INTRODUCTION:

The annealing process step that utilizes thermal treatment at sub-melt temperature for highly cross-linked ultra high molecular weight polyethylene (UHMWPE) is known to maintain mechanical properties. However, it is critical to use consistent starting virgin material and optimal temperature range for annealing to produce cross-linked material with desired properties [1]. Re-melting of highly cross-linked UHMWPE is known to reduce its tensile strength and elongation (toughness).

In this study, it is hypothesized that different starting virgin UHMWPE materials will produce highly cross-linked UHMWPE with different properties after the same cross-linking and annealing process. It is also hypothesized that re-melting of highly cross-linked UHMWPE can be detected and distinguished by small-punch (bi-axial) testing [2].

MATERIALS AND METHODS:

Consolidated UHMWPE from low- and mid-end of the acceptable ASTM density range were selected as starting material for a cross-linking and subsequent annealing treatment.

GUR 1020 and compression molding were the resin and consolidation method, respectively. Low-end density (LD) material was processed by a small cavity mold method (PPD Group, Waterville, Canada) while mid-end density (MD) material was produced from production-scale molding (Quadrant, Reading, PA, USA).

The cross-linking and annealing treatment consisted of three cycles of 30 kGy gamma irradiation and subsequent annealing (SXL) to accumulate gamma irradiation dose of 90 kGy in materials. Thermal treatment was carried out at 130 °C, below melting temperatures of both starting material groups. Typical physical properties of starting materials (LD and MD) are listed in Table 1.

Tensile (N=5) using mini tensile samples, small punch (N=5), and Crystallinity / Peak Melting temperature (N=5) were evaluated following ASTM D638, F2183, and F2625 standards, respectively. Student t-test was used to determine statistical significance.

RESULTS:

The lowest melting peak appeared at about 130 °C during differential scanning calorimetry (DSC) runs for LD-SXL (LD-SXL); thermographs of this material exhibited multi-modal melting peaks (Figure 1). These were not observed in either the starting LD and MD materials or MD-SXL (MD-SXL).

The MD-SXL showed significantly higher tensile properties than its LD-SXL counterpart (Table 2). No significant difference was found in bi-axial small punch peak load of MD-SXL and LD-SXL. However, a higher displacement and ultimate load was observed in small punch load-displacement curve (Figure 2) for LD-SXL. Note that except peak load, other metrics such as displacement and ultimate load cannot be derived for LD-SXL; tests were terminated manually as they reached set-up limits and exhibited evidences of slippage.

DISCUSSION:

We confirmed our first hypothesis that different starting virgin UHMWPE materials will produce highly cross-linked UHMWPE with different properties after the same cross-linking and annealing process.

Re-melting occurred in LD-SXL during the 130 °C annealing process. The low tensile properties that associated with re-melted highly cross-linked UHMWPE and multi-modal thermographs from DSC indicated that starting material with low-end density was not suitable for SXL 130 °C process. The use of consistent starting material is critical to produce best quality production scale highly cross-linked UHMWPE.

Future studies will investigate the possible effect of critical mass of materials during annealing process.

However, the second hypothesis, “re-melting of highly cross-linked UHMWPE can be detected and distinguished by small-punch (bi-axial) testing” was defeated.

The mechanical behavior of LD- and MD-SXL was similar during small punch test until the displacement was approximately 4.5 mm; our data also indicated that a fully re-melted highly cross-linked UHMWPE would fracture at similar displacement. Nevertheless, the small punch test for LD-SXL was terminated prematurely due to the triggering of a set-up limit (at 0.25 inches or 6.35 mm). This prevented damages to the testing fixtures.

On the other hand, the low properties obtained from tensile test correctly indicated that LD-SXL was re-melted during the 130 °C annealing process.

REFERENCES: