Reference Material 8457, Ultra High Molecular Weight Polyethylene 0.5 cm Cubes

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Introduction. Crosslink density is an important parameter that affects the wear resistance and mechanical properties of Ultra High Molecular Weight Polyethylene (UHMWPE). A new ASTM standard test method for determining the crosslink density of UHMWPE is in final revision. The method involves measuring dimensional changes in cubic samples while immersed in a swelling agent. NIST is providing its reference UHMWPE, RM 8456, in the form of 0.5 cm cubes to facilitate measurements using the new standard test method. The objective of this paper is to present the geometric properties of Reference Material 8457, Ultra High Molecular Weight Polyethylene 0.5 cm Cubes. These cubes, newly available from NIST, are for measurements of swelling which can be used to calculate crosslink density[1]. RM 8457 has not been crosslinked, however.

Materials and Methods. The cubes of RM 8457 were fabricated out of bars of UHMWPE RM 8456. The mechanical properties of RM 8456 were previously reported [2] and are given on the report of investigation that accompanies bars purchased from NIST. The UHMWPE used in RM 8456 and RM 8457 was donated by Poly Hi Solidur, Inc., MediTECH Division (Production Code PG9981, Premium Grade Ultra High Molecular Weight Polyethylene, Virgin UHMWPE Raw Material Lot No. 332945, source identified as TICONA GUR 1050 [##]). Spiegelberg et al.[1] used similar cubes, fabricated from the same bar as the cubes of RM 8457, to measure the volumetric swelling by in-situ measurements of the change in length between two opposing faces of the cubes. They calculated crosslink density by assuming isotropic swelling behavior. Crosslinking was induced by exposing the cubes to ionizing radiation. Preliminary measurements had shown that the swelling of the 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. The axial direction of the bars was chosen. 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 faces were the faces that are recommended for use in measurements of swelling and are the ones that were used by Spiegelberg et al[1]. Measurements of the surface roughness of the cubes were made with a Federal Products Surfanalyzer 5000[##], using an EPT-1040 probe having a probe range of ± 50 µm.

Results. The root mean square surface roughness (R_q) of the faces recommended for swelling measurements was found to be 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 0.2 µm. The cubes are nominally 0.50 cm on an edge. The mean and expanded uncertainty of the extrusion (axial) direction dimension was (0.4919 ± .005) cm and (0.5017 ± .006) cm in the perpendicular direction (the ± values represents a 95 % confidence interval). Edge dimensions were measured with a micrometer on 40 cubes randomly selected.

Discussion. RM 8457 is expected to exhibit identical response to gamma irradiation as the cubes of RM 8456 that were used in the round robin test evaluation of crosslinking[1]. The surface roughness of RM 8457 would have a negligible effect on the uncertainty of swell-ratio[1] measurements conducted using the new method. The method specifies the sensitivity of the measurement to be 1 % of the initial height of the sample (this is essentially the same as requiring a relative expanded combined uncertainty of no higher than 1 %). The root mean square surface roughness, 5.5 µm, corresponds to 0.1 % of the initial sample height of 0.5 cm. This relative uncertainty of 0.1 % provides a negligible contribution to an expanded combined uncertainty of 1 % for the measurement method. Further, an uncertainty analysis has shown that a relative sensitivity of 1 % will produce an expanded combined relative uncertainty of less than 10 % in crosslink density for samples that have swollen to a fraction 50 % beyond their initial height[3]. This is the amount of swelling found and, therefore, expected for samples exposed to typical irradiation doses used to improve wear[1]. Hence, a relative uncertainty of 0.1 % in surface roughness translates to only a 1 % uncertainty in crosslink density.

[##] Commercial products identified are neither endorsed by NIST nor claimed by NIST to be superior to others.

References

(1) Spiegelberg, S., Kurtz, S., Muratoglu, O., Greer, K., Costa, L., Wallace, S., Cooper, C., (2002). Interlaboratory Reproducibility of Swell Ratio Measurements for Cross-linked Polyethylene. 48th Annual Meeting of the Orthopedic Research Society, Dallas, TX
(2) Eichmiller, F.C.; Tesk, J.A. and 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, April 2001 St Paul, MN)
(3) Spiegelberg, S., private communication