Polymer Testing 27 (2008) 616–620

Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

Test Method

Evaluation of J-initiation fracture toughness of ultra-high-molecular-weight polyethylene used in total joint replacements

R. Varadarajan, C.M. Rimnac *

Musculoskeletal Mechanics and Materials Laboratories, Department of Mechanical and Aerospace Engineering, Case Western Reserve University, Cleveland, OH 44106, USA

ARTICLE INFO

Article history:
Received 22 February 2008
Accepted 25 March 2008

Keywords:
UHMWPE
Fracture toughness
CTOD
Total joint replacement

ABSTRACT

Fracture of ultra-high-molecular-weight polyethylene (UHMWPE) total joint replacement components is a clinical concern. Thus, it is important to characterize the fracture resistance of UHMWPE. To determine J-initiation fracture toughness (J_Q) for metals and metallic alloys, ASTM E1820 recommends a procedure based on an empirical crack blunting line. This approach has been found to overestimate the initiation toughness of tough polymers like UHMWPE. Therefore, in this study, a novel experimental approach based on crack tip opening displacement (CTOD) was utilized to evaluate J_Q of UHMWPE materials. J-initiation fracture toughness was experimentally measured in ambient air and a physiologically relevant 37 °C PBS environment for three different formulations of UHMWPE and compared to the blunting line approach. The CTOD method was found to provide J_Q values comparable to the blunting line approach for the UHMWPE materials and environments examined in this study. The CTOD method used in this study is based on experimental observation, and thus, does not rely on an empirical relationship or fracture surface measurements. Therefore, determining J_Q using the experimentally based CTOD method proposed in this study may be a more reliable approach for UHMWPE and other tough polymers than the blunting line approach.

1. Introduction

Fracture of ultra-high-molecular-weight polyethylene (UHMWPE) components which frequently compose one half of the bearing couple in total joint replacements is a clinical concern. Therefore, there has been much interest in the evaluation of the fracture toughness of different, clinically relevant UHMWPE formulations [1–5].

The fracture toughness, J_{IC}, for metals and metallic alloys can be determined based on the intersection of an empirical crack tip blunting line and the J–R curve (ASTM E1820 [6]). The blunting line relationship is given by

$$J = m\sigma_Y \Delta a \tag{1}$$

where m is the slope of the blunting line, σ_Y is the effective yield strength (an assumed value of uniaxial yield strength that represents the influence of plastic yielding upon fracture test parameters), and Δa is the crack extension. For tough polymers, Eq. (1) may overestimate the crack extension associated with crack tip blunting [7]. Therefore, for UHMWPE, a blunting line relationship based on ultimate tensile stress was recommended by Rimnac et al. [1].

Alternatively, J_{IC} can be determined using a procedure based on stretch zone width proposed by Kobayashi et al. [8]. In this method, the length of the stretch zone that appears during blunting is measured by microscopic examination of the fracture surface. J_{IC} is then determined using the following relationship:

$$J_{IC} = m\sigma_{flow} SZW \tag{2}$$

* Corresponding author. Tel.: +1 216 368 6442; fax: +1 216 368 3007.
E-mail address: clare.rimnac@case.edu (C.M. Rimnac).

0142-9418/$ - see front matter © 2008 Published by Elsevier Ltd.
doi:10.1016/j.polymertesting.2008.03.007

where m is the slope of the blunting line ($m = 2$), σ_{flow} is the stress midway between the yield stress and the ultimate tensile stress, and *SZW* is the stretch zone width.

Based on stretch zone width measurements, following the procedure recommended by Kobayashi et al. [8], Pascaud et al. [7] recommended the use of a blunting line for UHMWPE that utilized ultimate tensile strength [1] and maximum crack extension with a slope $m = 2$:

$$J = 2(UTS)\Delta a_{\max} \tag{3}$$

This recommendation was based on experiments conducted on one UHMWPE formulation (as-received, ram extruded Hostalen GUR 4150-HP, Westlake plastics, Lenni, PA) tested in ambient air conditions.

The applicability of the UHMWPE-specific blunting line relationship given in Eq. (3) to highly crosslinked and post-processed UHMWPE materials [9] is not known. Therefore, in this study, a novel experimental approach based on crack tip opening displacement (CTOD) was evaluated and compared to the blunting line approach to

(1) determine if the CTOD method can be used to obtain J_{IC} for UHMWPE; and
(2) determine the applicability of the blunting line approach for highly crosslinked UHMWPE materials.

2. Materials and methods

Three UHMWPE materials were examined in this study: sterilized (30 kGy gamma radiation in nitrogen packaging); highly crosslinked and annealed (65 kGy gamma radiation, 130 °C); and, highly crosslinked and remelted (65 kGy gamma radiation, 150 °C) [10]. All three materials were made from ram extruded, orthopedic grade, GUR 1050 UHMWPE (Orthoplastics, Lancashire, UK).

Disk-shaped compact tension specimens were machined from transverse cross-sections of extruded rod such that the crack propagated in the transverse plane of the rod. Specimen dimensions were selected as per ASTM E1820 [6], and specimen width (W) and thickness (B) were 40 and 10 mm, respectively. The specimens were initially notched to a depth of 16 mm ($a/W = 0.4$). After notching, the specimen surfaces were polished using a mechanical grinder/polisher (Ecomet 6, Buehler, Lake Bluff, IL) for better visualization of the crack tip. The polished specimens were then precracked by pressing a razor blade into the notch at a controlled displacement rate (0.06 mm/min) in a screw-driven materials testing machine, Instron 4411 (Instron Corporation, Canton, MA, USA). This method has been found to minimize damage at the crack tip [11]. A 2 mm long precrack was introduced into each specimen in this manner, resulting in an initial normalized crack length, $a/W = 0.45$. Specimens were stored at −20 °C until testing to minimize potential oxidation of the material [12].

Razor sharpened specimens were tested in a servo-hydraulic closed loop materials testing machine (Instron 8501, Canton, MA) in two environments: ambient air ($n = 2$–3/group) and a more physiologically relevant phosphate buffered saline with Ca and Mg (PBS, Sigma-Aldrich Inc, St. Louis, MO) bath at 37 °C ($n = 3$–4/group) [3]. This PBS solution provides an ionic strength similar to normal joint fluid (0.138 M NaCl and 0.0027 M KCl) [13]. Specimens tested in the PBS bath were pre-soaked in a PBS bath at 37 °C for 4 weeks after polishing and precracking. Specimens were kept submersed in the PBS bath maintained at 37 °C throughout testing.

The initial crack length was measured visually using a traveling microscope with a resolution of 0.01 mm (Gaertner Scientific Corporation, Chicago, IL). The specimens were monotonically loaded at a crosshead speed of 10 mm/min until crack extension in the range of approximately 1–3 mm was achieved. The crack tip was cooled by an air jet throughout the testing to minimize heating in the ambient air specimens. Ambient air specimens were tested at 23–24 °C.

To measure crack extension, the tested specimens were freeze fractured (cooled in liquid nitrogen) immediately following testing. The final crack length was calculated as per ASTM D6068 [14]. J was calculated according to ASTM D6068 [14]

$$J = \frac{\eta_{pl} U}{Bb} \tag{4}$$

where U is the energy required to extend the crack, B is the thickness of the specimen, b is the initial unfractured ligament length, and $\eta_{pl} = 2.5$ for a compact tension specimen. The energy required to extend the crack is given by the area under the load–displacement curve.

The J–R curve for each treatment group was generated from the specimen with the largest crack extension in the treatment group using the single specimen normalization method with a power law-based deformation function [3]. The conditional fracture toughness, J_Q, was also calculated using the blunting line approach (Eq. (3)) for each treatment group. The effect of the material and testing environment on J_Q was examined. J_Q is the specimen size-independent plane strain fracture toughness and can be considered to be J_{IC} only if the following specimen size criterion [15] is satisfied:

$$B, b \geqslant 17\left(\frac{J_Q}{\sigma_y}\right) \tag{5}$$

where σ_y is the effective yield stress (and may be replaced by the ultimate tensile stress for UHMWPE) and b_0 is the initial unfractured ligament length.

During monotonic loading, a small square region ($5 \times 5\,\text{mm}^2$) encompassing the crack tip was recorded in 'avi' file format at 30 frames per second using a traveling microscope (Gaertner Scientific Corporation, Chicago, IL) with a CCD camera (XC-75, Sony Corporation, Japan). The recorded 'avi' files were then processed by a custom image processing routine developed in Matlab v7.0.4 (The Mathworks Inc., Natick, MA) to obtain CTOD. The image processing routine grabs an image frame from the 'avi' file, thresholds the image according to a user-defined threshold level, identifies the crack tip and then computes CTOD at any given distance behind the crack tip. A resolution of 0.014 mm was achieved with this method. Thus, the CTOD at 0.28 mm (20 times the image resolution) behind the crack tip was computed from the 'avi' file for all the specimens. The horizontal distance between the crack tip and the loading axis (crack tip distance) was also measured throughout the test. Load and CTOD data were synchronized manually.

CTOD vs. applied crosshead displacement and crack tip distance vs. applied crosshead displacement for all specimens within a treatment group were pooled together. CTOD was found to increase at a steady rate until a particular applied displacement and then to continue to increase at a slower rate, thereby exhibiting a bimodal behavior (Fig. 1). The applied displacement value at which the change of slope occurs is denoted as v_{maxQ}. The load corresponding to v_{maxQ} is denoted as P_{maxQ}. With respect to crack tip distance, with specimen loading, it was observed that the crack tip distance initially decreased, then flattened, and then either increased with an increase in applied displacement (Fig. 1) or remained unchanged. The applied displacement value at which the CTOD vs. crack tip distance curve begins to become flattened is denoted as v_{minQ}. This was estimated by visual inspection of the crack tip distance vs. applied displacement curves.

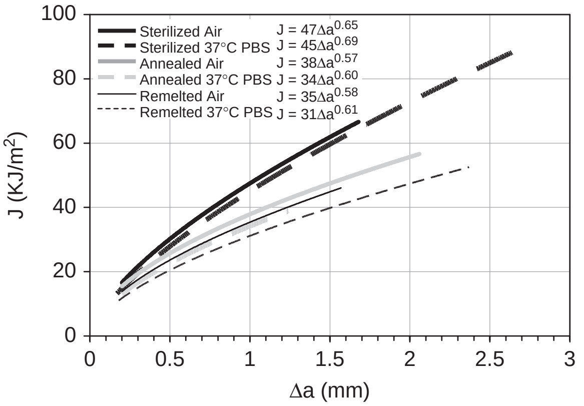

Fig. 1. *J–R* curves of all treatment groups [10].

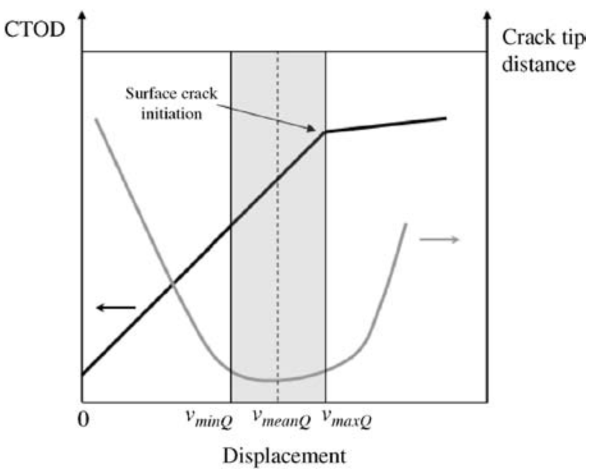

Fig. 2. Schematic of CTOD, crack-tip distance vs. displacement curves.

The load corresponding to v_{minQ} is denoted as P_{minQ}. The load P_{meanQ}, which corresponds to the displacement, v_{meanQ} ($v_{meanQ} = (v_{maxQ}+v_{minQ})/2$) was calculated. From these displacement and load values, a set of J_Q values, J_{maxQ}, J_{minQ}, and J_{meanQ}, were calculated using Eq. (4). J_{meanQ} was compared to J_Q determined using the blunting method for each treatment group (Fig. 2).

3. Results

J–R curves were successfully determined for each treatment group (Fig. 1, [10]). For all treatment groups, the criterion to obtain specimen size-independent plane strain fracture toughness as given by Eq. (5) was not met. Therefore, J_Q was determined for each treatment group. In the ambient air environment, J_Q for the annealed and remelted materials were 22% and 31% lower than that of the sterilized material, respectively (Table 1). In the 37 °C PBS environment, J_Q for the annealed and remelted materials were 29% and 42% lower than that of the sterilized material, respectively (Table 1). J_Q for the specimens tested in the 37 °C PBS environment were 3%, 12% and 18% lower than that of the specimens tested in ambient air for the sterilized, annealed and remelted materials, respectively (Table 1).

The change of slope in the CTOD vs. applied dis placement curve was distinct for all treatment groups (Figs. 3–7). For annealed specimens tested in 37 °C PBS environment, crack tip distance vs. applied displacement curves were not available due to experimental error. Accordingly, it was possible to determine J_{minQ}, J_{meanQ}, and J_{maxQ} for the sterilized, annealed, and remelted materials tested in air and the sterilized and remelted materials tested in 37 °C PBS (Table 1).

For the sterilized, annealed and remelted treatment groups tested in the ambient air environment, the J_Q values predicted by the blunting line approach (Eq. (3)) were 18%, 32% and 9% higher than the experimentally measured J_{meanQ} values, respectively (Table 1). For the sterilized and remelted treatment groups tested in the 37 °C PBS environment, the J_Q value predicted by the blunting line approach was 26% and 12% higher than the experimentally measured J_{meanQ} values, respectively.

In the ambient air environment, J_{meanQ} of the annealed and remelted materials were 30% and 26% lower than that of the sterilized material, respectively (Table 1). In the

Table 1
J_{minQ}, J_{meanQ}, and J_{maxQ} values obtained experimentally and J_Q values predicted by the blunting line for the UHMWPE materials

Environment	Material	J_Q (KJ/m^2)			
		J_{minQ}	J_{meanQ}	J_{maxQ}	Blunting line
Ambient air	Sterilized	20.59	27.08	34.07	32.00
	Annealed	13.65	18.96	24.89	25.00
	Remelted	15.51	20.10	24.78	22.00
37 °C PBS	Sterilized	19.70	24.66	30.95	31.00
	Annealed	–	–	–	22.00
	Remelted	13.07	16.06	19.31	18.00

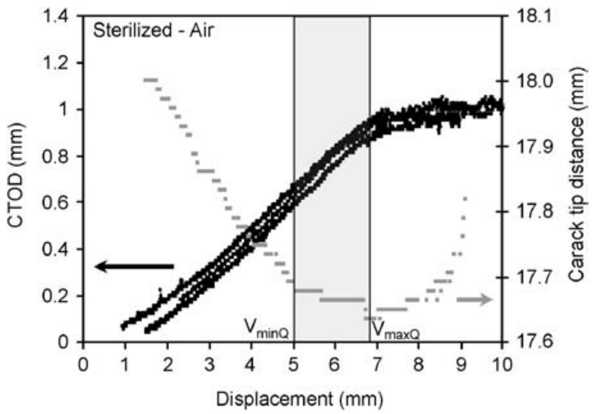

Fig. 3. CTOD and crack-tip distance as a function of displacement for sterilized materials tested in ambient air ($n = 3$). Shaded area is the region between v_{minQ} and v_{maxQ}.

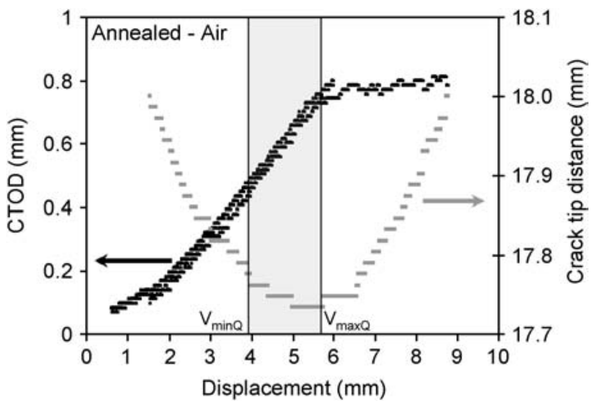

Fig. 4. CTOD and crack-tip distance as a function of displacement for annealed materials tested in ambient air ($n = 2$). Shaded area is the region between v_{minQ} and v_{maxQ}.

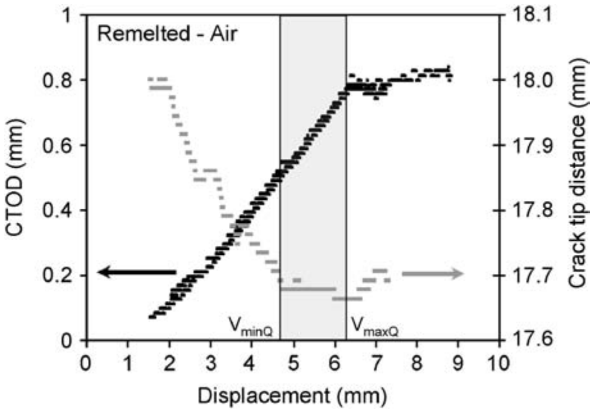

Fig. 5. CTOD and crack-tip distance as a function of displacement for remelted materials tested in ambient air ($n = 2$). Shaded area is the region between v_{minQ} and v_{maxQ}.

37 °C PBS environment, J_{meanQ} of the remelted materials was 35% lower than that of the sterilized material. J_{meanQ} of the sterilized and remelted materials tested in the 37 °C

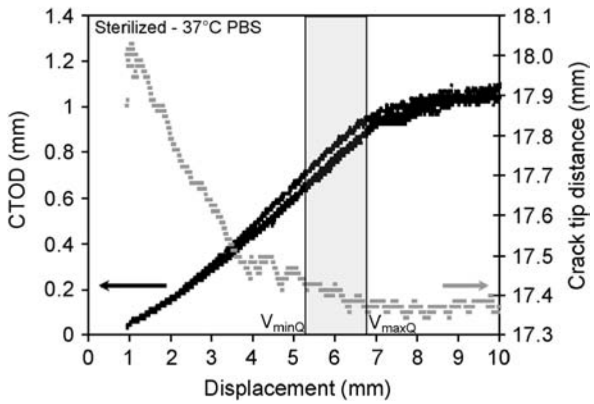

Fig. 6. CTOD and crack-tip distance as a function of applied displacement for sterilized materials tested in 37 °C PBS ($n = 3$). Shaded area is the region between v_{minQ} and v_{maxQ}.

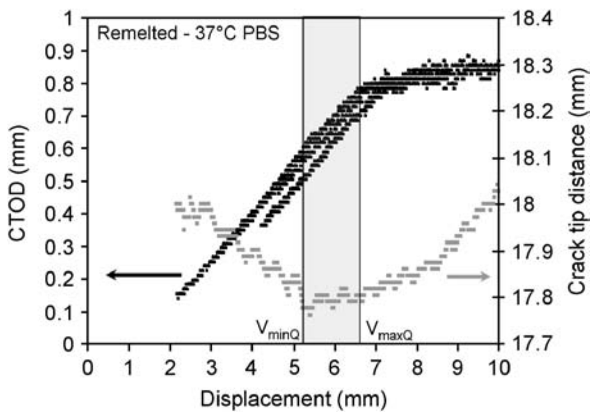

Fig. 7. CTOD and crack-tip distance as a function of displacement for remelted materials tested in 37 °C PBS ($n = 4$). Shaded area is the region between v_{minQ} and v_{maxQ}.

PBS environment were 9% and 20% lower than that in ambient air, respectively.

4. Discussion

The CTOD method utilized in this study provided J_Q values comparable to the blunting line approach for the UHMWPE materials and environments examined in this study. The change of slope for the CTOD vs. applied displacement curves can be attributed to crack initiation on the surface of the specimen (v_{max}, Fig. 2). The point at which the crack tip distance initially begins to stabilize (v_{min}) may be indicative of crack initiation in the mid-thickness of the specimen, which would not be realized by surface measurements. Hence, J_{meanQ} was computed as an average of J_{minQ} and J_{maxQ}. J_{meanQ} is believed to be a reasonable estimate of J_Q. For all the treatment groups it was observed that the experimentally measured J_{meanQ} was conservative when compared to J_Q obtained by the blunting line approach (Table 1). Interestingly, J_{maxQ} and J_Q obtained by the blunting line approach were very similar (within 0.1–13%) in all cases, despite J_{maxQ} being based on surface measurements.

The blunting line equation (Eq. (3)) recommended by ASTM E1820 [6] is strictly applicable only for metals. This equation has been found to be satisfactory for some polymers [16]; however, it has also been found to be unsatisfactory for other polymers [17–19]. The applicability of the blunting line approach depends on: (1) the fitting method; (2) the exclusion lines; and (3) the slope and stress value used to define the blunting line. Pascaud et al. [7] justified the validity of Eq. (3) for a UHMWPE material based on observations of the stretch zone caused by blunting of the crack tip. However, it may not always be straightforward to identify the stretch zone from the fracture surface. In addition, the stretch zone width measurements are sensitive to the initial notch root radius [7]. The accuracy of J_Q/J_{IC} predictions will depend on appropriate determination of stretch zone width measurement and selection of parameters in the blunting line equation. In contrast, the CTOD method used in this study is based on experimental observation and thus does not rely on an empirical relationship or fracture surface measurements. Therefore, determining J_Q using the experimentally based CTOD method proposed in this study may be a more reliable approach for UHMWPE and other tough polymers than the blunting line approach.

The lower J_Q, J_{meanQ} values for the highly crosslinked materials as compared to the sterilized material indicate that they have reduced resistance to crack initiation in both test environments. Both highly crosslinked UHMWPE materials showed a similar resistance to crack initiation. All three materials demonstrated a loss of crack initiation resistance in the 37 °C PBS environment as compared to ambient air. These findings are consistent with related studies on the fatigue-crack propagation resistance and the *J–R* behavior of these materials which demonstrate reduced resistance to cyclic and static fracture in a 37 °C PBS environment compared with ambient air [10,20].

5. Conclusions

The CTOD method is a useful experimental approach to determine J_Q of UHMWPE materials. The method provides J_Q values that are comparable to or more conservative than the blunting line approach.

References

[1] C.M. Rimnac, T.M. Wright, R.W. Klein, J integral measurements of ultra high molecular weight polyethylene, Polym. Eng. Sci. 28 (1988) 1586–1589.
[2] R.S. Pascaud, W.T. Evans, P.J. McCullagh, D.P. Fitz Patrick, Influence of gamma-irradiation sterilization and temperature on the fracture toughness of ultra-high-molecular-weight polyethylene, Biomaterials 18 (1997) 727–735.
[3] R. Varadarajan, C.M. Rimnac, Static fracture resistance of UHMWPE using single specimen normalization method, Polym. Test. 27 (2008) 260–268.
[4] J.C. Cole, J.E. Lemons, A.W. Eberhardt, Gamma irradiation alters fatigue-crack behavior and fracture toughness in 1900H and GUR 1050 UHMWPE, J. Biomed. Mater. Res. 63 (5) (2002) 559–566.
[5] A. Gomoll, T. Wanich, A. Bellare, J-integral fracture toughness and tearing modulus measurement of radiation cross-linked UHMWPE, J. Orthop. Res. 20 (2002) 1152–1156.
[6] ASTM, E1820-01 standard test method for measurement of fracture toughness, in: Annual Book of ASTM Standards, ASTM, West Conshohocken, 2001.
[7] R.S. Pascaud, W.T. Evans, Critical assessment of methods for evaluating Jic for a medical grade ultra-high molecular weight polyethylene, Polym. Eng. Sci. 37 (1997) 11–17.
[8] H. Kobayashi, K. Hirano, H. Nakamura, H. Nakazawa, A fractographic study on evaluation of fracture toughness, in: Proceedings of the Fourth International Conference Fracture, 1977.
[9] S. Kurtz, The UHMWPE Handbook, Elsevier Academic press, 2004.
[10] R. Varadarajan, C.M. Rimnac, The effect of fluid absorption on the fatigue crack propagation resistance conventional and highly crosslinked UHMW polyethylenes for orthopaedic implants, in: Fatigue 2006, Ninth International Fatigue Congress, May 14–19, 2006, Atlanta, GA, p. 345.
[11] E.K. Dapp, C.M. Rimnac, Effect of precracking method on the static (J-Integral) fracture resistance of UHMW polyethylene, in: Transactions of ASME, 1998.
[12] S.M. Kurtz, M. Herr, L. Ciccarelli, J. Bergstrom, C. Rimnac, A. Edidin, Thermomechanical behavior of virgin and highly cross-linked ultra-high molecular weight polyethylene used in total joint replacements, Biomaterials 23 (2002) 3681–3697.
[13] T. Kitano, G.A. Ateshian, V.C. Mow, Y. Kadova, Y. Yamano, Constituents and pH changes in protein rich hyaluronan solution affect the biotribological properties of artificial articular joints, J. Biomech. 34 (8) (2001) 1031.
[14] ASTM, D6068-96 standard test method for determining J–R curves for plastic materials, in: Annual Book of ASTM Standards, ASTM, West Conshohocken, 1996.
[15] S. Hashemi, J.G. Williams, The effects of specimen configuration and notch tip radius on the fracture toughness of polymers using J-c, Plast. Rubbers Process Appl. 6 (4) (1986).
[16] D.D. Huang, J.G. Williams, Fracture-toughness of impact modified polymers based on the J-integral-comments, Polym. Eng. Sci. 30 (21) (1990) 1341–1344.
[17] D.D. Huang, J.G. Williams, J. Testing toughened nylons, J. Mater. Sci. 22 (7) (1987) 2503–2508.
[18] Y.W. Mai, P. Powell, Essential work of fracture and J-integral measurements for ductile polymers, J. Polym. Sci. Part B Polym. Phys. 29 (7) (1991) 785–793.
[19] H. Swei, B. Crist, S.H. Carr, The J integral fracture-toughness and damage zone morphology in polyethylenes, Polymer 32 (8) (1991) 1440–1446.
[20] R. Varadarajan, C.M. Rimnac, The effect of fluid environment and crack closure on the fatigue crack propagation resistance of conventional and highly crosslinked UHMW polyethylenes for orthopaedic implants, in: Ninth International Fatigue Conference, Atlanta, 2006.