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1 **An investigation of the appearance of lustering on HMPE fiber ropes**

2

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10

11 Abstract

12

13 The low friction coefficient of HMPE fibers may be a disadvantage for some applications such as winches
14 but it can also be used to develop new technology. Recently this specific property was used in the design
15 of innovative pulleys for sailboats. The contact is then between a low friction metallic ring and a HMPE
16 fiber rope loop. During service, a specific phenomenon appears at the contact area on the HMPE fibers
17 called lustering due to its visual aspect. It is also observed in eye splice contact zones during tensile
18 testing. In the present work, an investigation was made to understand and explain the appearance of the
19 lustering of the fibers. Based on examination of different hypotheses, mechanical tests and a range of
20 microstructural observations, the conclusion is that the appearance of the lustering phenomenon is the
21 result of compaction of the filaments of the HMPE rope loop.

22

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24 Keywords: HMPE, rope, compaction, lustering, fiber, microstructure

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31 Understanding the internal deformation mechanisms of fibrous materials during complex mechanical
 32 loading is one of the key elements in predicting the mechanical behavior of ropes. High molecular weight
 33 polyethylene fibers are increasingly used in both the marine and ballistic protection sectors thanks to
 34 their high stiffness and strength, environmental resistance and light weight (Vlasblom, 2018). Another
 35 characteristic of HMPE fibers is their low friction coefficient. Various studies have been performed on
 36 HMPE friction, in particular to try to improve the adhesion properties of HMPE (Jin et al., 2016), but this
 37 low friction behavior has also been used to create novel lightweight textile pulleys, made of a low friction
 38 metallic ring and a HMPE rope loop (Figure 1.a.). The contact between these two components creates a
 39 complex mechanical loading: both tension and compression are present, together with temperature
 40 increases due to friction. Experience with these pulleys has revealed an interesting polishing phenomenon
 41 on the contact surface known as “lustering”. The latter results in a decrease in the friction of the HMPE
 42 fibers, and can also be observed in the contact region between the eye splices of tensile rope samples
 43 and steel loading pins. Figure 1.b. shows how the load needed to rotate the textile pulley changes with
 44 the number of cycles. The strength decrease is correlated to a friction coefficient decrease and the
 45 appearance of the lustering phenomenon (Bain, 2020).

46



Figure 1.a. Lightweight textile pulleys, made of a low friction ring and a HMPE rope loop

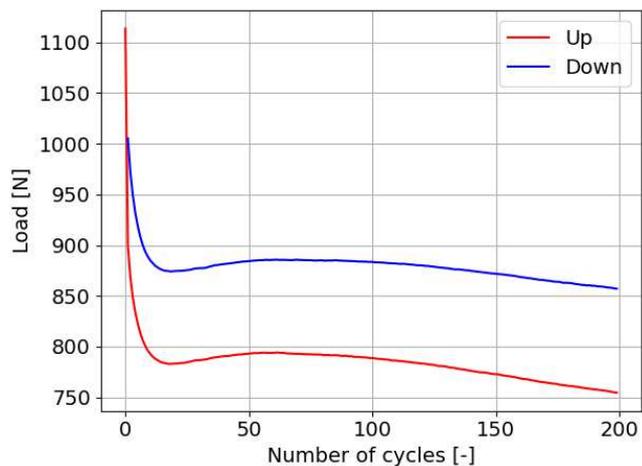


Figure 1.b. Change in the load needed to rotate a textile pulley versus the number of cycles under an 8kN static load (Bain, 2020)

47

48 The purpose of this study is to understand the appearance of this phenomenon. The lustering can be
 49 characterized by three aspects: darkening, a good optical reflection of the contact area, and a fiber
 50 cohesion that makes the fibers look like a homogenous polymer material.

51 Preliminary observations were made on this phenomenon. First it seems to be reversible; by applying a
52 multi-directional bending of the contact area, the lustering disappears and individual fibers re-appear.
53 Also, SEM (Scanning Electron Microscopy) observations were made on lustered fibers. The SEM images
54 show no sign of abrasion and a very low surface roughness for the lustered area. It is almost impossible
55 to distinguish the individual fibers in this region.

56

57 Based on these first observations, a literature review of previous studies was performed. Caldwell et al.
58 (2005) studied the orientation of fibers in wool fiber assemblies. Wool fibers are made of a multitude of
59 fiber scales from nanometer to around 20 micrometers diameter for the final fiber. Through tomography
60 observations, Caldwell et al. showed different wool fiber arrangements with respect to the neutral axis. A
61 specific fiber arrangement was observed in which wool fibers are compacted in their section in a prismatic
62 pattern and the wool filaments are parallel to one another. This kind of arrangement was also observed
63 by Bunsell (2018) for Dyneema™ SK76 fibers. For ballistic applications, Golovin & Phoenix (2016); McDaniel
64 et al. (2017) and Sockalingam et al. (2017) studied the effect of transverse compression on HMPE filament
65 behavior and on its meso- and nano-structure. McDaniel et al. (2017) observed a misalignment and a lack
66 of compaction of the fibrils which composed the HMPE filaments after a transverse compression. Also,
67 Golovin & Phoenix (2016) showed a drop in tensile strength of the HMPE filament after a transverse
68 compression caused by a decrease in crystallinity from 95 to 80 %, and a reduction in crystallite size by
69 about a factor of two. These conclusions have been supported with Differential Scanning Calorimetry
70 (DSC) analysis and birefringence images. Golovin & Phoenix (2016) observed that HMPE filaments become
71 optically transparent when they are thin enough. These previous studies on transverse compression were
72 made at ambient temperature. Some other studies focused on the microstructure of HMPE (Dyneema™)
73 fibers under the influence of the temperature and stretching. Tian et al. (2015) showed through wide-
74 angle X-ray scattering (WAXS) analysis and Raman spectroscopy that a macroscopic deformation induced
75 microstructural changes in HDPE films: a lamellar structure could transform into micro-fibrils with stress-
76 induced melting and crystallization mechanisms (Jiang et al., 2007; Tang et al., 2007). Also, Tian et al.
77 (2015) observed that a high temperature helps the formation of fibrillar crystals. The transition between
78 lamellar and "shish-kebab" states happens smoothly in SAXS observations. Forster et al. (2015)
79 demonstrated that HMPE fibers exposed to high temperatures may undergo shrinkage and loss of
80 orientation. McDaniel et al. (2015) compared several types of fibers with a high-resolution microscope and
81 WAXS observations. That study showed a modification in the microstructure of HMPE fibers during the
82 stretching process. Finally, through WAXS observations and Raman spectroscopy, Litvinov et al. (2011)
83 concluded that an increase of the fiber deformation during the stretching process leads to an increase in
84 the degree of crystallinity and an increase in the fiber modulus. Also, a specific process of hot compaction
85 to create HMPE fibers and polypropylene (PP) fiber composite was studied by Hine et al. (1993), Olley et

86 al. (1993), Kabeel et al. (1994), El-Maaty et al. (1996), Yan et al. (1997) and Morye et al. (1999). A specific
87 fiber arrangement has been observed by Olley et al. (1993), Kabeel et al. (1994), Yan et al. (1997) for HMPE
88 fibers and by El-Maaty et al. (1996) for PP fibers. This hot compaction process produces a homogeneous
89 material with similar original fiber properties. An optimal set of parameters (temperature, pression, time)
90 allows a portion of fibers to melt. On cooling, the molten material recrystallizes to form a "glue" to bind
91 the structure together. The thermomechanical loading is similar to that of the HMPE rope loop in the
92 contact area. For the HMPE rope loop, fibers are also stretched. The temperature for the hot compaction
93 process is near the melting temperature of HMPE fibers, which is higher than for the maximal service
94 temperature of the pulley or for a hot bedding-in. Kabeel et al. (1994) explained that a raised temperature
95 induces softness, so that the application of pressure compresses the fibers into a near-solid mass. He
96 observed that this produces hexagonal arrays. Olley et al. (1993) showed that at 138 °C, melting (and
97 recrystallization) only occurs in regions where molten polymer is not constrained to remain oriented.

98

99 Based on the first observations made and these previous studies on the microstructure of fibers, four
100 hypotheses can be made to explain the appearance of "lustering" on the contact surface of the HMPE
101 rope loop:

102

- 103 1. Coating migration to the outer surface of the HMPE loop
- 104 2. Fiber compaction in the transverse direction
- 105 3. Plasticity in HMPE fibers.
- 106 4. Microstructure modification due the complex thermomechanical loading undergone by the
107 HMPE loop

108

109 In order to test these hypotheses one by one, various observations and analyses have been made. Based
110 on the results, the discussion will provide conclusions about the validity of each of these hypotheses.

111

112 1. Material and Methods

113 1.1. Material

114

115 Different grades of Dyneema™ HMPE fibers were used during the study: Dyneema™ SK99 and SK78. First
116 observations were made on a Dyneema™ SK78 braided rope with a 500kN break load. Other observations
117 were made on Dyneema™ SK99 fibers with and without coating. The samples were provided by the
118 Lancelin Corderie (Ernée, France).

119

120 1.2. Samples and Experimental Devices



Figure 2.a. The different scales of the Dyneema™ SK99 braided rope of the study with a 4mm diameter, coating and a Mean Breaking Load (MBL) of 14.7kN.

Figure 2.b. The different scales of the Dyneema™ SK78 braided rope with a diameter of 22mm, coating and a MBL of 500kN (Sample K in red)

122

123 The braided ropes have different component scales, from the largest to the smallest as follows: the
 124 braided rope, the strand, the yarn and the filament (Figure 2.a). For bigger ropes, some extra scales can
 125 be added such as sub-strands and rope yarns. The fibers without coating are white in contrast to the
 126 coated ones which are grey. The composition of the coating is proprietary and confidential.

127

128 Different kinds of samples were employed in order to investigate the four hypotheses made to explain
 129 the appearance of the “lustering” phenomenon. These are listed below in Table 1:

130

Sample	Type	Scale (see Figure 2)	Fiber state (see Figure 3)	Loading (see Figure 4)
A	SK99	Part Textile axis	coated, lustered and not lustered	Dynamic loading
B	SK99	Part Textile axis	uncoated, lustered and not lustered	Dynamic loading
C	SK99	Strand from 4mm diameter rope	uncoated	None
D	SK99	Strand from 4mm diameter rope	coated	None

E	SK99	Strand from a textile axis	coated, lustered	Dynamic loading of 2kN
F	SK99	Strand from a textile axis	coated, lustered	Quasi-static loading of 8kN
G	SK99	Strand from a textile axis	coated, not lustered	Dynamic loading of 2kN
H	SK99	Strand from 4mm diameter rope	coated	bedding-in process at ambient temperature
I	SK99	Strand from 4mm diameter rope	coated	bedding-in process at 100°C
J	SK78	4mm diameter rope	coated, not lustered	None
K	SK78	Yarn from 22mm diameter rope	coated, lustered	Hot bedding-in
L	SK78	Yarn from 22mm diameter rope	not lustered	Hot bedding-in and de-lustering process

Table 1. Samples examined

131

132 The details of the samples are described according to the type of observation.

133 The difference between the fibers coated and uncoated can be seen on Figure 3.a and Figure 3.b.

134 Uncoated HMPE fibers are white while coated HMPE fibers for this study are gray.

135



Figure 3. a. Coated HMPE fiber rope, 4mm diameter

Figure 3. b. Uncoated HMPE fiber rope, 4mm diameter

136

137 Experimentally, two different methods are used to rotate the pulley: dynamic loading and quasi-static

138 lading. For both loading a specific device is needed. Figure 4.a and Figure 4.b show the different loadings.

139

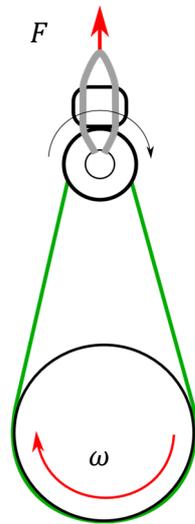


Figure 4.a. Dynamic loading

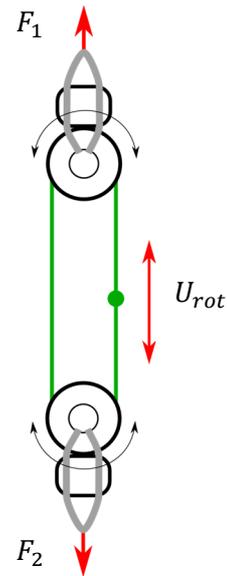


Figure 4.b Quasi-static loading

140

141 In the case of dynamic loading, the pulley is constantly rotating with an electric motor ($W = 51rpm$ which
 142 leads to a sliding speed around 0.2m/s) until the pulley reaches a stable temperature. The force F is
 143 applied by a tensile test machine.

144 For quasi-static loading, the force F is applied through a specially designed frame and a tensile test
 145 machine applies the displacement U_{rot} . The pulley is alternatively rotating with a displacement speed of
 146 20mm/min. The number of cycles was chosen arbitrarily, and fixed here at 100 cycles. The pressure results
 147 from the contact area, the geometry of the pulley and the global applied tension. A pressure of 71 MPa
 148 was calculated (based on measurements by Bain (2020)).

149 The bedding-in process at 100°C for sample I corresponds to one load/unload cycle at 20% stretching at
 150 100°C of a 4mm diameter rope. And for the bedding-in process at ambient temperature for sample H it
 151 corresponds to one load/unload cycle at 3% stretching at ambient temperature of a 4mm diameter rope.

152 A de-lustering protocol was applied to lustered sub-strands. The protocol is described on Figure 5. It
 153 consists of unpacking and tousling fibers by bending the lustered sub-strands in several directions.

154

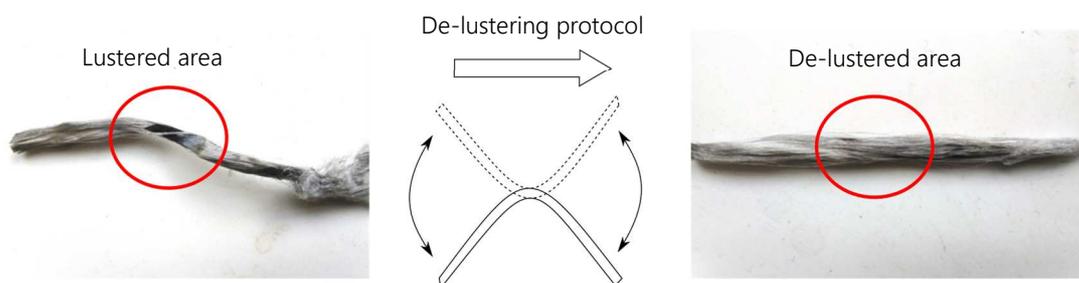


Figure 5. De-lustering protocol

155

156 1.2.1. Scanning Electron Microscope (SEM) samples

157

158 The zone of interest extracted from the rope for SEM depends on whether it is a single fiber, a strand or
159 an entire braided rope section. For this study strands, braided rope sections and parts of the textile axis of
160 the pulley were observed. In order to have the best image, the electron beam gun of the SEM has to be
161 relatively close to the samples. For this reason, in the case of the textile axis, the sample has the geometry
162 shown in Figure 6. The SEM used for these observations is a JEOL JSM-IT300 at ENSTA Bretagne.

163

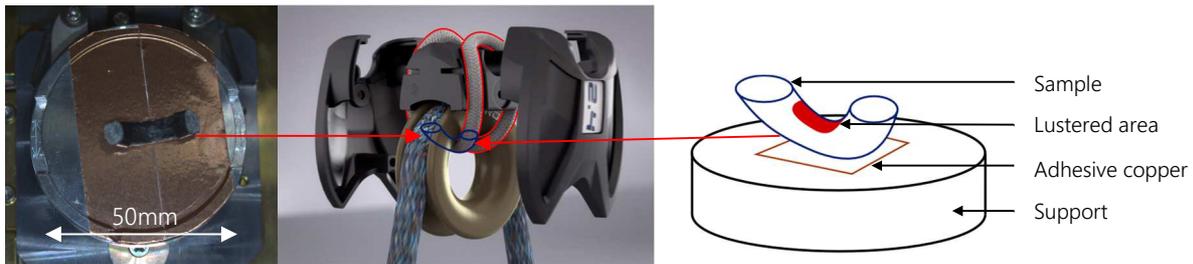


Figure 6. Sample A: Part of 4mm diameter textile axis with lustered area

164

165 The other samples are placed on a support as shown in Figure 7 (top view).

166

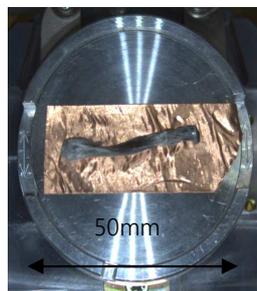


Figure 7. Sample K (see Table 1 and Figure 2b) before SEM observations, yarn diameter around 3mm

167

168 SEM settings differ according to the thickness of the sample and the density of the fibers. Good quality
169 images and a high magnification of the sample surfaces can be obtained. Furthermore, SEM is preferred
170 as HMPE fibers are transparent which makes optical microscopy observations difficult.

171

172 1.2.2. Digital microscopy samples

173

174 Because of the transparency of the HMPE fibers, digital microscopy was not used for surface
175 observations. However, it was used to observe the section of fibers, which were first cast in epoxy resin.
176 To have a good image quality, the observation area of the sample has to be systematically polished from
177 coarse to mirror finish (Figure 8.). Different samples were extracted from a 500kN break load braided rope

178 that experienced a hot bedding-in treatment during manufacture to stabilize its fiber assembly and
179 mechanical properties.

180 The observation of the 500kN braided rope was chosen as it included numerous lustered areas. The
181 section from a lustered textile axis made of Dyneema™ SK99 was examined to validate the observations
182 and the conclusions made for the Dyneema™ SK78 fibers extracted from the 500kN braided rope. A
183 disadvantage of the latter is that the level of the bedding-in load and the temperature history were
184 unknown.

185



Figure 8. Sample K cast in resin ready for digital microscope observations

186

187 1.2.3. Differential Scanning Calorimetry (DSC) samples

188

189 The crucibles of DSC analyzers are quite small. Two methods were used to place the fibers in the crucible,
190 depending on the kind of sample. If the fibers were not lustered, the strand was extracted. Then it was cut
191 into small pieces with a ceramic knife. If on the other hand the fibers were lustered, only the compacted
192 fibers from the upper surface were selected and placed in the crucible.

193

194 1.2.4. WAXS samples:

195

196 Wide Angle X-ray Scattering (WAXS) tests were performed at the Laboratory for Solid-state Physics at the
197 University of Paris-Sud. Strands were extracted from rope in different states. These samples were
198 positioned between two pieces of cardboard in order to be ready to put in the WAXS experimental set-
199 up (Figure 9).



Figure 9. WAXS sample

200

201 Diffraction patterns were recorded with a MAR345 detector mounted on a rotating anode X-ray
202 generator (copper anode, small focus, 40kV, 40mA). Multilayer optics are used to provide a
203 monochromatic and parallel beam (wavelength 0.1542nm). Acquisition times were of the order of a few
204 minutes.

205

206 2. Results

207 2.1. Visual and SEM observation

208

209 First, visual and SEM observations were made in order to understand this “lustering” phenomenon on
210 coated and uncoated specimens.

211

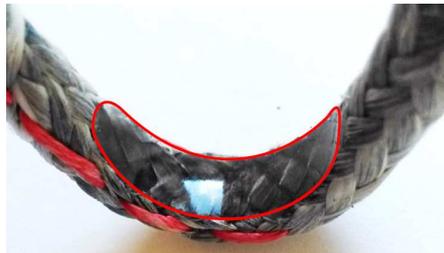


Figure 10. Visual observations of the contact surface between the textile axis and the ring of the pulley.

The “lustered” area is outlined in red.

212

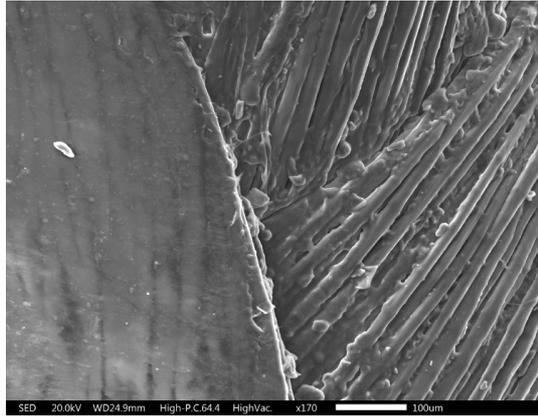


Figure 11. First SEM observations of lustered fibers (on the left side of the image).

213



Figure 12.a. Visual observations of sample B

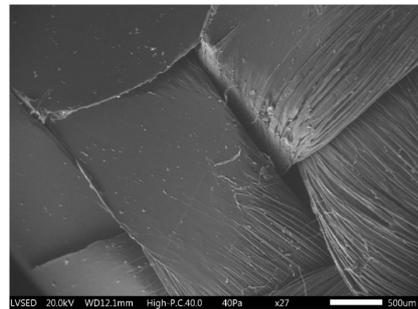


Figure 12.b. SEM observations of sample B

214

215 2.2. Digital microscope observations

216

217 Digital microscope observations were made on sub-strands extracted from the 500kN breaking load
 218 braided rope described previously (Sample K, see Figure 2.a) and on strand from 4mm diameter rope
 219 coming from the factory (Sample J, see Figure 2.b). The 500kN breaking load braided rope had
 220 undergone a hot bedding-in. The yarn from this rope, similar to the HMPE rope loop of the pulley, had
 221 been subjected to a complex mechanical loading with temperature rise, compression and tension loading.
 222 The lustered areas of sample K are located in the strands. The 4mm diameter rope coming from the
 223 factory had not been bedded-in. It can be considered that the filaments inside the sample J have not
 224 been subjected to any previous mechanical loading. Following the experimental protocol described
 225 earlier for SEM observations, Figure 13 and Figure 14 were obtained.

226

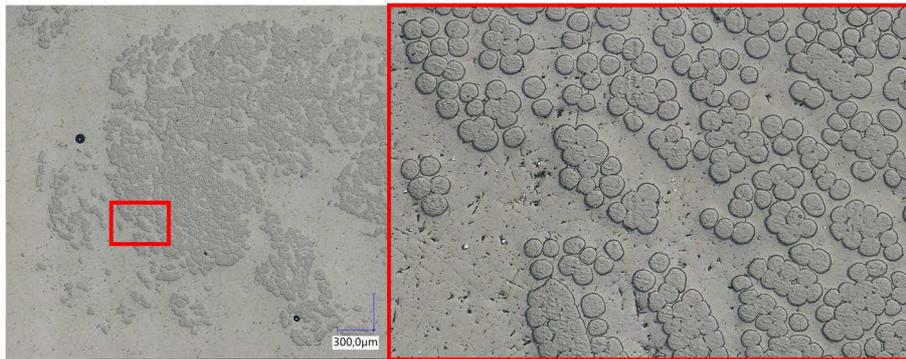


Figure 13. Digital microscope images of sample J

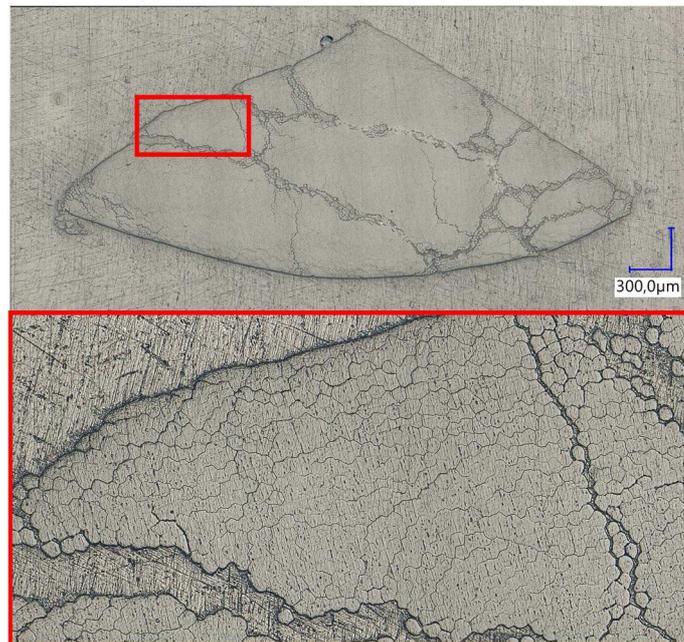


Figure 14. Digital microscope images of sample K

227

228 The section of a de-lustered area was observed with the digital microscope, cf Figure 15.

229

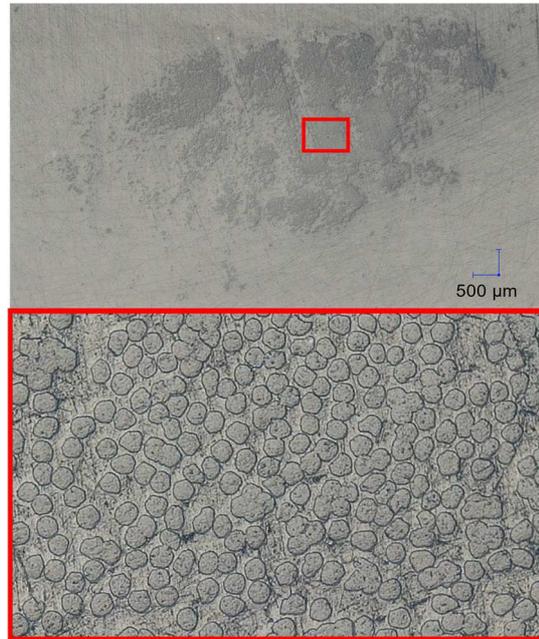


Figure 15. Digital microscope images of sample L after applying the de-lustering protocol.

230

231 After the de-lustering procedure, the fiber sections look very similar to their initial state. In order to
 232 quantify the evolution of the fiber's diameter and section, digital microscope measurements were made
 233 on several fiber sections (Figure 16.). Table 2. presents the results obtained.

234

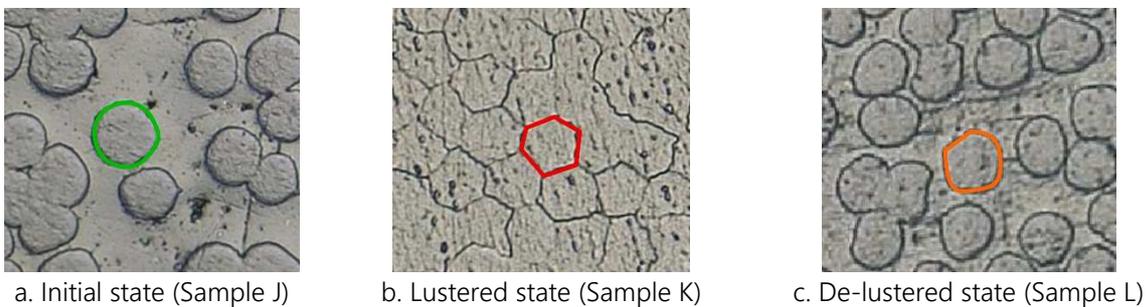


Figure 16. Evolution of the fiber sections for the different states

235

		Mean area in μm^2	Standard deviation on 10 measurements in μm^2	Mean equivalent diameter in μm
State	Initial	340	77.3	20.8
	Lustered	243	32.7	17.6
	De-lustered	313	49.7	20

Table 2. Mean areas of the fiber sections according to their state

236

237 2.3. DSC results

238

239 HMPE is known to be a fiber with a very high degree of crystallinity (greater than 90%). DSC (Differential
240 Scanning Calorimetry) measurements were performed for different HMPE fiber states. Figures 17 shows
241 the results for the different DSC samples. To refer to each sample, i is the number of the test.

242

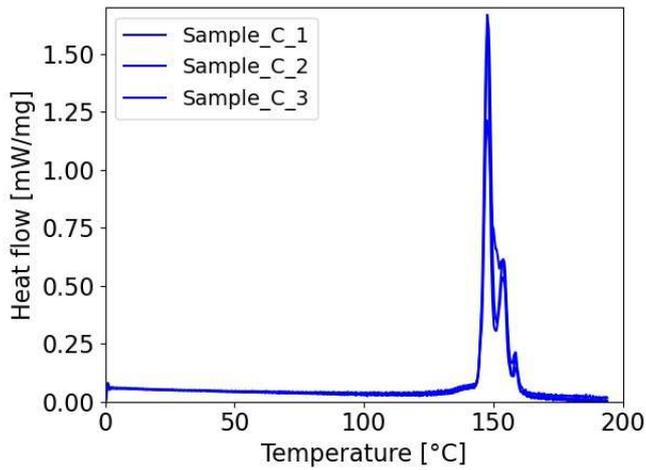


Figure 17.a. DSC results for samples D

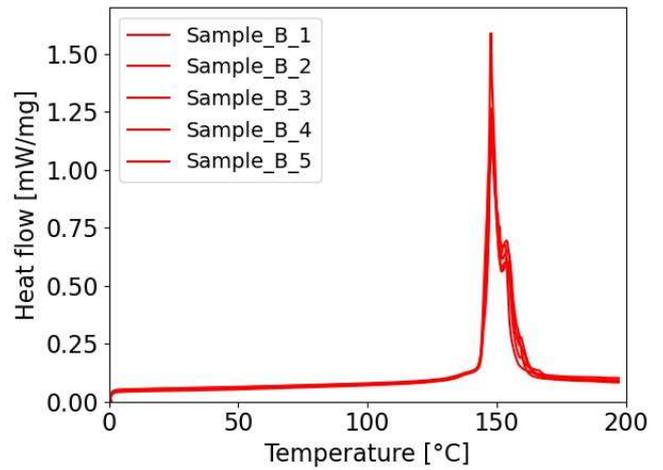


Figure 17.b. DSC results for samples C

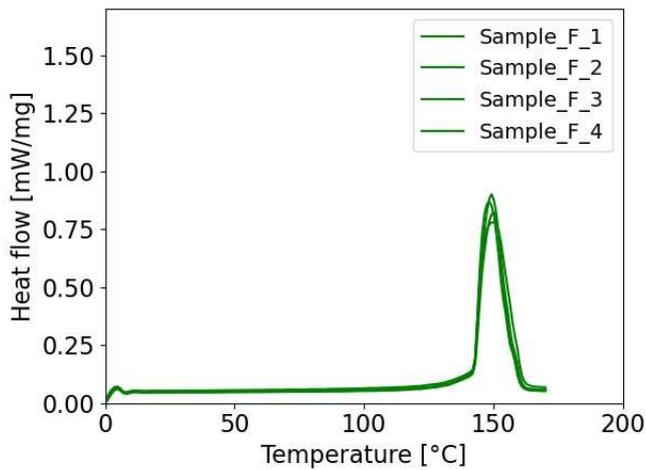


Figure 17.c. DSC results for samples G

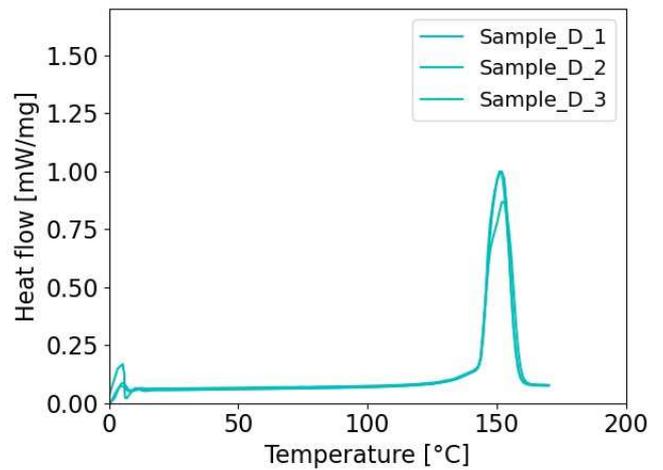


Figure 17.d. DSC results for samples E

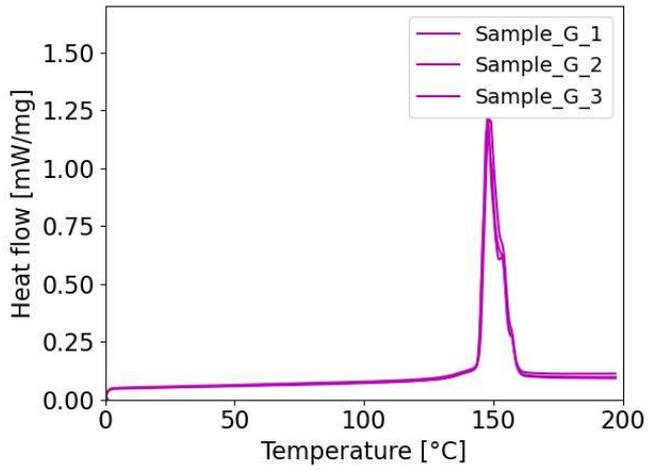


Figure 17.e. DSC results for samples H

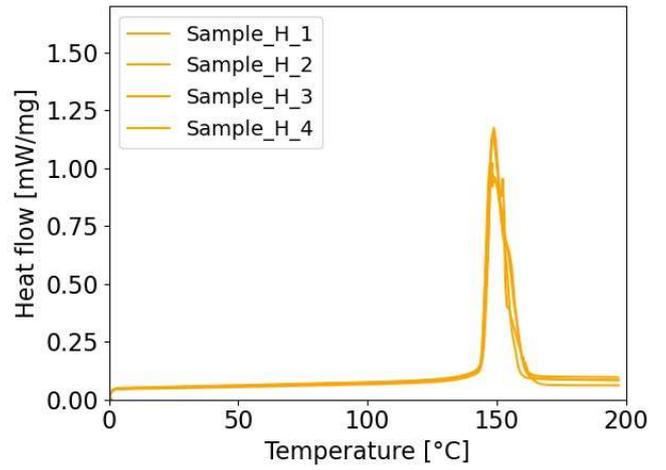


Figure 17.f. DSC results for samples I

243

244 2.4. WAXS results

245

246 WAXS (Wide Angle X-rays Scattering) was also performed for different HMPE fibers states. Figure 18

247 shows the results for the different WAXS samples.

248

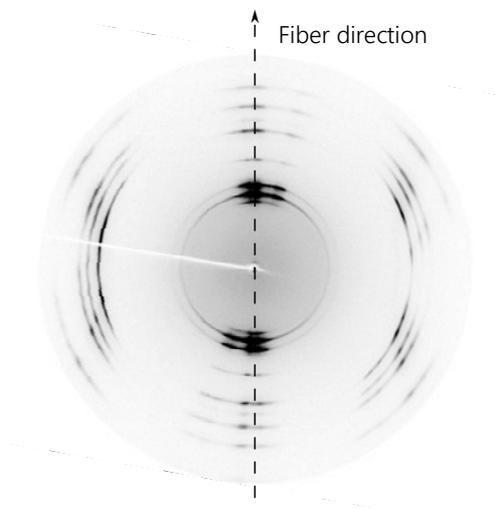


Figure 18.a. WAXS results for sample D

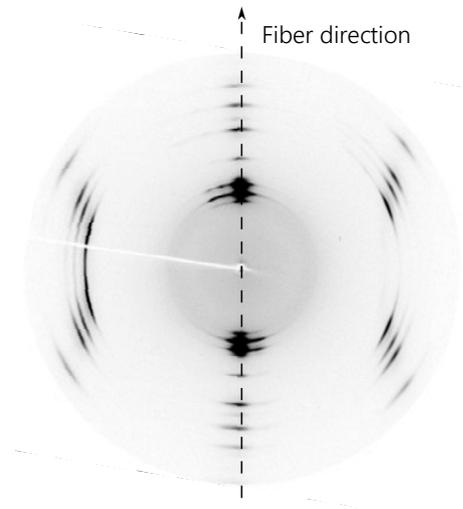


Figure 18.b. WAXS results for sample E

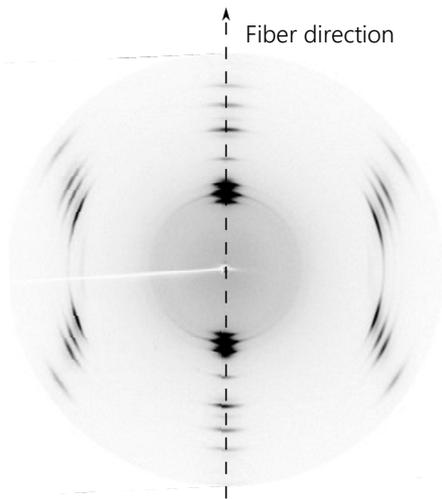


Figure 18.c. WAXS results for sample F

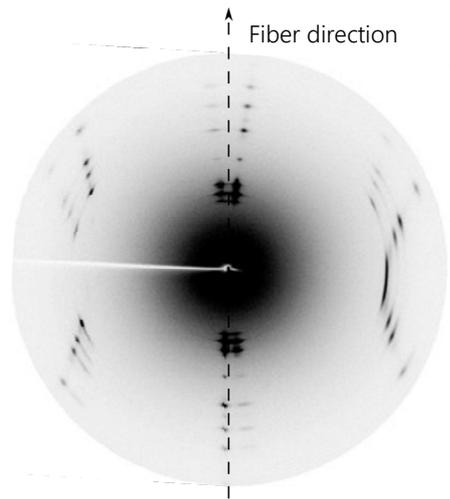


Figure 18.d. WAXS results for sample H

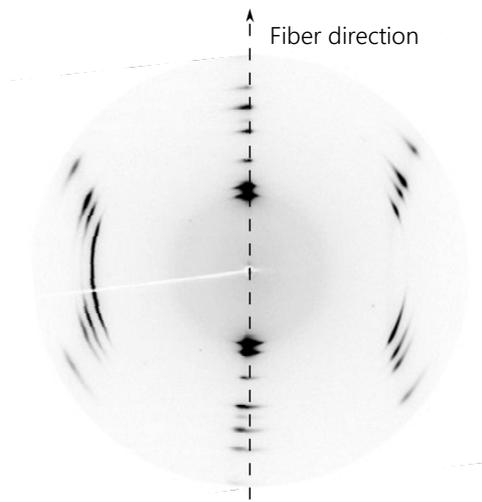


Figure 18.e. WAXS results for sample I

249

250 All these experimental results will now be discussed.

251

252 3. Discussion

253

254 First, visual and SEM observations were made in order to analyze this “lustering” phenomenon. As shown
 255 in Figure 10 and Figure 11, the lustered area can be characterized by several aspects such as:

256

257

- Darkening of the color and hardening of the contact area
- Optical reflectance for visual observations
- Difficulty to identify the individual fibers in the lustered area even with SEM observations.

258

259

260

261

262 SEM and digital microscope observations were used to examine three of the four hypotheses about the
263 appearance of “lustering” on the contact surface of the HMPE loop: the migration of the coating, the
264 compaction of the fibers, and the plasticity.

265

266 3.1. Migration of the coating

267

268 This first hypothesis was quickly checked by observing lustering on fibers without coating as shown on
269 Figure 12.a and 12.b. Darkening of the surface is less evident but the optical reflectance and the hardening
270 of the area were still identifiable. Also, on SEM images (Figure 12.b.) similar observations to those in
271 Figure 8. can be made, where fibers are difficult to identify separately in lustered areas.

272 Thus, it can be concluded that the migration of the coating is not the cause of the lustering phenomenon.
273 A coating may nevertheless be important in improving the abrasion resistance and affecting global
274 friction between fibers and other materials. For the other hypotheses, more detailed observations were
275 made.

276

277 3.2. Compaction

278

279 The observations of the cross sections of fibers with and without “lustering” on Figure 13 and Figure 14
280 are very interesting. The lustered sample shows smooth contours. The original circular sections of the
281 fibers are highly compacted until reaching a prismatic “paving” shape, nearly hexagonal. Therefore, fibers
282 tend to follow a spherical packing arrangement in which the fibers fill as much of the space as possible. It
283 is difficult to distinguish fiber sections in the packing area; the fibers are deformed to fill the remaining
284 space. Similar observations have already been made on wool fibers by Caldwell et al. (2005). This
285 arrangement was also visible on observations of Dyneema™ SK76 fibers made by Bunsell (2018). Olley et
286 al. (1993), Kabeel et al. (1994), Yan et al. (1997) for HMPE fibers and El-Maaty et al. (1996) for PP fibers,
287 observed similar fiber arrangements produced by a hot compaction process.

288 The temperature reached by the pulley near the contact area is around 70 °C during a dynamic loading of
289 2kN (Bain, 2020). This is significantly lower than the temperature used for hot compaction process. If the
290 compaction temperature is too low, there is insufficient melt to fill the interstices, the fibers deform into
291 polygonal shapes, and insufficient transverse strength is developed (Kabeel et al., 1994). Nevertheless,
292 some portion of HMPE fiber surfaces may melt at the contact area. Further investigations need to be
293 carried out to observe interstitial material between HMPE fibers, in a similar way to the work of Olley et al.
294 (1993).

295

296

297 3.3. Plasticity/ Permanent strain

298

299 Due to the heavy compaction that fibers undergo, the question of possible transverse plasticity/
300 permanent strain of the fibers can be posed. McDaniel et al. (2017) performed transverse compression
301 tests on Dyneema™ SK76 filaments with multiple load/unload cycles. The tests showed a non-linear
302 behavior with a stiffening under high strains. This increase in the stiffness can explain the apparent
303 hardening of the lustered area. During each unload, the filament does not return to its original diameter,
304 which indicates the presence of plasticity, cf Figure 19. However, the lustering phenomenon seems to be
305 reversible when a de-lustering protocol is used.

306

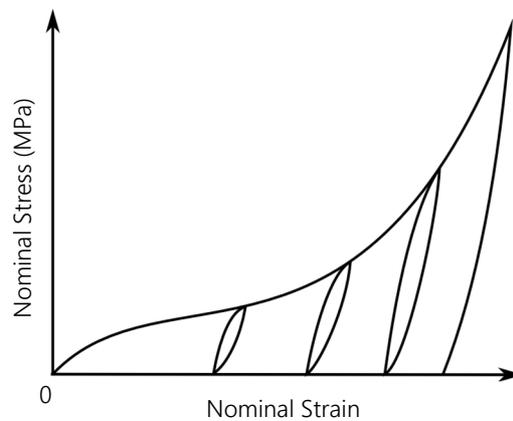


Figure 19. Mechanical response of HMPE filament with multiple char/discharge cycles on transverse compression (based on tests by McDaniel et al. (2017))

307

308 The evolution of the section of samples J, K and L can be observed and compared from Figure 17 and
309 Table 2, in order to characterize the transverse plasticity of HMPE filaments. After applying the de-
310 lustering protocol, the fibers seem to have returned to their initial state, cf Figure 13. The fibers undergo a
311 transverse deformation of around 28.5% between their initial state and the lustered state. These fibers
312 had a residual diametral strain of around 4% compared to their initial shape. This plasticity can be
313 explained by the fact that the filament is also itself an assembly but at a smaller scale. SEM observations
314 were made on the disassembly of a HMPE filament (Figure 20), and even smaller fibrillar elements, known
315 as fibrils, can be observed.

316

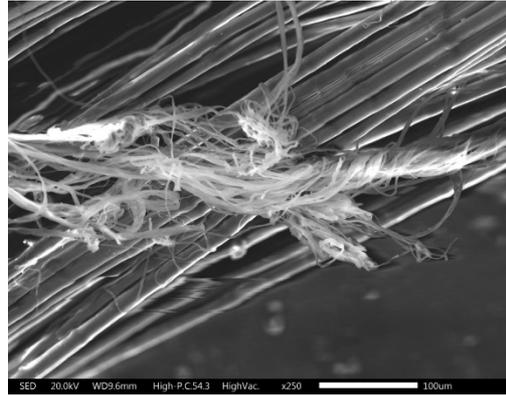


Figure 20. Disassembly of a HMPE filament

317

318 The filament is a complex structure of fibrils and voids. This suggests that there may be no plasticity
 319 within the fibrils but only suppression of the inter-fibril voids during contact, i.e. a change in the internal
 320 filament organization. The de-lustering protocol can make it possible to restore voids within and between
 321 the filaments.

322

323 An additional argument to rule out plasticity as a major factor of the analysis is to evaluate what would be
 324 the longitudinal strain induced by the radial strain observed, if plasticity was the main cause. In the case
 325 of pure plastic material, the volume is kept constant during the transformation, which leads to the
 326 relation:

327

328

$$\lambda_1 \lambda_2 \lambda_3 = 1$$

329

330 With:

331

- $\lambda_1 = L/L_0$ stretch in fiber direction with L deformed length and L_0 original length.

332

- $\lambda_2 \lambda_3 = (R/R_0)^2 = (\phi_{mean}/\phi_{init})^2 = (17.6/20.8)^2 = 0.716$ with λ_2 and λ_3 stretch in radial direction, R deformed radius, R_0 original radius, ϕ_{mean} mean equivalent diameter at de-lustered state and ϕ_{init} mean equivalent diameter at initial state.

333

334

335

336 Then for the case of constant volume strain, the elongation in the fiber direction is:

337

338

$$\lambda_1 = 1/0.716 = 1.40$$

339

340 This means that the induced axial deformation would be around 40%. However, the results obtained
 341 under monotonic tension tests show that the filament strain at break is around 4% (Figure 21). These two
 342 values therefore seem inconsistent and plasticity does not seem to be a key factor involved in the

343 lustering phenomenon. However, the high compaction and/or alignment of the fibrils within the filament
344 does have a strong influence on the creation of this lustering phenomenon.
345

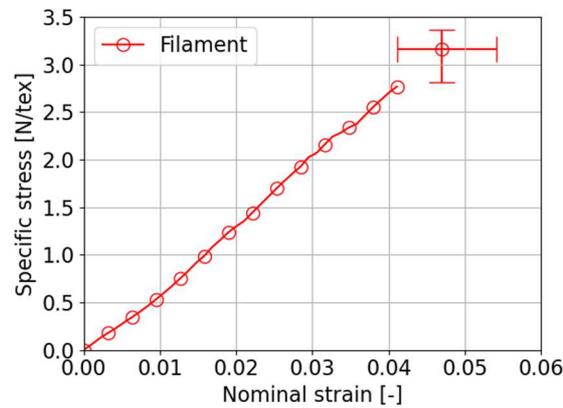


Figure 21. Monotonous axial tensile loading on filament of Dyneema™ SK99 (Mean behavior from 3 tests), Bain et al. (2020)

346

347 3.4. Modification of the microstructure

348

349 The final hypothesis to explain the lustering phenomenon is a modification of the microstructure
350 of the HMPE fibers. HMPE is known to be a fiber with a very high degree of crystallinity (greater than
351 90%). In order to examine the hypothesis that this changes during lustering, DSC (Differential Scanning
352 Calorimetry) measurements and WAXS (Wide Angle X-rays Scattering) were performed.

353

354 3.4.1. DSC analysis

355

356 The degree of crystallinity χ_c is defined by the ratio between the fusion enthalpy of the polymer ΔH_f and
357 the theoretical fusion enthalpy of the 100% crystalline material ΔH_f^0 :

358

$$359 \chi_c = \Delta H_f / \Delta H_f^0$$

360

361 3.4.1.1. Coating influence

362

363 Figure 17.a., Figure 17.b. and Table 3. show the results for the various measurements carried out on
364 Dyneema™ SK99 coated (sample D) and uncoated (sample C).

365

366 Examining the heat flow curves as a function of temperature, we note that the trend is the same whether
 367 for coated or uncoated HMPE fibers. There is a main peak and two secondary peaks. The main peak
 368 characterizes the melting temperature of the main polymer chain. The melting temperatures are:

369

- 370 • $T_{fusion_D} = 147.6\text{ }^{\circ}\text{C}$ with a standard deviation of 0.12 °C from 3 tests
- 371 • $T_{fusion_C} = 147.9\text{ }^{\circ}\text{C}$ with a standard deviation of 0.06 °C from 5 tests

372

373 According to the manufacturer of these fibers, the melting point is between 144 °C and 152 °C. The
 374 temperatures obtained experimentally are within the range of the expected values.

375

376 With the mean values of fusion enthalpy obtained for HMPE fibers in sample C and B, and with a fusion
 377 enthalpy of 100% crystalline polyethylene ΔH_{fPE}^0 taken equal to 285 J/g. The degrees of crystallinity
 378 obtained are as follows:

379

- 380 • $\chi_{c_D} = 85.3\%$ with a standard deviation of 0.8% over 3 tests
- 381 • $\chi_{c_C} = 95.3\%$ with a standard deviation of 2.9% over 5 tests

382

383 There is a significant difference in the degrees of crystallinity of the two materials. This can be explained
 384 by the coating (C). As the latter contributes to sample weight but is not a material with a high degree of
 385 crystallinity, it reduces the apparent degree of crystallinity of Dyneema™ SK99.

386

Test number	Sample D	Sample C
1	242.0	280.4
2	241.5	273.5
3	245.6	277.8
4	/	261.7
5	/	264.6
Mean	243.0 J/g	271.6 J/g

Table 3. Fusion enthalpies in J/g for the two samples C (coated) and D (uncoated)

387

388 3.4.1.2. Lustering influence

389

390 Figure 17.c., Figure 17.d. and Table 4. show the results for the various measurements carried out on
 391 Dyneema™ SK99 fibers coated, not lustered, and extracted from a textile axis (Sample G) and Dyneema™

392 SK99 fibers coated, lustered and extracted from the same textile axis (Sample E). The samples were taken
 393 from an INO-BLOCK® pulley textile axis that underwent a dynamic loading. As a result, fibers had a
 394 complex mechanical loading together with heating. Extracting the two samples from the same loop
 395 reduces the influence of temperature on the results.

396
 397 The heat flows for samples (G) and (E) are very similar. However, there is a change in the shape of the
 398 melting peaks compared to samples (D) and (C). This difference may indicate that changes have taken
 399 place in the crystalline phase of the material. The melting temperature are:

- 400
 401 • $T_{fusion_G} = 151.9\text{ }^{\circ}\text{C}$ with a standard deviation of $0.36\text{ }^{\circ}\text{C}$ for 3 tests
 402 • $T_{fusion_E} = 149.3\text{ }^{\circ}\text{C}$ with a standard deviation of $0.83\text{ }^{\circ}\text{C}$ for 4 tests

403

Test number	Sample G	Sample E
1	263.8	254.1
2	267.4	266.0
3	273.6	261.4
4	/	273.0
Mean	268.3 J/g	263.6 J/g

Table 4. Fusion enthalpies in J/g for the two states G (not lustered) and E (lustered)

404
 405 The degrees of crystallinity obtained are as follows:

- 406
 407 • $\chi_{c_G} = 94.1\%$ with a standard deviation of 2.78% over 3 tests
 408 • $\chi_{c_E} = 92.5\%$ with a standard deviation of 2.79% over 4 tests

409
 410 The crystallinity ratios obtained for the two materials are quite close but slightly higher for the lustered
 411 zone. The fiber used for the INO-BLOCK pulley loops is coated. If we compare the values of the
 412 crystallinity ratios obtained for samples G and E to the coated raw material from the factory (D); there is a
 413 notable difference. This increase in the rate of crystallinity of the HMPE loop can be explained by various
 414 factors. First, the complex mechanical loading can lead to reorientation of the fibers and crystallization
 415 (Tian and al., 2015). Second, the temperature rise on the pulley during loading can also induce the
 416 creation of crystals. This is why further analyses were carried out to understand the influence of
 417 mechanical loading and temperature.

418

419 Furthermore, in comparison to DSC results from Hine et al. (1993), no low-melting peak can be observed.
 420 It can be concluded that no significant portion of fibers has been melted. Indeed Hine et al. (1993)
 421 showed the appearance of a low-melting peak on the DSC melting endotherm when only 8% of fibers
 422 had been melted. So, either the melted fibers in the lustered cannot be measured through DSC or there
 423 are none. A morphological study could be performed to look at the regions between fibers in more
 424 detail.

425

3.4.1.3. Influence of Bedding-in and temperature

427

428 Figure 17.e., Figure 17.f. and Table 5. show the results for the various measurements carried out on the
 429 coated SK99 fibers with a bedding-in process at ambient temperature (Sample H) and Dyneema™ coated
 430 SK99 fibers with a bedding-in process at 100 °C (Sample I). The samples I were taken from a braid which
 431 had undergone a monotonic tensile test at a temperature of 100 °C without going to failure but which
 432 exceeded 20% nominal strain. The samples H were obtained from a braid which had undergone
 433 monotonous tension loading at room temperature until failure (nominal strain around 4%). This
 434 difference in deformation should be considered in the analyses. However, the goal here is not to quantify
 435 the contributions of temperature and mechanical loading on the increase in the degree of crystallinity.
 436 For this, additional analyses would be required.

437

438 The evolution of the heat flows for samples H and I are very similar. However, a change in the shape of
 439 the melting peaks can be noted, compared to the material D where 3 distinct peaks are observed. For
 440 samples H and I, these 3 peaks tend to form only one. In addition, using these curves, the melting
 441 temperature can be determined for the different materials:

442

- 443 • $T_{fusion_H} = 146.7 \text{ °C}$ with a standard deviation of 1.81 °C for 3 tests
- 444 • $T_{fusion_I} = 144.9 \text{ °C}$ with a standard deviation of 0.75 °C for 3 tests

445

Test number	Sample H	Sample I
1	251.1	277.9
2	252.3	264.2
3	264.7	269.0
Mean	256.0 J/g	270.4 J/g

Table 5. Fusion enthalpies in J/g for the two samples H and I

446

447 The degrees of crystallinity obtained were as follows:

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- $\chi_{cH} = 89.8\%$ with a standard deviation of 2.64% for 3 tests
- $\chi_{cI} = 94.9\%$ with a standard deviation of 2.44% for 3 tests

The bedding-in at ambient temperature results in a 4.5% increase in the crystallinity ratio, while the bedding-in at 100 °C results in an increase of 9.5%. These increases are significant. The application of mechanical loading increases the degree of crystallinity within these fibers. Carrying out mechanical loading at a higher temperature facilitates the movement of molecular chains (Tian et al., 2015) and thus increases the degree of crystallinity for the bedding-in at 100 °C.

3.4.2. WAXS analysis

The analysis of 2D images from WAXS observations can be difficult to interpret for the untrained eye but the data obtained by X-ray diffraction are rich. Through measurements of the angle and the intensity of diffracted X-rays, it is possible to characterize the crystalline phase. For this study, the radial scan diffractogram is not analyzed. Only the diffraction pattern is used to characterize the microstructure of the fibers. Figure 22 illustrates the specific data of interest for this paper:

- the width “e” of a specific diffraction line is inversely related to the average dimension of crystallites along the considered direction: in other terms, the smaller the thickness “e”, the larger this average dimension. This analysis is limited by the intrinsic resolution of the set-up that can be estimated using a powder made of macroscopic crystallites (typically quartz or silicon powders). In the present case, the width for all observed diffraction lines is resolution-limited that means that average crystallite dimensions are for all cases too large to be evaluated with the present set-up.
- the angular spread “ $\Delta\alpha$ ” of the azimuthal lines characterizes the orientation of the crystallites with respect to the fiber axis. The smaller the spread, the more the molecular chains are oriented in the average fiber direction. Care has to be taken that the observed angular spread is a convolution of the crystallite orientation within the individual fibers with the orientation of the individual fibers with respect to the average fiber direction.

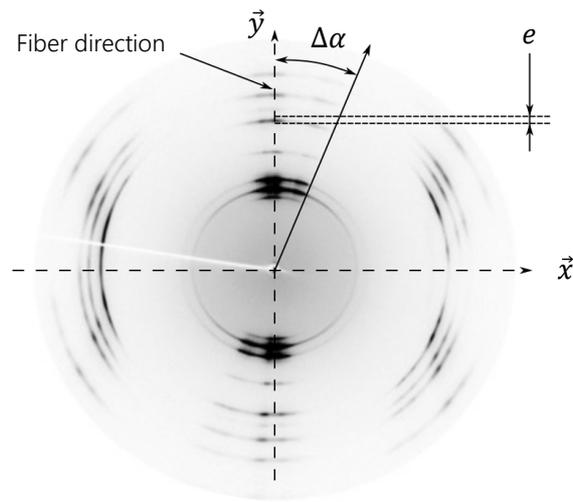


Figure 22. Data of interest on 2D image from WAXS observation

480

481 It would also be possible to analyze crystallinity ratio and crystal size based on these diffractograms, but
 482 that is not the aim here. It should be noted that during these measurements, the filaments making up the
 483 observed yarn are not perfectly aligned in the yarn direction. In addition, the network of chains that the
 484 X-rays must cross is complex. This explains the presence of other diffraction spots. For the case of a
 485 totally amorphous polymer material, a WAXS analysis would give a very diffuse ring of large thickness. For
 486 all the measurements performed, the presence of interrupted rings can be observed. This is characteristic
 487 of a crystal structure with a strong orientation of the crystallites in the fiber direction. We also note that
 488 the thickness of the rings is quite small, which confirms the presence of a material with a well-defined
 489 crystalline arrangement.

490

491 WAXS results on lustered fibers (see Figure 18.b and 18.c) show a slightly less diffuse ring with a smaller
 492 angle α than for the virgin fibers (see Figure 18.a.). The thickness e , is constant. These observations lead to
 493 the conclusion that lustering does not modify the crystal structure of HMPE fibers. The slight change in
 494 the crystal orientation in the fiber direction can be explained by the application of a tension on the fibers.
 495 To verify this hypothesis, two others samples were observed: sample H and sample I.

496 WAXS results from the bedded-in fibers at ambient temperature and at 100 °C (see Figure 18.d and 18.e),
 497 show a strong decrease in the alpha angle, especially for the case of bedding-in at ambient temperature.
 498 There is therefore a strong reorientation of the molecular chains in the direction of the fiber during
 499 bedding-in, that is not only affecting the rope construction. Another conclusion can be made from DSC
 500 measurements: sample H has a lower degree of crystallinity but the orientation distribution of the
 501 crystallites in the stretching axis seems to be stronger than for sample I. It seems that the higher
 502 temperature induces a higher crystallinity ratio but allows a faster relaxation of the crystals.

503

504 These WAXS analyses help to demonstrate the absence of change in microstructure between lustered
505 HMPE fibers and HMPE fibers that experienced a similar tension mechanical loading without lustering
506 (comparison between samples E and I). Indeed, the changes occurring in the microstructure of the
507 lustered fibers are mainly explained by the mechanical loading applied to them when the pulley is
508 tensioned. It has also been shown that it is still possible to improve the orientation of grade SK99
509 Dyneema™ fibers by applying a bedding-in (comparison between samples E and H). In order to optimize
510 the bedding-in protocol of ropes, it would be interesting to perform fiber analyses at different controlled
511 bedding-in levels and different temperatures, in order to analyze the evolution of the microstructure of
512 the fibers during the process.

513

514

515 Conclusion

516

517 This paper investigates the lustering phenomenon observed in the contact area between textile ropes and
518 a pulley ring. This phenomenon results in spectacular changes to the optical aspects of the contact area
519 as well as greatly improving the friction performance of the pulley. The lustering phenomenon is often
520 observed but is very poorly documented, and hardly reported for HMPE fibers. The complex
521 thermomechanical loading on the loop seems to be the main cause of the appearance of the lustering
522 phenomenon. Based on published literature on HMPE, several hypotheses are suggested to explain
523 lustering; coating effects, fiber compaction, fiber plasticity or changes to the crystalline microstructure.
524 Various samples have been studied, showing lustering or not, obtained under several mechanical loading
525 and thermal conditions. First, SEM observations showed that the “lustering” phenomenon is visible on
526 both coated and uncoated fibers, ruling out this hypothesis. Then, WAXS and DSC measurements showed
527 that while the loading conditions can induce a change of crystallinity ratio and crystal orientation, these
528 variations are related to the thermal and mechanical loading conditions but do not show any correlation
529 to the fact that the samples were lustered or not. Finally, digital microscope observations of the cross
530 section of fibers in the lustered area showed that fiber compaction is the main feature which is modified
531 when lustering occurs. This compaction seems not to be induced by any fiber plasticity but rather by a
532 rearrangement of the microfibrils. In conclusion, the high tension in the filaments of the loop as well as
533 the flexure and compression applied by the pulley ring, the sliding of the ring relative to the loop, and the
534 increase in the temperature lead to a specific arrangement of the filaments. This rearrangement leads to
535 lustering and to the strong improvement of the friction properties. It is now possible to define lustering
536 areas. However, it is difficult to quantitatively characterize the level of lustering that is achieved and
537 hence, the optimal service or process parameters required to induce it. To do so, additional investigations

538 would be helpful, such as micro-indentation tests to characterize the hardness of the lustered fiber
539 regions.

540

541

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543

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546

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