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**Extended Abstract**

# The Role of Microstructure on the Fatigue and Fracture Properties of Medical Grade Ultra High Molecular Weight Polyethylene

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## ABSTRACT

This work examines the role of microstructure on the fatigue and fracture properties of ultra high molecular weight polyethylene (UHMWPE). The aim of this work is to develop optimized microstructures that provide both fatigue and wear resistance for implant applications. Recent studies have shown that bulk cross-linking improves the wear resistance of UHMWPE. However, cross-linking degrades fracture properties such as ultimate tensile stress and strain, J-integral fracture toughness and fatigue crack propagation resistance. One problem with bulk cross-linking is that they employ a melting process. Recrystallization after melting is obtained without any application of pressure and results in a decrease in crystallinity and concomitant mechanical properties. Crystallinity can be restored with the utilization of to utilize high-pressure crystallization on the cross-linked UHMWPE. High crystalline PE has been shown to have a substantial increase in fracture and fatigue crack propagation resistance. This work examines the coupled effects of cross-linking and enhanced crystallinity via high-pressure methods to improve the mechanical properties of UHMWPE. The role of various microstructures on the fatigue and fracture properties are examined and discussed in the context of total joint replacement design.

## INTRODUCTION

Fatigue resistance of medical grade ultra high molecular weight polyethylene (UHMWPE) remains a clinically relevant problem for the medical community. UHMWPE is used to replace cartilage in damaged joints and serves as one of the bearing surfaces in total joint replacements (TJR). The articulating surfaces can experience large fluctuations in stress, and in total knee replacements these stresses can range from 40 MPa of compression through 10 MPa of tension. This large cyclic contact stress has been implicated in a number of device failures exhibiting delamination or fatigue wear mechanisms. Much of the recent polymer science research in the orthopedic community has been devoted to minimizing wear in total joint replacements. This has resulted in the development of highly crosslinked UHMWPE resins for use in TJR. A consequence of this crosslinking has been the reduction of toughness and fatigue crack propagation resistance (Baker et al, 2003). This loss of mechanical integrity may limit the

application of these resins in total knee replacements where cyclic stresses are highly demanding. Further investigation is needed to develop a polymer that is resistant to both fatigue and wear under high cyclic stresses. This study investigates the use of an enhanced crystallinity, which has been previously shown to improve fatigue crack propagation resistance (Baker et al. 1999) along with crosslinking which has been shown to improve wear resistance of UHMWPE (Wang et al. 1997). High pressure crystallization was used to increase crystallinity and lamellar size. Gamma radiation with an annealing step was used to crosslink the polymer. Fatigue crack propagation studies were performed in order to assess the resistance of this material to growth of flaws under cyclic loading. The aim of this work is to optimize the polymer microstructure for resistance to both fatigue damage and wear processes in order to extend the life of total joint replacements.

**EXPERIMENTS**

The base material used in this study was a GUR 1050 resin (Hoechst Celanese/Ticona). The Commercial GUR 1050 rod stock of 5" diameter was subjected to 50 kGy gamma radiation (Isomedix Inc., Northborough, MA). Thereafter the rod stock was maintained at 170C for 4 hours in an oven and slowly cooled to room temperature to quench the free radicals and complete the crosslinking. Control and crosslinked UHMWPE rods were then machined into 2" long cylinders of 1/2" diameter to snugly fit into a high pressure cell. The high pressure cell was heated to 180C (control UHMWPE) or 240C (50 kGy UHMWPE). A pressure of 300 MPa (control UHMWPE) or 500 MPa (50 kGy UHMWPE) was applied. After a period of 1 hour, the sample was cooled to room temperature under pressure and then the pressure was released. Six material groups were examined: (PE) control (GUR 1050); (XPE) 50 kGy crosslinked UHMWPE; (HP-PE) 180°C 300 MPa (uncrosslinked); (HP-XPE) 240°C 500 MPa (crosslinked); (Q-PE) PE quenched in liquid nitrogen from 170 C; and (Q-XPE) XPE quenched in liquid nitrogen from 170 C.

The six material groups used in this study were machined into notched micro-tensile specimens from processed rod stock. Notches were machined and pre-cracked with a surgical blade. An initial a/w of 0.3 was utilized for the fatigue tests. All fatigue tests were performed on an Instron servohydraulic materials testing machine in room temperature air at a frequency of 5 Hz. The fatigue fracture tests were run with a load ratio R=0.1. Crack length was monitored optically with number of cycles to determine crack propagation rates, da/dN, as a function of stress intensity range, ΔK. For the crack propagation studies, the stress intensity range necessary to generate crack growth at a rate of approximately $10^{-7}$ mm/cycle was measured and defined as a near-threshold $\Delta K_{incept}$. The stress intensity was calculated using fracture mechanics analysis of a single edge notched specimen (Irwin 1973):

$$\Delta K = \Delta\sigma\sqrt{\pi a} \bullet F(a/w);$$

where

$$F(a/w) = 1.12 - 0.231(a/w) + 10.55(a/w)^2 - 21.72(a/w)^3 + 30.39(a/w)^4 .$$

USAXS was performed at the UNI-CAT beamline of the Advanced Photon Source, Argonne National Laboratory, using a Bonse-Hart ultra-small-angle scattering instrument. The beam cross-sectional area was 2.0 mm x 0.6 mm and 10 kEV x-rays were used. UHMWPE specimen thickness was 1.5nmm. Data were collected in the form of absolute

intensities I (cross section, $cm^{-1}$) as a function of the scattering vector q ($nm^{-1}$) where q is defined as: $q = (4\pi/\lambda)\sin\theta$, such that $\theta$ equals one half of the scattering angle, and $\lambda$ is the wavelength of X-rays. An angular scattering range where $q_{min}=0.001$ [$nm^{-1}$] and $q_{max}=1.0$ [$nm^{-1}$] was measured. The USAXS scattering functions were converted to paired distance distribution functions using a previously established inverse Fourier transform technique developed by Glatter (1977). The long period, or inter-lamellar distance, was obtained from the first maximum of the distance distribution function.

DSC was performed using a Perkin Elmer Pyris instrument. Percent crystallinity was calculated by normalizing the heat of fusion of each sample to that of polyethylene crystal (293 J/g). Crystallinity was determined from the average of 3 samples for each sample group.

## RESULTS AND DISCUSSION

A summary of the crystallinity, USAXS long period, and near threshold ($10^{-7}$ mm/cycle) fatigue crack inception value are provided in Table 1. As expected, high pressure crystallization increased both crystallinity and lamellae size. Crystallinity was increased from 53 % to 70.9% for the uncrosslinked resin, and similarly increased from 46.3% to 67.5% for the crosslinked resin. This crystallinity is brought about via an enhancement of lamellae size as corroborated by USAXS. For the quenched groups, the crystallinity is substantially decreased to 35.7% for the noncrosslinked group and 30.1% for the crosslinked group.

**Table I. Summary of material and mechanical properties**

| Group | Crystallinity (%) | Lamellar Size (nm) | E (MPa) | Yield Strength (MPa) | $\Delta K_{th}$ ($MPa*m^{1/2}$) | Hardness $H_v$ (MPa) |
|---|---|---|---|---|---|---|
| Q-PE | 35.7 | 10.2 | 186 | 16.6 | 0.97 | 53.7 |
| PE | 50.2 | 28.11 | 288.22 | 21.30 | 1.14 | 66.48 |
| HP-PE | 70.9 | 131.17 | 341.22 | 23.43 | 1.57 | 88.55 |
| Q-XPE | 30.1 | --- | --- | --- | 0.77 | 53.5 |
| XPE | 46.2 | 23.1 | 224.59 | 19.01 | 0.92 | 57.59 |
| HP-XPE | 67.5 | 50.63 | 346.39 | 20.65 | 1.03 | 74.16 |

Figure 1 shows high resolution field emission scanning electron micrographs of the six material groups. The lamellae microstructures were revealed by using a permanganate etch.

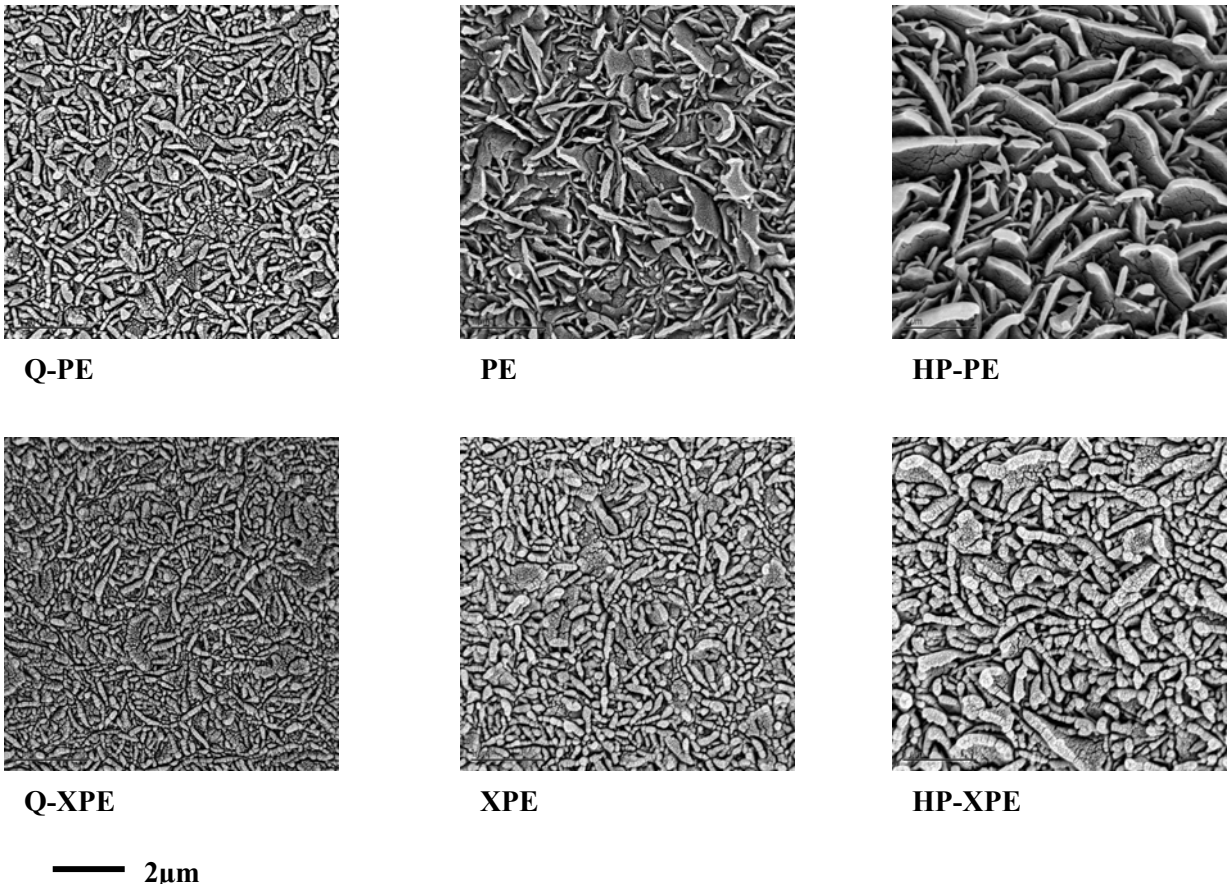


**Figure 1.** Field emission scanning electron micrographs of the microstructures for the 6 material groups. All images at 20,000 x.

Fatigue crack propagation inception values for all the material groups are shown in Table 1. Crosslinking and quenching resulted in a decreased resistance to fatigue crack propagation while high pressure treatments resulted in an increased resistance to fatigue crack inception. The effect of high pressure crystallization is known to improve fatigue crack propagation resistance of uncrosslinked UHMWPE and has been documented previously (Baker et al. 1999, Baker et al., 2003). It is thought that the improved fatigue resistance is associated with enhanced lamellae size and associated improvements in mechanical properties. The enhanced lamellae size may aid in crack tip deflection processes that could reduce the effective stress intensity ahead of the crack tip. It has been postulated (Suresh, 1993) that the threshold for onset of fatigue crack growth may occur when crack tip opening displacement ($\delta_t$) attains a value comparable to a critical microstructural dimension:

$$\Delta\delta_t = \Delta K^2 / 2\sigma_Y E$$

$$\Delta K_{th} \propto \sqrt{(\sigma_Y E\, l^*)}$$

where $\sigma_Y$ is the yield strength, E is the elastic modulus, and L8 is the critical microstructural length scale. For this study there is a strong linear correlation ($R^2$= 0.89) between crack inception and critical crack tip opening displacement. Thus, the use of enhanced crystallinity and tailored microstructure may provide an effective mechanism for improving fatigue crack propagation behavior of crosslinked resins used for bearing and orthopedic applications.

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