

## IMPACT OF THERMAL TREATMENTS APPLIED TO PET VASCULAR PROSTHESES ON FABRIC CONTEXTURES AND FIBER SURFACE FEATURES

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### ABSTRACT

For more than sixty years textile vascular prostheses have saved millions human lives but achieving attributes of a native artery still remains a challenge. Graft failures have been, in part, assigned to the prosthesis finishing processes, generally based on thermal treatments. These ones enable to reduce the water permeability and fix the wavy form of prosthetic tube walls involved by crimping processes. Four tubular structures have been woven with various polyester yarns (Setila, Dacron, Diolen and Viscosuisse) and then thermally crimped according to a predefinite protocol. The aim was to investigate the impact of crimping thermal treatments on the prosthetic fabric contextures and the fiber surface features. Optical microscopy showed tight compact warp-faced structures which became less porous after finishing treatments. Thermomechanical crimping constraints gave rise to some flattening traces, observed by SEM on Setila and Diolen fibers. All filaments presented smooth surfaces and round sections except Dacron ones which have irregular diameter and helical configuration resulting from texturing processes. This aspect decreased after prosthesis finishing, indicating an attenuation of the yarn texturing effect. Dacron filament surface is also partially marked by annular streaks, slightly protruding with pointed and sharp edges. Such a shape could be explained by the "skin-core" structure of these fibers, but its mechanical impact on the prosthesis performance remains unknown.

### KEYWORDS

Fiber morphology; heat setting; PET polyester; SEM; vascular prosthesis.

## 1. INTRODUCTION

The polyethylene terephthalate (PET) is widely used in several medical devices such as prosthetic vascular grafts, sutures and wound dressings (commercially known as Dacron® the first to be FDA approved). Despite the presence of hydrolytically cleavable ester linkage, PET is relatively stable *in vivo* mostly owing to the high crystallinity and hydrophobicity. It is one of the two standard biomaterials of prosthetic vascular grafts used clinically. A key requirement of materials in cardiovascular applications is the "3b": biocompatibility, biostability and biofunctionality. PET is recognized to be well suited for large vessels (Chevallier et al., 2003; Ben Abdessalem et al., 1999) with high flow rates and low resistance (notably aortic and carotid arteries). Although its tensile strength, tensile modulus and compliance are relatively lower than those of PET, expanded polytetrafluoroethylene (ePTFE) is currently the only synthetic substitute to bypass smaller vessels (less than 8 mm diameter) (Chlupác et al., 2009; Modjarrad, Ebnesajjad, 2014). It is produced by a series of extrusion, stretching and heating processes to create microporous structure imparting low thrombogenicity to graft wall.

Manufactured from multifilament yarns, PET prostheses are commonly available as woven or warp-knitted fabrics, which will determine the porosity of the graft as well as its mechanical properties and transmural

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blood extravasation levels. Anyway, textile implants show better aptitude for sutures than PTFE molded ones. They are particularly used in sites needing long grafts with satisfactory bending properties such as the above-knee femoropopliteal bypass. Since water permeability of textile structures, especially knitted ones, is too high to be pulled down only by subsequent impregnation, the compaction of these fabrics is necessary to bring their porosity within acceptable levels. This can be accomplished by either chemical or thermal treatments. We proved in previous works (Azaiez et al., 2010; Ben Abdesslem et al., 2009) that all compaction systems induce loss of the mechanical properties due to loss of crystalline orientation, chain scission and breaking intermolecular bonds in the polymer. Concurrently or subsequently to compaction, textile prostheses are commonly crimped to circumferentially stiffen their walls and increase their elongation potential. These "accordion" corrugated pleats enable the surgeon to control the longitudinal tension and improve the resistance to kinking, as well. Textile vascular prostheses are then sealed by coating with albumin, gelatin, collagen or other biodegradable synthetic materials before conditioning and sterilization (by alkylating gas treatment,  $\gamma$  radiation or plasma technologies).

Many advances have been achieved in last decades, notably in terms of biocompatibility (Chevallier et al., 2003; Chakfe et al., 1993), thrombo-resistance (Tara et al., 2014) and structural design (Mary et al., 1997; Kancevica et al., 2011) of PET implants. However, the ability to match attributes of a vascular substitute to those of a native artery still remains a challenge. Cases of prosthetic failure are regularly reported in the medical literature (Chakfe et al., 2001; Riepe et al., 1997; Zilla et al., 2007). Long-term degradation remains a problem for vascular surgeons. The etiology of late degradation is probably related to multiple factors such as the design of the textile structure (Dieval et al., 2003, 2008), alterations of the prosthesis during the manufacturing process (Dieval et al., 2012), and, possibly, secondary physicochemical alterations when the prosthesis is exposed to the systolic-diastolic arterial stress (Chaouch et al., 2009). These complications may lead to dilation and even rupture of the prosthesis.

Several researchers (Feldstein, Pourdeyhimi, 1990; Pourdeyhimi, 1986) demonstrated that the finishing treatments result in significant physical changes affecting the mechanical performance of PET yarns used in vascular grafts, which seriously degrade their healing process (Pourdeyhimi, 1987). Guidoin *et al.* (1992) proved that the filament swelling induced by compaction treatments is so strong that often individual fibers of a multifilament-yarn bundle are pressed against each other to such an extent that the yarns become mellow and show flat surfaces at the contact points. It is therefore suggested that the failure of these materials may be in part due to fiber alterations caused by prosthesis finishing treatments. However, the impact of these treatments on physical, chemical and morphological fiber properties has been rarely discussed in the literature.

In an earlier series of papers (Khlif et al., 2012; Azaiez et al. 2010), microstructural changes of PET fibers following prosthesis thermal finishing processes have been analyzed. In the present work, some fabric structures and fiber surface morphological evolutions related to prosthesis crimping treatments have been studied.

## **2. MATERIALS AND METHODS**

### **2.1. Materials**

In order to study morphological impacts of thermal finishing treatments applied to textile vascular grafts, four seamless tubular plain-weave fabric samples (F1-F4) have been manufactured. In such a pattern, weft yarns lie alternately over and under warp ones thus conferring maximum interlacing points on fabric and therefore best mechanical performances among all types of weaves (high burst strength and low tendency to fatigue) (Ben Abdesslem, Mokhtar, 2006; Mokhtar et al., 2010). To ensure minimal water permeability, loom settings, particularly pick counts, have been maximized so achieving weavability saturation. Plain weave fabrics with warp and weft cover factors of 12 in both directions are easy to weave. Thereafter, weaving becomes generally more difficult and for fabrics having cover factors of about 14 + 14, which is our case (Table 1), fairly strong weaving machines are required (Gandhi, Sondhelm, 2016). A narrow-fabric needle loom NFJM2 (Jakob Müller, Switzerland) has been therefore used for fabric sampling.

Table 1: Loomstate parameters of woven prosthetic tubes

Fabric structure	F1	F2	F3	F4
Weft Yarn/Warp Yarn	Y1/Y5	Y2/Y5	Y3/Y5	Y4/Y5
Warp count (ends cm <sup>-1</sup> )	28.0	27.3	27.3	27.3
Weft count (picks cm <sup>-1</sup> )	32.8	28.7	33.8	33.8
Warp cover factor <sup>a</sup> (ends cm <sup>-1</sup> tex <sup>0.5</sup> )	14.8	14.4	14.4	14.4
Weft cover factor <sup>a</sup> (picks cm <sup>-1</sup> tex <sup>0.5</sup> )	13.9	11.7	12.6	12.9
Area density (g m <sup>-2</sup> )	150.3	136.6	140.4	142.4
Thickness (mm)	0.25	0.25	0.24	0.25
Porosity <sup>b</sup> (%)	56.1	59.7	59.1	59.1
Ribbon width (mm)	32 x 2			

<sup>a</sup> calculated by multiplying the yarn count (per cm) by the square root of the yarn linear density (in tex) and dividing by 10 (Denton, Daniels, 2002)

<sup>b</sup> measured according to ISO 7198:2016 (gravimetric method)

Woven prostheses, commercially available, are generally manufactured with 90-320 dtex (10<sup>-4</sup> g m<sup>-1</sup>), either single or plied, polyester filament yarns (Guidoin et al., 1992). To both avoid poor sheds inducing fabric defects and fulfill raised longitudinal strength of prosthetic tubes, high-tenacity yarns are greatly recommended for warp. However, radial graft compliance is mainly dependent of mechanical and thermal behavior of weft yarns (Singh et al., 2015). The whole weaving has been performed with a single low-shrinkage 280 dtex warp (Y5) and a different weft has been used for each fabric (Table 1). Provided by several fiber spinning mills, weft yarns (Y1-Y4: Dacron and other PET commercial fibers) include similar fineness (140-180 dtex) but various structures (flat, textured, intermingled, twisted), different prior heat histories and hence different thermal behaviors (Table 2).

Table 2: Physical parameters of yarns used for fabric samples

Yarn specifications	Weft				Warp
	Y1	Y2	Y3	Y4	Y5
Trade name	Setila	Dacron	Diolen 57T	Viscosuisse	Diolen 61ST
Fiber manufacturer	Nouvelle Setila France	Invista USA	PHP Fibers Germany	Tersuisse Switzerland	PHP Fibers Germany
Yarn count (dtex) / No. of filaments / Twist per meter	180/88/0	167/68/0	140/24/20	145/30/100	280/48/130
Linear filament density (dtex)	2.05	2.46	5.83	4.83	5.83
Manufacturing process <sup>a</sup>	POY	DTY	FDY	FDY	FDY
Yarn type	flat	textured	intermingled	low-twisted	intermingled
HAS <sup>b</sup> at 180 °C – 2 min	70%	15%	10%	5%	2%
Breaking strength (cN tex <sup>-1</sup> )	20	38	57	41	65
Cross-section	round				
Luster	semi-dull (< 0.05% titanium dioxide)				

<sup>a</sup> POY: Partially Oriented Yarn, DTY: Draw Textured Yarn and FDY: Fully Drawn Yarn.

<sup>b</sup> Hot Air Shrinkage: the decrease in length of yarns caused by a treatment in hot air, expressed as a percentage of length of untreated yarns; the lengths are measured, before and after treatment, under 0.5 cN tex<sup>-1</sup> pretension.

## 2.2. Thermal treatments

After removal from loom, woven tubes have been prewashed, mainly to eliminate contamination introduced accidentally or deliberately during previous manufacturing operations, whose traces could be permanently attached to textile armature at subsequent heat setting, which absolutely cannot be tolerated on the finished product (Guidoin et al., 1992). Fabric specimens were then helically crimped according to the protocol suggested in Table 3. In this investigation, an approach combining compaction and crimping

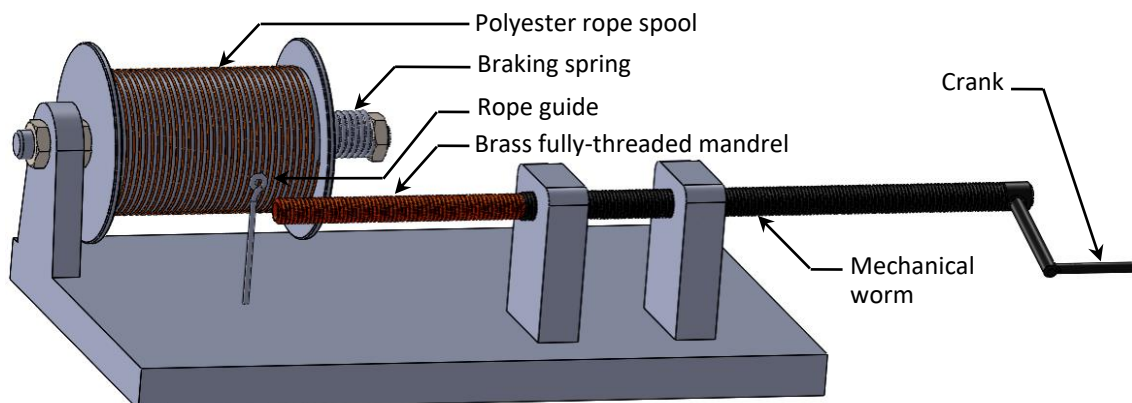
processes has been adopted to minimize alterations in the macromolecular order due to repeated thermal interventions on yarns (Azaiez et al., 2010; Guidoin et al., 1992).

Table 3: Procedural protocol adopted for the crimping of textile vascular prostheses

Stages	Processes	Equipment
0 Visual inspection	Examination, generally without magnification, of the tubular fabric to eliminate all imperfections such as stains and structure discontinuities (holes, knots, etc.) that could be source subsequent problems	Intense direct or indirect lighting
1 Tube cutting	Cut length is about twice the final length desired for the crimped tube	–
2 De-oiling/fabric relaxation	40 °C - 30 min washing by using a non-ionic detergent (Materge AP, 1 g l <sup>-1</sup> ), followed by thorough rinsing, spinning and open air flat drying	Automatic front loading washing machine
3 Helical pleating	Accordion corrugation performed by winding, at preset tension, polyester rope around prosthetic tube through which a threaded mandrel have been previously fitted (Fig. 1.a) (Khelif et al., 2012)	Rope-winding device designed for prosthesis creping (Fig.1.b) (Khelif et al., 2011)
4 Heat setting (according to a, b or c processes)	a. Dry heat treatment at temperatures (180-220 °C) and durations (2-10 min) adapted to prosthesis textile structure b. Overheated vapor treatment at temperatures (130-170 °C) and durations (15-60 min) adapted to prosthesis textile structure c. Autoclaving at temperatures (120-140 °C) and durations (10-30 min) adapted to prosthesis textile structure	Fixing stenter Steamer Autoclave
5 Quenching and retrieving crimped tubes	Immediate immersion in cold water (15 °C) to keep permanent wavy configuration on tube walls. The winding rope is then removed and the crimped tubes are gently unscrewed from threaded mandrels	–



(a) The accordion pleating formation



(b) The rope-winding device

Figure 1: Helical crimping of textile tube by fitting it on a threaded mandrel and winding a polyester rope all around prior to heat setting treatment

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As the action mode of each heat carrier (hot air, overheated vapor and pressurized superheated water) differs from one process to another, treatment conditions that are specific to one process may not be optimal for others (Table 3 – stage no. 4). However, treatment durations that we choose after several preliminary tests exceed those encountered in textile industrial practices, since the fabric part, located at the mandrel thread cavities, is obviously less exposed to heat carrier and thus needs much more time to reach set temperatures.

### **2.3. Morphological characterization**

Microscopic observations have been fulfilled to characterize both fabric texture and fiber morphology. After fabric opening flat, a crimp-removal technique based on stretching prosthetic samples at an appropriate tension by using band nippers permitted to flatten their walls when examined under optical microscope (Leica Microsystems, Switzerland). However, for fiber examination, filament specimens have been softly extracted from yarns randomly taken among prosthetic fabric samples. These manipulations, requiring much dexterity and precision in order to avoid any mixture of warp and weft filaments, were performed manually, eventually by means of a fabric decomposition needle. Any filament considered damaged during handling was systematically rejected.

Fiber dimensional measurements have been also performed by an optical microscope (Motic B1-211A, Hong Kong) connected to a computerized fiber fineness analysis system (YG002C). A magnification of 1000x was adopted and the measurements have been repeated (50 to 100 measurements per fiber bundle) until a 5% maximum practical error limit was achieved.

Besides, scanning electron microscopy (SEM) was applied to investigate the impact of prosthesis crimping processes on fiber morphology. The surface features of fiber samples were studied under a vacuum using an SEM device (FEI ESEM Quanta 200, USA) operating at 10 kV accelerating voltage. Prior to analysis, filaments have been sputter-coated with 6 nm gold/palladium layer in an ion coater. Such a thin conductive layer increases beam stability and improves image quality without risk of masking the fiber topography.

## **3. RESULTS AND DISCUSSION**

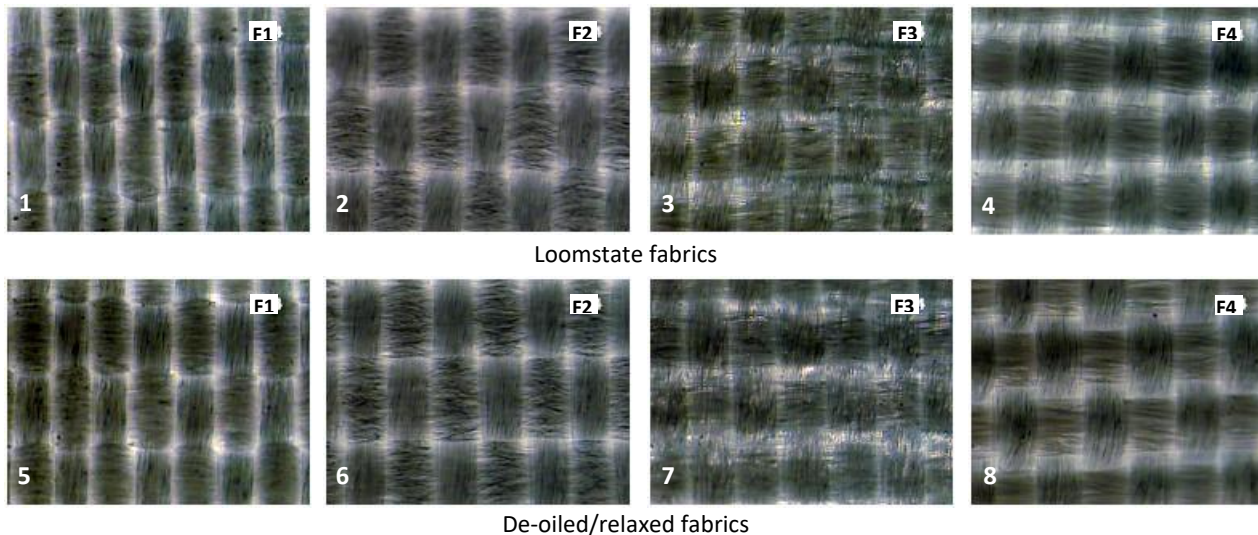
As expected, all fabrics, especially crimped ones, show close compact structures without any particular marking or apparent defect due to weaving or even crimping operations (Fig. 2). Loomstate fabrics have a much higher warp cover factor than weft one (Table 1). They display warp-faced plain structures. The cover factor indicates indeed the extent to which the area of a fabric is covered by one set of yarns. During prosthesis crimping process, woven tubes have been tightly held to a threaded mandrel by means of a strong rope, which greatly limited warp shrinkage that could take place after heat setting. Fabrics did not therefore manifest significant change in their pick count and generally exhibited a pronounced warp crimp (waviness) when removed from crimping device (Fig. 2.9-16). At heating, indeed, weft shrinkage had been much greater than warp one as the latter had been pre-stabilized.

A complementary study of treated-fabric specifications allowed the calculation of cover factors for heat set samples (those chosen in Fig. 2, no. 5-16) by measuring the new yarn counts (according to NF EN ISO 2060:1995) and deducing the new warp and pick counts from the evaluation of thermal fabric shrinkage in both directions. The fabrics porosity percentages have also been determined according to gravimetric method by fiber density measurements performed on a density gradient column (Table 4).

Table 4: Treated fabric specifications

Treated specimens <sup>a</sup>	Warp			Weft			Fabric cover factor <sup>b</sup> (yarns cm <sup>-1</sup> tex <sup>0.5</sup> )	Porosity <sup>c</sup> (%)
	Count (ends cm <sup>-1</sup> )	Linear density (dtex)	Cover factor	Count (picks cm <sup>-1</sup> )	Linear density (dtex)	Cover factor		
5	29.8	272	15.6	32.8	185	14.1	29.6	51.6
6	28.2	272	14.7	28.7	171	11.9	26.6	59.7
7	28.2	272	14.7	33.8	149	13.0	27.7	58.6
8	28.2	272	14.7	33.8	147	12.9	27.7	58.5
9	30.8	284	16.4	33.2	159	13.2	29.7	42.7
10	29.1	284	15.5	29.1	146	11.1	26.7	52.1
11	29.1	284	15.5	34.2	139	12.8	28.3	55.1
12	29.1	284	15.5	34.2	136	12.6	28.1	48.6
13	30.8	284	16.4	33.2	155	13.1	29.5	42.7
14	29.1	284	15.5	29.1	179	12.3	27.8	53.1
15	29.1	284	15.5	34.2	154	13.4	28.9	53.6
16	29.1	284	15.5	34.2	147	13.1	28.6	49.6

The increase of fabric cover factors following thermal treatments, essentially due to the increase of warp cover factors (Tables 1 and 4), confirms the highly compact character already observed on crimped fabric samples presented in Fig. 2. Such cover factors, varying from 26.2 to 29.5, are considered as very high (Gandhi, Sondhelm, 2016) and contribute to limit the prosthetic fabrics permeability to liquids. Changing the cover factors and/or the area density may affect the strength, thickness, stiffness, stability, porosity, fluid permeability and abrasion resistance of fabrics. By using appropriate cover factors and adequate yarns, most of the abrasion on warp-faced fabrics can be concentrated on the warp yarns which are stronger, while the weft will be protected (Gandhi, Sondhelm, 2016). On the other hand, fabric porosities have largely decreased in crimped structures where swelled filaments are often packed against each other to create tight compact surfaces. This inevitable loss of porosity (Guidoin et al., 1992) is considerable for the F1 fabric (-24%, from 56.1% at loomstate to 42.7% after stenter or autoclave treatments) whose weft is made from POY having a great heat shrinkability (Table 2) and less spectacular for the F2 and F3 fabrics, respectively manufactured with textured and intermingled warp yarns which are generally more voluminous than twisted ones employed in the F4 fabric (Tables 1 and 4). However, comparable porosity percentages have been recorded for structures crimped according to two different heat-setting methods (specimens 9 and 13; 10 and 14; 11 and 15; 12 and 16). It turns out that porosity is more influenced by the yarn properties and the textile structure employed in weaving than by crimping processes. A great fabric-porosity (preferably more than 50% for woven structures) is, in all cases, crucial to ensure good endothelial colonization *in vivo* and thus a perfect healing of the prosthesis (Guidoin et al., 1992; Tian-Jian, 1991).



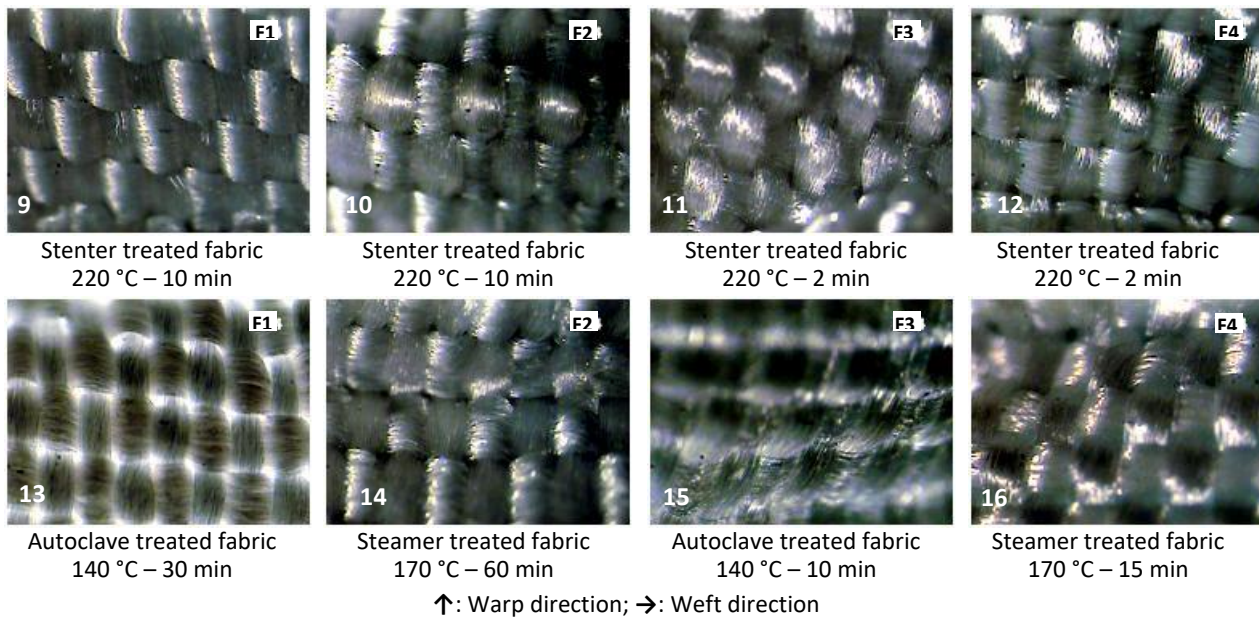
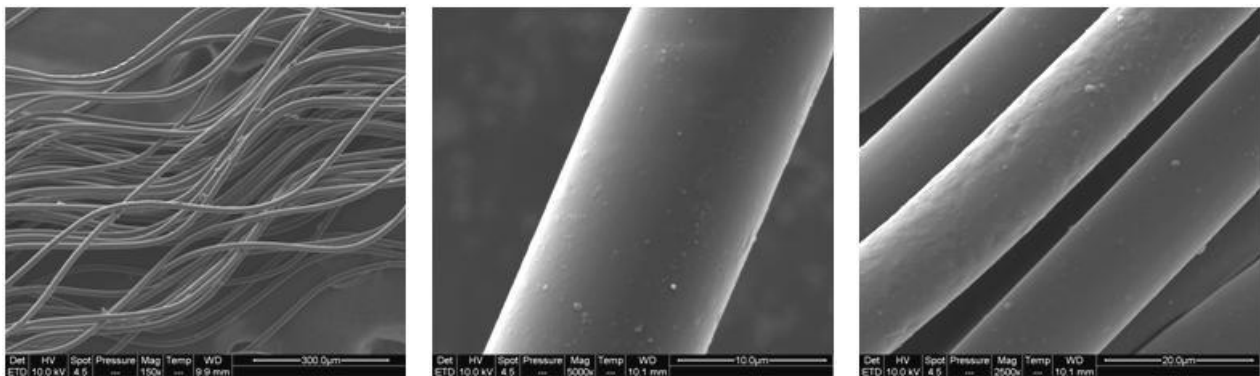
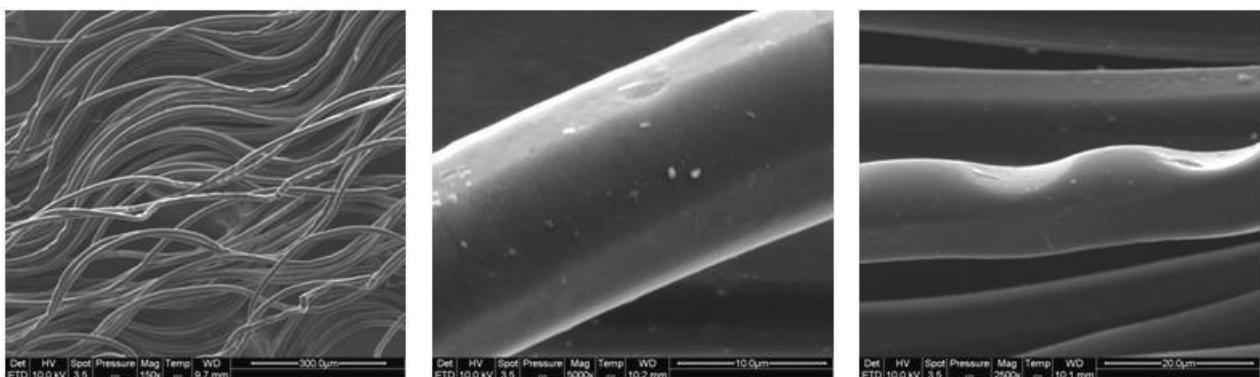


Figure 2: Optical microscopic views (x 80) of luminal surfaces for a selection of prosthetic specimens before and after thermal crimping treatments

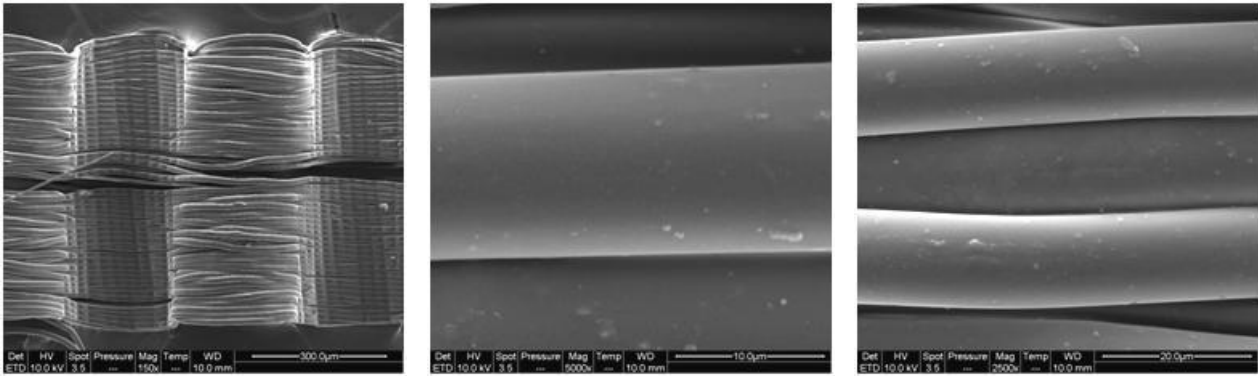
SEM micrographs (Fig. 3-7) recorded with three magnification levels (150x, 600 or 2500x and 5000x) confirm that all fibers have originally smooth surfaces and round sections except Dacron ones (Y2) which particularly have a variable diameter and a helical configuration resulting from the torsional and bending stresses that could cause the yarn to develop crimp and contract at texturing. In fact, when a fully extended stretch yarn is allowed to contract by 10 to 20%, the filaments follow helical paths, which alternate from right-handed to left-handed (Hearle et al., 2001). The Dacron filament spiral shape tends to straighten and its torsion marks diminish after prosthesis crimping treatments, which ties in with the findings of Guidoin *et al.* (1992).



(a) Filaments extracted from Y1 yarns in turn removed from the loomstate fabric F1



(b) Filaments extracted from Y1 yarns in turn removed from the fabric F1 crimped in stenter at 220 °C - 10 min

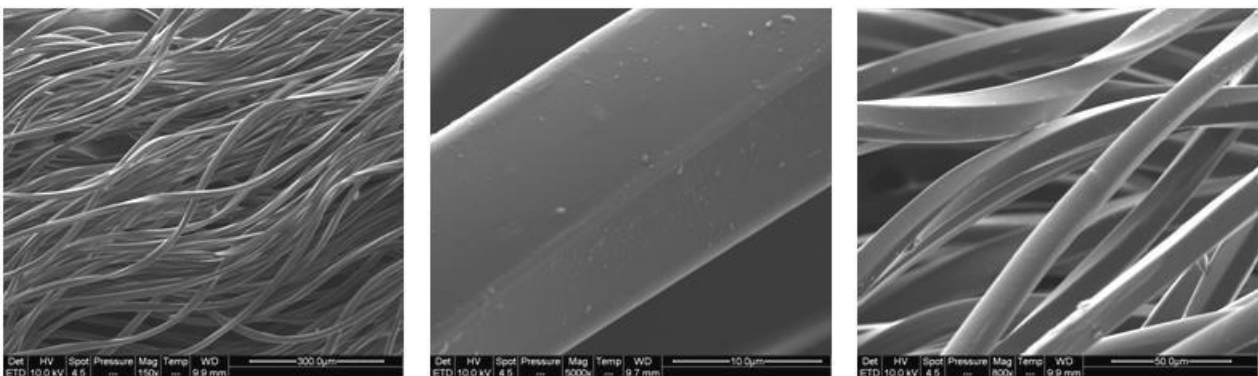


(c) Filaments extracted from Y1 yarns in turn removed from the fabric F1 heat set in stenter without crimping (free-annealed) at 220 °C - 10 min

Figure 3: SEM micrographs of PET filaments extracted from Y1 yarns (Setila, POY 180f88/0) in turn removed from the prosthetic fabric F1 before and after heat setting, with and without crimping

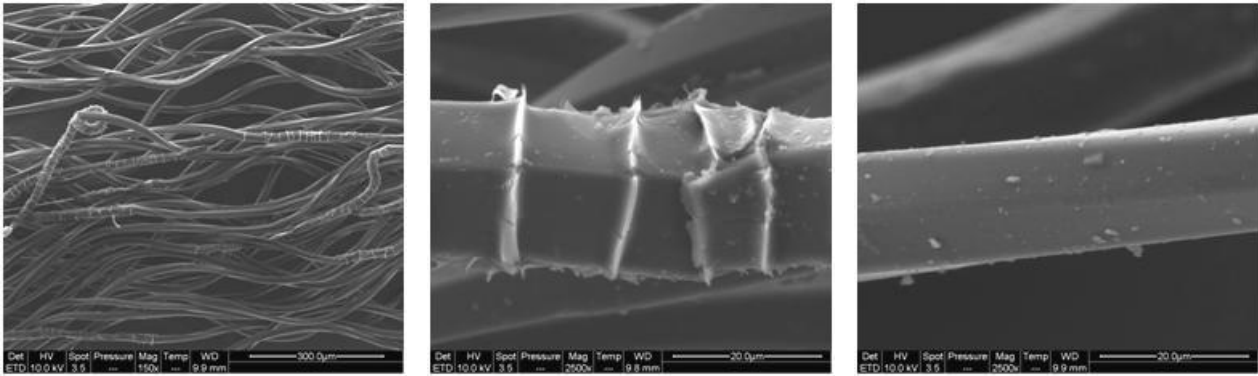
We also note the presence of oligomers, especially in Viscosuisse filaments (Y4 yarn) which have distinctly quite large crystals (Fig. 6.a). Referring to the works of Kassenbeck and Marfels (1977) concerning the oligomers distribution in polyester fibers, it could be estimated that these filaments (Viscosuisse) were probably treated at about 205 °C during usual procedures eliminating latent-tensions initially created at spinning-drawing. However, the other fibers (those of Y1, Y2, Y3 and Y5 yarns) have smaller oligomers that become, in contrast, big and numerous after thermal treatments. These oligomers, generated by polymerization secondary reactions and comprising few monomer units, contain linear oligoesters generally having several melting temperatures all lower than PET one. At spinning, oligoesters migrate to the fibril surfaces, thereby creating protrusions which can dislocate at contact with heated draw rolls thus forming craters (Reese, 2003). These irregularities could degrade the yarn friction properties and thus induce breaks, which could affect the behavior of the prosthesis *in vivo* (Guidoin et al., 1992). However, solid state polycondensation could limit the oligomers mass concentration in PET fibers to 1% (Reese, 2003).

The smooth character of fibers topography remains unchanged after heat treatments except for the Dacron filaments (constituting Y2 yarns), whose surface is marked, on some parts, by a kind of annular streaks, slightly protruding with pointed and sharp edges, following both hot air and vapor heat settings (Fig. 4.b-c). This phenomenon, appearing only on the Dacron filaments having undergone a texturing treatment, could be explained by the "skin-core" structure of these fibers.

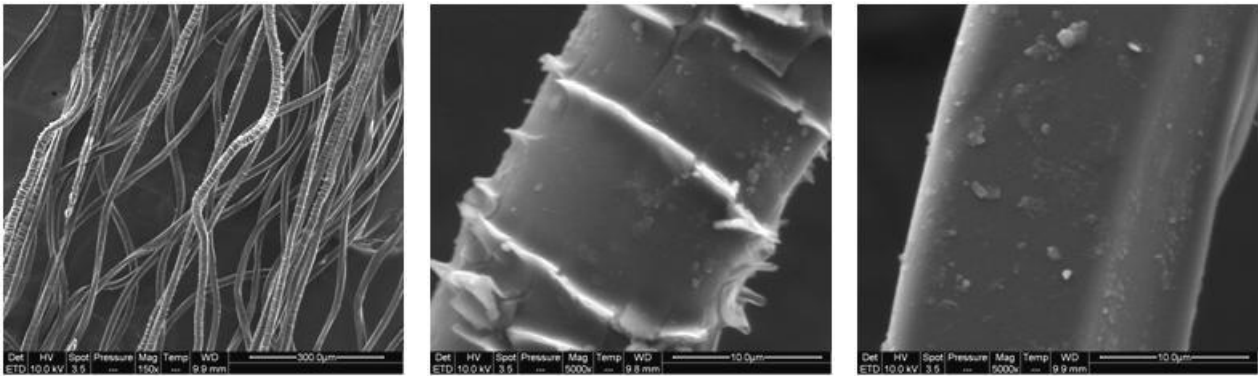


(a) Filaments extracted from Y2 yarns in turn removed from the loomstate fabric F2



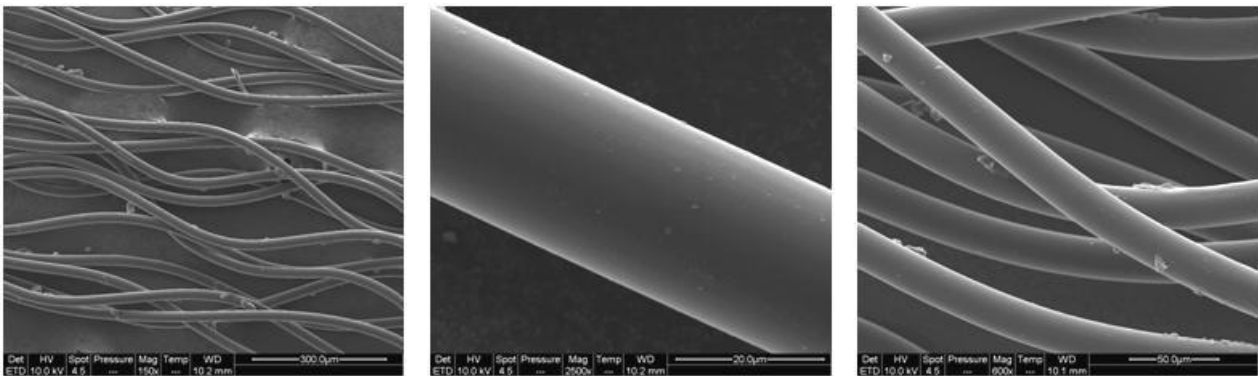


(b) Filaments extracted from Y2 yarns in turn removed from the fabric F2 crimped in stenter at 220 °C - 10 min

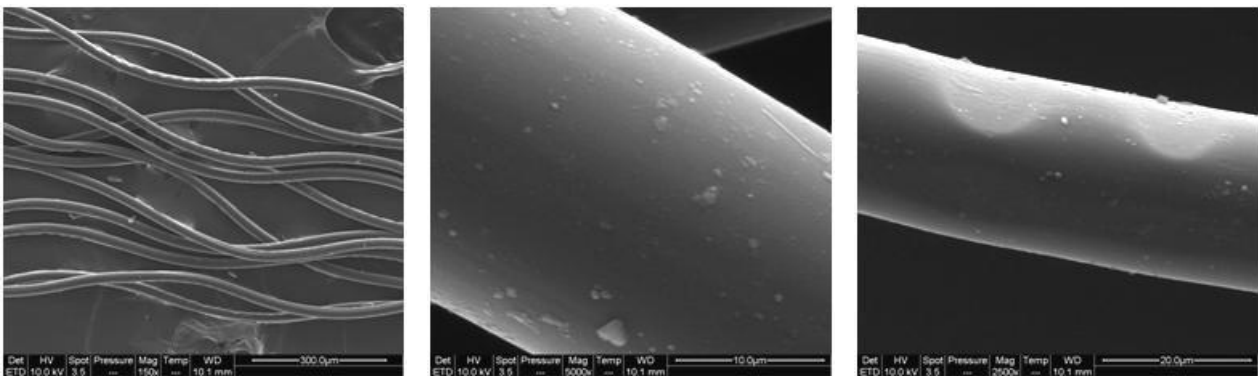


(c) Filaments extracted from Y2 yarns in turn removed from the fabric F2 vaporized at 170 °C - 60 min

Figure 4: SEM micrographs of PET filaments extracted from Y2 yarns (Dacron, DTY 167f68/0) in turn removed from the prosthetic fabric F2 before and after crimping treatments

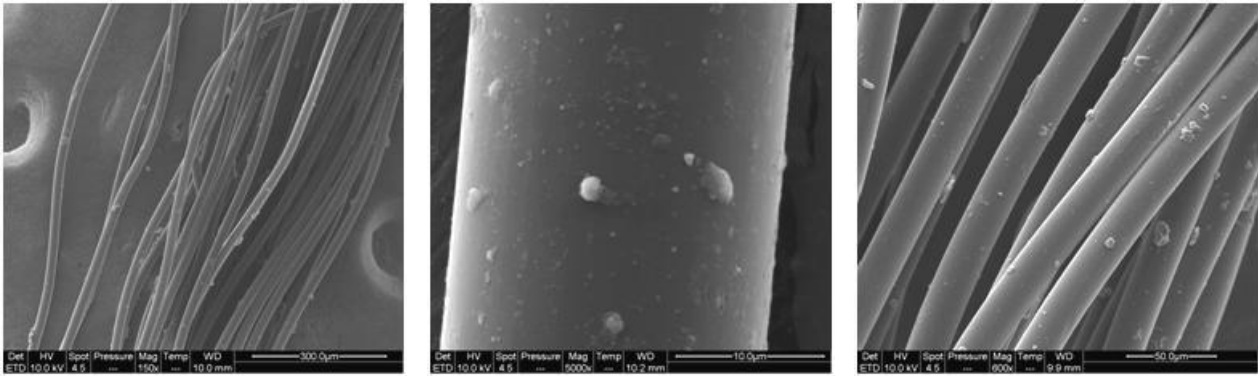


(a) Filaments extracted from Y3 yarns in turn removed from the loomstate fabric F3

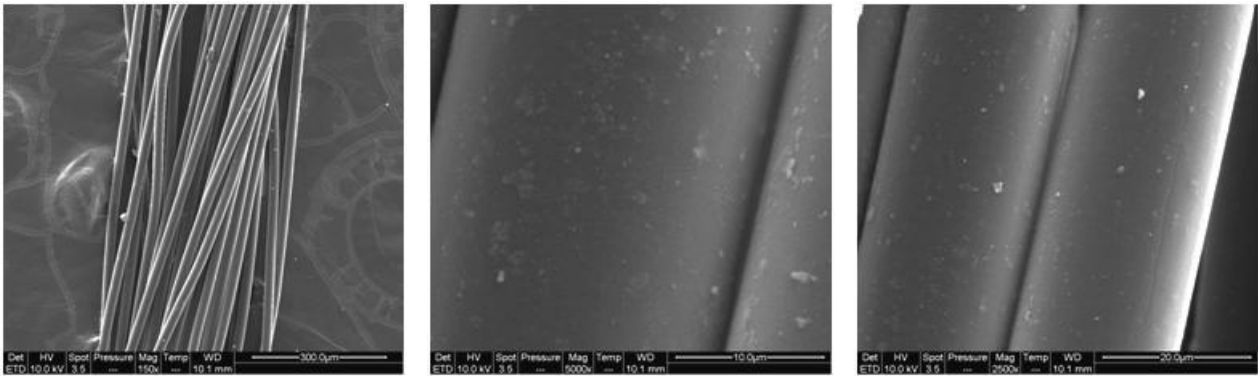


(b) Filaments extracted from Y3 yarns in turn removed from the fabric F3 vaporized at 170 °C - 15 min

Figure 5: SEM micrographs of PET filaments extracted from Y3 yarns (Diolen 57T, FDY 140f24/20) in turn removed from the prosthetic fabric F3 before and after crimping treatment



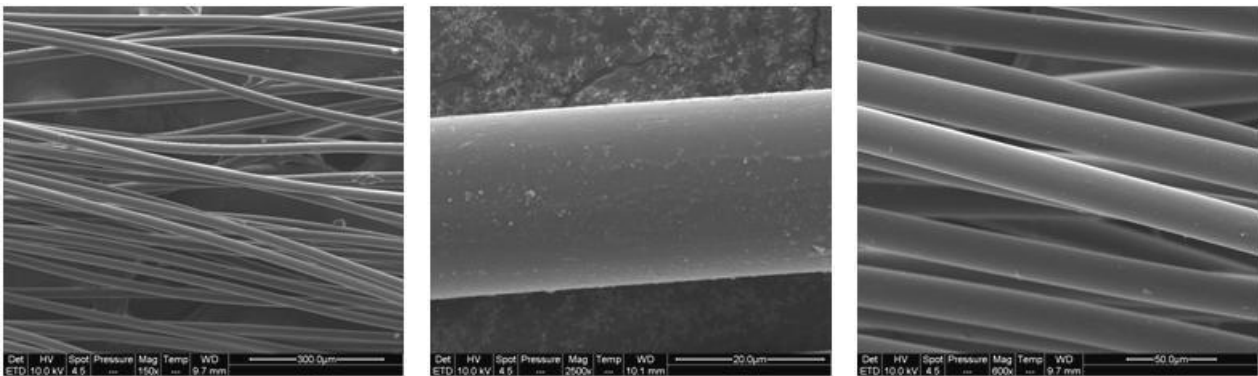
(a) Filaments extracted from Y4 yarns in turn removed from the loomstate fabric F4



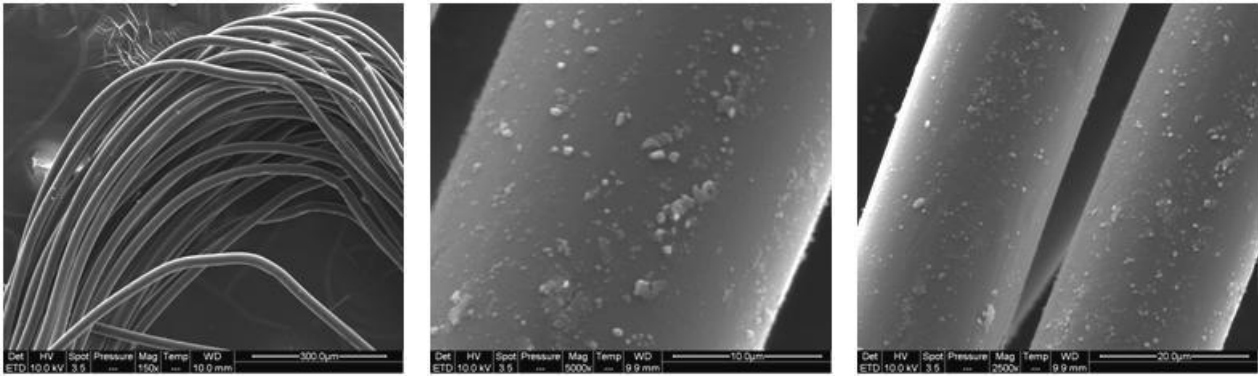
(b) Filaments extracted from Y4 yarns in turn removed from the fabric F4 autoclaved at 140 °C - 10 min

Figure 6: SEM micrographs of PET filaments extracted from Y4 yarns (Viscosuisse, FDY 145f30/100) in turn removed from the prosthetic fabric F4 before and after crimping treatment

Further examination of the Dacron yarns shows a high bulk and low stretch structure. This configuration is usually achieved by false-twist texturing (Yildirim et al., 2009; Reese, 2003; Hearle et al., 2001; Kassenbeck, Marfels, 1977) applied to spun continuous-filaments of POY by the simultaneous actions of three main parameters, which are called 3t: tension (drawing), twist, and temperature. The filament bundle is subsequently untwisted and then heated under controlled partial relaxation. The yarn fixing is most often done by heat conduction at temperatures close to the melting point. It could also occur by radiation, convection or combination of these processes. Whatever the heat source, the heating time is, for economic reasons, too short (0.3-0.4 s) (Weidmann, 2010). This ultra-fast treatment makes the fiber periphery ("skin"), directly exposed to the heat source, retract completely while the more central part ("core") fails to release all residual stresses (Demir, Behery, 1997). This difference in shrinkage power could explain the salient shape of the Dacron fibers topography, generated following thermal treatments of prosthesis crimping (the core retracts, but the skin, already stabilized, does not retract). However, the impact of this annularly-salient shape on the future mechanical behavior of Dacron fibers *in vivo* remains unknown and requires further investigation.



(a) Filaments extracted from Y5 yarns in turn removed from the loomstate fabric F2



(b) Filaments extracted from Y5 yarns in turn removed from the fabric F2 crimped in stenter at 220 °C - 10 min

Figure 7: SEM micrographs of PET filaments extracted from Y5 yarns (Diolen 61ST, FDY 280f48/130) in turn removed from the prosthetic fabric F2 before and after crimping treatment

The prosthesis crimping affected the sectional circularity of Setila fibers (Fig. 3-b) and, to a lower degree, Diolen (57T) ones (Fig. 5-b). Some deformation has also been fixed on both shrinkable fibers (those constituting Y1 and Y3 yarns). These marks reasonably resulted from the firm fabric-compression between the threaded mandrel and the winding rope at prosthesis creping. Furthermore, such deformations did not appear anywhere on Setila fibers heat set at the same conditions (hot air at 220 °C - 10 min) but in the free state (without tension or creping). These fibers show indeed a very regular appearance (Fig. 3-c).

Apart from a variation of diameter which will be studied in the following, the prosthesis crimping treatments gave rise to neither alarming defects nor significant changes in the morphology of both Viscosuisse and Diolen 61ST filaments. These exhibit well-rounded sections which have not been compromised by the temperature or the severe prosthesis crimping stresses (Fig. 6-7).

In addition, we note that the three heat setting methods, accomplished by stenter, steamer or autoclave, give, at this level of the analysis, almost similar fiber aspects.

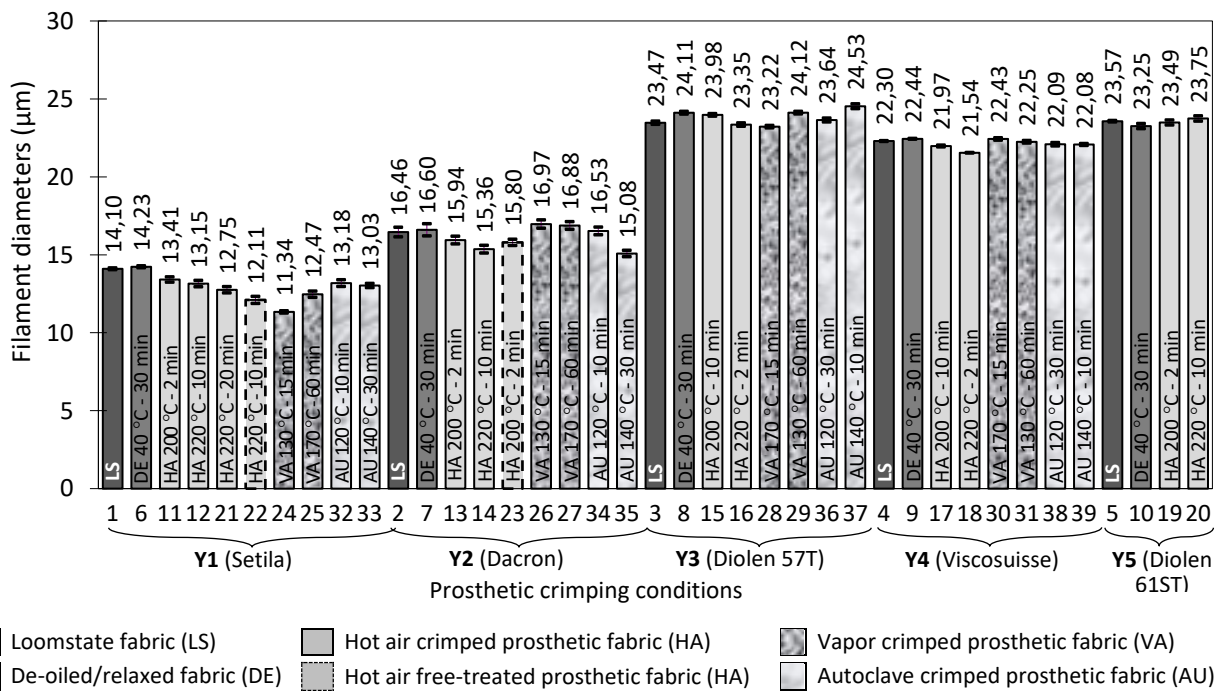


Figure 8: Diameter measurements of filaments extracted from Y1-Y5 yarns in turn removed from prosthetic fabrics (F1-F4) for different treatment conditions

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According to Fig. 8 presenting fibers diameter measurements, we note the presence of two fineness ranges: fine fibers, Setila and Dacron (those of Y1 and Y2 yarns), whose diameters turn around 14-16  $\mu\text{m}$  and thicker fibers, Diolen and Viscosuisse (those of Y3, Y4 and Y5 yarns), whose diameters rises to 22-23  $\mu\text{m}$ . Their specific surfaces (lateral surfaces per unit volume) are thereby significantly different, giving them different degrees of exposure to heat during prosthesis crimping processes.

The de-oiling treatment, being carried out at a low temperature (40  $^{\circ}\text{C}$ ), had no significant impact on the fiber diameters. The heat setting did not significantly alter the Diolen and Viscosuisse fiber diameters, in contrast, decreases of 5 to 20% in Setila partially oriented fibers and up to 8% in Dacron textured fibers were recorded after crimping.

According to Fig. 8, the Setila fibers removed from the 220  $^{\circ}\text{C}$  - 10 min hot air crimped fabric (test no. 12) recorded a 13.15  $\mu\text{m}$  average diameter (a decrease of 7% compared to the loomstate fabric), while those withdrawn from fabrics treated under the same conditions but in the free state (without crimping, test no. 22) had a 12.11  $\mu\text{m}$  diameter (a decrease of 14% compared to the loomstate fabric). This could be explained by the biphasic structure of all pre-oriented polyester filaments with, on the one hand, an amorphous phase and, on the other hand, a mesophase whose importance and degree of orientation increase with the winding speed applied at spinning (Bouriot et al., 1980). When heated, the POYs are subjected to two competitive phenomena: the shrinkage whose importance increases with the pre-orientation rate and the crystallization which is triggered at a temperature as lower as the pre-orientation is important. A preponderance of crystallization on the shrinkage is generally observed from spinning speeds of 2500-3000  $\text{m min}^{-1}$  (Bouriot et al., 1980). In this case, the crystallization, by its rapidity, seems to inhibit the shrinkage and transform part of the mesophase into an unfolded chain configuration, which results in a decrease of the fiber diameter. The high-tension heat-setting of POYs (the case of prosthesis crimping treatments) probably favored a transition of a part of the completely disoriented amorphous zones to meso zones, thus giving the Setila fibers a greater diameter than that obtained with tensionless heat setting.

Dacron filaments have also undergone a substantially identical diameter decrease for fabrics treated with dry heat at 200  $^{\circ}\text{C}$  - 2 min with (test no. 13) or without tension (test no. 23) from 16.46  $\mu\text{m}$  to 15.94  $\mu\text{m}$  and 15.80  $\mu\text{m}$ , respectively. Presenting a three-phase structure (amorphous, meso and crystalline phases), the Dacron filaments decreased in diameter, after heat setting, because of the new mesomorphic zones rearrangement (alignment) (Gupta, Kumar, 1981).

By assimilating the polyester fiber to a homogeneous cylinder of diameter  $d$  ( $\mu\text{m}$ ) and density  $\rho$  ( $\text{g cm}^{-3}$ ), its count  $t$  (dtex) can be written:

$$t_{\text{dtex}} = \frac{\rho \pi d^2}{400} \quad (1)$$

The formula (1) could explain the yarn count evolution (registered in Tables 1 and 4) directly related to the changes in their fiber diameters after crimping thermal treatments. Due to fiber diameter decrease, a reduction in Setila yarn count of about 12% after stenter heat setting (specimen 9) and 14% after autoclaving (specimen 13) has been noted, despite a slight increase in the fiber density (about 1%) (Khlif, 2017). However, Dacron, Diolen and Viscosuisse experienced a slight decrease of their yarn count for dry heat prosthetic crimping (Table 4: specimens no. 10-12) and a count rise for wet heat crimping performed in steamer and autoclave (Table 4: specimens no. 14-16) due to fiber shrinkage (diameter increase, Fig. 8: tests no. 27, 30 and 37) under the direct action of water as a heat carrier which seems to be more "aggressive" than hot air (Khlif et al., 2011, 2012).

#### 4. CONCLUSIONS

The aim of this study was to investigate the impact of crimping thermal treatments applied to PET textile vascular prostheses on the fabric structure and the fiber surface characteristics. For this purpose, four tubular fabric samples have been woven with various PET multifilament yarns and thermally crimped according to a predefinite protocol applied with several treatment conditions.

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Optical microscopy showed tight compact warp-faced structures without any particular marks due to the mechanical pressure to which prosthetic fabrics have been subjected during crimping. These treatments brought the factors of coverage to quite high levels, which greatly improve the fabrics waterproofness (Mokhtar et al., 2010). Nevertheless, the fabric walls stiffen, and their porosities reduce as well. This degrades therefore both the mechanical compliance and the incorporation capacity of the prosthesis.

The SEM micrographs show that the fibers are parallel and clean without any contaminant traces (oiling or others), nor major damage resulted from the operations of finishing or even manufacturing following, for example, mechanical shocks caused by contact with an inappropriate rigid surface (comb mark or heddle friction) as already reported by Guidoin *et al.* (1992) (fiber damage caused by knitting needles). However, some flattening traces have been observed on Setila and Diolen 57T fibers due to the severe mechanical constraints imposed by the prosthesis crimping device. The whole filaments present smooth surfaces and round sections except Dacron ones which particularly have a variable diameter and a helical configuration resulting from texturing process. This twisted shape decreased after prosthesis finishing, indicating an attenuation of the yarn texturing effect and therefore its extensibility and voluminosity. To preserve this effect, an adequate choice of thermal finishing temperature according to that of texturing is necessary (the first temperature must never exceed the second one) and a perfect knowledge of the fiber spinning history is crucial for a good adjustment of the prosthesis finishing conditions.

The smooth character of fibers topography remains unchanged after heat treatments except for Dacron filaments, whose surface is marked, on some parts, by a kind of annular streaks, slightly protruding with pointed and sharp edges. This special shape could be explained by the "skin-core" structure of textured Dacron fibers, but its impact on the future prosthetic mechanical behavior *in vivo* remains unknown and requires further investigation.

The presence of oligomers has been noted with different degrees on all the fibers, whether they are treated or not and their concentration should be minimized since the polymerization step. Furthermore, fiber diameters, overall, showed a slight decrease after fabric crimping using dry heat thus inducing changes in yarn counts. This is explained by the microstructural changes that the fibers have experienced under the combined effect of extreme heat and high mechanical stress (Khlif et al., 2012; Khlif, 2017).

These obvious changes in the PET fiber structure have a great impact on the mechanical and chemical behavior of implants and affect prosthesis lifetime in the human body. Consequently, thermal treatments conditions have to be rigorously optimized during graft finishing.

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