# THE EFFECT OF ACCELERATED AGING ON MECHANICAL PROPERTIES AND OXIDATION OF UHMWPE LINERS GAMMA-STERILIZED IN NITROGEN

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**Introduction**

Recently, the ASTM subcommittee on polyethylene started working to define a standard accelerated aging protocol for the analysis of ultra high molecular weight polyethylene (UHMWPE) acetabular liners. A multi-laboratory round robin study is in progress, whereby oxidation indices, as determined from optical techniques, are measured on UHMWPE samples aged by various protocols. Of considerable interest is the comparison of accelerated aged specimens, through oxygen-bombs, with shelf-aged products, in order to determine the conditions that most closely replicate the latter's history. Sulzer Orthopedics was the first company to sterilize UHMWPE acetabular liners in the presence of nitrogen (1986), and consequently has access to specimens that have been shelf-aged for over 10 years. We present results of an accelerated aging protocol (1), and compare them with data collected from shelf-stored specimens sterilized in the presence of nitrogen. These results will be of use towards defining an accepted accelerated aging standard.

**Material/Methods**

We examined 13 shelf-stored cups (5-10 years) with a PE thickness of 10 mm, sterilized with gamma rays in nitrogen up to 2.5 Mrads. They were compared to accelerated aged products (5 specimen each) also with a thickness of 10 mm that had also been sterilized with gamma rays in nitrogen. Specimens underwent an accelerated-ageing protocol to different lengths of time in an oxygen bomb at 70°C, 5 atm $O_2$ (1). All cups and test-specimens are machined from compression molded GUR 1020.

The oxidation index was measured by means of Fourier Transform Infrared Spectroscopy (FTIR) in accordance with the German standard DIN 53383-2 (extinction ratio between the IR absorption band at 1720 $cm^{-1}$ and the absorption band existing in the UHMWPE at 2020 $cm^{-1}$). Uniaxial tensile testing was conducted on machined dogbones according to ISO 5834 and ISO 527 to assess the change in mechanical properties with aging. Charpy impact tests were conducted according to ISO 11542.

**Results and Discussion**

The oxygen levels found *in vivo* correlates quite well to shelf-stored samples packaged in nitrogen. Consequently, the temperature-dependent kinetics of aging should have good correlation for the two media. In Figure 1, the bulk oxidation index is shown as a function of aging time. The dashed line shows the results for the shelf-aged specimen (10 years). The single point oxidation index measured on the shelf-aged specimens allows comparison of the aging protocols. The results show that approximately 15 days of accelerated aging in $O_2$, 5 bar, 70 °C corresponds to 10 years real-time aging and compare well with full oxidation profiles collected previously (2). Furmann (3) investigated the change in density following aging in $O_2$, 5 bar, 70°C. With extrapolation of his real-time aging diagram, the period of 14 days in $O_2$, 5 bar, 70 °C corresponds to about 10 years, which correlates very well with the results described here. The oxidation index of the accelerated specimens show a very small standard deviation, which indicates the good reproducability of that method.

From the mechanical results shown in Figure 2 for bomb-aged specimens, there is little difference in ultimate tensile stress or % elongation to break after 15 days of accelerated aging. After this length of time, however, both these parameters decrease rapidly. Clearly, the oxidation observed in Figure 2 leads to sufficient chain scission as to affect the decrease in mechanical properties shown here. The additional results shown in Table 1 indicate the further change in mechanical properties as a function of sterilization time.

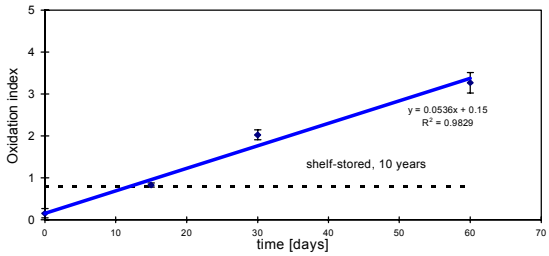


Figure 1: Bulk oxidation as a function of accelerated aging time

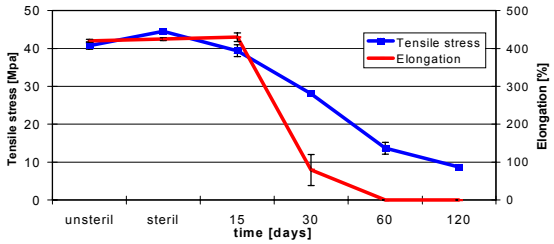


Figure 2: Mechanical properties as a function of accelerated aging time

Table 1: Mechanical properties as a function of aging time

| Aging [days] | Young's Modulus [Mpa] | Yield Stress [Mpa] | Impact Strength [kJ/m²] |
|---|---|---|---|
| Unsterilized | 1005 ± 17 | 40 ± 1 | 187 ± 7 |
| Sterilized (0) | 1085 ± 12 | 43 ± 1 | 120 ± 5 |
| 15 | 1101 ± 24 | 38 ± 2 | 72 ± 7 |
| 30 | 1488 ± 42 | 27 ± 0 | 12 ± 2 |
| 60 | 3200 ± 2 6 | 13 ± 2 | *** |
| 120 | 3276 ± 118 | 8 ± 0 | *** |

***too fragile for testing

**Conclusion**

- Approximately 15 days accelerated aging in $O_2$, 5 bar, 70 °C correspond to 10 years real-time aging *in vivo* and shelf stored. This accelerated aging protocol is recommended for product investigation.
- Mechanical analysis of artificially-aged specimens show that after 30 days of bomb-aging, little mechanical strength remains in the specimens, but that the 15 day aged specimens, corresponding to 10 years of shelf-storage, exhibit good mechanical properties.
- These results present valuable information for determining the optimal pre-treament conditions for acetabular liners used for hip simulator wear studies.

**References**

1 W.M. Sanford and K.A. Saum; Accelerated oxidative Aging Testing of UHMWPE; ORS 1995, Orlando, Florida
2 Spiegelberg S.H. and Schaffner S.R.; Oxidation profiles in shelf stored UHMWPE samples; Society for Biomaterials, Providence 1999
3 Furmann B.D. et al.; Kinetics, Chemistry and calibration of UHMWPE accelerated aging methods; ORS 1998, New Orleans, USA

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