



	<b>Experiment title:</b> Effect of compaction processes on the microstructure of single oriented UHMWPE fibers	<b>Experiment number:</b> ME-1187
<b>Beamline:</b> ID-11	<b>Date of experiment:</b> from: 7/09/2005 to: 10/09/2005	<b>Date of report:</b> 4/08/2006
<b>Shifts:</b> 12	<b>Local contact(s):</b> Dr. Silvia Capelli	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): Liron Shavit-Hadar <sup>*1</sup> , Dmitry M. Rein <sup>1</sup> , Rafail Khalfin <sup>1</sup> , Yachin Cohen <sup>*1</sup> , Ann E. Terry <sup>2</sup> , Guido heunen <sup>2</sup>  <sup>1</sup> Department of Chemical Engineering, Technion-Israel Institute of Technology, Haifa Israel 32000  <sup>2</sup> Dept. of Chemical Engineering, Eindhoven University of Technology, P.O. Box 513, 5600 MB Eindhoven, The Netherlands		

In this study we investigated the effect of melting and re-crystallization processes on the microstructure and orientation in high-performance fibers of ultra-high molecular weight polyethylene (UHMWPE), across the breadth of the fiber. This information is relevant to a novel process for fabrication of self-reinforced composites by compaction of such fibers using judicious application of a pressure-temperature protocol. Here we report some of the preliminary results.

Figure 1 shows X-ray patterns obtained from original fiber scanned at room temperature (a) and at 136°C (b). Comparing these two patterns, it is possible to note the usual melting behavior of the fiber, as the crystals at the edges of the fiber melt at a lower temperature than those at the fiber's center.

From the following formulas we calculated the orientation factor:

$$\langle \cos^2 b_{hkl} \rangle = \frac{\int_0^{\frac{\pi}{2}} \exp \left[ - \left( \frac{\cos(\alpha)}{b_{hkl}} \right)^2 \right] \cdot \sin(\alpha) \cdot \cos(\alpha)^2 \cdot d\alpha}{\int_0^{\frac{\pi}{2}} \exp \left[ - \left( \frac{\cos(\alpha)}{b_{hkl}} \right)^2 \right] \cdot \sin(\alpha) \cdot d\alpha}$$

$$\langle \cos^2 \phi_{c,Z} \rangle = 1 + \frac{\langle \cos^2 b_{110} \rangle - \langle \cos^2 b_{200} \rangle \cdot \left( \frac{d_{110}}{d_{200}} - 1 \right)}{\left( \frac{d_{110}}{2 \cdot d_{200}} \right)^2 - 1}, \quad f = \frac{3 \cdot \langle \cos^2 \phi_{c,Z} \rangle - 1}{2}$$

$b_{hkl}$  is the orthorhombic  $hkl$  peak half width in azimuth

$d_{110}$  and  $d_{200}$  are the d-spacing of the orthorhombic 110 and 200, respectively

$f$  is the Herman's orientation function

This Calculation in those two cases showed a constant value all across the fiber's breadth.

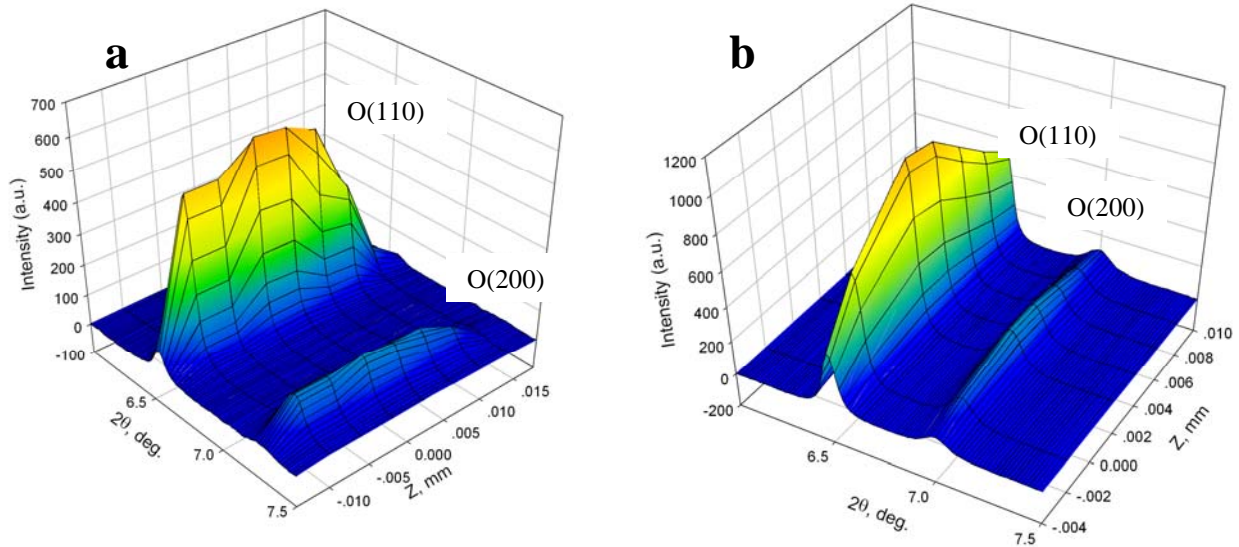


Figure 1: Three-dimensional representation of X-ray diffraction patterns of original UHMWPE fiber (Dyneema SK75) scanned along its breadth: (a) scanning at room temperature. (b) scanning at 136°C.

Comparing single fibers compacted under various conditions showed differences between the melting of the fiber's center and the fiber's edge. For example, some characteristic values rising from the diffraction pattern of a fiber compacted under 600 bars and 150°C are summarized in the following Figures:

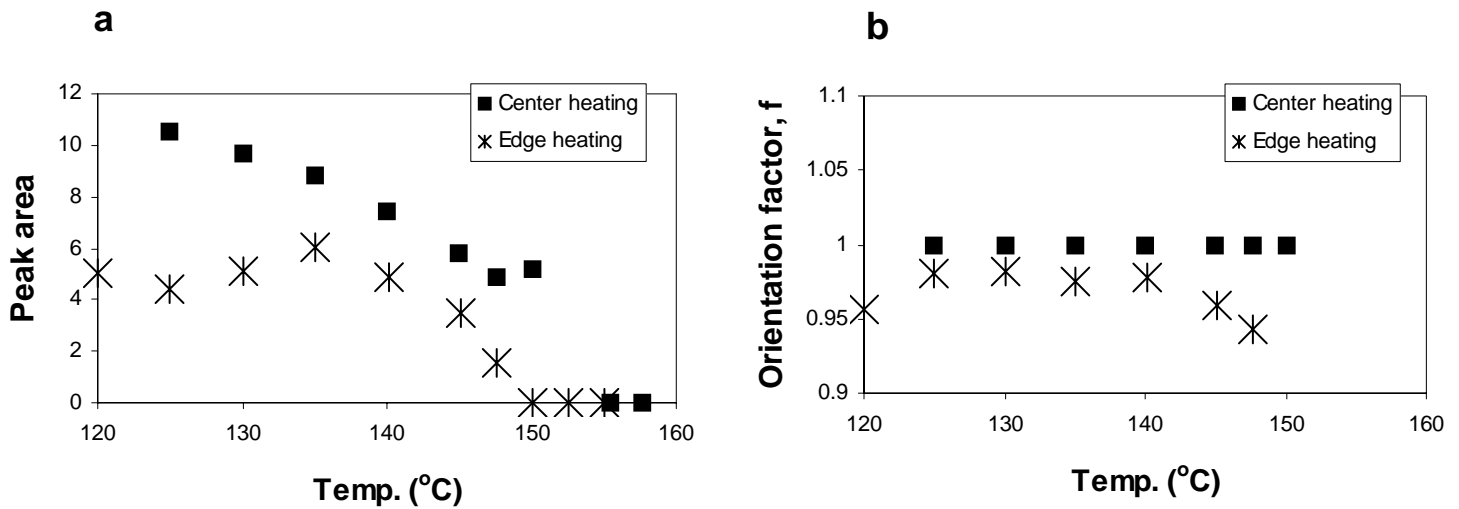


Figure 2: Melting of UHMWPE fiber compacted under 600 bars and 150°C following its central reflections (center heating) and in a subsequent experiment following the reflections from his edges (edge heating). (a) O(110) peak area as a function of temperature (b) Herman's orientation factor as a function of temperature.

Figure 2a might suggest that there is a recrystallization process occurring at the edge of the fiber while melting, whereas this phenomena is not observed in the fiber's center. In addition, it is possible to note the difference in the melting point between these two cases. Figure 2b shows that while orientation is preserved completely at the fiber center until full melting occurs, there is a different behavior at the fiber edge. There, orientation seems to increase slightly during first heating, then remains fairly constant, and finally decreasing at higher temperatures until full melting occurs. Similar effects where revealed in single fibers compacted under different conditions. Further analysis of these samples, currently in progress, may reveal how fibers are fused together in the compaction process, thus pointing to the optimal compaction conditions.