Super Ductile UHMWPE by High Temperature Melting

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Introduction

Radiation cross-linking and subsequent thermal treatment improve the wear resistance and oxidation stability of UHMWPE while decreasing its mechanical properties [1]. Both the strength and ductility of the cross-linked polymer decline with increasing cross-link density. One strategy to offset this decline is to improve the strength and ductility of the polymer before cross-linking. The ductility of cross-linked UHMWPEs was highly improved by melting at temperatures up to 325 °C with elongation over 800% [2]. We hypothesized that the ductility and toughness of unirradiated UHMWPE could be enhanced significantly by high temperature melting prior to radiation cross-linking due to the higher mobility of chains in the un-cross-linked state. The overall goal is to combine this method with subsequent crosslinking to obtain a ductile and wear resistant UHMWPE.

Materials and Methods

UHMWE was melted in an inert convection oven (Despatch, MN) at 280, 300, and 320 °C for 2, 5, and 12 h, respectively. These samples were designated as UH T-t (T is temperature and t is time). Microtomed thin slices (150 µm) were characterized by Fourier Transform Infrared Spectroscopy (FTIR) to investigate the formation of vinyl end groups (absorbance 909 cm⁻¹) which were indexed (VI) as an indicator of chain scission in UHMWPE. The peak melting points and percentage crystallinity were measured by using a Q-1000 differential scanning calorimeter (TA, DE) at 10 °C/min.

Thermogravimetric analysis (TGA) was performed at a heating rate of 20°C/min (Q500, TA Instruments, Newark, DL) on UHMWPE samples, which were then held at 280, 300, 320 and 340°C for 24 hours.

Type IV tensile specimens (n=5) according to ASTM-638 were stamped out of 3.2 mm thick sections for tensile testing. Double notched bars (63.5 x 12.7 x 6.350 mm) were used for load impact strength tests at Orthoplastics Inc. (UK) according to ASTM F-648. Freeze-fractured surfaces of UHMWPE before and after high temperature melting were visualized by scanning electron microscopy.

Bi-directional pin-on-disc (POD) wear tests [1] were conducted in bovine serum at a frequency of 2 Hz for 1 million cycles (MC). The weight loss at every 0.147 MC was measured and wear rates were calculated as a linear regression against number of cycles from 0.5 to 1 million cycles.

Results and Discussion

TGA results (not shown) indicate that UHMWPE did not decompose at 320 °C and below over a 24 hour time period. High temperature melting (HTM) induced chain scission [3], characterized by the vinyl index (VI), especially at higher temperatures and longer times (Fig 3). While increasing chain scission was correlated strongly with increasing elongation to break (Fig 1c), the ultimate tensile strength (UTS) was only significantly affected at VI larger than 0.07 (Fig 1a). Moreover, work to failure (WF), an indicator of tensile toughness, was improved until a VI of 0.06 (Fig 1b). Therefore, the ductility and toughness was improved for all samples except those with severe chain scission. The double-notched Izod impact strength increased from 127 kJ/m² for UHMWPE to 175 kJ/m² for UH 280-12, but it was not reduced significantly until a VI of 0.05 (Fig 1d). Interestingly, the tensile toughness and strength were not improved for UH 280-12 despite significantly.

High temperature melting also increased chain diffusion across grain boundaries (Fig 2), resulting in thinner crystalline lamellae with decreased peak melting temperature but increased crystallinity (Table 1). These thin crystal lamellae may have improved plasticity [4] in comparison to conventional UHMWPE crystals and may have contributed to EAB up to 1100% that we observed in high temperature melted UHMWPEs.

Conclusion

Our hypothesis that high temperature melting would improve the ductility and toughness of unirradiated UHMWPE can be improved without sacrificing wear properties significantly. This material can then be cross-linked to improve wear resistance while maintaining improved toughness and ductility over the current state-of-the-art radiation cross-linked UHMWPEs.

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References


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References