017 ± 0.006	

Table 2. Information Values for Cross-linking Caused by Gamma Irradiation [2] based on Round Robin Test Results

Summary of Mean (± S_R, Absolute Interlaboratory Uncertainty) Swell Ratio (q), Cross-link Density (υ_d), and Molecular Weight Between Cross-links (M_c) for the Four Sample Sets

Dose (kGy)	Swell Ratio (q)	Cross-link Density (υ_d) [mol/dm^3]	Molecular Mass (Weight) Between Cross-links (M_c) [g/mol]
54.2	3.37 ± 0.26	0.133 ± 0.017	7 650 ± 1 010
71.5	3.12 ± 0.24	0.151 ± 0.021	6 720 ± 920
89.2	3.12 ± 0.24	0.152 ± 0.020	6 700 ± 890
110.1	2.97 ± 0.31	0.170 ± 0.032	6 150 ± 1 190

Table 2 is based on a round robin study conducted in 2001 involving four sets of ultra high molecular weight polyethylene test samples tested by six laboratories [2]. For all sets of samples, all the specimens were prepared at the same time by the same laboratory. Each laboratory tested three specimens from each set of samples. Samples from NIST Reference UHMWPE, RM 8456, were machined into 5 mm cubes, packaged in nitrogen, and sample sets were irradiated with one of four gamma irradiation doses (54.2, 71.5, 89.2, and 110.1) kGy. The data indicate that for the range of irradiation doses currently used to produce highly cross-linked UHMWPE for orthopedic applications, the swell ratio measurement is associated with interlaboratory expanded relative uncertainty of 8 % to 11 %.

Table 3. Information Values for Mechanical Properties of RM 8456 [1]

Property	Mean	U	U [(a)]	Units
Young's Modulus	1 258	22	44	MPa
Yield Strength	23.56	0.33	0.66	MPa
Ultimate Tensile Strength	45.8	3.0	6.0	MPa
Elongation	460	20	40	% (Percent)

[(a)] The expanded uncertainty is computed as $U = 2u$ to approximate a 95 % confidence interval [3].

Table 4. Information Value for Total Trace Element Concentration

Total trace element concentration	46 mg/kg

REFERENCES

[1] Eichmiller, F.C.; Tesk, J.A.; Croarkin, C.M.; *Mechanical Properties of Ultra High Molecular Weight Polyethylene NIST Reference Material RM 8456*; Transactions of the Society for Biomaterials; p. 472, 27th Annual Meeting, St Paul, MN (April 2001).

[2] Spiegelberg, S.; Kurtz, S.; Muratoglu, O.; Greer, K.; Costa, L.; Wallace, S.; Cooper, C.; *Interlaboratory Reproducibility of Swell Ratio Measurements for Cross-linked Polyethylene*; 48th Annual Meeting of the Orthopedic Research Society; Dallas, TX (2002).

[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed June 2012); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed June 2012).

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