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ESCUELA TÉCNICA SUPERIOR DE
INGENIEROS DE CAMINOS, CANALES Y PUERTOS

**Multiscale analysis of the mechanical
behaviour of needle-punched nonwoven
fabrics**

TESIS DOCTORAL

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Ingeniera Industrial

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Departamento de Ciencia de Materiales

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*A Nicola,
a mi hermana María,
y a mis queridos padres,
Francisco Javier y María Caridad.*

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*Francisca Martínez Hergueta
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Resumen

Los fieltros son una familia de materiales textiles constituidos por una red desordenada de fibras conectadas por medio de enlaces térmicos, químicos o mecánicos. Presentan menor rigidez y resistencia (al igual que un menor coste de procesado) que sus homólogos tejidos, pero mayor deformabilidad y capacidad de absorción de energía. Los fieltros se emplean en diversas aplicaciones en ingeniería tales como aislamiento térmico, geotextiles, láminas ignífugas, filtración y absorción de agua, impacto balístico, etc. En particular, los fieltros punzonados fabricados con fibras de alta resistencia presentan una excelente resistencia frente a impacto balístico, ofreciendo las mismas prestaciones que los materiales tejidos con un tercio de la densidad areal. Sin embargo, se sabe muy poco acerca de los mecanismos de deformación y fallo a nivel microscópico, ni sobre como influyen en las propiedades mecánicas del material. Esta carencia de conocimiento dificulta la optimización del comportamiento mecánico de estos materiales y también limita el desarrollo de modelos constitutivos basados en mecanismos físicos, que puedan ser útiles en el diseño de componentes estructurales.

En esta tesis doctoral se ha llevado a cabo un estudio minucioso con el fin de determinar los mecanismos de deformación y las propiedades mecánicas de fieltros punzonados fabricados con fibras de polietileno de ultra alto peso molecular. Los procesos de deformación y disipación de energía se han caracterizado en detalle por medio de una combinación de técnicas experimentales (ensayos mecánicos macroscópicos a velocidades de deformación cuasi-estáticas y dinámicas, impacto balístico, ensayos de extracción de una o múltiples fibras, microscopía óptica, tomografía computarizada de rayos X y difracción de rayos X de gran ángulo) que proporcionan información de los mecanismos dominantes a distintas escalas. Los ensayos mecánicos macroscópicos muestran que el fieltro presenta una resistencia y ductilidad excepcionales.

El estado inicial de las fibras es curvado, y la carga se transmite por el fieltro a través de una red aleatoria e isótropa de nudos creada por el proceso de punzonamiento, resultando en la formación de una red activa de fibra. La rotación y el estirado de las fibras activas es seguido por el deslizamiento y extracción de la fibra de los puntos de anclaje mecánico. La mayor parte de la resistencia y la energía disipada es proporcionada por la extracción de las fibras activas de los nudos, y la fractura final tiene lugar como consecuencia del

desenredo total de la red en una sección dada donde la deformación macroscópica se localiza. No obstante, aunque la distribución inicial de la orientación de las fibras es isótropa, las propiedades mecánicas resultantes (en términos de rigidez, resistencia y energía absorbida) son muy anisótropas. Los ensayos de extracción de múltiples fibras en diferentes orientaciones muestran que la estructura de los nudos conecta más fibras en la dirección transversal en comparación con la dirección de la máquina. La mejor interconectividad de las fibras a lo largo de la dirección transversal da lugar a una esqueleto activo de fibras más denso, mejorando las propiedades mecánicas. En términos de afinidad, los fieltros deformados a lo largo de la dirección transversal exhiben deformación afín (la deformación macroscópica se transfiere directamente a las fibras por el material circundante), mientras que el fieltro deformado a lo largo de la dirección de la máquina presenta deformación no afín, y la mayor parte de la deformación macroscópica no es transmitida a las fibras.

A partir de estas observaciones experimentales, se ha desarrollado un modelo constitutivo para fieltros punzonados confinados por enlaces mecánicos. El modelo considera los efectos de la deformación no afín, la conectividad anisótropa inducida durante el punzonamiento, la curvatura y re-orientación de la fibra, así como el desenredo y extracción de la fibra de los nudos. El modelo proporciona la respuesta de un mesodominio del material correspondiente al volumen asociado a un elemento finito, y se divide en dos bloques. El primer bloque representa el comportamiento de la red y establece la relación entre el gradiente de deformación macroscópico y la respuesta microscópica, obtenido a partir de la integración de la respuesta de las fibras en el mesodominio. El segundo bloque describe el comportamiento de la fibra, teniendo en cuenta las características de la deformación de cada familia de fibras en el mesodominio, incluyendo deformación no afín, estiramiento, deslizamiento y extracción. En la medida de lo posible, se ha asignado un significado físico claro a los parámetros del modelo, por lo que se pueden identificar por medio de ensayos independientes. Las simulaciones numéricas basadas en el modelo se adecúan a los resultados experimentales de ensayos cuasi-estáticos y balísticos desde el punto de vista de la respuesta mecánica macroscópica y de los micromecanismos de deformación. Además, suministran información adicional sobre la influencia de las características microestructurales (orientación de la fibra, conectividad de la fibra anisótropa, afinidad, etc) en el comportamiento mecánico de los fieltros punzonados.

Abstract

Nonwoven fabrics are a class of textile material made up of a disordered fiber network linked by either thermal, chemical or mechanical bonds. They present lower stiffness and strength (as well as processing cost) than the woven counterparts but much higher deformability and energy absorption capability and are used in many different engineering applications (including thermal insulation, geotextiles, fireproof layers, filtration and water absorption, ballistic impact, etc). In particular, needle-punched nonwoven fabrics manufactured with high strength fibers present an excellent performance for ballistic protection, providing the same ballistic protection with one third of the areal weight as compared to dry woven fabrics. Nevertheless, very little is known about their deformation and fracture micromechanisms at the microscopic level and how they contribute to the macroscopic mechanical properties. This lack of knowledge hinders the optimization of their mechanical performance and also limits the development of physically-based models of the mechanical behavior that can be used in the design of structural components with these materials.

In this thesis, a thorough study was carried out to ascertain the micromechanisms of deformation and the mechanical properties of a needle-punched nonwoven fabric made up by ultra high molecular weight polyethylene fibers. The deformation and energy dissipation processes were characterized in detail by a combination of experimental techniques (macroscopic mechanical tests at quasi-static and high strain rates, ballistic impact, single fiber and multi fiber pull-out tests, optical microscopy, X-ray computed tomography and wide angle X-ray diffraction) that provided information of the dominant mechanisms at different length scales. The macroscopic mechanical tests showed that the nonwoven fabric presented an outstanding strength and energy absorption capacity.

It was found that fibers were initially curved and the load was transferred within the fabric through the random and isotropic network of knots created by needlepunching, leading to the formation of an active fiber network. Uncurling and stretching of the active fibers was followed by fiber sliding and pull-out from the entanglement points. Most of the strength and energy dissipation was provided by the extraction of the active fibers from the knots and final fracture occurred by the total disentanglement of the fiber network in a given section at which the macroscopic deformation was localized. However, although the initial fiber orientation distribution was isotropic, the mechanical properties (in terms

of stiffness, strength and energy absorption) were highly anisotropic. Pull-out tests of multiple fibers at different orientations showed that structure of the knots connected more fibers in the transverse direction as compared with the machine direction. The better fiber interconnection along the transverse direction led to a denser active fiber skeleton, enhancing the mechanical response. In terms of affinity, fabrics deformed along the transverse direction essentially displayed affine deformation –i.e. the macroscopic strain was directly transferred to the fibers by the surrounding fabric–, while fabrics deformed along the machine direction underwent non-affine deformation, and most of the macroscopic strain was not transferred to the fibers.

Based on these experimental observations, a constitutive model for the mechanical behavior of the mechanically-entangled nonwoven fiber network was developed. The model accounted for the effects of non-affine deformation, anisotropic connectivity induced by the entanglement points, fiber uncurling and re-orientation as well as fiber disentanglement and pull-out from the knots. The model provided the constitutive response for a mesodomain of the fabric corresponding to the volume associated to a finite element and is divided in two blocks. The first one was the network model which established the relationship between the macroscopic deformation gradient and the microscopic response obtained by integrating the response of the fibers in the mesodomain. The second one was the fiber model, which took into account the deformation features of each set of fibers in the mesodomain, including non-affinity, uncurling, pull-out and disentanglement. As far as possible, a clear physical meaning is given to the model parameters, so they can be identified by means of independent tests. The numerical simulations based on the model were in very good agreement with the experimental results of in-plane and ballistic mechanical response of the fabrics in terms of the macroscopic mechanical response and of the micromechanisms of deformation. In addition, it provided additional information about the influence of the microstructural features (fiber orientation, anisotropic fiber connectivity, affinity) on the mechanical performance of mechanically-entangled nonwoven fabrics.

CHAPTER 1

Introduction

1.1 Nonwoven fabrics

Some of the most common synthetic materials used in our daily life are based on small diameter fibers (ceramic, carbon, glass, polymeric, etc). They are normally used in composite materials embedded in a matrix (polymeric, metallic or ceramic), whose primary mission is to orient the fibers along particular directions and to protect them against chemical and environmental effects. Structural composite materials are currently used in engineering applications driven by weight reduction and their demand in aerospace, automotive, energy and sports industry is increasing very rapidly. However, the presence of a continuous matrix is not required in some applications such as, for instance, ballistic protection, thermal and sound insulation, filtration, etc, resulting in a new set of engineering materials known as dry fabrics, see Fig. 1.1.

Fibers can be woven to form 2D or 3D architectures or randomly distributed in a network structure, leading to a nonwoven fabric. The network structure of a nonwoven fabric is formed from a random dispersion of fibers that are partially connected by local fiber bonds during the manufacturing process, Fig. 1.1(b) and (d). Nonwoven fabrics have emerged as very promising materials for different industrial applications due to the lower processing costs as compared with their woven counterparts.

Components made up of fiber networks can be found in nature (muscles and vascular tissues, cells, etc), and they have been manufactured since long time. Paper is an ancient

1.1 Nonwoven fabrics

Figure 1.1: Woven and nonwoven dry fabrics: (a) Plain weave fabric made up by Kevlar KM2 fibers, (b) Nonwoven fabric made up by Kevlar fibers (courtesy of Professor Sergei B. Sapozhnikov), (c) 3 harness satin fabric made up by Dyneema SK65 fibers (UHMWPE Ultra High Molecular Weight Polyethylene fibers), (d) Nonwoven fabric made up by Dyneema SK75 fibers.

example of a fibrous material made up by cellulose fibers connected by hydrogen bonds forming a random network (Cox, 1952; Bronkhorst, 2003; Isaksson and Hägglund, 2007). Cotton and wool fabrics can eventually being processed by carding to form fiber felts.

Several definitions are given by the industry for nonwoven fabrics. All of them classify a nonwoven as a web or batt layer of fibers linked together by friction, cohesion or adhesion mechanisms. Nonwoven technology has grown during the last years by combining developments from textile, paper and polymer industries. One of the main advantages of nonwovens is the possibility to tailor their properties by fiber selection, network density and interfiber bond quality. Nowadays nonwovens can be manufactured using a large variety of fibers leading, therefore, to many possible applications. Different fibers (polymer, ceramic, carbon nanotubes) can be bonded in networks to produce flexible, resistant, damage tolerant, permeable and energy absorbent materials depending on the final application.

From the viewpoint of structural performance, nonwovens present lower stiffness and strength than their woven counterparts. However, they can sustain large deformations be-

fore failure (higher than the strain-to-failure of the fibers) because of their random structure and fiber interconnections. As a result, they show enhanced ductility, damage tolerance and defect insensitivity (Ridruejo et al., 2015) and are very promising as shock absorbers in automotive and aerospace, applications, portable electronics or in ballistic protection against small fragments. Additionally, geotextile and geomembranes are often used in soil reinforcement and erosion control.

In addition to their mechanical properties, nonwoven fabrics present a porous structure and can work as efficient filters for liquids. In this regard, it is important to mention hygiene and sanitary applications (disposable diapers, sanitary napkins, wraps, etc.) which constitute around the $\approx 50\%$ of the nonwoven global market (Russell, 2007). They are also used as acoustic and thermal insulators as well as for fire protection.

1.2 Manufacturing of nonwoven fabrics

Nonwoven fabrics have been conventionally manufactured from dry-laid carded webs using converted textile processing machinery, replacing the weaving of yarns in traditional textiles by fiber bonds. The manufacturing of nonwoven fabrics can be divided in three different stages classified according to Russell (2007) into: web formation, web bonding and fabric finishing.

1.2.1 Web formation

The purpose of web formation is to mix individual fibers to form a homogeneous web with uniform density. Thus, individual fibers are transformed into a two-dimensional (web) or a three-dimensional web assembly (batt), which is the preform of the final fabric. Web formation processes are classified into dry-laid, wet-laid or polymer-laid.

- Dry-laid manufacturing. Carding and air laying are the most important methods used in dry-laid. The main difference between them consists of the fiber deposition method and the final fiber orientation distribution. The carding machine is fed by fiber staples which cross the carding toothed rollers in the machine direction, while air laying involves the uniform dispersion of individual fibers onto the web using air

1.2 Manufacturing of nonwoven fabrics

Figure 1.2: Operation of a simple needle-punching machine (Russell, 2007).

stream. Air laying fiber networks exhibit more isotropic structures in comparison to carded webs which show anisotropic fiber distributions along the machine (MD) and transverse directions (TD).

- Wet-laid process is closely related to the manufacturing of paper. The web precursor is a dispersion of fibers in a liquid. The dispersion is deposited into a manufacturing line, pressed and dried to form the final web. This technique allows to use brittle fibers as zirconia, alumina, glass or whiskers.
- Polymer-laid nonwovens are produced by extrusion and spinning of thermoplastic blends. The thermoplastic is melted, extruded, quenched, drawn and finally, deposited into the fiber web. Intermediate steps are eliminated, leading to the most cost efficient method to produce nonwoven fabrics. The two main commercial polymer-laid processes are spun-bonding and melt-blowing and the main difference between them is found in the fiber deposition method. Fibers in the spun-bonding process are extruded from a spinneret and deposited with the aid of a specially designed aerodynamic device. Melt-blowing fibers are blown using high velocity air streams onto a collector conveyor. High molecular weight and medium melt viscosity polymers are preferred for spun-bonding, while low molecular weight polymers are used in melt-blowing.

1.2.2 Web bonding

Raw mats are consolidated by bonding processes to produce mechanical percolation loading paths. The degree of bonding is a primary factor affecting the mechanical and physical properties of nonwoven fabrics, including stiffness, strength, thermal and electrical conductivity, etc. Bonding is generally carried out in line with web formation and more than one bonding process can be used in some fabric constructions. Consolidation methods include chemical, thermal and mechanical bonding. The selection and intensity of the bonding method should be carried out taking into account the final performance required.

- Chemical bonding uses adhesive binders in emulsion by saturating, spraying, printing, or foaming techniques. Binders can easily permeate thick porous nonwoven fabrics due to their low viscosity. After binder application, the layer is dried and adhesive bonds at fiber intersections are created.
- Thermal bonding involves the use of heat and pressure to weld fibers together. Thermal bonding requires a thermoplastic component, which can be the fiber itself, or a secondary component specifically introduced for this purpose. Heat is applied until the thermoplastic component becomes viscous, flowing by surface tension and external pressure to the fiber crossovers where bonds are formed during the subsequent cooling.
- Mechanical bonding form mechanical entanglements between fibers. Mechanical consolidation methods include stitchbonding, needlepunching and hydroentangling. Stitchbonding creates a fiber reinforcement in the through-the-thickness direction similar to the manufacturing process of 3D woven preforms. In needlepunching, fibers are mechanically entangled by reciprocating barbed needles through the moving batt, see Fig. 1.2. The barbed needles, see Fig. 1.3, are clamped into a board which oscillates vertically between two fixed plates containing the moving batt introduced by a feed system consisting of nip rollers. As the web moves through the loom, more fibers are progressively entangled by the needle barbs and a coherent fabric structure is formed. Hydroentangling consolidates the web by means of high-velocity water jets. A curtain of multiple high-pressure columnar water jets is produced by pumping water through capillary cone-shaped nozzles in a jet strip clamped into an injector. These high-velocity jets are directed at a pre-formed web supported on a

1.3 Mechanical response of nonwovens

Figure 1.3: Action of a barbed needle (Russell, 2007).

moving conveyor, which may be a flat bed or cylindrical surface. Fiber entanglements are introduced by the combined effects of the incident water jets and the turbulent water created in the web which intertwines neighboring fibers.

1.2.3 Web finishing

The finishing of nonwoven fabrics is of increasing importance as manufacturers try to add value to the final product by increasing the functionality, appearance or aesthetics. There are no standard finishing routes for nonwoven fabrics and the selection of processes and the finishing effects depend on the particular end-use application. Traditionally, finishing is classified as either wet finishing (washing, chemical impregnation, dyeing and coating) or dry finishing (calendering, embossing, emerising and microcreeping). Nonwoven fabrics can also be printed, flocked or combined with other fabrics, films and foils to form laminates, combining the properties of each nonwoven layer.

1.3 Mechanical response of nonwovens

Nonwoven fabrics are a new set of dry fabrics of increasing technological relevance due to their reduced manufacturing costs as compared with the more traditional woven counterparts. It is difficult to establish a general classification of nonwoven fabrics from the viewpoint of their mechanical behaviour, but it is commonly accepted to establish two major categories, namely bonded and entangled networks.

1.3 Mechanical response of nonwovens

Figure 1.4: Failure mechanisms in a Vetrotex M123 bonded nonwoven subjected to in-plane tensile deformation (Ridruejo et al., 2010): (a) Bond fracture micromechanisms and (b) Nominal stress (force per unit width) vs. nominal strain. Failure mechanisms in a PP polypropylene Typar SF32 bonded nonwoven subjected to in-plane tensile deformation (Ridruejo et al., 2012a): (c) Bond fracture micromechanisms. (d) Nominal stress (force per unit width) vs. nominal strain.

The micromechanisms involved in the deformation of nonwoven fabrics are intrinsically linked to the stress state attained in the fibers and the way individual fibers interact with the network through the bonds. Generally speaking, stronger bonds, as in chemical or thermal bonding, lead to higher stiffness and strength, although with lower ductility. Meanwhile, the behaviour of entangled nonwovens and, more specifically, needle-punched nonwovens is controlled by the fiber disentanglement process and the subsequent relative sliding and pull-out resulting in materials with high ductility and superior energy dissipation capability.

The onset of damage in bonded networks is essentially controlled by the strength of the bonds (Ridruejo et al., 2010; Farukh et al., 2014b). Ridruejo et al. (2010) determined the response of a bonded nonwoven fabric manufactured with bundles of E-glass fibers

1.3 Mechanical response of nonwovens

(Vetrotex M123, Saint Gobain) distributed isotropically in the plane, see Fig. 1.4(a). The behavior of the material was essentially linear and elastic up to the onset of damage which occurred by the brittle failure of the bonds between cross fiber bundles, see Fig. 1.4(b). Bundle reorientation and additional bond failure triggered the localization of the damage in a wider area containing oriented fiber bundles spanning the macroscopic crack. The final residual strength was provided by friction between bundles, which also led to an additional energy dissipation. Ridruejo et al. (2012a) also characterized the mechanical behavior of a thermally bonded polypropylene nonwoven (Tytar SF32 manufactured by Dupont). In this material, the fibers underwent plastic strains prior to bond failure, see Fig. 1.4(c), and this effect led to a large non-linearity in the nominal stress vs. strain curve prior to the maximum load, see Fig. 1.4(d).

The central feature governing the mechanical behavior of entangled networks is the formation of temporary contacts and entanglements between fibers. While the initial mechanical response of bonded networks is usually approximated by a linear-elastic law, entangled networks behave as non-linear elastic solids where the initial deformation is usually endorsed to fiber straightening and slack mechanisms (Masse and Poquillon, 2013). As soon as the entanglement strength is reached, fibers start to slide with respect to each other leading to large and non-recoverable strains even prior to material failure. Hence, the micromechanical response of these set of materials is much more complex than the response of the bonded networks previously described, due to the relevance of two additional mechanisms: fiber sliding and fiber disentanglement.

Fiber sliding is defined as the relative displacement among fibers, which modifies the distance between end points of the fiber, see Fig. 1.5. Progressive fiber sliding leads to fiber disentanglement, see Fig. 1.5(b) and (c). As a result, the microstructure in entangled nonwoven fabrics strongly evolves with the applied deformation due to fiber rotation, straightening, sliding and final disentanglement.

Fiber sliding is well documented in woven fabrics (Kirkwood et al., 2003) and several numerical models of pull-out failure were proposed in the past to address this mechanism (Zhu et al., 2011; Parsons et al., 2013). It has been shown that an increment of the pull-out force leads to an enhancement of in-plane properties (Dong and Sun, 2009). The pull-out or disentanglement force is highly dependent on different parameters such as fabric architecture, fiber length, clamping pressure, surface friction and loading conditions. For

Figure 1.5: Main deformation micromechanisms involved in the global deformation of entangled nonwovens: a) Reference configuration of the fiber inside the network. b) Fiber sliding. c) Fiber disentanglement. d) Dyneema Fraglight nonwoven subjected to in-plane tensile deformation showing extensive fiber disentanglement and pull-out (Martínez-Hergueta et al., 2015)

instance, Nilakantan and Gillespie (2013) determined that increasing the fabric pre-tension resulted in an increment of the pull-out force in woven materials. Friction between fibers seems to be one of the most relevant parameters and has to be carefully addressed in the modelization of woven fabrics. A low friction behavior decreases the ballistic performance of a Kevlar KM2 woven fabric while the opposite may lead to premature yarn rupture (Rao et al., 2009; Duan et al., 2005).

Nonwoven fabrics are highly inhomogeneous materials at the microscopic level. As a result, the macroscopic deformation under some circumstances is not effectively transmitted to all the fibers of the network and non-affine deformation modes may become dominant (Ward and Sweeney, 2004). Affine deformation is found in high density nonwovens with rigid bonds introduced by chemical or thermal treatments (Farukh et al., 2014a; Demirci et al., 2012; Hou et al., 2009; Sabuncuoglu et al., 2012) or by electrospinning (Silberstein et al., 2012; Pai et al., 2011). In this case, the local kinematic constraints introduced by the rigid bonds make the network to deform uniformly at the microscopic level. However, the lack of fiber constraint in compliant and low-density fiber networks results in local fluctuations of fiber displacements also known as non-affine deformations. This effect is illustrated in Fig. 1.6 where the elastic modulus of paper is plotted as a function of the fiber length and network density (Picu, 2011). There is a lack of efficiency in the strain transfer from the macroscopic to the microscopic level in low density networks very close

1.4 Mechanical behaviour of nonwovens modeling

Figure 1.6: Variation of the elastic modulus of paper with density for two different fiber lengths (Picu, 2011).

to the percolation limit¹, and this lead to an elastic modulus of the network well below the affine predictions assuming homogeneous deformation.

Non-affine deformation can also develop in certain nonwovens due to the stress transmission mechanisms by friction (Jeon et al., 2014) because increasing contacts between fibers results in a better stress transmission. Above a certain limit, the contact efficiency becomes almost perfect and the displacement continuity is achieved, leading to affine deformation at the macroscopic level. However, if the contact surface is lower than a given threshold, the stress transmission efficiency decreases and only a part of the macroscopic deformation is transferred to individual fibers, leading to non-affine deformation, which is accommodated by fiber slippage.

1.4 Mechanical behaviour of nonwovens modeling

The mechanical performance of fiber networks has attracted the interest of many researchers in the field of paper and textile manufacturing during the last decades. In contrast to the woven counterparts, the fiber network response depends not only on the relevant fiber mechanical properties as the stiffness, strength and toughness, but also on the fiber-

¹The formation of critical loading paths in fiber networks gives rise to a discontinuous change in mechanical properties (elastic modulus and strength) of the network at the so-called percolation threshold.

1.4 Mechanical behaviour of nonwovens modeling

to-fiber interactions and load transfer mechanisms. Due to the enormous difficulty of the proposed problem, different approaches have been developed depending on the method used to account for the fiber interaction in the network.

Phenomenological approaches are efficient methods to tackle the mechanical response of nonwoven fabrics. They rely on the use of modified non-linear elastic, plastic and damage models that are fitted against experimental results under simple loading conditions, as in-plane tension and shear. They can be easily used as constitutive equations in finite element models which are calibrated or tuned from experiments but should be limited to loading paths not excessively deviated from test data. In addition, the main limitation of phenomenological models is the obvious lack of micromechanical information arising from the fiber-to-fiber interactions affecting the current deformation and fracture mechanisms which in turn depend on the fibers used and on the manufacturing method.

Models which represent the microstructure of the nonwoven fabric and its evolution during deformation, including damage and fracture, are not often found in the literature due to the complexity of the modelization of the fiber-to-fiber interactions. A pioneering work was due to Cox (1952) who treated analytically the problem of a random network of elastic, infinite and non-interacting fibers subjected to in-plane deformation. Cox's model is based on the introduction of a uniform strain field in the nonwoven area so each fiber cross is uniquely dictated by the average strain field (also known as affine deformation). As a result, the rotation of the fiber at cross points is neglected and each single fiber is subjected only to uniaxial loading.

The introduction of the kinematic hypothesis of non-interacting fibers in the model allowed an easy analytical integration of stresses to determine the elastic constants of the fiber network. The elastic constants of the network, namely the elastic and shear moduli, depended consequently on the fiber elastic modulus as well as on the network density (total fiber length per unit area). In addition, finite length fibers can be included in Cox's model by reducing the fiber elastic modulus according to the shear stress transfer with the network neighborhood following the shear-lag theory.

Cox's model of non-interacting fibers is considered the upper bound for the elastic constants of a fiber network and it is only attained in the case of perfectly straight and infinite fibers randomly distributed (see Fig. 1.6 for the elastic modulus of low density paper networks (Picu, 2011)). Nevertheless, real non-wovens are manufactured using finite

Figure 1.7: Numerical predictions of the Von Mises stress (MPa) in a glass fiber nonwoven in a notched specimen ($a_0/W=0.4$) loaded in tension (Ridruejo et al., 2010).

length, curly fibers with initial slack, linked and anchored by chemical and/or mechanical bonds which clearly deviate from the ideal uniform strain kinematics in Cox's model. In addition, high density networks are also able to transmit bending and shear forces through the relative rotation of the bonds at the crossing points between fibers.

The successful application of Cox's model to predict stiffness and strength of paper drove the model extension to other nonwovens and textiles, see, for instance, the seminal work of Kallmes and Corte (1960), or other more recent works (Bronkhorst, 2003; Liu et al., 2013; Isaksson and Hägglund, 2009). Classical laminate theory developed for fiber-reinforced polymer lay-ups has also been applied to model nonwoven microstructures by assuming that fibers in each orientation behave as a single unidirectional and orthotropic ply following a given orientation fiber distribution function (Bais-Singh et al., 1998; Liao and Adanur, 1997). Coupling between plies is established by imposing perfect bonding between them and a ply strength criterion can be applied to determine the nonwoven laminate strength.

1.4 Mechanical behaviour of nonwovens modeling

Nevertheless, the enormous complexity of the fiber-to-fiber interactions and deformation modes in nonwovens requires the development of more advanced and refined models to account for microstructural evolution during the deformation, including fiber rotation and straightening, fiber failure, disentanglement and relative sliding. A first class of models in the literature is aimed at explicitly representing the actual microstructure of the fiber network: individual fibers or fiber bundles interacting among them by means of different mechanisms located at the cross overs or entanglement points. The main advantage of such *computational micromechanics* models is, hence, the possibility of studying non-local variations of the mechanical variables, as the fiber network density or strain localizations due to damage. Notables examples can be found in the literature (Ridruejo et al., 2010) for straight E-glass fiber bundles, which includes the bond strength and the friction among bundles. Fiber bundles were treated as straight elastic Timoshenko beams and the bundle network was created through connector elements which allow bond failure by shear loading. These authors studied the behavior of unnotched and notched rectangular specimens subjected to uniaxial tension to determine the tensile strength as well as the notch sensitivity. The stress redistribution due to bond failure at the crack tip allowed to relax the stress concentrations due to the notch over a wider material zone, see Fig. 1.7. Similar methodologies were also used for thermal-bonded nonwoven fabrics (Farukh et al., 2014b, 2013; Demirci et al., 2012; Sabuncuoglu et al., 2012) where the application of a heated rolled calendar onto the fiber web produce polymer melted monolithic regions which are connected by individual fibers that are simulated using straight trust elements.

However, the simulation of the entire nonwoven structure becomes computationally prohibitive when the ratio between the specimen dimensions and the average fragment length of the nonwoven becomes large. Homogenization models emerge as the most efficient methods. *Computational homogenization* theory assumes that the behavior of the nonwoven fabric can be obtained by submitting an RVE (representative volume element in the statistical sense) to different loading conditions, for instance, tension, shear or a combination of them, Fig. 1.8. The stress and strain fields at the microlevel are solved by means of the finite element method under the appropriate boundary conditions while the mechanical field variables, as stress and strain tensors, are homogenized in the RVE by volume integration in the whole domain. The selection of the appropriate size of the RVE should be taken into account and the microstructural length scale should be small as

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Figure 1.8: Computational micromechanics representative volume element: (a) 2D RVE (Heyden, 2000) and (b) 3D RVE (Dirrenberger et al., 2014).

compared with the domain size, (Dirrenberger et al., 2014; Fliegner et al., 2013; Isaksson, 2011).

For instance, Hearle and Stevenson (1964) studied the effect of fiber curl and slack on the elastic behavior of periodic bidimensional networks. Fiber bonds were rigid in this case and the corresponding curled segments were modeled by means of beam elements with a modified elastic behaviour to account for the local curvature radius. Heyden (2000) used a similar approach but extended to a 3D RVE to study the mechanical properties of cellulose fiber fluffs. Åström and Niskanen (1991) and subsequently Räisänen et al. (1997) studied the fracture behavior of rigid bonded bidimensional fiber networks made up by elastoplastic fibers and concluded that the response of the network was similar, in shape, to the stress-strain relationship of the single filament irrespective of the network density. The direct translation of the stress-strain curve shape of the single filament into macroscopic curve of the nonwoven is only valid when fibers do not exhibit initial slack, bonding between filaments is perfect and the fibers essentially sustain axial loads. Nonetheless, these hypothesis are not totally fulfilled in mechanically-entangled fiber networks where initial slack and relative fiber sliding becomes a predominant deformation mechanism. When an entangled fiber network is strained and sliding occurs, the stress translated into the fibers is dominated by fiber sliding and affine deformation is no longer true. The loss of the proportional relation between macroscopic and microscopic strains, known as non-affine

Figure 1.9: (a) Experimental and (b) numerical deformed configuration of a TyparSF33 polypropylene nonwoven notched specimen subjected to tensile loading simulated by a multiscale constitutive model (Ridruejo et al., 2012b).

deformation, makes the simulation very difficult from the computational viewpoint as the fiber-to-fiber contacts should be tracked during the deformation history (Barbier et al., 2009; Durville, 2005; Kulachenko and Uesaka, 2012; Rodney et al., 2005; Pourdeyhimi et al., 2006).

A simplified attempt to reduce the computational cost of the numerical homogenization of an RVE is the development of lattice structures to represent the local kinematics deformation of the nonwoven material. For instance, Silberstein et al. (2012) proposed a triangular unit cell to represent a electrospun mat network where each member of the triangular cell represented a single filament. This model was useful to provide a rough estimation the elastic energy stored in axial and bending deformation modes but it has to relay in many fitting parameters without a clear physical meaning. Along the same lines, Pai et al. (2011) used a 4-fiber lattice RVE using curved fibers and included torsional springs to capture the effect of bond rotation. This model was able to yield estimations of the influence of the curly fibers on the elastic behavior of the network but was not robust enough to fully describe their overall mechanical response. Diamond lattices were developed by Hearle and Purdy (1978) which included fiber slippage as the main deformation mechanism.

The computational models described above are restricted to low density networks containing a moderate number of fibers and fiber-to-fiber contacts (Ridruejo et al., 2010),

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and become prohibitive in high density nonwoven fabrics. Moreover, numerical difficulties associated with the problem discretization can constraint the internal rotations of the web and produce spurious local increments of the stiffness of the material, which can trigger damage localization (Ridruejo, 2011). To overcome such limitations, *multiscale homogenization* was developed to determine the macroscopic behavior while capturing the main microstructural features.

Multiscale homogenization in nonwovens is carried out by the combination of two basic ingredients: fiber and network features. The first one includes the mechanical response of a single fiber and the interactions with its neighborhood while the second encompasses the fiber spatial distribution, the fiber orientation distribution (ODF) and the bond distribution. They are used to obtain an average response of the mechanical field variables by combining all fiber directions according to the orientation distribution. As a result, the homogenization scheme is able to determine the local averaged response of the nonwoven fabric for a given strain tensor at the Gauss point level of the finite element model and makes the technique special suited to work as a constitutive equation in commercial finite element codes programmed as material user subroutines. Ridruejo et al. (2012a) developed a constitutive equation for polypropylene thermal bonded nonwovens (Ridruejo, 2011) under finite strains that account for fiber rotation and plasticity. The model is based on an expansion of the Cox developments under finite strains and rotations which was originally applied to biological structural tissues by Planas and Elices (2007). In addition, Ridruejo et al. (2012a) included the damage mechanisms observed experimentally, bond and fiber failure, fiber sliding and pull-out, following an stochastic approach based on Monte Carlo lottery. The model allowed to determine the stress-strain curves and damage mechanics of plain and notched specimens of a polypropylene nonwoven fabric, Fig. 1.9. Similar *multiscale homogenization* schemes were developed by other authors. See, for instance the work carried out to study the mechanical response of nonwoven fabrics made up of ultra high molecular weight polyethylene fibers (UHMWPE) (Chocron et al., 2002) by Raina and Linder (2014) and Jearanaisilawong (2008).

One of the main disadvantages of multiscale homogenization is related to the large number of parameters in the model. While in *computational homogenization*, the material response is fully described by the fiber constitutive equation and the bond behaviour, *multiscale homogenization* requires to establish *a priori* the kinematics of the fiber embedded

in the network and the interaction with the neighborhood material. These hypotheses are crucial to accurately describe the deformation and damage mechanisms of the nonwoven fabric, specially in entangled fiber networks where fiber sliding plays an important role. Furthermore, the experimental characterization of this complex response is not straightforward and numerical predictions are often fitted to experimental results (Raina and Linder, 2014).

1.5 Objectives

The mechanical response of nonwoven fabrics may differ depending on the bond nature, resulting in different dominant deformation and fracture mechanisms. Most of the relevant scientific work in this area has been carried out for thermal or chemical bonded networks. In these materials, the sequential evolution of the microstructure is well understood. Fiber bonds dictate the strength of the material which is followed by the subsequent fiber redistribution and pull-out. However, entangled networks exhibit a much more complex mechanical behavior and the influence of fiber slack, reorientation, sliding and final disentanglement at finite strains is not well established. Most of the results in the literature are related to commercially-available nonwoven fabrics and the mechanical characterization is often guided to the final end-user application, lacking a comprehensive knowledge of the deformation mechanisms involved.

This is precisely the first objective of this doctoral thesis: to set-up the appropriate experimental techniques and microstructural characterization methods to ascertain the sequence of deformation and failure processes in a commercial-available entangled nonwoven. This work provides a comprehensive study of the mechanical response of a mechanically-entangled nonwoven fabric produced by needlepunching and subjected to in-plane deformation and impact loads.

Nevertheless, experimental characterization is not enough to provide a complete insight of the physical processes involved in this complex problem. Simulation can be very useful to understand the interaction among the different mechanisms and provide quantitative estimations of the effect of the microstructure on the mechanical response. Thus, the second objective of this thesis was to develop a physically-based multiscale constitutive model to predict the macroscopic mechanical response of the mechanically-entangled nonwoven fab-

1.5 Objectives

ric by means of the finite element method. The main assumptions of the model in terms of dominant deformation and fracture mechanisms should be obtained from the experimental analyses. In addition, the model parameters should have a clear physical meaning and should be identified by means of independent tests as far as possible. Finally, the model should be able to reproduce the mechanical behavior under different loading conditions (in plane deformation, ballistic impact, etc.) and be affordable from the computational viewpoint.

This thesis dissertation is divided into two parts. The first one is devoted to the experimental characterization of the needle-punched nonwoven fabric and includes the description and the microstructural analysis of the material. It encompasses the material description and the experimental techniques (Chapter 2), the microstructural and micromechanical characterization (Chapter 3), the in-plane characterization in quasi-static and dynamic regimes including the evolution of the microstructure with the deformation (Chapter 4) and the ballistic characterization (Chapter 5). The second part of the dissertation addresses the development of the constitutive model and its validation with in-plane and ballistic experimental results (Chapter 6 and 7).

CHAPTER 2

Material and experimental techniques

The material and the different experimental techniques used in the thesis are presented in this chapter. The nonwoven fabric is a commercial needle-punched felt made up of ultra high molecular weight polyethylene fibers (UHMWPE). It was fully characterized from the microstructural and mechanical viewpoints using various techniques. In particular, the complex nonwoven fiber network was revealed by means of X-ray Computer Tomography (XCT) and 2D Wide-angle X-ray diffraction (WAXD). The influence of the microstructure on the final mechanical performance was addressed by means of a combination of mechanical characterization tests at different length scales ranging from individual tensile fiber and fiber pull-out tests to conventional coupon tests as well as dynamic and ballistic tests.

2.1 Material

The material used in this study is a needle-punched nonwoven fabric manufactured by DSM under the commercial trademark Fraglight NW201, Fig. 2.1(a). The fabric is made up of Dyneema SK75 ultrahigh molecular weight polyethylene fibers (UHMWPE) of ≈ 60 mm in length, which often form two-fiber bundles of $\approx 11 \mu\text{m}$ in diameter, Fig. 2.1(b). The nonwoven fabric presents a highly entangled fiber network, which is well suited for appli-

2.1 Material

Figure 2.1: (a) Scanning Electron Microscope (SEM) micrograph of the Fraglight NW201 nonwoven fabric. (b) Typical Dyneema SK75 two fiber bundle.

cations driven by energy absorption as, for instance, personal protection against ballistic impact of small fragments.

The thickness (1.5-2.3mm) is not an objective parameter to characterize the fabric because it depends on the clamping forces used during the measurement (ASTM D5729-97). Instead, the fiber content measured as the total fiber length per unit area of the nonwoven is used because the stiffness and the strength scale with this parameter (Picu, 2011), which is given by the ratio between the areal weight of the fabric, ρ (190-220 g/m²) and the density of the polyethylene fiber, ρ_f (970 Kg/m³).

Fraglight is manufactured by the continuous deposition of single filaments (two-fiber bundles) on a moving bed surface, forming a batt. The batt is needlefelted, with the aid of the oscillatory application of barbed needles, producing fiber loops and mechanical entanglements among fibers (Russell, 2007). The short needle-to-needle distance, as well as its continuous and repetitive oscillatory motion through the batt thickness consolidates the fiber network to its final configuration. The manufacturing process introduces two principal material directions frequently known as machine (MD) and transverse (TD) directions which follow the bed displacement and the perpendicular direction, respectively, see Fig. 2.2. More specific details of manufacturing methods of needlepunched nonwovens were reviewed in Chapter 1.

The nonwoven material was supplied in a roll of 1 m width. Specimens for testing and characterization were cut with a hot-wire because knives, scissors and blades are not effec-

Figure 2.2: Main material orientations of the fabric.

tive because of early edge blunting. The in-house hot-wire system was built by connecting a straight copper wire to an electrical power supply to reach the desired temperature ($\approx 180^\circ\text{C}$) by controlling the voltage between the wire edges. The temperature of the wire was higher than the melting temperature of the polyethylene fibers leading to a clean edge cut albeit additional bonds between fibers were introduced in the edges due to fiber melting.

2.2 Experimental techniques

2.2.1 Microstructural characterization

X-Ray Computed Tomography

X-Ray Computed Tomography (XCT) is a non-destructive inspection technique in which the 3D reconstruction of the volume is obtained from the X-ray images acquired at different angles by rotating the specimen. Fig. 2.3 shows the principle of operation of X-ray tomography system. X-rays are generated by the acceleration of electrons towards a target made of tungsten or molybdenum. The electrons extracted from the filament (cathode) are focused and centered by electromagnets and travel inside the vacuum tube towards the target (anode), generating X-rays. The sample is positioned at a certain angle θ between the X-ray source and the detector which records a radiography. The angle of the sample is modified in order to record a set of radiographies which are subsequently post-process to recreate a 3D volume.

2.2 Experimental techniques

Figure 2.3: Scheme of the working principle of an open X-ray tube with a transmission target.

The sample can be modeled as a two or three-dimensional distribution of the X-ray attenuation coefficient, $\mu(x, y)$, which is a property that characterizes the ability of the material to absorb X-ray from the beam source. The radiation intensity, I , transmitted through a layer of material, see Fig. 2.4, is related to the incident intensity, I_0 according to Lambert-Beer's law, Equation 2.1. This equation relates the total attenuation $p(t)$ (ratio of the transmitted to the incident intensity) through the X-ray absorption coefficient of the material, $\mu(x, y)$

$$p(t) = \ln \frac{I}{I_0} = \exp \left[\int_{\Gamma} \mu(x, y) ds \right] \quad (2.1)$$

where the line integral represents the total attenuation suffered by the X-ray beam traveling along a straight path $s(x, y)$ through the cross-section of the object and t is the distance from each ray of a parallel beam to the center of rotation, see Fig. 2.4. The procedure for the reconstruction of a sample volume from the radiographies collected at different angles θ of rotation is explained on a parallel beam configuration for simplicity. During the radiography collection, the sample rotates around the z axis (perpendicular to the paper). The cross-section of the sample is described by the function $f(x, y)$. The X-ray beam is assumed to be formed by parallel rays. When each ray passes through the sample, part of the radiation is absorbed and the attenuated intensity, $p(t, \theta)$, is collected in the detector.

Figure 2.4: Principle of tomography and illustration of the Fourier slice theorem.

The attenuation will depend on the absorption coefficient of the material and on the length of the path, s , through the sample.

Once the different projections are recorded for a set of rotation angles, the next step is to obtain the tomographic reconstruction of the original object. The object is reconstructed by means of the projection-slice theorem (Herman, 1980; Kak and Slaney, 1987). This theorem establishes that the reconstruction of the object $f(x, y)$ is possible from the X-ray attenuation projections acquired at infinite rotation angles, $p(t, \theta)$. This function $p(t, \theta)$ is also known as the Radon transform. The projection-slice theorem states that it is possible to reconstruct the cross-section of the object $f(x, y)$ by finding the inverse Radon function of $p(\omega, \theta)$ (Fourier inverse transform) and the total volume of the object is obtained by stacking up a number of slices corresponding to different cross-sections. Unfortunately, the inverse Radon transform is extremely unstable with respect to noisy data and, in practice, a stabilized and discretized version of the inverse Radon transform (known as the Filtered Back Projection algorithm) is used. The idea of the Filtered Back Projection algorithm is to assign to each point of the object the average intensity of all the projections that pass through that point. The back projected image is, however, a blurred version of the original object. To overcome this effect, the reconstructed object is filtered using a high pass filter. Finally, the object is reconstructed by means of specific interpolation techniques.

2.2 Experimental techniques

XCT is recommended for the microstructural characterization of nonwovens in the case of dense fabrics where conventional SEM imaging is not enough to reveal the microstructure (Jubera et al., 2014; Hou et al., 2009; Isaksson and Hägglund, 2007). XCT is able in this case to provide the reconstructed geometry of the fibers within the network in a three dimensional domain (Ridruejo, 2011).

The network structure of the Fraglight nonwoven was studied by means of X-ray computed tomography. Tomograms were obtained with a Phoenix Nanotom 160NF at 50 kV and 275 μA using a Mo target in Mode 1. A set of 1700 radiographies were acquired for each tomogram using an exposure time of 750 ms. The tomograms were reconstructed from these radiographies through an algorithm based on the filtered back-projection procedure for Feldkamp cone beam geometry. The reconstructed volume encompassed a nonwoven area of $\approx 5 \times 5 \text{ mm}^2$ with a resolution of 3 $\mu\text{m}/\text{voxel}$. Even though the scanned area of the fabric was smaller than the average fiber length (60 mm), it provided useful information about the nonwoven morphology, specially at the fiber entanglement regions close to the needlepunch points.

2D Wide Angle X-Ray Diffraction

2D Wide Angle X-Ray Diffraction is a non-destructive inspection method that can be used to study the structure of semicrystalline polymers. This technique allows to determine with accuracy the crystal phase, the texture of a polymer film (including the preferred alignment direction of crystallites), the crystallite size and the presence of film stresses (Kittel, 1996). Fig. 2.5 shows a schematic representation of a general set-up of a diffractometer used in WAXD experiments. The sample is mounted on a holder, in between the collimator and the detector. The material is exposed to a focused monochromatic beam of X-rays and the intensity of the diffracted beam is recorded in the detector for a given incident angle.

The diffraction peaks are found when the reflections from parallel planes of atoms lead to a constructive interference and, thus, the phase difference between reflections is proportional to 2π . This condition is expressed by Bragg's law according to

$$2d \sin \theta = n\lambda \quad (2.2)$$

Figure 2.5: 2D Wide Angle X-Ray Diffraction set-up.

Figure 2.6: Representation of the Bragg's law. The atomic planes are spaced d . The path difference for rays reflected from adjacent planes is $2d \sin \theta$. Constructive interference occurs when this length is proportional to the wavelength of the radiation λ .

where d is the spacing between the atomic planes, θ the incident angle of the beam, n an integer, and λ the wavelength of the beam, see Fig. 2.6.

Bragg reflection can occur only for $\lambda \leq 2d$, so the wavelength λ has to be of the same order as the crystal spacing (0.1 to 10 nm), so X-rays are used for this purpose. Every crystal has a unique pattern of d spacings, which is a 'fingerprint' for the crystal. Solids with the same chemical composition but different phases can be identified by their pattern of d -spacings. 2D diffractograms are composed by concentric rings at a certain distance from the beam center on a background surface. The distance of the ring from the beam center depends on the spacing between diffracting planes d . The intensity along

2.2 Experimental techniques

the rings depends on the alignment of the polymer chains, leading to a constant intensity if the chains are isotropically oriented in all directions or to discrete spots in the case of the alignment of the molecular chains along a preferential direction. In addition, WAXD also provides information about the evolution of the semi-crystalline microstructure during material straining.

Dyneema fibers are obtained by severe drawing of ultra high molecular weight polyethylene. The molecular chains within the fiber are almost perfectly aligned with the fiber axis. 2D WAXD of these fibers are characterized by discrete spots which indicate the fiber axis. This information can be used to track the fiber orientation within the nonwoven fabric and relate this information with its overall microstructure (Russell et al., 2013).

Wide Angle X-Ray Diffraction (WAXD) experiments were carried out at the Institute of Polymer Science and Technology of CSIC (Consejo Superior de Investigaciones Científicas). Diffraction patterns of the nonwoven fabric were acquired using a flat plate camera attached to a Phillips 2 KW tube X-ray generator with monochromatic nickel-filtered Cu K_α radiation, $\lambda = 1.5 \text{ \AA}$ wavelength with an exposure time of 1 minute. The inspection spot size was $\approx 1 \text{ mm}^2$. It should be remarked that the WAXD technique was able to deliver valuable information of the fiber orientation distribution even though the inspection window was much smaller than the average fiber length of the nonwoven.

The scanning records were centered in the range of diffraction angles $15^\circ < 2\theta < 25^\circ$ which covered the diffraction peaks corresponding to the (110) and (200) planes of the orthorhombic polyethylene crystal. The orientation of these planes could be easily used to determine the fiber tangent distribution and the corresponding changes in fiber orientation due to the deformation. Considering a curved fiber, as the one depicted in Fig. 2.7, the average tangent direction, $\langle \vec{t} \rangle$, can be determined as

$$\langle \vec{t} \rangle = \frac{1}{L} \int_A^B \vec{t} ds = \frac{\vec{r}_B - \vec{r}_A}{L} \quad (2.3)$$

which can be understood as a measure of the orientation angle, β in Fig. 2.7(a), of a fictitious straight fiber joining the fiber ends (end-to-end vector). Therefore, the distribution of average orientation vectors within the inspection spot can be obtained by means of WAXD and this information can be considered representative of the overall fiber orientation in the nonwoven material, Fig. 2.7(b).

Figure 2.7: (a) Planar representation of a curved fiber within the inspection spot of the WAXD measurement. The end-to-end orientation vector, $\langle \vec{t} \rangle$, can be used to track fiber orientation distribution. (b) Distribution of end-to-end vectors in the nonwoven material.

The image treatment of the diffraction patterns was carried out with the scientific software FIT2D from ESRF (European Synchrotron Radiation Facility) (Hammersley, 1995). The intensity of the diffraction planes was measured for a certain angle in order to identify properly the position of the diffraction peaks with respect to the beam center. The orientation distribution functions (ODF) of the fibers was determined by measuring a circular section of 30 pixels of width around the main diffraction peak in 360 points homogeneously distributed in the planar area, as for instance in Fig. 2.8. The equivalent intensity used for the comparison of the patterns was the result of the diffraction intensity minus the intensity of a background area of the diffractogram to remove the influence of differences in fiber density or exposure time. The methodology was applied to the diffraction peaks corresponding to both (110) and (200) of polyethylene, and resulted in equivalent fiber orientation distribution functions.

Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) is a thermo-analytical technique used to measure phase changes. The DSC apparatus operates on the basis of the 'null balance' principle. Within the system, there are two small sample holders: one for the sample to be tested and the other with a reference material with a well-defined heat capacity at constant pres-

2.2 Experimental techniques

Figure 2.8: Example diffractogram treated with FIT2D for an UHMWPE fiber bundle aligned along the horizontal direction. Discrete diffraction spots corresponding to the (110) plane appeared parallel to the fiber axis. Diffraction intensity is measured in the circular section drawn around the peaks.

sure C_p in the temperature range of operation. The sample and the reference material are maintained at nearly the same temperature throughout the test and the enthalpy variation, ΔH , can be computed for the reference material in the temperature range T_1 and T_2 as

$$\Delta H = \int_{T_1}^{T_2} C_p dT. \quad (2.4)$$

A DSC system (Q200 TA Instruments) was used to determine the glass transition temperature of Dyneema fibers extracted from the nonwoven in nitrogen atmosphere. Prior to any test, specimens were weighted ($\approx 3\text{--}5$ mg) and, then, placed in an aluminium pan perfectly sealed with an aluminium cover. The specimen temperature was first stabilized at 20°C for 10 min, and then heated at a rate of $10^\circ\text{C}/\text{min}$ up to 200°C . The fusion temperature of the Dyneema fibers was in this case determined from the heat-flow curve obtained from the DSC system.

2.2.2 Mechanical characterization

In-plane quasi-static tests

Rectangular specimens were subjected to in-plane uniaxial loading along the different material directions. To this end, a pair of two flat steel plates fastened with regular screws were used to clamp the fabric by means of frictional forces. The grips were connected to the

actuator and the cross-head of an Instron 3384 electromechanical testing frame. The grips allowed the easy characterization of Fraglight using coupons with different widths ranging from 35-300mm. The maximum displacement of the cross head of the testing frame enabled also straining the material up to deformations in excess of 100%.

The optimum specimen dimensions were determined by systematically testing coupons with different heights and widths in order to avoid any possible interference of the fiber length or of the clamping system on the mechanical response. For instance, the response of very short specimens was distorted because of parasitic fiber clamping between both grips. It was found that the influence of the fiber length could be neglected when the coupon height was larger than half the average fiber length (30 mm).

The full-field displacement and deformation fields of the specimens subjected to in-plane loading was determined by means of Digital Image Correlation (DIC) using the commercial software VIC2D (Correlated Solutions, Inc.). DIC is based on tracking the local gray intensity distribution on the specimen surface by direct comparison with the corresponding undeformed configuration. The full field displacement is obtained and, subsequently, the strain tensor can be determined from the derivatives of the displacement field. More details of the experimental technique can be found in (Canal, 2011; Totry et al., 2008b,a). The technique has been previously applied to reveal deformation patterns in nonwovens (Ridruejo et al., 2015; Isaksson and Häggglund, 2009). The quality of the gray contrast between individual fibers is enough to capture the non-homogeneous strain field during mechanical loading, although it can be enhanced by introducing randomly distributed dark spots with a ink pen.

In-plane dynamic tests

The Split-Hopkinson bar is the standard device for testing materials at high strain rates. The Split-Hopkinson bar apparatus is composed by a loading bar, an incident or input bar and a transmission or output bar, see Fig. 2.9. The specimen is mechanically gripped between the input and the output bar. The loading bar is then pulled to produce an elastic tensile pulse, which propagates along the input bar towards the specimen. When the incident pulse reaches the specimen, part of this pulse is reflected into the input bar due to the impedance mismatch at the bar-specimen interface. The remaining intensity

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Figure 2.9: Scheme of a conventional Split-Hopkinson pressure bar.

Figure 2.10: Split-Hopkinson bar design for tensile testing of mechanically-entangled nonwoven fabrics. The system is composed by two hollow input and output bars.

of the pulse is transmitted into the specimen and, finally, into the output bar. Axial strain gages are mounted on the surfaces of the input and output bars providing time-resolved measurements of the traveling elastic pulses. Assuming a perfect one-dimensional propagation of the stress waves along the bars, the nominal strain rate $\dot{\epsilon}(t)$ in the specimen can be computed as

$$\dot{\epsilon}(t) = \frac{2c_0}{L} \epsilon_r(t) \quad (2.5)$$

where L is the initial specimen length, $\epsilon_r(t)$ the time-resolved strain of the reflected pulse in the incident bar and c_0 is the wave velocity in the bars. Direct integration of equation 2.5 provides the time-resolved axial strain of the tested specimen $\epsilon_t(t)$. Finally, the nominal axial stress of the specimen σ can be determined as

$$\sigma(t) = \frac{A_0}{A_S} E \epsilon_t(t) \quad (2.6)$$

where A_S is the cross-sectional area of the specimen and A_0 and E are the cross-sectional area and the elastic modulus of the bar material, respectively.

Measuring the high strain rate tensile response of nonwovens presents considerable technical difficulties for several reasons. Firstly, low impedance materials result in a very low response in the transmission bar, which is difficult to measure. Secondly, their inherent low wave speed makes the transit time much longer than in metallic materials and an equilibrated stress state may not be reached during dynamic deformation unless the duration of the stress pulse is long enough. Thirdly, mechanically-entangled nonwovens can withstand very large strains before failure and it is generally not possible to obtain the whole stress-stretch curve in a single pulse. Thus, the bars and the incident pulse need to be long enough to achieve the required displacement. And, finally, the minimum specimen size to characterize the fiber network depends on the fiber length which is usually very large as compared with the standard sizes used in the Split-Hopkinson bar. To overcome these problems, several solutions were reported in the literature including miniaturized Hopkinson bars (Luo et al., 2009), lighter hollow transmission bars (Chen et al., 1999) or low impedance solid metallic (Shergold et al., 2006; Pellegrino et al., 2015) and polymer bars (Wang et al., 1994; Shim and Mohr, 2009; Rao et al., 1997).

To overcome these difficulties, a novel Split-Hopkinson bar system was designed in collaboration with the Impact Engineering Laboratory of the University of Oxford. Aluminium 7075-T6 alloy hollow tubes of 50.8 mm in outer diameter, 1.651 mm thickness and 2.7 m length were used, see Fig. 2.10. Hollow tubes were used instead of traditional solid rods to increase the transmitted signal during the tests. The hollow tubes were also light enough with reduced inertia in order to attain the required specimen elongation. The striker bar was also modified to obtain a longer pulse, using a U-shaped bar of 2.5 m length manufactured with Ti-6Al-4V alloy. Further details for the Split-Hopkinson Tensile Bar apparatus are summarized in Appendix A.

Ballistic tests

The Fraglight nonwoven fabric was subjected to ballistic impact loading using a gas gun. To this end, specimens of 500 x 500 mm² were first clamped along their four edges using an aluminum rigid rig. A total of 16 steel screws with 6 mm diameter were used to clamp the fabric to the frame. The four edges of the fabric were previously impregnated with Derakane 8084 epoxy vinylester resin to avoid sliding. The resin was cured at room

2.2 Experimental techniques

Figure 2.11: Experimental set-up for ballistic tests.

temperature for 24 hours. The free surface of the fabric within the frame occupied $350 \times 350 \text{ mm}^2$.

Ballistic tests were carried out using a pneumatic launcher SABRE A1 + gas gun, see Fig. 2.11 available at the Department of Material Science of the Polytechnic University of Madrid. The projectile used was a steel sphere of 5.5 mm in diameter (caliber 0.22) and 0.706 g of mass which was fit on a barrel of 7.62 mm in diameter using a plastic sabot. The impact velocity applied was in the range 270 to 400 m/s, leading to impact energies between 25 and 55 J. A Phantom V12 high-speed video camera was used to measure the initial and residual velocities of the projectile and obtain the energy absorbed by the fabric during impact. Images were acquired at a frame rate of 40.000 fps, using resolution of 512×256 pixels with an exposure time of $23.45 \mu\text{s}$.

Micromechanical testing

Two kind of tests at the fiber scale were carried out in this thesis: direct mechanical characterization of single fibers extracted from the fiber felt and the pull-out tests dedicated to determine the resistance exhibited by individual fibers and fiber bundles when are extracted from the bulk fabric.

These tests were performed using a MTS Nano Bionix system (MTS Systems Corporation) equipped with a 1N load cell with a nominal load and an extension resolution of 50 nN and 35 nm, respectively. The experiments were carried out at the Department of Material Science of the Polytechnic University of Madrid. The Nano Bionix system is, essentially, composed by two supporting arms, a fixed one which is connected to the load cell and a

Figure 2.12: Experimental set-up (MTS Nano Bionix) for micromechanical tests on individual fibers or fiber bundles.

moving arm where displacements are applied using an endless screw, see Fig. 2.12. The load cell is very fragile, and special precautions were taken when handling the specimen during the connection to the load cell in order to prevent damage of the system.

Individual tests on single fibers or two-fiber bundles were carried out using the cardboard method. Fibers were first extracted from the fabric by carefully pulling them with tweezers. Fibers were dyed with a blue ink for a better visualization during the tests. The edges of the fiber were immediately bonded to a cardboard frame using a cyanoacrylate adhesive to prevent fiber damage during manipulation, see Fig. 2.13. The open window of the cardboard was selected within the range of 10-20 mm fixing *a priori* the gage length of the tested fiber. Afterwards, the cardboard was connected to the testing machine clamping system at the bonding points and the cardboard arms were carefully cut so the load was entirely transmitted through the fiber. Tensile tests were carried under stroke control at normal nominal strain rates of 10^{-4}s^{-1} .

The stress carried by the fiber was computed from the applied load F and the cross-section of the fiber A which was measured with an optical microscope (Zeiss EVO MA-15). The fiber strains were measured using the compliance methodology. To this end, fibers with different gage lengths were tested to remove the influence of the machine compliance on the measured load-displacement curve. The total elongation of a fiber with gage length L is $\Delta L = FL/EA + C$ where F , E and A stand for the applied force, the fiber elastic

2.2 Experimental techniques

Figure 2.13: UHMWPE fiber bonded onto a cardboard for mechanical tensile testing.

Figure 2.14: Pull-out tests carried out in the NanoBionix system. (a) Single fiber pull-out tests. (b) Multiple-fiber pull-out test.

modulus and the fiber cross-section, respectively, while C is the machine compliance. A set of two tests with different fiber length is enough to determine the machine compliance. Afterwards, engineering strains were computed using the elastic elongation and the gage length of the fibers.

In addition, pull-out tests were performed using the same Nano Bionix testing system to determine the load-displacement curve when fibers or fiber bundles were pulled out from the bulk felt network. This curve provided valuable information regarding the sliding mechanisms in mechanically-entangled nonwovens as well as the pull-out strength and the energy dissipated during the process.

2.2 Experimental techniques

Single fiber or fiber bundles were extracted carefully from the edge of a folded specimen of $80 \times 80 \text{ mm}^2$, see Fig. 2.14(a). The folded fabric was then attached to the lower grip of the testing frame while the individual fiber or fiber bundle was bonded with cyanoacrylate to an acetate film connected to the load cell. The initial and previously extracted fiber length ranged between 15-20 mm of the total fiber length (60 mm). Experiments were performed under stroke control at cross head speeds in the range of 0.01 to 1.0 mm/s to capture any possible strain rate effects.

In addition, multiple fiber bundles pull-out tests were performed using double-edge notched tension specimens (DENT) of $50 \times 75 \text{ mm}^2$ oriented along MD and TD directions, see Fig. 2.14(b). Two deep symmetric notches were introduced perpendicular to the loading direction in each specimen leaving around ≈ 15 to 50 filaments connecting both halves of the specimen. The number of filaments was determined using a magnifying glass after testing. The pull-out extraction velocity was set to 1.0 mm/s in all the tests. The multiple fiber bundle pull-out configuration was envisaged to reduce the experimental scatter with respect to the single fiber bundle tests and to determine more precisely the influence of the material orientation on the extraction mechanisms. The number of fibers tested in each of the DENT specimens was large enough to capture the essentials of the disentanglement and sliding mechanisms providing quasi-independent values of bundle stiffness, strength and energy dissipated regardless of the number of fibers bridging the halves of the specimen.

CHAPTER 3

Microstructural and micromechanical characterization

The next section is devoted to the description of the needle-punched nonwoven fabric in microstructural and micromechanical terms. Material microstructure was ascertained by means of optical microscopy, X-Ray Computed Tomography and 2D Wide Angle X-Ray Diffraction. The use of this latter experimental technique applied to the characterization of nonwovens is one of the novelties of this doctoral thesis. Besides, deformation and failure micromechanisms were determined by micromechanical testing. Quantification of the energy dissipation was carried out by fiber tensile tests in the case of fiber breakage and by pull-out experiments in the case of fiber sliding and disentanglement. Single bundle pull-out tests were also useful to characterize the response of individual fibers inside the structure and the percentage of detached fibers from the network skeleton. Additional multiple bundle pull-out tests were accomplished to analyze any orientation dependency. This study pretends to be the first attempt to characterize these pull-out curves for needle-punched nonwoven fabrics. The material parameters obtained from the micromechanical analysis were subsequently used for the development of a multiscale material constitutive model in Chapter 6.

3.1 Microstructural characterization

A qualitative assessment of the microstructure of the nonwoven fabric by Scanning Electron Microscopy (SEM) could be found in Fig. 2.1(a), which did not show any directionality of the fiber distribution. A first approximation to the morphology of the nonwoven material was obtained by a direct optical microscopy inspection, see Fig. 3.1(a). The needle-punch points introduced by the barbed needles during manufacturing were marked with ink to facilitate the quantitative analysis. The pictures were post-processed by the commercial software ImageJ, see Fig. 3.1(b) and a Matlab script (MathWorks, Inc.) was used to calculate the entanglements distribution and the punched density, see Fig. 3.1(c). The punch density was $\approx 13\text{-}14\text{ cm}^{-2}$ and knots were distributed following a random pattern.

A more detailed inspection of the fabric microstructure was carried out by XCT. The tomography of a thin slice of $\approx 100\text{ }\mu\text{m}$ of thickness parallel to the fabric plane from the as-received fabric showed a quasi-planar distribution of curved fibers, see Fig. 3.2(b), which in some cases presented a shape close to a circumference in the plane of the batt, see Fig. 3.2(c). This pattern was locally distorted around the needle-punch points, in which the fiber network showed a through-the-thickness stitched-like structure, Fig. 3.2(a). The barbed needles opened the fabric in the through-the-thickness direction forcing the fiber bundles to move from the web top to the bottom surface. They remained in such position after the needles returned and the oscillatory movement led to the mechanical entanglement of the fiber bundles in the network.

More valuable quantitative information about the orientation and curvature angle of individual fiber bundles could be obtained from these tomographies. Both parameters, orientation and curvature, of individual fiber bundles were measured using the same criteria: the curvature angle θ (Fig. 3.3) was measured from the coordinates of two extreme points and a central one of the circular arc of each fiber while the orientation with respect to MD, β , was given by the angle between chord direction of the circular arc and MD, Fig. 3.3. Both parameters were measured in approximately 100 fiber bundles from five different tomographies. It should be noticed that these measurements were carried out over fiber fragments as the total fiber length was well above the size of the analyzed region.

Figure 3.1: Needle-punch pattern of a specimen oriented at MD. (a) Original picture. (b) ImageJ postprocessing. (c) Matlab implementation.

Figure 3.2: (a) Through-the-thickness tomography of the fiber distribution around a needle-punch point. (b) 3D tomography, showing the position of the knots and the fiber curvature. (c) Tomography parallel to the fabric plane in the as-received condition, showing the fiber distribution in the fabric plane.

3.1 Microstructural characterization

Figure 3.3: (a) Cumulative density function for the fiber orientation angle, β , for the as-received condition. b) Cumulative density function for the fiber curvature angle, θ , for the as-received condition.

The cumulative density function (CDF) of the orientation angle, β , is shown in Fig. 3.3(a). It follows a straight line (diagonal line in Fig. 3.3(a)), indicating that the initial fiber orientation within the plane of the batt was isotropic. The CDF corresponding to the curvature angle, θ , is plotted in Fig. 3.3(b) and showed a similar probability of finding fibers with all curvature angles.

The microstructure was also analyzed by means of two dimensional Wide Angle X-Ray Diffraction (WAXD). Fig. 3.4(a) shows the diffraction pattern of a straight polyethylene fiber bundle extracted from the nonwoven fabric. An azimuthal arc on the poles is observed and the intensity peaks correspond to the diffraction planes (110) and (200) of the orthorhombic polyethylene crystals oriented parallel to the fiber axis. The wide intensity peak can be attributed to the slight misalignment of the fiber during the experiments as well as to the small misorientation of polyethylene crystals with respect to the fiber axis. The diffraction pattern of the nonwoven fabric in the as-received condition, Fig. 3.4(b), shows two circular rings with constant intensity corresponding to the (110) and (200) crystallographic planes. This pattern indicates that fiber orientation distribution was isotropic within the fabric plane.

Figure 3.4: Two-dimensional WAXD patterns of (a) Dyneema fiber bundle. (b) Non-woven fabric in the as-received condition.

These findings are in perfect agreement with the observations carried out by XCT presented in Fig. 3.3. It should be noted that the information obtained by WAXD encompasses the information of both fiber rotation and fiber straightening and it is not possible to deconvolve these effects. Nevertheless, the information obtained by XCT (the CDFs of the orientation angle β and of the curvature angle θ) can be used to reconstruct the orientation of the diffraction planes and compare the diffraction patterns from the reconstructed fabric with those obtained by 2D WAXD in the deformed fabric. The reconstruction of the diffraction planes was carried out by assigning an equal probability to the orientation of the diffraction angles within the interval $\beta \pm \theta/2$. The intensity of the diffraction pattern obtained from XCT data of the fiber orientation and fiber curvature angles in the interval $[0, \pi/2]$ has been compared with the intensities obtained by WAXD in Figs. 3.5. The results are in good agreement and showed in both cases an initial isotropic fiber orientation distribution function (ODF).

3.2 Micromechanical characterization

Figure 3.5: Orientation distribution functions of fiber bundles obtained from WAXD and XCT for the as-received condition. The horizontal line of intensity $1/2\pi$ corresponds to the isotropic distribution.

3.2 Micromechanical characterization

3.2.1 Fiber tensile tests

10 tensile tests of single fiber bundles were carried out following the experimental procedure described in Section 2.2.2. The mechanical behavior of the polyethylene fibers was essentially linear elastic until failure, Fig. 3.6(a), and it was characterized by the elastic modulus, $E_f = 39.7 \pm 8.9$ GPa, the tensile strength, $\sigma_f = 2.45 \pm 0.75$ GPa and the strain-to-failure, $\epsilon_f = 4.80 \pm 1.05$ %. These properties are in agreement with values reported in the literature for SK75 Dyneema fibers (Russell et al., 2013; Kromm et al., 2003). The total energy dissipated by fiber breakage was measured by the area under the load-displacement curve, resulting in ≈ 8.1 mJ/m of elastic energy stored per unit fiber length in Dyneema Polyethylene fibers at failure, which was comparable with previous reported value of ≈ 66 kJ/kg in Karthikeyan et al. (2013).

Figure 3.6: (a) Tensile stress-strain curve of Dyneema fibers at a nominal strain rate of $10^{-4} s^{-1}$. (b) Representative pull-out force-displacement curves of single fiber bundles extracted from the nonwoven fabric.

3.2.2 Pull-out tests

Single and multiple fiber bundle pull-out tests were carried out using the experimental set-ups described in previous Section 2.2.2 to determine the contribution of each deformation mechanisms on the mechanical response of the material and quantify micromechanical parameters such as stress to initiate sliding and the energy dissipated by frictional processes.

Single fiber bundle pull-out test

23 pull-out tests of single fiber bundles were carried out. They showed three distinct responses, denominated anchored, slippage and untangled. Representative force-displacement curves of each one are plotted in Fig. 3.6(b). The initial response was equivalent in all cases and corresponded to fiber uncurling. The sliding resistance at the physical contacts with other fibers is overcome at very low loads (of the order of 10 mN) in the case of untangled fibers, which are extracted from the fabric. The pull-out load decreased with the sliding distance as the length of the embedded fiber and the number of fiber-to-fiber contacts decreased and was practically zero when the displacement reached 30 mm,

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which was very close to the actual fiber length of 60 mm. This behavior was observed for approximately 40% of the single fiber bundles extracted from the fabric. On the contrary, 20% of the fibers were anchored to the fabric and presented a very stiff response, with no traces of fiber slippage. Finally, 40% of the fiber bundles presented an intermediate behavior. The load-displacement curve was initially similar to that of the anchored fibers but a sudden reduction in the load –accompanied by an increase in the fiber displacement– occurred in the range of 30 to 60 mN. The process was repeated several times during the fiber pull-out, and it was attributed to the successive fiber knot disentanglement which caused extra elongation without the application of additional load. The load was reduced to practically zero after the fiber has disentangled from all the knots.

The statistical distribution of the maximum load measured during the pull-out tests (excluding anchored fibers) is plotted in Fig. 3.7(a) for tests carried out at pull-out velocities of 0.01, 0.1 and 1.0 mm/s. The results are presented in a box plot format which shows in boxes the 50% of the results for the first Q1 and third Q3 quartile and the median value Q2 with an horizontal line. The whole variability of the test population is finally represented by vertical lines. The scatter in the maximum pull-out load was very large due to the nature of the fiber network and no big differences were found with the pull-out velocity although the central quartiles and the median increased with the pull-out velocity very likely due to the rate dependency of the friction coefficient between fibers.

The energy dissipated during fiber pull-out per unit length of fiber extracted was computed from the area under the pull-out load-displacement curve divided by the total fiber length extracted and the statistical results are plotted in Fig. 3.7(b) for tests carried out at pull-out velocities of 0.01, 0.1 and 1.0 mm/s. They showed similar trends to those reported above for the maximum pull-out load: large scatter and limited influence of the pull-out velocity on the energy dissipated. The average values of the energy dissipated by fiber sliding and pull-out in Fig. 3.7(b) increased from ≈ 9.7 mJ/m at 0.01 mm/s to ≈ 18.1 mJ/m at 1.0 mm/s and both were larger than the ≈ 8.1 mJ/m of elastic energy stored per unit length in Dyneema Polyethylene fibers until failure by fiber breakage.

The single fiber bundle pull-out tests provided very valuable information about the failure micromechanisms and energy dissipation in the needle-punched nonwoven. Only a minor fraction of the fibers ($\approx 20\%$) were actually anchored in the fabric network and were able to carry the maximum load dictated by the strength of the polyethylene fibers.

Figure 3.7: (a) Maximum pull-out load (excluding anchored fibers) during single fiber bundle tests as a function of the pull-out velocity. (b) Energy dissipated during pull-out tests per unit length of fiber extracted.

As a result, fiber breakage was not the principal energy dissipation mechanism during deformation and most of the energy was dissipated by fiber sliding and pull-out from the fabric. Moreover, a significant fraction of fibers ($\approx 40\%$) was not connected to the fiber network and did not contribute to the strength. Thus, these results indicated that loading paths were created within the network during deformation by fiber bundles that were effectively connected to each other. Additional straining led to fiber breakage or fiber sliding in the bundles within these loading paths.

Nevertheless, the micromechanical tests presented above were not enough to shed light on the dependency of the failure mechanisms with respect to the material direction. Of course, the experimental scatter in the single fiber bundle pull-out tests may mask valuable information regarding the effect of fiber orientation on the sliding process but fiber orientation cannot be tracked in the pull-out test because the fiber is fully embedded within the fabric and its actual orientation is not known.

3.2 Micromechanical characterization

Figure 3.8: Pull-out test carried out in a double-edge notch specimen loaded in tension along MD. The arrow shows the needle-punch knot extracted during the test.

Multiple fiber bundle pull-out tests

Multiple fiber bundle pull-out tests with double-edge notched specimens were carried out to overcome the previous limitation. In this case, the introduction of a very deep notch in the specimen led to an orientation dependency as most of the active fibers that carried load through the central ligament should be aligned at the loading direction, Fig. 2.14(b).

Representative force *vs.* pull-out length curves of the multiple fiber bundle tests are plotted in Figs. 3.9(a) and (b) for specimens loaded along TD and MD, respectively, at a pull-out velocity of 1.0 mm/s. A representative curve of a single fiber pull-out test is also plotted for comparison. The curves corresponding to single fiber and multiple fiber pull-out tests were qualitatively similar: an initial elastic region followed by a serrated shape caused by the progressive fiber knot disentanglement. Nevertheless, the initial linear region was stiffer in the case of the multiple fiber pull-out tests because it accounted for two different mechanisms: uncurling of the multiple fiber bundle and bulk fabric deformation. The second effect was negligible for single fiber tests but became more important with the number of fibers in the bundle. In addition, the initial stiffness along TD was 98 ± 24 mN/mm, approximately twice higher than that along MD, which only attained 48 ± 18

Figure 3.9: Force *vs.* pull-out length curves of multiple fiber bundle tests carried out at a pull-out velocity of 1.0 mm/s. (a) Loading along TD. (b) Loading along MD.

mN/mm. This stiffness was independent on the number of fibers involved in the test (in the range 10 to 45), and this suggested that the main contribution to the stiffness was related to the bulk deformation of the fabric at the microscopic level, which was different along TD and MD orientations. It is worth noting that the needlepunch knots were extracted from the fabric in several occasions during the pull-out tests along MD (Fig. 3.8) but this never happened along TD, in which fiber extraction took place without displacement of the knots. These differences indicated that the fiber pull-out mechanisms at the knots depended on the orientation due to the particular structure of the fiber entanglement created by the barbed needles.

The serrated path of the load-displacement curves of the multiple fiber bundle pull-out tests was due to progressive fiber disentanglement during pull-out. The maximum load attained during the tests as well as the dissipated energy per unit length of extracted fiber (computed from the area under the force *vs.* pull-out length curve divided by the final pull-out length) are plotted in Figs. 3.10(a) and (b), respectively. The results for single fiber pull-out tests are also plotted for comparison. It is worth nothing that the sliding resistance as well as the dissipated energy per unit fiber length decreased with the number of fibers and this behavior can be attributed to two different causes, namely the stochastic nature of the disentanglement process and the lack of an efficient load transfer mechanism as the

3.3 Concluding remarks

Figure 3.10: (a) Sliding resistance of multiple fiber bundles per number of fibers. (b) Energy dissipated during multiple fiber bundle pull-out tests per unit length of pull-out fiber.

number of fibers in the network increased. The first mechanism was associated with the higher probability of finding weakest paths in the network for fiber sliding as the number of fibers increase. The second one was associated with the lack of load transfer efficiency for networks of increasing complexity. Therefore, the higher the bundle interconnections, the lesser the efficiency for load transfer during the multiple fiber pull-out tests. Neither the sliding load nor the dissipated energy presented a strong dependence with the loading direction either MD or TD.

3.3 Concluding remarks

The microstructure at the as-received condition of the needle-punched nonwoven fabric was determined and main deformation and failure mechanisms were characterized. Overall structure of the nonwoven was composed by a fiber network constituted by bundles generally made up by two Dyneema fibers with very large aspect ratio. Fibers were connected by mechanical entanglements in discrete knots with fibers oriented at the through-the-thickness direction. Knots were randomly distributed all over the planar area. Fiber curvature was very heterogeneous with a very strong variance, from totally straight fibers

to curly fibers forming marked loops. Fibers were randomly oriented within the plane of the batt. Same results were obtained by both, XCT and 2D WAXD for the fiber ODF.

Regarding the deformation and failure mechanisms, fiber breakage was characterized by fiber tensile testing, while the load-displacement curves for the extraction of fibers were determined by a series of pull-out tests. The used of these tests in nonwoven fabrics is a novel contribution of the doctoral thesis.

Single fiber bundle pull-out tests were useful to quantified the percentage of fibers undergoing different responses, namely anchored, untangled and slippage. As it was mentioned previously, only a minor portion of the fibers ($\approx 20\%$) underwent the required strain to failure, presenting the anchored response. A significant percentage of the fibers ($\approx 40\%$) were untangled to the network skeleton, posing negligible resistance to fiber extraction. Finally, the most representative percentage of fibers ($\approx 40\%$), presented a slippage response and slided with respect to each others, being fiber disentanglement the main failure mechanisms found for this certain material. The comparison between the energy dissipated by pull-out and fiber breakage revealed that the frictional processes might dissipate at least twice the energy of the elastic energy stored in the fiber.

Multiple fiber pull-out tests showed no significant effect of fiber orientation, MD or TD, on the sliding resistance as well as on the dissipated energy by fiber disentanglement. However, there was a difference in the micromechanical stiffness of the bulk material when straining bundles of fibers at a certain direction, being the bulk stiffness along TD double than along MD even if the initial fiber ODF was isotropic. Furthermore, during the extraction of fibers along MD, in certain cases some of the tests ended with the extraction of the needlepoint demonstrating the differences in the interconnection among the fibers due to the generation of weaker points. Thus the fiber interconnection in the transverse direction TD seems to be more efficient than the machine direction MD.

Single and multifiber bundle pull-out tests also indicated that the sliding resistance as well as the dissipated energy per fiber length was strongly affected by the number of fibers tested. During single pull-out tests, the stress was transferred directly to the extracted fiber and the dissipated energy was the maximum for each of the fiber configurations within the network. However, when loading a controlled bundle of fibers, the stress was not efficiently transferred within the bundle, which possessed a specific network configuration by itself.

3.3 Concluding remarks

As a result, the load transfer efficiency was reduced resulting in a lower sliding resistance and dissipated energy.

CHAPTER 4

In-plane mechanical properties

In-plane macromechanical characterization of the needle-punched nonwoven material is exposed in this Chapter. Quasi-static characterization along main material directions (TD, MD and 45D) and dynamic characterization along the stiffest direction (TD) were accomplished. DIC was used in both cases to obtain the strain distribution along the surface material. Dynamic characterization was carried out with a novel Split-Hopkinson Tensile Bar (SHTB) apparatus specially designed for soft materials.

Microstructure evolution was ascertained by means of X-Ray Computed Tomography and 2D Wide Angle X-Ray Diffraction. The comparison of as-received and pre-deformed specimens shed light into the deformation micromechanisms inherent of needlepunched nonwovens. Fiber orientation distribution functions for each deformation state were easily obtained by WAXD. Failure micromechanisms individually identified by the previous micromechanical characterization (Chapter 3) were found during the macromechanical testing.

By these experiments the physics involved in the deformation of the material at quasi-static and high strain rates were determined. The ductile response and high energy absorption capacity of the material were justified by the complex interaction between micromechanisms. The material parameters obtained during the experimental campaign were used for the development of a multiscale material constitutive model in Chapter 6.

4.1 Quasi-static properties

Strain rate (s^{-1})	Orientation	$S_{max}(\text{kN/m})$	ϵ_{max}	Toughness (kN/m)
0.01	MD	12.9 ± 0.8	1.22 ± 0.07	8.7 ± 1.8
0.01	TD	37.1 ± 4.4	0.59 ± 0.04	16.6 ± 2.6
0.01	45°D	21.8 ± 0.5	0.84 ± 0.01	15.7 ± 4.6
0.08	MD	14.5 ± 0.7	1.25 ± 0.07	9.9 ± 1.5
0.08	TD	35.7 ± 0.7	0.55 ± 0.01	16.1 ± 2.8

Table 4.1: In-plane tensile mechanical properties of the nonwoven fabric as a function of the orientation and strain rate.

4.1 Quasi-static properties

Plain square specimens were subjected to initial uniaxial tension under stroke control along MD, TD and 45°D at engineering strain rates of 0.01 and 0.08 s^{-1} following the experimental technique exposed in the Section 2.2.2. Between 2 and 4 tests were carried out for each orientation and strain rate. Representative nominal stress (force per fabric width) *vs.* engineering strain curves in the three orientations are plotted in Fig. 4.1. The average values of the nominal strength, S_{max} , the strain at maximum load, ϵ_{max} and the toughness (area under the nominal stress-strain curve) are depicted in Table 4.1, together with the standard error, as a function of the loading orientation and strain rate.

The shape of the stress-strain curves was analogous but the values of the nominal strength and of the strain at the maximum load differed among loading orientations. The nonwoven fabric was stiff and strong along TD and compliant and weak along MD, while the response along 45°D was in between them. The initial part of the stress-strain curve was almost linear with a moderate slope and it was controlled by the uncurling/sliding of the individual fibers within the fabric. It was followed by progressive increase in stiffness. A rigid skeleton, connecting the entanglement points through straight fibers, was formed and the fibers rotated to be aligned with the loading axis. The maximum load bearing capacity of the nonwoven fabric was attained at very large engineering strains ($\approx 50\%$ and $\approx 125\%$ for the TD and MD, respectively). This ductile behavior cannot be explained by simple affine stretching of individual fiber bundles within the fabric and fiber sliding should be considered as the main deformation mechanism at the maximum load and afterwards. After the peak, the load carried by the nonwoven fabric was progressively reduced, leading to a typical bell-shaped curve. Damage localized in a given area of the specimen in this stage

Figure 4.1: Representative nominal stress *vs.* engineering strain curves for the specimens tested in tension at strain rates of 10^{-2} s^{-1} along different directions

and large changes in the density were observed with the naked eye. They were attributed to the development of fiber pull-out mechanisms which controlled the deformation of the material in this last regime. The ability of the nonwoven architecture of sustain load at very large strains leads to an exceptional energy absorption capability which makes this material specially suited for ballistic protection against fragments. Failure strain for TD and MD orientations were characterized as 100% and 160%, respectively.

The dissipated energy was dependent on the material orientation. It was measured as the energy dissipated by one layer of the fabric per crack length. For the specimens oriented at MD and TD directions the crack length was considered the width of the sample w , meanwhile for the 45°D specimens the crack length was considered the diagonal of the sample $\sqrt{2}w$. A comparison between the total absorbed energy for all the configurations is shown in Fig. 4.2(a). Testing at TD orientation dissipated double of the energy of the sample oriented at MD, being the 45°D sample and intermediate case. A rough approximation of the energy dissipated per fiber was also obtained. After strain localization, pull-out took place monotonically in a given section of the specimen until the total separation of both parts of the specimen was done. Those fibers clearly pulled-out in that section, were

4.1 Quasi-static properties

Figure 4.2: (a) Absorbed energy by crack length *vs.* material orientation. (b) Lateral contraction *vs.* longitudinal deformation for specimens oriented at TD and MD.

carefully cut with scissors and weighted with a scale. Then, the energy dissipated per fiber length was obtained from the area below the load-displacement curve after the peak and the total length of the extracted fibers. The results yield $\approx 0.1 \text{ mJ/m}$ of dissipated energy for both orientations TD and MD, a value well below the energy ranges obtained with the single and multiple fiber pull-out tests, but in agreement with the aforementioned reduction of efficiency of load transmission, see Chapter 3.

The poisson coefficient of the nonwoven was independent on the stretching direction as the ratio between deformation for two perpendicular directions was similar, Fig. 4.2(b). However there was a different initial slope for the specimens stretched at MD causing a delay of the lateral contraction. Once the damage started, the perpendicular deformation reached a plateau at 50% of contraction for TD and at 70% of contraction for MD due to the higher strain to failure.

Tests with periodic unloading-reloading cycles at TD and MD directions were also carried out to ascertain the contribution of elastic and inelastic strains to the total deformation, see Fig. 4.3(a) and (b) respectively. They indicate that most of the deformation was irrecoverable even at very low strains and the mechanical behavior of the nonwoven fabric was pseudo-plastic. Moreover, unloading did not modify the network structure nor contribute to damage.

Figure 4.3: Nominal stress *vs.* engineering strain behavior under monotonic and cyclic deformation. (a) Along TD and (b) Along MD.

The full-field of the logarithmic strain along the loading axis obtained by digital image correlation is shown in Fig. 4.4 for the specimens tested at 0.08 s^{-1} along TD, MD and 45°D for different values of the applied strain. All specimens adopted a hourglass shape as a result of the constraint in the grips and the large finite deformations and this effect was more marked in MD and 45°D because of the larger failure strain. Although strong strain gradients developed near the grips, the strain was fairly homogeneous in the central region of the specimens tested along TD and MD before the maximum load, Figs. 4.4(a) and (d). Damage localization and failure was triggered afterwards in between the grips and the center of the specimen in these samples, Figs. 4.4(c) and (f). In the specimen tested along 45°D , most of the load was carried along TD and the strain was localized in this direction before the maximum load, Fig. 4.4(g) and (h). This behavior was dictated by the strong anisotropy between TD and MD and final fracture occurred near the grips in this case.

The pull-out length was carefully measured for tests along TD and MD orientations by DIC, see Fig. 4.5(a) and (c) respectively, and by post-mortem fractographies, Fig. 4.5(b) and (d). Fiber pull-out length varied from 45 to 20 mm, which was higher than half of the total fiber length. Considering that the fiber pull-out started at the onset of damage for in-plane testing, the mean pull-out length could be determined from the force-displacement curves of the tensile tests (36.93 mm for TD and 34.78 mm for MD).

4.1 Quasi-static properties

Figure 4.4: Contour plots of the logarithmic strain in the loading direction at different applied strains of three specimens deformed along different orientations. (a) MD, 80%. (b) MD, 100%. (c) MD, 150%. The lateral contraction of $\approx 70\%$ is remarkable. (d) TD, 55%. (e) TD, 70%. (f) TD, 85%. (g) 45°D, 30%. (h) 45°D, 55%. (i) 45°D, 110%.

Figure 4.5: Specimens deformed in tension just before fracture. The contour plots of the deformation along the loading axis (obtained by means of digital image correlation) could not be obtained in the central region, in which the extensive pull-out destroyed the pattern used to correlate the displacements. The approximate length of the pulled out region is marked in both orientations. (a) and (b) Engineering strain of 140% along TD just before and after fracture. (c) and (d) Engineering strain of 170% along MD just before and after fracture.

4.2 Microstructure evolution

Microstructure evolution during deformation was analyzed for in-plane loads at TD and MD orientations. Tomographies of thin slices ($\approx 50 - 100 \mu\text{m}$ in thickness) parallel to the fabric plane are shown in Figs. 4.6(a) and (b) after deformation of 40% along MD and TD, respectively. Inspection was carried out at unloading state due to the pseudo-plastic response of the material. The qualitative analysis of these tomographies clearly indicates that deformation along TD was accompanied by fiber straightening and orientation in the loading direction, Fig. 4.6(b). On the contrary, fiber re-orientation along the loading axis was limited after 40% of strain along MD, Fig. 4.6(a).

The comparison of the cumulative density functions (CDFs) between the as-received and the deformed conditions is shown in Fig. 4.7. The methodology for the data treatment was exposed in previous Chapter 3. CDFs of the orientation angle, β , are shown

4.2 Microstructure evolution

Figure 4.6: X-ray tomographs thin slices parallel to the nonwoven fabric plane. (a) After 40% deformation along MD. (b) After 40% deformation along TD.

in Fig. 4.7(a). During deformation, the fibers were rapidly aligned parallel to the loading axis when the fabric was deformed along TD, while fiber re-orientation was very limited upon deformation along MD and only a minor fraction of them were able to rotate and carry load. The CDFs corresponding to the curvature angle, θ , are plotted in Fig. 4.7(b) for the same conditions and show similar trends. Fiber uncurling and straightening (which is characterized by a reduction in θ) developed more rapidly when the fabric was strained along TD in comparison with MD.

These results clearly indicated that the differences in fiber uncurling and re-orientation during deformation were closely related to the anisotropy in the mechanical properties between TD and MD. The fibers tended to align with respect to the loading axis when the fabric was deformed in the TD direction and this re-orientation, together with the straightening of the fibers, was directly responsible of the higher stiffness and strength. In contrast to this behavior, fiber orientation was mildly influenced by the macroscopic deformation along MD and only a minor fraction changed orientation and was able to support more loads. Moreover, fiber straightening was also limited. These latter observations seem to suggest that anisotropic response of the nonwoven fabric can be attributed to the lack of efficient mechanisms to transfer the load to the whole fiber network along MD. Thus,

Figure 4.7: (a) Cumulative density function for the fiber orientation angle, β , in the as-received condition and after 40% strain along MD and TD. (b) Cumulative density function for the fiber curvature angle, θ , for the as-received condition and after 40% strain along MD and TD.

the mechanisms of fiber sliding and pull-out around the needle-punch points in different orientations seem to be critical to understand the mechanical behavior of the nonwoven fabric.

The evolution of the microstructure with the applied deformation was also analyzed by means of two dimensional Wide Angle X-Ray Diffraction (WAXD). Fig. 4.8 shows the evolution of the WAXD patterns with the deformation. Fig. 4.8(a) shows the diffraction pattern of a straight polyethylene fiber bundle and Fig. 4.8(b) shows the diffraction pattern of the as-received conditions. The diffraction patterns of the samples deformed up to 40% strain along TD and MD are depicted in Figs. 4.8(c) and (d), respectively. The intensity of the circular rings after deformation along MD was fairly constant, indicating that the fiber orientation distribution remained very isotropic. On the contrary, the anisotropy in the fiber orientation due to stretching along TD was clearly observed in the corresponding diffraction pattern.

These findings are in perfect agreement with the observations carried out by XCT presented in Fig. 4.9. XCT data received the same treatment as in previous Chapter 3. The intensity of the diffraction pattern obtained from XCT data of the fiber orientation and fiber curvature angles in the interval $[0, \pi/2]$ has been compared with the intensities obtained by WAXD in Figs. 4.9(a) and (b) for the fabric deformed up to 40% along TD

Figure 4.8: Two-dimensional WAXD patterns of (a) Dyneema fiber bundle. (b) Nonwoven fabric in the as-received condition. (c) Nonwoven fabric deformed 40% along TD. (d) Nonwoven fabric deformed 40% along MD.

Figure 4.9: Orientation distribution functions of fiber bundles obtained from XCT and WAXD. (a) Specimens strained up to 40% along TD. (b) Specimens strained up to 40% along MD. The horizontal line of intensity $1/2\pi$ corresponds to the isotropic distribution.

and MD, respectively. Both results are in good agreement and demonstrate the ability of these techniques to track the microstructural changes (in terms of fiber uncurling and fiber re-orientation) during deformation of nonwoven fabrics.

After the validation of the WAXD, the microstructure evolution at different stages of the deformation was analyzed up to the maximum peak load (125% of deformation along MD and 50% of deformation along TD). The results along MD are plotted in Fig. 4.10. The corresponding ODFs are shown in a polar representation. The fibers were progressive oriented with the applied strain along the loading direction (0 and π) and reached a maximum of alignment for an applied strain of 100%. Further straining up to 120% led to a reduction in anisotropy of the fiber ODF, which also became asymmetric because of the onset of damage.

To compare the maximum ratio of fiber alignment of each direction (MD and TD) a WAXD inspection before the onset of damage was carried out. The diffraction patterns for MD and TD are depicted in Figs. 4.11(b) and (c), respectively and the corresponding ODFs are compared in Fig. 4.11(a) in polar plots. As it was seen before, fibers were aligned at the loading direction for each case. Furthermore, the degree of orientation was very similar in both cases although the sample was stretched up to 40% along TD and up to 100% along MD. In terms of microstructure evolution, same alignment level is reached in both cases, but with a strong delay when stretching along MD.

Figure 4.10: Polar representation of the fiber ODF as a function of the applied strain along MD.

Figure 4.11: Comparison between maximum fiber alignment for each direction. (a) Polar representation of the fiber ODF after 100% deformation along MD and 40% deformation along TD. (b) 2D WAXD pattern of a sample deformed 100% along MD. (c) 2D WAXD pattern of a sample deformed 40% along TD.

4.3 Dynamic properties

4.3.1 Dynamic load

Dynamic testing at high strain rates was carried out at the Split-Hopkinson Tensile Bar (SHTB) apparatus exposed in Appendix A following the experimental procedure described in Section 2.2.2. The wave propagation in the bars is analyzed in Fig. 4.12. The stress pulse generated had an approximate trapezoidal shape with a duration of 1 ms, and the hollow output tube was sensitive enough to register the transmitted wave. Negligible noise in the signal was found, see Fig. 4.12(a). A final peak was observed in the input signal as a consequence of the mismatch impedance at the input bar/specimen interface, which caused the overlapping of the incident and reflected waves, see Fig. 4.12(b). The strain rate reached was almost constant for more than 0.5 ms, in the range of $300 - 400s^{-1}$, 3 orders of magnitude higher than for quasi-static tests.

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Figure 4.12: Wave propagation in the SHTB system. (a) Voltage *vs.* time registered for the 3 strain gauges. The square shaped stress pulse of 1 ms of duration is appreciated. (b) Wave reflections in time at the gauges of the bar.

Figure 4.13: Analysis of the dynamic force equilibrium. Forces measured in the input and output bars. Dynamic equilibrium was not achieved during the experiment.

Even though the pulse duration was 5 times longer than conventional pulses for SHTB, the dynamic force equilibrium was not achieved. The time evolution of the forces measured in the input and output bars is depicted in Fig. 4.13. Although the duration of the pulse was about 1 ms, the time overlapping of the forces was of less than 0.5 ms. By the time the transmitted wave arrived to the output bar, the force in the input bar was almost zero. This result was unexpected. In previous studies (Chocron et al., 2002) the initial propagation wave velocity was $c = 600$ m/s, so the travelling time along the sample was less than 0.07 ms for an initial gauge length of $L_0 = 40$ mm. Due to this fact, a time pulse of 1 ms is supposed to be considered enough to reach the dynamic force equilibrium after several back and forth reflections.

4.3.2 DIC validation

The deformation mechanisms were analyzed by Digital Image Correlation (DIC) in order to explain the lack of force equilibrium during the experiment. Input and output velocities were correlated with the data registered by the strain gauges to prove the reliability of the DIC measurements. The velocity of the input bar, v , can be calculated as

$$\sigma = v c \rho \quad (4.1)$$

4.3 Dynamic properties

Figure 4.14: Validation of the DIC measurements with the experimental velocities registered by the strain gauges at the interfaces between specimen and bars. (a) Velocities at the input and output interfaces registered by the gauges. DIC pictures correspond to the measurements at (b) $t = 3.00$ ms, (c) $t = 3.40$ ms, (d) $t = 3.60$ ms and (e) $t = 4.00$ ms.

where $\sigma = 0.11$ GPa is the input pressure, $\rho = 2800 \text{ kg/m}^3$ is the aluminium density and $c = 5070$ m/s is the propagation velocity. The resulting bar velocity was approximately $v = 7.75$ m/s. Due to the strong impedance mismatch between the aluminium bar and the specimen, nearly all the incident traction pulse was reflected at the output bar/specimen interface and the particle velocity was slightly lower than $2v$ (< 15.5 m/s). This result was in good agreement with the strain gauges measurements, see Fig. 4.14(a), where an approximately constant input velocity of 14.5 m/s was registered. The output velocity was almost zero for the crossing of the first pulse. All these values agreed with the results obtained from the DIC images. The evolution of the velocity gradient is shown in Figs. 4.14(b), (c), (d) and (e). A strong gradient (from 14 to 0 m/s) appeared on the specimen during the first

microseconds, see Fig. 4.14(b) and after that the gradient decreased rapidly with time. The stress pulse crossed the sample and the final velocity along the specimen became close to zero at 4 ms, see Fig. 4.14(e).

4.3.3 Experimental results

The analysis of the full-field displacements provided the most important hints of the dynamic deformation mechanisms of the nonwoven. The evolution of the longitudinal engineering strains is shown in Fig. 4.15. Time $t = 0$ indicated the instant just before the loading pulse arrived at the incident bar/specimen interface. The incident bar end moved and pulled the nonwoven from the right side. Only the portion of specimen in contact with the incident bar was stretched at the very beginning of the loading ($t = 200 \mu s$) as indicated in Fig. 4.15(a). The deformation propagated towards the other side of the specimen during the test, but after a certain instant ($t = 600 \mu s$) the strain at the left edge got frozen and could only evolved at the right side of the specimen, see Fig. 4.15(d). The heterogeneous strain field was the responsible of the lack of force equilibrium during the experiment.

The strong longitudinal strain gradient was due to the variation in stiffness (and, thus, variation in the wave propagation speed) with the applied strain. For instance, Chocron et al. (2002) reported an approximately linear increase in the wave propagation velocity in these materials from 600 m/s in as-received condition up to 4000 m/s after 25% of deformation. The wave rebounds are analyzed following the scheme shown in Fig. 4.16. The wave generated at the first instant of the test with a propagation velocity c_0 , see Fig. 4.16(a), travels along the sample from the input interface to the opposite side (output bar/specimen interface), and it is reflected with a velocity $2c_0$ due to the strong mismatch impedance between the nonwoven fabric and the output bar. At the same time, faster waves appear at the input interface as a result of the increment of stiffness of the material with the deformation ($c_1 > 2c_0$), see Fig. 4.16(b). Once the two waves (the reflected and the transmitted) travelling at different velocities arrive at the same material point, they create a wave front with an impedance mismatch due to the variation of stiffness with the strain, avoiding the propagation of higher strain waves into the left side of the material, Fig. 4.16(c). The wave interference is repeated all over the simulation with waves of higher velocity ($c_2 > 2c_1$). The result of this iterative process is a strong strain gradient as shown in

Figure 4.15: Evolution of the longitudinal engineering strain E_{xx} during SHTB test, determined by a Lagrangian formulation. (a) $200\mu s$. (b) $400\mu s$. (c) $600\mu s$. (d) $900\mu s$. Higher strains are registered in the side of the specimen closer to the input bar.

Fig. 4.15(d), with a heterogeneous strain evolution over the specimen. Similar phenomena have been observed in metallic foams (Lopatnikov et al., 2003; Elnasri et al., 2007) or epoxy (Song et al., 2006), where a shock front is created and a partial permanent deformation appears depending on the initial impact velocity. This propagation demonstrated the existence of a non-uniform deformation region in the specimen for high strain rates, while a homogeneous plane strain state was originated before the onset of damage in quasi-static tests, see Chapter 4.

Figure 4.16: Wave rebound in the specimen during dynamic testing. $c_0 < c_1 < c_2$. (a) Initial wave propagation at velocity c_0 . (b) Reflection of the first wave arriving at the output bar with a velocity $2c_0$, lower than c_1 . (c) Interference of both waves at an intermediate point of the specimen, while new waves with velocity $c_2 > 2c_1$ are created at the input bar interface.

The dynamic stress-strain curve was finally built up combining the force-displacement data registered by the gauges, and the relation between bar displacement and maximum strain measured by DIC, taking into account the slippage of the nonwoven from the grips. DIC only registered the first crossing pulse, so the dynamic stress-strain curve was only characterized up to 40% of deformation. This treatment results in a series of discrete points following the recording frequency of the high speed camera. Dynamic results are compared with the quasi-static curve in Fig 4.17. The nonwoven presented a strain-rate independent mechanical behaviour with a similar non-linear pseudo-elastic response in the range from $\dot{\epsilon} = 0.01\text{--}400 \text{ s}^{-1}$ up to 40% of strain. The dynamic strength could not be determined due to the limitations of the experimental set-up. Surprisingly, there was not a difference between frictional processes associated with quasi-static or dynamic tests.

4.4 Concluding remarks

Figure 4.17: Comparison between the stress-strain curve for tensile test along TD orientation under quasi-static and dynamic conditions at strain rate $\dot{\epsilon} = 400 \text{ s}^{-1}$ up to 40% of deformation.

4.4 Concluding remarks

Experimental characterization of the in-plane response of the needle-punched nonwoven fabric for quasi-static and dynamic regime was accomplished. The macroscopic quasi-static tests showed that the nonwoven fabric presented a considerable strength and an outstanding energy absorption capacity and that the mechanical behaviour along TD and MD was highly anisotropic: the stiffness, strength (by a factor of 3), and energy dissipated (by a factor of 2) along TD were much higher than those along MD. The anisotropy during deformation has to be attributed to the features of the fiber entanglements introduced in the network during needle-punch consolidation. The rough measurement of the energy dissipated by length of extracted fiber also agreed with the conclusions obtained in Chapter 3, where it was proven that the load transfer efficiency was reduced by increasing the number of extracted fibers, resulting in a lower sliding and dissipated energy per individual fibers.

The evolution of the microstructure was ascertained by means of XCT and WAXD. Fibers were initially curved and randomly distributed within the plane of the batt, see Chapter 3. As a result of needle-punching, mechanical entanglements were established in

a fraction of the fibers well above the mechanical percolation threshold and the applied load was transferred to the fabric through a set of fibers linked to the entanglement points, which form an active skeleton. The stiffness along TD was much higher than along MD and the analysis of the fiber orientation with deformation showed that the active skeleton included many more fibers in the former, which have rotated towards the loading axis as a result of the load transfer. Uncurling and stretching of the active fiber was followed by fiber sliding and pull-out from the entanglement points. The strength and energy dissipation was provided by fibers which were progressively extracted from the entanglement points and final fracture occurred by the total disentanglement of the fiber network in a given section at which the macroscopic deformation was localized. The higher strength and energy dissipation along TD has to be mainly attributed to the higher fraction of fibers contributing to the deformation in this orientation, as compared with MD. This result –origin of the anisotropic mechanical behavior of the nonwoven fabric– was caused by the features on the entanglement points: the fiber interconnection along TD seems to be more efficient than along MD, leading to a contribution of a larger fraction of fibers during deformation along TD. In terms of affinity, TD-orientated fibers basically displayed affine deformation –i.e. most of the macroscopic strain was transferred to the fibers by the surrounding nonwoven–, while MD-oriented fibers underwent non-affine deformation, and most of the macroscopic strain was not transferred to the fibers.

Strain rate independence was found in the range $\dot{\epsilon} = 0.01 - 400s^{-1}$ up to 40% of deformation. Dynamic testing was carried out in a novel Split-Hopkinson Tensile Bar apparatus specially designed for testing soft materials. Dynamic strength of the material was not determined due to the limitations of the experimental set-up. The only difference between quasi-static and dynamic regime was the strain gradient in the surface of the sample. While homogeneous deformations were observed until the onset of damage in quasi-static tests, strong strain gradients appeared in dynamic testing due to the non-linear nature of the stress-strain curve, finding higher deformations close to the input bar.

CHAPTER 5

Ballistic performance

The goal of this chapter is the ballistic characterization of the needle-punched nonwoven fabric and the analysis of the influence of the fiber orientation on the ballistic performance of the material. To this end, ballistic tests were carried out in as-received and pre-deformed specimens along the machine (MD) or the transverse directions (TD). The fiber ODF was obtained by means of WAXD at different points to assure a homogeneous microstructure all over the surface of the specimen. The influence of the fiber orientation distribution on the ballistic performance was measured in terms of ballistic limit and absorbed energy. Failure mechanisms under impact were analyzed and compared with the micromechanisms found for in-plane tensile tests.

These experiments were subsequently used to prove the reliability of the constitutive model implemented in Chapter 6 by the correlation between impact and finite element simulations exposed in Chapter 7.

5.1 Ballistic characterization

5.1.1 Pre-deformed specimens

Specimens of 500 mm in width (with 300 mm between grips) and different heights were deformed up a final length, l_f , of 500 mm following the procedure describe in Section 2.2.2.

5.1 Ballistic characterization

Figure 5.1: Nominal stress *vs.* engineering strain curves of the specimens pre-deformed along TD and MD. The solid circles in each curve indicate the pre-deformation level of each sample.

The initial length of the sample, l_0 , was selected according to $l_0 = l_f/(1 + \epsilon)$ so after stretching, the sample was deformed up to an engineering strain ϵ of 10%, 20%, 40%, 60%, 80%, 100% or 120% along MD and up to 10%, 20% or 40% along TD. The corresponding nominal stress (force per unit width) *vs.* engineering strain curves for both orientations are plotted in Fig. 5.1 and they show the strong anisotropy in the mechanical response of the nonwoven fabric. In all cases, the fabric was unloaded once the required stretch level has been reached during the test, and thus before the onset of damage localization in a given region of the fabric appeared except in the case of samples with 120% of strain along MD where cracks already started to propagate. The pseudo-plastic deformation of the nonwoven even at very low strains inhibit the recovery of the strain given during tensile testing, see Chapter 4.

The shape of the specimens (in which the width was much longer than the initial height) and the smaller width of the grips (as compared with the specimen width) reduced the transversal contraction of the needle-punched fabric during tensile deformation, see Fig. 5.2. As a result, the central region of the sample was subjected to a homogeneous strain along the loading direction and was not influenced by the lateral contraction. Thus, the microstructure evolution as a result of stretching could be considered homogeneous in this central region, leading to a constant reduction of the areal density as well. Under this hypothesis, and taking into account that the deformation of the nonwoven fabric is pseudo-

Figure 5.2: (a) Specimen tested up to 40% engineering strain along TD. (b) Schematic geometry of the specimen before and after 40% deformation along TD.

plastic (Chapter 4), the areal density, ρ , in the central region of the deformed specimens can be computed as

$$\rho = \frac{\rho_0}{1 + \epsilon} \quad (5.1)$$

where $\rho_0 = 200 \text{ g/m}^2$ was the areal density of the as-received material.

The microstructure evolution in the central region of the stretched specimens was determined by means of the fiber ODF obtained by WAXD in order to assess whether it was homogeneous over the surface of the sample. WAXD patterns were obtained at four different positions of the specimen deformed up to 40% along MD and TD, which are indicated in Fig. 5.3(a). The corresponding fiber ODFs are plotted in Fig. 5.3(b) for TD and Fig. 5.3(c) for MD together with that of an isotropic material. Fiber re-orientation in the case of the sample stretched along MD was moderate, so quasi-isotropic ODF was found along all the surface of the specimen, see Fig. 5.3(c). In the case of deformation along TD fibers were aligned in the central region of the samples deformed by 40% in the loading direction, see Fig. 5.3(b). The differences among the spots *b*, *c* and *d* were negligible, supporting the assumption that the deformation in the central region of the specimen was homogeneous, while the fiber ODF corresponding to the spot *a*, which was within the region unconstrained by the grips, was different. Thus, a central region of $350 \times 350 \text{ mm}^2$ presented a homogeneous microstructure with the same areal density and fiber ODF.

5.1 Ballistic characterization

Figure 5.3: (a) Position of the spots scanned by WAXD in the sample deformed up to 40% along TD. Fiber orientation distribution function of the spots indicated in (a) for (b) deformation along TD and (c) deformation along MD. The horizontal line stands for the theoretical fiber ODF of an isotropic material, which is very close to that of the as-received fabric.

Figure 5.4: Deformation of the as-received nonwoven fabric during impact at 305 m/s. (a) $t = 200 \mu\text{s}$, (b) $t = 900 \mu\text{s}$ and (c) $t = 2150 \mu\text{s}$. The projectile is stopped by the fabric.

Figure 5.5: Deformation of the as-received nonwoven fabric during impact at 360 m/s. (a) $t = 50 \mu\text{s}$, (b) $t = 100 \mu\text{s}$ and (c) $t = 175 \mu\text{s}$. The projectile penetrates the fabric.

5.1.2 Deformation and failure mechanisms under impact

Ballistic impacts were carried out in as-received and pre-deformed specimens. The deformation of the as-received nonwoven fabric during impact at 305 m/s (below the ballistic limit) and at 360 m/s (above the ballistic limit) is shown in Figs. 5.4 and 5.5, respectively. Energy dissipation during impact was due to the transfer of momentum from the projectile to the fabric and to the strain energy dissipated by the fabric due to the in-plane deformation. The in-plane longitudinal wave traveling through the nonwoven made the fibers move radially towards the impact point, leading to the formation of a conical region of strained material. The deformation in this region was accommodated by the same mechanisms observed during in-plane tensile deformation at quasi-static and dynamic regime, which included uncurling and re-orientation of the active fibers followed by fiber sliding and pull-out from the entanglement points, leading to a permanent global deflection of the

5.1 Ballistic characterization

target until the projectile was stopped (Fig. 5.5), causing a similar strain gradient to the one found for dynamic tensile tests, see Chapter 4.

In contrast with the ballistic performance of woven fabrics (Chocron et al., 2010), the nonwoven counterparts showed a much larger out-of-plane deformation, which comes as a result of the lower stiffness and higher strain to failure. The base of the cone was not circular but elliptical due to the different stiffness of the nonwoven fabric along TD and MD. It should be noted that the stiffness of the material (and, thus, the wave propagation speed) varied with the applied strain and the orientation. For instance, Chocron et al. (2002) reported an approximately linear increase in the wave propagation velocity in these materials from 600 m/s of the as-received condition up to 4000 m/s after 25% of stretching.

The energy absorbed before fabric piercing was directly proportional to the progress of the transverse strain wave, or in other words, proportional to the square root of the strained area deflected by the transverse wave. In Fig. 5.6 it is shown the discrete results of absorbed energy *vs* the deflected area adjusted by a square root fitting. The deflected area was approximated to the area of the elliptical cone,

$$S = 2a\sqrt{b^2 + h^2}E\left(\sqrt{\frac{1 - \frac{b^2}{a^2}}{1 + \frac{b^2}{h^2}}}\right) \quad (5.2)$$

where a is the semimajor axis, b is the semiminor axis, h is the height of the cone and $E(m)$ is a complete elliptic integral of the second kind (Abramowitz and Stegun, 1972).

$$E(m) = \int_0^{\frac{\pi}{2}} \sqrt{1 - m^2 \sin^2 \theta} d\theta \quad (5.3)$$

In the tests carried out above the ballistic limit, the final penetration of the target was accomplished by tearing as the fibers were pulled out from the entanglement points or broken near to the impact point, see Fig. 5.5. It should be noted that fiber fracture was not found to be a dominant fracture mechanism during in-plane tensile deformation (Chapter 3). Thermal softening of the Dyneema fibers, due to adiabatic heating, may be responsible of fiber fracture during impact. To check this hypothesis, as-received fibers (carefully extracted from the nonwoven fabric with tweezers) and fibers attached to the projectile after the impact were analyzed by means of differential scanning calorimetry,

Figure 5.6: Absorbed energy *vs* deformed area for all penetrating impacts. Absorbed energy was directly proportional to the square root of the deformed area.

described in Section 2.2.1. In the case of the fibers attached to the projectile, the whole fiber was analyzed (average length ≈ 60 mm) although only a small part of the fiber was in contact with the projectile of 5.5 mm in diameter.

The thermograms of the as-received and impacted fibers are shown in Fig. 5.7. The melting enthalpy of the as-received and impacted fibers were 293 J/g and 204 J/g, respectively. Taking into account that the higher melting enthalpy, the higher crystallinity, these results point to a local melting of the fiber during impact. The ratio between the peaks at 147°C and 156°C varied significantly among impacted samples, mainly due to the heterogeneous temperature profile during impact as well as during cooling after the impact fiber. Nevertheless, the thermogram clearly indicates that impacted fibers were heated at temperatures higher than 160°C, leading to the partial melting of the fiber. It should be noted that this phenomenon was only found in fibers extracted from specimens that underwent penetration.

5.2 Ballistic performance

Figure 5.7: Differential scanning calorimetry thermograms of as-received and impacted fibers.

5.2 Ballistic performance

The ballistic performance of the as-received and pre-deformed nonwoven fabric was assessed in terms of the ballistic curves depicted in Figs. 5.8 (a), (b) and (c). The solid and broken lines in these figures correspond to the Lambert formula,

$$V_{residual} = (V_{initial}^n - V_{50}^n)^{1/n} \quad (5.4)$$

where the ballistic limit V_{50} and the exponent n were obtained for each set of experimental results by the least squares fitting. The best fit to the experimental data was obtained with $n = 5$ for the as-received and the specimens pre-deformed along TD and with $n = 3$ for the specimens pre-deformed along MD. The higher the value of n , the lower the ductility of the material. It should be noted that the gas gun did not allow to launch projectiles with speeds lower than 250 m/s. If the ballistic limit was above this speed, the experimental values were in very good agreement with V_{50} in equation (5.4). Four specimens (those pre-deformed along TD by 20% and 40% and along MD by 100% and 120%) presented a ballistic limit below 250 m/s that could not be reached experimentally. In these cases, the ballistic limit was given by V_{50} , as obtained by the fitting of equation (5.4) to the available experimental results.

Figure 5.8: Experimental results of the ballistic impact tests for the nonwoven fabric. (a) As-received nonwoven fabric and specimens pre-deformed along TD by 10%, 20% and 40%. (b) Specimens pre-deformed along MD by 10%, 20% and 40 %. (c) Specimens pre-deformed along MD by 60%, 80% 100% and 120%. The solid and broken lines stand for equation (5.4) for each set of experimental results.

5.2 Ballistic performance

Regardless of the scatter of the experimental data in Fig. 5.8, it is obvious that pre-deformation of the nonwoven fabric along TD led to a marked reduction in the ballistic performance, Fig. 5.8(a). Similar results were obtained when the nonwoven fabric was pre-deformed more than 60% along MD, Fig. 5.8(c), but pre-deformation by 10% or 20% along MD did not degrade the ballistic performance within the experimental scatter, Fig. 5.8(b). A clearer view of the effect of pre-deformation on the ballistic performance can be found in Fig. 5.9(a), in which the ballistic limit V_{50} of the as-received and pre-deformed samples is plotted as a function of the areal density, determined according to equation (5.1). Pre-deformation of the fabrics led to a reduction in the ballistic limit approximately linear with the areal density for both MD and TD. However, the reduction was higher for the specimens pre-deformed along TD. It should be noted, however, that the specific ballistic limit, V_{50}/ρ , increased from 1.7 m³/gs to 2.6 m³/gs when the fabric was stretched by 100% along MD, and decreased slightly (up to 1.7 m³/gs) after 40% deformation along TD, Fig. 5.9(b).

The improvement in the ballistic performance was not just in terms of specific ballistic limit V_{50} and furthermore a remarkable increment of the ductility was noticed. To analyze the ductility of the material, the absorbed energy *vs* the initial energy of the impact is shown in Fig 5.10. As-received nonwoven and samples with a 20% and a 40% of deformation along MD are included. The red line represents the 100% of energy absorption, the orange line corresponds to the 75% and the yellow line, the 50%. The energy absorption for the as-received material for velocities above the ballistic limit was lower than the 50% of the maximum absorbed energy in the ballistic limit V_{50} , meanwhile the samples deformed at the MD were capable to absorb a higher percentage of energy, from 75% to 50%. These results point out the improvement of the ductility of the material due to the changes in the ODF.

Figure 5.9: (a) Ballistic limit, V_{50} of the nonwoven fabric as a function of the areal density, ρ . (b) Specific ballistic limit, V_{50}/ρ and aspect ratio (a/b) of the deformed region after impact in the specimens pre-deformation along MD and TD directions.

Figure 5.10: Ballistic performance. Absorbed energy by the target *vs.* initial kinetic energy of the impact.

5.2 Ballistic performance

Figure 5.11: Deformation of the nonwoven fabric pre-deformed 60% along MD during impact at 322 m/s. (a) $t = 150 \mu\text{s}$, (b) $t = 225 \mu\text{s}$ and (c) $t = 350 \mu\text{s}$. Notice the large volume fraction of fibers pulled out from the fabric after penetration.

Figure 5.12: Deformation of the nonwoven fabric pre-deformed 20% along TD during impact at 260 m/s. (a) $t = 75 \mu\text{s}$, (b) $t = 125 \mu\text{s}$ and (c) $t = 175 \mu\text{s}$. Notice the fiber slippage around the projectile.

As indicated above, ballistic impact on the nonwoven fabric led to the formation of a cone of deformed material whose cross-section was elliptical. The ratio between the major and minor semi-axes of the ellipse, a/b , was measured for all the specimens that underwent penetration and the average value (together with the standard deviation) is plotted in Fig. 5.9(b) as a function of the pre-deformation of the fabric. a/b was ≈ 2 in the as-received fabric, increased up to ≈ 3.5 when the fabric was pre-deformed by 40% along TD and decreased up to ≈ 1.2 when the fabric was pre-deformed along MD by 80%. Further pre-deformation along MD did not modify the aspect ratio of the ellipse. These results point out again the influence of the fiber orientation and of in-plane isotropy in the mechanical properties on the ballistic performance. In this respect, it should be noted that stretching along MD from 100% to 120% modified neither the fiber orientation, (Chapter 4, Fig. 4.10), nor the shape of the elliptical zone that contributed to the deformation, nor the specific ballistic limit, Fig. 5.9(b).

Thus, fiber orientation by pre-deformation along MD and TD had different effects on the specific ballistic limit and on the energy absorbed per unit mass of the fabric and they have to be traced to change in the dominant deformation and fracture mechanisms. The deformation during impact of the nonwoven fabrics pre-deformed along MD by 60% and along TD by 20% is depicted in Figs. 5.11 and 5.12, respectively. Both cases correspond to impact tests at speeds above the ballistic limit that led to full penetration of the fabric by the projectile. The impact gave rise to the formation of a cone of strained fabric in both cases but the final fracture of the specimen pre-deformed along MD was accompanied by extensive fiber pull-out, Fig. 5.11(c), which was not found in the sample pre-deformed along TD, Fig. 5.12(c). Fiber pull-out from the fabric was identified as one the most important energy absorption mechanisms during in-plane deformation of the needle-punched nonwoven fabric and was responsible for the changes in the ballistic behavior between the specimens stretched along MD and TD.

These changes in the failure mechanisms can be traced to the anisotropic deformation of the nonwoven fabric along TD and MD, which led to different mechanical responses under impact. Pre-deformation of the fabric along TD was accompanied by the preferential orientation of the fibers along the loading direction, Chapter 3, Fig. 4.11(a) and (c). The resulting microstructure can be found in the tomograms obtained by XCT in Fig. 5.13. The as-received material presents an isotropic fiber orientation, see Fig. 5.13(a), but most of the fibers have been oriented along TD after 40% of deformation along this orientation, see Fig. 5.13(b). As a result, the mechanical anisotropy of the nonwoven fabric is maximum because very few fibers remain oriented along MD. During impact, the projectile was able to slip through the fibers because the resistance to deformation along MD was negligible, and the overall energy absorption capability decreased. On the contrary, the fiber ODF was isotropic after pre-deformation along MD by 60% because most of the macroscopic strain was not transferred to the fibers in this orientation. Further straining led to preferred fiber alignment along MD, indicating that the entanglement points became effective to transfer the load to the fibers and, thus, the in-plane mechanical behavior of the fabric was more isotropic. This statement is supported by the almost circular shape of the elliptical zone that contributed to the deformation during impact, Fig. 5.9(b). The resistance to impact was similar along MD and TD, and penetration took place by fiber pull-out from the entanglement points rather than by slippage through the fiber network.

5.3 Concluding remarks

Figure 5.13: XCT images of the needle-punched nonwoven fabric. (a) As-received conditions. (b) After 40% deformation along TD, showing the preferential fiber orientation along TD. The entanglement points are marked with black circles.

5.3 Concluding remarks

The impact behavior of the needle-punched nonwoven was analyzed after stretching the fabric along TD and MD to ascertain the effect of the fiber orientation on the ballistic properties. During impact, the energy of the projectile was accommodated by the formation of a cone of deformed material with an elliptical cross-section due to the different wave propagation speed along MD and TD. Energy dissipation during impact was due to the transfer of momentum from the projectile to the fabric and to the strain energy dissipated by the fabric due to the in-plane deformation. The deformation was accommodated by the same mechanisms observed during in-plane tensile deformation for quasi-static and dynamic regime, which included uncurling and stretching of the active fibers connected by the entanglement points followed by fiber sliding and pull-out, leading to a permanent global deflection of the target until the projectile was stopped. Stress concentration appeared in the same way as in dynamic testing, causing a strong gradient with maximum deformation at the impact point.

Additionally, the influence of the fiber alignment on the ballistic performance of the nonwoven was analyzed. The impact mechanisms in the pre-deformed specimens were similar but it was found that pre-stretching the nonwoven fabric along MD (the softest direction) up to 100% led to a continuous increase in the specific ballistic limit (minimum penetration velocity divided by the areal density) of the fabric and of the energy dissipated per unit weight. On the contrary, both the specific ballistic limit and the energy dissipated per unit weight decreased when the nonwoven fabric was pre-deformed along TD. Moreover,

the elliptical zone of deformed material around the impact point became close to a circle when the fabric was pre-deformed along MD, while the aspect ratio of the ellipse increased if the material had been pre-deformed along TD. Penetration in the former situation (pre-deformation along MD) was accompanied by extensive fiber pull-out around the projectile but this phenomenon was not observed in the latter. Pre-deformation of the fabric along TD was accompanied by the preferential orientation of the fibers along the loading direction, enhancing the mechanical anisotropy of the nonwoven fabric because very few fibers remained oriented along MD. During impact, the projectile was able to slip through the fibers because the fabric resistance to deformation along MD was negligible and the overall energy absorption capability decreased. On the contrary, pre-deformation along MD led to a more isotropic in-plane mechanical behavior of the fabric, which improved the impact resistance as the projectile could not slip between fibers, which had to be pulled out from the fabric. These results show that careful design of the nature of the entanglement points and of the fiber orientation can be used to design nonwoven fabrics with improved energy absorption capability by maximizing energy dissipation by fiber pull-out and avoiding projectile penetration by slippage among fibers.

CHAPTER 6

Multiscale constitutive model for needlepunched nonwoven fabrics

The aim of this chapter is the development of a multiscale constitutive model for the mechanically entangled nonwoven fabric previously characterized, including the dominant deformation and failure mechanisms at different length scales (fiber, bundle and network). The model accounts for the effects of non-affine deformation, anisotropic connectivity induced by the entanglement points, fiber uncurling and re-orientation as well as fiber disentanglement and pull-out from the knots. The model provides the constitutive response for a mesodomain of the fabric corresponding to the volume associated to a finite element. As far as possible, a clear physical meaning is given to the model parameters, so they can be identified by means of independent tests, see Chapter 3. The model was validated against the experimental results of the tensile response of plain specimens along the two main material directions shown in Chapter 4.

6.1 Description of the model

The model presented in this section describes the mechanical response of a nonwoven fiber network including the effect of non-affine deformation, anisotropic connectivity, fiber uncurling and re-orientation as well as fiber disentanglement and pull-out from the knots.

6.1 Description of the model

The model is intended to be used within the framework of the finite element method and provides the constitutive response for a mesodomain of the fabric corresponding to the volume associated to a finite element. As far as possible, a clear physical meaning is given to the model parameters, so they can be identified by means of independent tests. The model is divided in two blocks. The first one is the network model which establishes the relationship between the macroscopic deformation gradient $\mathbf{F}$ and the microscopic response obtained by integrating the response of the fibers in the domain under study within a rigorous continuum tensorial formulation. The second one is the fiber model, which takes into account the deformation features of each set of fibers, including non-affinity, uncurling, rotation, pull-out and disentanglement.

6.1.1 Network model

The structure and deformation of the fiber network follows the model developed by Ridruejo et al. (2012a), which was successfully applied to simulate mechanical behavior of thermally-bonded polypropylene nonwoven fabrics (Ridruejo et al., 2012a,b). The model considers a mesodomain formed by a square planar region of arbitrary size containing a random network of long, curly, noninteracting fibers, Fig. 6.1. Due to the small fabric thickness, most of the fibers were oriented in the fabric plane, with the exception of the entanglement points, where fiber stitching in the through-the-thickness direction was promoted by the barbed needles. However, the contribution of the out-of-plane fibers to the in-plane mechanical response of the fabric can be neglected, leading to a 2D model (Termonia, 2003). Fiber stretching was negligible as compared to uncurling, so the fibers were considered inextensible, and the macroscopic strain transmitted into the fiber only modified the curvature and orientation within the network. Following Petterson (1958), it was assumed that the changes in curvature and orientation of one fiber did not influence the orientation of the adjacent fibers within the mesodomain.

Each fiber is characterized by a vector $\mathbf{N}$ whose orientation is given by the two extremes of the fiber, P and Q , according to Fig. 6.1,

$$\mathbf{N} = \frac{\overline{PQ}}{\|P_0Q_0\|} \quad (6.1)$$

Figure 6.1: Schematic network mesodomain formed by different sets of curly fibers.

where P_0 and Q_0 stand for the positions of the two extremes of the fiber in the initial, unstressed configuration, which is taken as reference. $\mathbf{N}$ forms an angle β with respect to an arbitrary, privileged direction (e.g. the transverse direction TD of nonwoven fabric) in the initial configuration.

If the mesodomain is subjected to a certain imposed deformation, given by the deformation gradient $\mathbf{F}$, the equivalent fiber extension will be given by $\lambda = \|\mathbf{N}\|$ according to

$$\lambda = \sqrt{\mathbf{C}\mathbf{N} \cdot \mathbf{N}} \quad (6.2)$$

where $\mathbf{C} = \mathbf{F}^T \mathbf{F}$ is the right Cauchy-Green strain tensor.

Curly fibers in the undeformed configuration are assumed to be formed by successive semi-circumferences with a radius R_0 and a curvature angle $\theta_0 = \pi$ (Fig. 6.2). As a result of deformation, the radius R and the curvature angle θ change according to

$$n \|\overline{PQ'}\| = 2nR \sin(\theta/2) \quad (6.3)$$

where $n = L_{fiber}/R_0\theta_0$ and L_{fiber} the fiber length. The progressive uncurling of the fiber as a result of deformation is related to the curvature angle θ according to

$$\lambda = \frac{\pi}{\theta} \sin \frac{\theta}{2} \quad (6.4)$$

6.1 Description of the model

Figure 6.2: Sketch of a curly fiber. (a) Reference configuration. (b) Current configuration.

and the fiber is fully straightened when $\lambda = \frac{\pi}{2}$. As the fiber is assumed to be inextensible, further fiber stretching from this point is understood as a movement of the straight fiber which is pulled out from the entanglement points.

The stress of the fiber is dependent on the fiber extension and is originated by shear forces transmitted by friction among fibers, resulting in an instant force T , which can be transformed into engineering stresses with the fiber cross section Ω_0

$$\sigma_f = \frac{T(\lambda)}{\Omega_0} = \sigma_f(\lambda) \quad (6.5)$$

As the fiber doesn't stretched, the cross section remains constant during all the deformation process. The mechanical power per unit volume of fiber $\dot{\omega}$ associated to the force T acting on the fiber yields,

$$\dot{\omega} = \frac{T\dot{\lambda}}{\Omega_0} \quad (6.6)$$

Using the time derivative of equation (6.2) in equation (6.6), the mechanical power per unit volume of fiber, $\dot{\omega}$ can be written in the reference configuration as

$$\dot{\omega} = \sigma_f(\lambda)\dot{\lambda} = \sigma_f(\lambda)\frac{\dot{\mathbf{C}}\mathbf{N} \cdot \mathbf{N}}{2\sqrt{\mathbf{C}\mathbf{N} \cdot \mathbf{N}}}. \quad (6.7)$$

Recalling as

$$\dot{\omega} = tr \left[\sigma_f(\lambda) \frac{(\mathbf{N} \otimes \mathbf{N})\dot{\mathbf{C}}}{2\sqrt{\mathbf{C}\mathbf{N} \cdot \mathbf{N}}} \right]. \quad (6.8)$$

Taking into account all the fibers in the material, the last expression is able to define the mechanical power per unit volume $\dot{\omega}_{fabric}$ of a fabric

$$\dot{\omega}_{fabric} = tr \left[\sum_{\beta_0} \Psi(\beta_0) \sigma_f(\lambda) f_f(\beta_0) \frac{(\mathbf{N} \otimes \mathbf{N})}{2\sqrt{\mathbf{C}\mathbf{N} \cdot \mathbf{N}}} \dot{\mathbf{C}} \right] \quad (6.9)$$

where $\Psi(\beta_0)$ is the fiber orientation distribution function (ODF) at the reference configuration, $\sigma_f(\lambda)$ is the stress carried by the fiber, $f_f(\beta_0)$ the distribution function of the active fiber length engaged in the deformation process, and β_0 is the fiber orientation in the reference configuration with respect to the privileged direction.

Besides, continuum models are written in terms of the Cauchy stress tensor in the current configuration as

$$\boldsymbol{\sigma} = \frac{1}{J} \mathbf{F} \mathbf{S} \mathbf{F}^T \quad (6.10)$$

where J , the Jacobian, is the determinant of $\mathbf{F}$ and $\mathbf{S}$ is the second Piola-Kirchoff stress tensor, which considers forces and areas in the reference configuration.

Considering a homogeneous solid, subjected to an arbitrary deformation given by $\mathbf{C}$, the mechanical power per unit volume due to deformation written in the reference configuration in terms of the second Piola-Kirchoff stress tensor is given by

$$\dot{\omega} = tr \left[\frac{1}{2} \mathbf{S} \dot{\mathbf{C}} \right] \quad (6.11)$$

and the integration of the mechanical power corresponds to the total internal energy consumed

$$W = \int_t \dot{\omega} \quad (6.12)$$

Finally the mechanical equivalence between the microscopic and the continuum mechanical models will be satisfied by the following condition

$$tr \left[\frac{1}{2} \mathbf{S} \dot{\mathbf{C}} \right] = tr \left[\sum_{\beta_0} \Psi(\beta_0) \sigma_f(\lambda) f_f(\beta_0) \frac{(\mathbf{N} \otimes \mathbf{N})}{2\sqrt{\mathbf{C}\mathbf{N} \cdot \mathbf{N}}} \dot{\mathbf{C}} \right] \quad (6.13)$$

for all possible arbitrary deformation velocities given by $\dot{\mathbf{C}}$. This expression is equivalent in terms of mechanical power stored or dissipated by the system. Thus, the constitutive

6.1 Description of the model

behavior of the nonwoven fabric can be expressed in terms of the second Piola-Kirchoff stress tensor and the right Cauchy-Green strain tensor as

$$\mathbf{S} = \sum_{\beta_0} \Psi(\beta_0) \sigma_f(\lambda) f_f(\beta_0) \frac{(\mathbf{N} \otimes \mathbf{N})}{\sqrt{\mathbf{C}\mathbf{N} \cdot \mathbf{N}}} \quad (6.14)$$

where the information concerning fiber orientation should be also provided in the reference configuration. Therefore the constitutive equation of the fabric over the planar area can be expressed as

$$\mathbf{S} = \int_{-\frac{\pi}{2}}^{\frac{\pi}{2}} \Psi(\beta_0) \sigma_f(\lambda) f_f(\beta_0) \frac{(\mathbf{N} \otimes \mathbf{N})}{\sqrt{\mathbf{C}\mathbf{N} \cdot \mathbf{N}}} d\beta_0 \quad (6.15)$$

The fiber ODF in the reference configuration $\Psi(\beta_0)$ is defined as the probability for a fiber to be oriented in the interval $(\beta_0, \beta_0 + d\beta_0)$. Obviously,

$$1 = \int_{-\frac{\pi}{2}}^{\frac{\pi}{2}} \Psi(\beta_0) d\beta_0. \quad (6.16)$$

The experimental results summarized in Chapter 3 indicated that the fraction of active fibers (actually engaged in the deformation process) was different along MD and TD. The total fiber volume per unit area of the fabric is given by ρ/ρ_f , where ρ is the areal density of the fabric and ρ_f is fiber density¹. The needlepunching process induces an anisotropic connectivity in the network so the active fiber length that participates in the deformation depends on the orientation. This is accounted in the model through the active fiber length function, $f_f(\beta_0)$, which expresses the fiber volume per unit area of the fabric that contributed to the deformation in the orientation $(\beta_0, \beta_0 + d\beta_0)$. It is assumed to be independent of the applied strain and it has to fulfill the condition

$$\frac{\rho}{\rho_f} = \int_{-\frac{\pi}{2}}^{\frac{\pi}{2}} f_f(\beta_0) d\beta_0 \quad (6.17)$$

¹ ρ/ρ_f has dimensions of length and indicates the total fiber length that has to be laid per unit area to manufacture the fabric. This parameter, and not the fiber volume fraction or the nonwoven thickness, is normally used to define the fiber content because the nonwoven thickness is very difficult to be determined as it depends on the compaction pressure during the measurement.

6.1.2 Fiber model

The mechanical response of an active fiber (or of a set of active fibers with the same initial orientation) within the network was characterized by means of pull-out tests, see Chapter 3. The load - displacement curves presented three regions, corresponding to different deformation mechanisms. The initial part was controlled by the uncurling of the fibers and by the stiffness of the fabric around the fiber or set of fibers which was pulled out. Moreover, the initial slope of the load - displacement curve depended on the orientation and was approximately twice higher along TD than along MD. Because of the nature of the entanglement points, the strain of the fibers pulled out along TD was fully transmitted to the bulk, leading to affine deformation, while non-affine deformation was found along MD. Afterwards, the pulled out fibers began to slide from the entanglements points and the load - displacement curve presented a serrated shape as the fiber was progressively disentangled from successive knots. Finally, the load was reduced to very low values as the fiber was completely disentangled and extracted from the fabric.

These mechanisms were introduced in a phenomenological model that characterizes the stress (σ_f) - stretch (λ) behavior of the fiber according to

$$\begin{aligned} \sigma_f &= K_{po} [\eta(\beta_0)(\lambda - 1)]^3 & \sigma_f < \sigma_{po} \\ \sigma_f &= (1 - d)K_{po} [\eta(\beta_0)(\lambda - 1)]^3 & \sigma_f > \sigma_{po} \end{aligned} \quad (6.18)$$

where K_{po} is the pull-out stiffness, σ_{po} the pull-out strength, $\eta(\beta_0)$ the affinity factor and d the damage parameter that accounts for the progressive reduction in the load carried by the fiber during extraction from the fabric. The initial fiber stiffness ($\lambda \rightarrow 1$) was considered negligible (due to the curl of the fibers (González and Llorca, 2005)) and increased afterwards due to the contribution of the fabric. Following Picu (2011), it was considered to evolve with the third power of the fiber stretch, which is a reasonable value for entangled materials with curly fibers. The stiffness was also dependent on the initial fiber orientation, β_0 , through the affinity parameter η , as shown in Fig. 6.3. It was assumed that all the deformation was transmitted to the bulk along TD and thus $\eta(0) = 1$ (affine deformation) while $\eta(\pi/2) = 0.5$ (non-affine deformation) along MD because the initial bulk stiffness during multiple fiber bundle pull-out tests along MD was half of that along TD, indicating that a significant part of the deformation was not transmitted to the fibers. The transition

6.1 Description of the model

Figure 6.3: Evolution of the affinity parameter η with the initial fiber orientation β_0 . $\beta_0 = 0$ corresponds to TD and $\beta_0 = \frac{\pi}{2}$ to MD. The magnitude η is symmetric for the interval $[-\frac{\pi}{2}, 0]$.

between both extremes was interpolated by means of a cubic Hermite polynomial which provides a plateau around the $\beta = 0$ and $\beta = \pi/2$ and a soft transition between them.

Once σ_{po} is reached, the pull-out process begins and the load carried by the fiber decreases progressively until is completely disengaged from the fabric. The stress carried by the fibers during this stage is determined by means of a continuum damage model which depends on the energy dissipated during fiber pull-out and is detailed in Appendix B.

The force necessary to extract a fiber from the fabric is given by the pulley theory according to

$$T(L) = T_0 e^{\mu\alpha} \quad (6.19)$$

where $T(L)$ is the force to extract a fiber which is embedded a length L into the fabric, T_0 the hold force, μ the friction coefficient and α the contact angle. The contact angle α can be considered proportional to the fiber curvature θ (eq. 6.4) and to the number of entanglement points $n = L/L_c$, where L_c is the contour fiber length between entanglements, according to

$$\alpha = \theta \frac{L}{L_c} \quad (6.20)$$

6.1 Description of the model

The energy dissipated to extract the fiber from the fabric, W , is given by

$$W = \int_0^{L_{po}} T(L) \, dL = T_0 \frac{L_c}{\mu\theta} (e^{\mu\theta L_{po}/L_c} - 1) \quad (6.21)$$

where L_{po} is the fiber length extracted from the fabric by pull-out. T_0 can be obtained from the condition that $T(L_{po}) = \sigma_{po}\Omega_f$, where Ω_f is the fiber cross section. Thus, the energy dissipated during the pull-out of one fiber is given by

$$W = \sigma_{po}\Omega_f \frac{L_c}{\mu\theta} (1 - e^{-\mu\theta L_{po}/L_c}) \quad (6.22)$$

and the corresponding fracture energy (per unit surface of the fiber), G , is

$$G = \frac{\sigma_{po}L_c}{\mu\theta} (1 - e^{-\mu\theta L_{po}/L_c}) = \frac{\sigma_{po}L_c}{\mu\theta} (1 - e^{-\mu\theta n_{po}}) \quad (6.23)$$

where $n_{po} = L_{po}/L_c$ is the upper bound of mechanical entanglements involved in the pull-out process.

The micromechanical tests presented in Chapter 3 showed that the knots presented different resistance to the extraction of the fibers. Some knots were open and the fiber could be pulled out from them very easily while others were very tight and the extraction of the fiber required higher stresses. In this model, it is assumed that these differences in the knot behavior are due to the strains at the knot. If the fibers connected in a knot are subjected to tensile strains, the knot will be locked and will contribute to the pull-out forces. However, if the fibers in the knot are subjected to compressive strains, the knot will be unlocked and the resistance to the pull-out will be much smaller. This mechanism is included in the model by assuming that actual number of active knots, n_{po} , depends on the macroscopic strain according to

$$n_{po} = \frac{L_{po}}{L_c} \sqrt{\frac{\hat{U}_{hyd} - \hat{U}_{unl}}{1 - \hat{U}_{unl}}} \quad (6.24)$$

where $\hat{U}_{hyd} = (\hat{U}_{11} + \hat{U}_{22})/2$ and

$$\begin{aligned} \hat{U}_{11} &= U_{11} & \text{if } U_{11} \leq 1 & \text{ or } \hat{U}_{11} = 1 & \text{ otherwise} \\ \hat{U}_{22} &= U_{22} & \text{if } U_{22} \leq 1 & \text{ or } \hat{U}_{22} = 1 & \text{ otherwise} \end{aligned} \quad (6.25)$$

6.2 Numerical implementation

where U_{11} and U_{22} stand for the components of the right stretch tensor $\mathbf{U}$. $\hat{U}_{unl}$ is the critical value of $\hat{U}_{hyd}$ at which the contribution of the knots to the pull-out process becomes negligible. Thus, energy dissipated by friction during pull-out decreases if one of the principal stretches is compressive and the reduction increases with the compressive strain.

6.2 Numerical implementation

The constitutive model developed in the previous section has been implemented as a VUMAT subroutine in Abaqus/Explicit. The numerical simulations were carried out within the framework of quasi-static deformation and large displacements and rotations with the initial unstressed state taken as reference. Abaqus/Explicit provides the right stretch tensor $\mathbf{U}$ to compute the stretch λ of each set of fibers, eq. (6.2). To this end, the right stretch tensor $\mathbf{U}$ is transformed into the right Cauchy-Green strain tensor $\mathbf{C}$ taking into account the corotational formulation of the VUMAT, $\mathbf{R} = \mathbf{I}$.

The fiber stretch λ was used to compute the fiber curvature θ according to eq. 6.4 and the updated orientation β was determined as

$$\beta = \arctan \left(\frac{U_{22} + U_{12}}{U_{11} + U_{12}} \tan \beta_0 \right) \quad (6.26)$$

where U_{11} , U_{12} and U_{22} are the components of the right stretch tensor $\mathbf{U}$.

Each mesodomain of the fiber network (that coincides with a finite element with one Gauss point) was described by 33 sets of fibers with different orientation in the range $(-\frac{\pi}{2}, \frac{\pi}{2})$. The second Piola-Kirchoff nominal stress tensor, $\mathbf{S}$, was obtained by integrating eq. (6.15) along the different orientations. The numerical integration was carried out using the Clenshaw-Curtis quadrature, which is based on an expansion of the integrand in terms of Chebyshev polynomials (Abramowitz and Stegun, 1972).

The fiber stretch λ was used to compute the trial stress σ_{trial} as well as fiber curvature θ . If $\sigma_{trial} \leq \sigma_{po}$, $\sigma_{trial} = \sigma_f$ according to equation (6.18), where σ_{po} was computed at the beginning of the simulation for each fiber set using a Monte Carlo lottery. If $\sigma_{trial} > \sigma_{po}$, the actual fiber stress σ_f was provided by the continuum damage mode in Appendix B depending on the damage variable d the fracture energy G and the softening parameter A , which ensures that the energy dissipated is always equal to G and independent of the

finite element discretization, as shown in Appendix C. A damage variable D was defined for each finite element (Gauss Point) as the average damage of all fibers sets in the elements. If $D > 0.99$, the element was removed from the simulations to avoid excessive element distortion.

6.3 Results and discussion

The model presented above was used to simulate the tensile deformation along TD and MD of the Fraglight NW201 nonwoven fabric. The numerical simulations followed the experimental results in Chapter 4. Square specimens of $100 \times 100 \text{ mm}^2$ were discretized with M3D4R membrane elements, with reduced integration (1 Gauss point per element), enhanced hourglass control and second order accuracy. Out-of-plane shear stresses were neglected, as in the Euler-Bernoulli beam formulation. The displacements of the upper and lower boundaries of the square domain were fully constrained, while the lateral boundaries were stress-free. The tensile test was simulated by imposing a vertical velocity of 0.5 m/s to the upper boundary of the square. This velocity was much higher than the experimental one to save computer time. Nevertheless, selected simulations carried out at 0.05 m/s provided equivalent results and showed that inertia and dynamic effects could be neglected. In addition, simulations carried out with different element lengths (4, 2 and 1 mm) gave similar results and demonstrated the objectivity of the numerical implementation, as shown in Appendix C. It should be noted that the default bulk viscosity option in Abaqus was used in the simulations to eliminate instabilities in the numerical model at the onset of damage. In addition, the minimum time step in the numerical simulation was fixed to $\Delta t = 10^{-5} \text{ ms}$, which is around two orders of magnitude smaller than the minimum stable time increment in the reference configuration. The minimum stable time step decreased during the analysis because of the distortion of the elements but it was never below 10^{-5} ms with the exception of the finest mesh (1 mm element length). In these cases, the total mass was increased by 2% to ensure stability without penalizing the time increment.

6.3 Results and discussion

6.3.1 Parameter identification

The constitutive model presented above relies in a number of parameters with a physical meaning that determine the behavior of the nonwoven fabric which were determined by means of independent tests in Chapters 3 and 4 whenever possible and are summarized here.

The initial fiber ODF was measured by means of XCT and WAXD in Chapter 3 and it was isotropic, so $\Psi(\beta_0) = 1/\pi$. However, the orientation distribution of active fiber length $f_f(\beta_0)$ in eq. (6.15) took into account the anisotropy induced by the entanglement point between MD and TD. It was assumed to be proportional to the affinity parameter η according to

$$f_f(\beta_0) = b\eta(\beta_0) - a \quad (6.27)$$

where $a=0.0836$ and $b=0.223$ are two parameters selected taking into account the condition imposed by equation (6.17). These values of a and b lead to a ratio of 5 between the active fiber length along TD and MD ($f_f^{TD}/f_f^{MD} = 5$) and are responsible for the differences in the strength between both orientations.

The fiber stress-stretch relationship depends on three parameters: the pull-out stiffness K_{po} , the pull-out strength σ_{po} and the energy dissipated during disentanglement and pull-out G . The pull-out stiffness $K_{po} = 9.0$ GPa was selected to be in good agreement with the initial stiffness of the fabric along TD. The strength to pull-out of each set of fibers σ_{po} was determined from the results of the single fiber pull-out tests presented in Chapter 3, which showed a large scatter. In order to reproduce more accurately the actual microstructure, it was decided to assign a value of σ_{po} for each fiber set within each mesodomain by means of a Monte Carlo lottery at the beginning of the simulations. The maximum and minimum values of σ_{po} were in the range $[0.3, 1.0]$ GPa, which are within the experimental scatter.

The energy dissipated during pull-out, G , given by eq.(6.23) depends on a number of parameters. The friction coefficient, $\mu = 0.1$, was obtained from the literature (Shen and Dumbleton, 1974), while the initial fiber curvature, θ_0 , was assumed to be equal to π . The pull-out length, $L_{po} \approx 35$ mm, could be roughly estimated as one half of the length of the pulled out zone just before the complete fracture in the specimens loaded along TD and MD, see Fig. 4.5 in Chapter 4. Finally, the contour length L_c and the parameter $\hat{U}_{unl}$ (the critical value of U_{hyd} at which the contribution of the knots to the pull-out process

Figure 6.4: Stress-stretch curves of fibers according to eq. (6.18) for an element length of 2 mm and for different values of the pull-out strength, σ_{po} , the affinity factor, η and the number of active knots along the fiber, n_{po} . (a) Stress-stretch curves for the maximum value of pull-out strength, $\sigma_{po} = 1$ GPa and (b) Stress-stretch curves for the minimum value of pull-out strength, $\sigma_{po} = 0.3$ GPa. Solid lines stand for affine deformation ($\eta = 1$) and broken lines for non-affine deformation ($\eta = 0.5$).

becomes negligible) were chosen as 2.0 mm and 0.85, respectively, to fit the experimental stress-strain curves.

Representative stress-stretch curves of fibers, according to eq. (6.18), for a finite element of 2 mm in length are plotted in Fig. 6.4. They present an initial non-linear behavior due to the progressive fiber straightening followed by a linear region that ends when the critical pull-out stress, σ_{po} , is attained. Afterwards, the stress decreases smoothly with the applied stretch due to the fiber pull-out. The main parameters that control the fiber behavior are σ_{po} and n_{po} that dictate the maximum load carried by the fiber and the energy dissipated during pull-out, respectively. In the latter case, the higher the number of active knots, n_{po} , the larger the energy dissipated by friction during pull-out. The affinity factor η determines the amount of the applied stretch λ which is transmitted to the fiber and thus controls the initial stiffness and the stretch at which the pull-out occurs. However, the influence of η on the amount of energy dissipated during pull-out is minor.

Figure 6.5: Nominal stress *vs.* engineering strain curves for specimens of 100 x 100 mm² deformed in tension along TD and MD. The solid lines correspond to the numerical simulations while the broken lines stand for the experimental results in Chapter 4.

6.3.2 Results and discussion

The tensile deformation of the nonwoven fabric was simulated and compared with the experimental results presented in Chapter 4. The numerical and experimental nominal stress (force per unit width) *vs.* the engineering strain is plotted in Fig. 6.5 for the specimens deformed along TD and MD. The numerical results in this figure were obtained with a mesh of an initial finite element length of 2 mm. Simulations carried out with meshes with different sizes (in the range 1 to 4 mm) are shown in Appendix C and presented equivalent results. The experimental curves in Fig. 6.5 stand for the upper and lower bounds of the experimental data in each orientation. The numerical curves in each orientation differ after the peak load because each simulation presented a different distribution of the pull-out strength in each fiber set, as dictated by the Monte Carlo lottery. Numerical and experimental curves were in very good agreement and the different regions in the nominal stress-strain curves were accurately predicted, including the initial non-linear region, the peak strength and the softening after the peak. Moreover, the model was able to reproduce the large differences in the mechanical behavior between TD and MD although the initial fiber orientation distribution was isotropic. The differences in the strength between both

orientations were due to the anisotropic connectivity, as given by $f_f(\beta_0)$, which determined the differences in the fraction of active fibers along TD and MD. The differences in the stiffness between TD and MD were mainly controlled by the non-affine response of the fiber network along MD because the applied stretch was only partially transferred to the fibers in this orientation. The combination of both factors led to the strong anisotropy in the mechanical response of the material, as shown in Fig. 6.5.

The initial deformation mechanisms in both orientations were fiber straightening and rotation towards the loading axis. Deformation along TD was affine and all the strain was directly transferred to the fibers. A large fraction of the fibers was already oriented along TD (due to the anisotropic connectivity) and fiber straightening and rotation were exhausted for applied strains around $\approx 40\%$. Thus, further deformation had to be accommodated by fiber pull-out leading to fracture by localization of damage in a given section of the specimen. Deformation along MD was, however, non-affine and only part of the applied strain was transferred to the fibers. In addition, the fraction of active fibers along MD was smaller (due to the anisotropic connectivity) and part of the applied deformation was spent in fiber rotation towards MD. As a result, fiber straightening and rotation took place up to applied strains around $\approx 100\%$ and damage was triggered beyond this point.

The accuracy of the model was also checked by comparing the numerical and experimental results of the shape of the deformed specimen as well as the distribution of the logarithmic strain in the loading direction, which was obtained in the experiments by means of digital image correlation. They are depicted in Figs. 6.6 and 6.7 for TD and MD orientations, respectively. The numerical simulations captured the hourglass shape and homogeneous deformation of the specimens in both orientations before the onset of damage (up to 50% along TD and 100% along MD) as well as the onset of damage localization at a given section of the sample at higher strains. They also reproduced the differences in the lateral contraction between specimens deformed along TD and MD, which was larger in the latter. Nevertheless, the model underestimated the absolute value of the lateral contraction in both cases because local wrinkling of the membrane elements was not considered in the model.

It is interesting to emphasize at this point the influence of the parameter $\hat{U}_{hyd}$ in eq. (6.24) on the energy dissipated during pull-out along TD and MD. Fiber disentanglement and pull-out occurs at applied strains around 50-60% along TD (Fig. 6.6c) and 100-120%

Figure 6.6: Experimental (a, c, e) and numerical (b, d, f) contour plots of the logarithmic strain along the loading axis (TD) at different values of the applied strain. (a) and (b) 30%. (c) and (d) 50%. (e) and (f) 70%.

Figure 6.7: Experimental (a, c, e) and numerical (b, d, f) contour plots of the logarithmic strain along the loading axis (MD) at different values of the applied strain. (a) and (b) 40%. (c) and (d) 100%. (e) and (f) 120%.

6.3 Results and discussion

along MD (Fig. 6.7c). The number of active entanglements points, n_{po} , in both orientations decreased as U_{11} or U_{22} decreased. As U_{11} was much lower when stretching along MD than U_{22} when stretching along TD at the onset of damage, the energy dissipated by friction was also smaller along MD, in agreement with the experimental results.

Another important outcome of the numerical model was the evolution of the fiber orientation with the applied strain. The orientation of each fiber set, β , was computed according to equation (6.26) as a function of the deformation gradient, and the fiber curvature, θ , was computed according to equation (6.4) as a function of the fiber stretch λ . This information was used to build the fiber ODF Ψ as function of the applied strain, following the same methodology employed in Chapters 3 and 4 for the treatment of the XCT data. The reconstructed fiber ODF could be compared with the corresponding experimental distribution obtained by WAXD in Chapter 4 and the polar representations of the numerical and experimental fiber orientation distributions at the center of the specimens deformed along TD and MD are plotted in Figs. 6.8 and 6.9, respectively. The experimental results demonstrate the progressive fiber rotation along the loading direction with the applied strain for the samples loaded along TD and MD and the numerical results corroborate this information. This leads to an anisotropic fiber distribution that is more marked along TD than along MD at 40%. This behavior can be attributed to both the higher affinity (more strain is transferred to the fibers) and the higher active fiber volume fraction (more fibers are affected by the applied strain) along TD. A marked anisotropic fiber orientation also develops after deformation along MD but at much larger strains (100%). It is worth noting that the numerical model was able to capture these microstructural changes that are critical to explain the behavior of the nonwoven fabric.

6.3.3 Influence of affinity and connectivity on the mechanical response

The experiments and simulations results presented above indicated that the strong anisotropy in the mechanical response of the mechanically-entangled nonwoven was triggered by the differences in network affinity and connectivity between both orientations. In order to clarify these points, which are relevant for nonwoven fabrics, a parametrical study was carried out to assess the effect of both parameters on the mechanical behavior.

Figure 6.8: Numerical and experimental polar representation of the fiber orientation distribution Ψ at the center of the specimen after 40% deformation along TD.

The nominal stress *vs.* engineering strain curves along TD and MD are plotted in Fig. 6.10(a) for a nonwoven fabric characterized by affine deformation ($\eta = 1$) and an anisotropic fiber connectivity in both orientations, as given by f_f in equation (6.27). The corresponding curve for affine deformation and isotropic fiber connectivity ($f_f = \frac{1}{\pi} \frac{\rho}{\rho_f}$ according to equation 6.17) is also plotted for comparison. It shows that the strength differential between the TD and MD is controlled by the differences in the active fiber length between both orientations, while the strength of a nonwoven fabric with isotropic connectivity is in between both. Of course, the anisotropic connectivity also influences the initial stiffness and, thus, the strain to failure is smaller in the stiffest orientation.

The effect of the anisotropic affinity between TD and MD is depicted in the nominal stress *vs.* engineering strain curves plotted in Fig. 6.10(b) for a nonwoven fabric characterized by an isotropic fiber connectivity ($f_f = \frac{1}{\pi} \frac{\rho}{\rho_f}$ according to equation 6.17). The curves corresponding to the TD and MD orientations were obtained with the function $\eta(\beta_0)$ shown in Fig. 6.3, which assumed affine deformation along TD and non-affine deformation along MD. The non-affine deformation along MD reduced the initial stiffness of the nonwoven and increased the strain to failure but it did not influence the strength, that was equivalent in both orientations and similar to the affine nonwoven with isotropic fiber connectivity.

Figure 6.9: Numerical and experimental polar representation of the fiber orientation distribution Ψ at the center of the specimen after deformation along MD. (a) 40%. (b) 60%. (c) 80%. (d) 100%.

Figure 6.10: Parametric study of the influence of the network affinity and connectivity on the nominal stress - engineering strain curve of the nonwoven fabric along TD and MD. (a) Affine model ($\eta = 1$) with anisotropic fiber connectivity given by f_f in equation 6.27. (b) Non-affine model (with η as shown in Fig. 6.3) with isotropic fiber connectivity ($f_f = \frac{1}{\pi} \frac{\rho}{\rho_f}$). The results for an affine model ($\eta = 1$) with isotropic fiber connectivity ($f_f = \frac{1}{\pi} \frac{\rho}{\rho_f}$) are plotted in both figures for comparison.

6.4 Concluding remarks

A physically-based micromechanical model was developed to simulate the in-plane deformation of mechanically-entangled nonwoven fiber networks. The model was intended to be used within the framework of the finite element method and provides the constitutive response for a mesodomain of the fabric corresponding to the area associated to a finite element. Each mesodomain was formed by a square planar region of arbitrary size containing a network of long, curly, non-interacting fiber sets with different orientation. The model was divided in two blocks. The first one was the network model which established the relationship between the macroscopic deformation gradient $\mathbf{F}$ and the microscopic response obtained by integrating the response of each fiber set within a rigorous continuum tensorial formulation. The network model also took into account that the needlepunching process induced an anisotropic connectivity in the network so the active fiber length that participates in the deformation depended on the orientation.

6.4 Concluding remarks

The second block was the fiber model, that took into account the deformation features of each fiber set. Fiber stretching was considered negligible, and the macroscopic strain transmitted into the fiber was dedicated to modify its curvature and orientation within the network as well as to pull-out the fibers from the knots in the fabric. These mechanisms were taken into account in a constitutive model for each fiber set that included the non-linear deformation and energy dissipation due to fiber uncurling as well as fiber disentanglement and pull-out from the knots. Moreover, the fiber model also accounted for the development of affine or non-affine deformation as a function of the initial fiber orientation in the fabric.

The model was used to simulate the tensile deformation along two perpendicular orientations (TD and MD) of a mechanically-entangled nonwoven fiber network, whose mechanical behavior at the micro and macroscale was previously characterized. The model parameters had a clear physical meaning and were obtained from independent tests whenever possible. The results of the finite element simulations of the tensile deformation of the nonwoven fabric along TD and MD were in very good agreement with the experimental data in terms of the nominal stress-strain curves, the shape deformed specimens and the evolution of fiber orientation distribution with the applied strain. Moreover, the model captured the large anisotropy in stiffness and strength between both orientations although the initial fiber orientation distribution was isotropic. The higher strength along TD, as compared with MD, was caused by the differences in the fiber connectivity between both orientations induced by the needlepunch process. Deformation along TD was affine and all the strain was directly transferred to the fibers. Fiber uncurling and rotation were exhausted for applied strains around $\approx 40\%$ and further deformation had to be accommodated by fiber pull-out leading to fracture by localization of damage in a given section of the specimen. On the contrary, deformation along MD was not affine and only part of the applied strain was transferred to the fibers. As a result, the stiffness along this orientation was much lower and the strain to failure increased as the exhaustion of the mechanisms of fiber uncurling and rotation occurred for applied strains around $\approx 100\%$ and damage was triggered beyond this point. These results show the potential of this physically-based model to reproduce the complex deformation and fracture micromechanisms of needle-punched nonwoven fabrics.

CHAPTER 7

Numerical simulation of the ballistic response

The goal of this chapter is the study of the deformation and failure micromechanisms during impact of the needlepunched nonwoven fabric by numerical simulation. The constitutive model developed in previous Chapter 6 was used to simulate the impact on as-received and pre-deformed specimens exposed in Chapter 5. Correlation between experiments and simulation was carried out in terms of ballistic limit and absorbed energy. Localization of the damage due to impact and anisotropy stiffness of the specimens were also compared. Finally, the model was used to explore the influence of non-affine deformation and anisotropy in the fiber connectivity on the impact response.

7.1 Numerical Simulations

The in-plane deformation and the behavior of the nonwoven fabric against ballistic impact exposed in Chapter 5 were simulated using the finite element method. To this end, the constitutive model presented in Chapter 6 was implemented as a VUMAT subroutine in Abaqus/Explicit. The numerical simulations were carried out within the framework of large displacements and rotations with the initial unstressed state taken as reference.

7.1 Numerical Simulations

Each mesodomain of the fiber network (that coincides with a finite element with one Gauss point) was described by 65 sets of fibers with different orientation in the range $(-\frac{\pi}{2}, \frac{\pi}{2})$.

Square specimens of 300 mm width and variable length, l_0 , (from 228 mm up to 500 mm) were discretized with M3D4R membrane elements, with reduced integration (1 Gauss point per element), enhanced hourglass control and second order accuracy. Out-of-plane shear stresses were neglected, as in the Euler-Bernoulli beam formulation. This geometry was large enough to accurately simulate the impacts on the clamped fabric with a free area of 350 x 350 mm². The mesh was finer around the impact zone (finite elements of 1 mm²) and the element size increased progressively with the distance from the impact zone to reduce the computational time.

The simulations were carried out in two successive steps. The samples were initialized stretched along either TD or MD to reach the final length of 500 mm. The displacements of the upper and lower boundaries of the samples were fully constrained, while the lateral boundaries were stress-free. The tensile test was simulated by imposing a vertical velocity of 1 m/s to the upper boundary of the square. This velocity was much higher than the experimental one to reduce the computing time but it was able to reproduce the quasi-static deformation because dynamic and inertia effects were negligible. The second step of the simulation was the impact of the fabric with a steel sphere of 5.5 mm in diameter and 7.85 g/cm³. All the boundaries of the specimen were fully constrained during the impact simulation. The steel sphere was modelled as a rigid solid and the friction coefficient between the sphere and the fabric was 0.1.

Quasi-static material parameters were used for the impact simulation, as the nonwoven was considered strain rate independent up to a 40% of deformation, see Chapter 4. Exact values of the material parameters are shown in Table 7.1. The only differences with the parameters used in quasi-static simulations are the fiber pull-out length, L_{po} , which was increased up to L_{fiber} because of the experimental observations in the ballistic tests where whole extraction of the fiber by the projectile is appreciated (Fig. 5.11), and the pull-out strength, σ_{po} , which was in the range [0.3 - 1.7] GPa for the ballistic simulations, slightly higher than the range [0.3 - 1.0] GPa chosen to simulate the quasi-static deformation. Nevertheless, both ranges are within the experimental range measured in the fiber pull-out tests, see Chapter 3.

Fiber density (ρ_f)	970 kg/m ³
Areal density (ρ)	0.2 kg/m ²
Fiber stiffness (K_{po})	9.25 GPa
Initial fiber curvature, (θ_0)	π
Pull-out strength (σ_{po})	[0.3, 1.7] GPa
Fiber pull-out length (L_{po})	60 mm
Contour length (L_c)	2.0 mm
Critical value of the hydrostatic strain ($\hat{U}_{unl}$)	0.85
Friction coefficient (μ)	0.1

Table 7.1: Model parameters for impact test.

7.2 Results and discussion

7.2.1 As-received nonwoven

The constitutive model presented above was used to simulate the impact behavior of the as-received nonwoven fabric at different impact velocities. The results of the simulations, in terms of the residual velocity, V_{res} , as a function of the initial velocity, V_{ini} , are plotted in Fig. 7.1. It should be noted that several simulations were carried out for the same initial velocity because the model predictions presented some scatter due to the changes in the fiber pull-out strength of each fiber. The experimental results presented in Chapter 5 are also plotted in Fig. 7.1. The comparison between both shows that the numerical simulations were able to capture the ballistic limit as well as the increase in the residual velocity just above the ballistic limit. Nevertheless, the model underestimated the residual velocity for impact at velocities above the ballistic limit. It should be noted that the final penetration of the target in the tests carried out above the ballistic limit was accomplished by tearing as the fibers were pulled out from the entanglement points or broken near to the impact point due to thermal softening, see Chapter 5. This latter mechanism of fiber fracture was not considered in the constitutive model and may account for the differences with the experiments above the ballistic limit.

The numerical simulations were useful to understand deformation mechanisms around the impact point. The in-plane stresses were transmitted by a longitudinal wave and the nonwoven fabric was stretched towards the impact point, generating complex stress and strain fields (Fig. 7.2). The regions affected by the longitudinal waves and by the (slower)

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Figure 7.1: Experimental and simulation results of the ballistic tests on the as-received nonwoven fabric.

transverse waves were ellipsoidal with the longest axis parallel to TD because the wave speed along TD was higher than along MD as a result of the mechanical anisotropy of the fabric. The in-plane stresses were rather similar along TD and MD far away from the impact point. The in-plane stresses around the impact point along TD were, however, higher than those along MD, Figs. 7.2(b), (d) and (f). These differences between TD and MD were more marked in the strain fields, Figs. 7.2(a), (c) and (e). The strains along MD were much higher because the deformation of the fabric in this orientation was non-affine. Thus, only a fraction of the strain was actually transferred to the fabric in this orientation and much higher strains were necessary to reach the stress level at the impact point. These differences have been experimentally observed during impact on anisotropic plates (Seidt et al., 2013).

The transverse wave led to the global deflection of the fabric (Fig. 7.3), and generated a cone of deformed material with an elliptical cross-section. The ratio of the major to the minor axis of the cross-section in the simulations was 1.6 as a result of the anisotropic mechanical properties induced by the entanglement points. This ratio is close to the experimental value of 2 reported in Chapter 5 and the differences can be attributed to the

Figure 7.2: Contour plot of the strain and stress fields for the as-received nonwoven fabric impacted at 300 m/s 125 μ s after the impact. (a) Maximum principal logarithmic strain, ϵ_I . (b) Von Mises stress, σ_{VM} . (c) Logarithmic strain along TD, ϵ_{TD} . (d) Stress along TD, σ_{TD} . (e) Logarithmic strain along MD, ϵ_{MD} . (f) Stress along MD, σ_{MD} . The broken lines in the figures stand for the boundary of the propagation of the transverse waves.

limitations of the continuum model to accurately reproduce the wave propagation along a mechanically-entangled fiber network. Finally, both experiments and simulation showed that penetration took place by the complete disentanglement of the fiber network at the impact point.

The constitutive equation also provided information about the energy dissipation processes in the fabric during impact. Outside of the impact point, the fabric was mainly stretched in tension along TD and MD (Fig. 7.2) and the deformation micromechanisms were similar to those found under in-plane tensile deformation: fiber uncurling and rotation was followed by fiber sliding and pull-out from the entanglement points, although these

Figure 7.3: Lateral view of the as-received fabric during the impact at 360 m/s. (a) Experimental photograph at 100 μ s. (c) 175 μ s and (e) 250 μ s. (b) Contour plot of the damage variable D at 100 μ s. (d) 175 μ s and (f) 250 μ s. The zones in red in the contour plots are representative of a fully disentangled fabric.

latter mechanisms were only active around the impact point. It should be noted that the extracted fiber length (≈ 60 mm) was much larger than the length of the damage zone (of the order of the diameter of the projectile, 5.5 mm), indicating that the strain gradient perpendicular to the fabric around the impact region was very large. Nevertheless, energy was not only dissipated in this region because the behavior of the fabric was pseudo-plastic in the whole deformation range (elastic, recoverable deformations were always negligible, see Chapter 4). Thus, the whole volume of deformed fabric contributed to absorb the energy of the projectile.

Figure 7.4: Locations of points X and Y with respect to the impact point.

Figure 7.5: Polar representation of the fiber ODF Ψ before and 125 μs after the impact at 300 m/s on the as-received nonwoven fabric. (a) Point X and (b) Point Y in Fig. 7.4.

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More details about the microstructure evolution can be obtained by the reconstruction of the fiber ODF during the impact test. The orientation of each fiber set, β , was computed according to eq. (6.26) as a function of the deformation gradient and the fiber curvature, θ , was determined according to eq. (6.4) as a function of the fiber stretch λ . This information was used to build the fiber ODF, Ψ , following the same methodology employed in Chapter 3 for the treatment of the XCT data. The polar representation of the fiber ODF at two points X and Y located at 15 mm from the impact point along TD and MD (Fig. 7.4) are plotted in Fig. 7.5, 125 μ s after the impact at 300 m/s. The original, isotropic ODFs are also plotted for comparison. The maximum strain was oriented along TD in point X and along MD in point Y and thus fibers within the fabric rotated towards different direction in both points. Fibers in point X tended to be oriented along TD ($\beta = \pi/2$) while those in point Y were aligned towards MD ($\beta = 0$). The degree of orientation depended on two factors: the volume fraction of fibers involved in the deformation process (which was larger along TD than MD due to the anisotropic connectivity of the entangled fiber network) and the applied strain (which was larger along MD due to the non-affine deformation in this orientation). Both contributions led to a slightly higher re-orientation along MD in point Y than along TD in point X , but the differences were not very large.

7.2.2 Pre-deformed specimens

Previous experimental results in Chapter 5 have shown that pre-deformation of the nonwoven fabric along TD and MD led a reduction in the ballistic limit. They are compared with the results of the numerical simulations in Figs. 7.6 and 7.7 for the specimens pre-deformed along TD and MD, respectively. The solid lines in these figures stand for the fitting of the simulations results to the Lambert formula,

$$V_{res} = (V_{ini}^n - V_{50}^n)^{1/n} \quad (7.1)$$

where V_{ini} and V_{res} are the initial and residual velocity of the projectile, respectively. The ballistic limit, V_{50} , and the exponent n were obtained by the least squares fitting of the simulation results. The simulations provided very accurate predictions of the reduction in the ballistic limit with pre-deformation in both orientations and of the ballistic curves along MD up to a pre-deformation of 80%. The predicted values of the residual velocity above

Figure 7.6: Experimental and simulation results of the ballistic tests in the specimens pre-deformed along TD. (a) 20% pre-deformation. (b) 40% pre-deformation.

the ballistic limit for the specimens pre-deformed along TD and for those pre-deformed along MD more than 100% were slightly lower than the experimental data, very likely for the same reasons indicated for the as-received specimens.

The experimental and the predicted variations of the ballistic limit with the pre-deformation strain along TD and MD are plotted in Fig. 7.8. Experimental results were taken from Chapter 5 while V_{50} for the simulations were obtained from the ballistic limits given by the Lambert formula. The agreement between numerical and experimental results is very good and both show an approximately linear reduction of the ballistic limit with the applied pre-strain. However, deformation along TD led to a much higher reduction in the ballistic limit than along MD.

The information provided by the numerical model can be very useful to understand these large differences in the ballistic performance as a function of the pre-deformation along TD and MD. The contour plots of the maximum principal logarithmic strain, ϵ_I , and of the Von Mises stress, σ_{VM} , are plotted in Fig. 7.9 for the specimens pre-deformed by 40% along TD and MD 125 μ s after the impact at 300 m/s. Pre-deformation along TD by 40% led to the preferential orientation of the fibers along TD, as shown in the fiber ODF in Fig. 7.10(a) and (b), increasing the anisotropy of the fabric. This is shown in the shape of the pseudo-ellipse that delineates the boundary of the transverse wave in Fig. 7.9(a) and (b). However, the strain and stress fields in the fabric were rather homogeneous and higher

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Figure 7.7: Experimental and simulation results of the ballistic tests in the specimens pre-deformed along MD. (a) 20% pre-deformation. (b) 40% pre-deformation. (c) 60% pre-deformation. (d) 80% pre-deformation. (e) 100% pre-deformation. (f) 120% pre-deformation.

Figure 7.8: Experimental and simulation results of the ballistic limit, V_{50} , of the non-woven fabric as a function of the pre-deformation along MD and TD. Solid symbols stand for the experimental results and open symbols for the numerical simulations. The straight lines stand for the least squares fitting of the experimental ballistic limit.

stresses were only found very close to the impact region. Moreover, further re-orientation of the fibers along TD or MD due to the impact stresses was very limited, see Fig. 7.10(a) and (b). These data indicate that pre-deformation along TD consumed a large fraction of the energy dissipation capacity of the fabric and reduced the efficiency of the fabric against ballistic impact. In addition, the experimental observations in Chapter 5 also pointed out that the large anisotropy in the fiber orientation induced by the pre-deformation along TD facilitated the penetration of the projectile through the fabric: very few fibers remained oriented along MD and the projectile was able to slip through the fibers because the fabric resistance to deformation along MD was negligible.

Pre-deformation by 40% along MD led to a preferential fiber orientation along MD, Fig. 7.10(c) and (d). This promoted a more isotropic mechanical response, as shown by the shape of the boundary that delineated the propagation of the transverse wave, which

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Figure 7.9: Contour plot of the strain and stress fields for the pre-deformed fabrics impacted at 300 m/s 125 μ s after the impact. (a) Maximum principal logarithmic strain, ϵ_I , in the sample pre-deformed by 40% along TD. (b) Von Mises stress, σ_{VM} in the sample pre-deformed by 40% along TD. (c) Maximum principal logarithmic strain, ϵ_I , in the sample pre-deformed by 40% along MD. (d) Von Mises stress, σ_{VM} , in the sample pre-deformed by 40% along MD.

was close to a circle, Fig. 7.9(c) and (d). Nevertheless, pre-deformation by 40% along MD did not exhaust the energy absorption capacity of the fabric, as shown by the strain and stress concentrations coming from the impact point 125 μ s after the impact, Fig. 7.9(c) and (d), as well as by the re-orientation of the fibers along TD (point X) and MD (point Y) as a result of the impact, see Fig. 7.10(c) and (d). Thus, the reduction in the energy absorption capacity of the nonwoven fabric pre-deformed along MD was smaller. The experimental observations during experiments indicated that slippage of the projectile between the fibers was more difficult in this case (because of the isotropic mechanical response). Moreover, interaction of the projectile with the fiber network, which led to more extensive fiber pull-out during penetration, enhanced the energy dissipation capacity of the fabric.

Figure 7.10: Polar representation of the fiber ODF Ψ before and $125 \mu\text{s}$ after impact at 300 m/s in the specimens pre-deformed by 40% along TD and MD (a) Point X and (b) Point Y in Fig. 7.4 in the specimen pre-deformed along TD. (c) Point X and (d) Point Y in Fig. 7.4 in the specimen pre-deformed along MD.

7.2 Results and discussion

Figure 7.11: Parametrical study of the influence of the network affinity and connectivity on the ballistic response of the nonwoven. Affine and non-affine deformation are characterized by $\eta = 1$ and $\eta = 0.5$ in eq. (6.18), respectively. Isotropic and non-isotropic fiber connectivity are given by $f_f = \frac{1}{\pi} \frac{\rho}{\rho_f}$ and f_f in eq. (6.27), respectively.

7.2.3 Influence of affinity and connectivity on the impact response

The constitutive model presented above can be very useful to understand the influence of non-affine deformation and of anisotropic fiber connectivity on the ballistic performance of this material. To this end, the ballistic curve and the ballistic limit were computed for four different *virtual* fabrics with different affinity and/or fiber connectivity. The first one was an affine, isotropic nonwoven fabric, characterized by $\eta = 1$ in eq. (6.18) and isotropic fiber connectivity $f_f = \frac{1}{\pi} \frac{\rho}{\rho_f}$. The second material presented a non-affine, isotropic behavior, with $\eta = 0.5$ in eq. (6.18) and $f_f = \frac{1}{\pi} \frac{\rho}{\rho_f}$. The third nonwoven showed affine deformation and anisotropic connectivity given by $\eta = 1$ in eq. (6.18) and f_f in eq. (6.27) computed with the values of $\eta(\beta_0)$ shown in Fig. 6.3, while the fourth material presented non-affine deformation with $\eta = 0.5$ in eq. (6.18) and anisotropic connectivity given by f_f in eq. (6.27).

The ballistic performance of the four nonwoven fabrics is plotted in Fig. 7.11. These results clearly show that non-affine deformation leads to a large improvement in the ballistic performance, while the fiber connectivity (either isotropic or anisotropic) had a negligible influence. The origin of these differences can be analyzed in the contour plots 125 μ s after the impact of the maximum principal logarithmic strain, ϵ_I , and of the Von Mises stress, σ_{VM} , in the virtual fabrics impacted at 300 m/s, Fig. 7.12. Wave propagation as well as the strain and stress fields presented radial symmetry from the impact point in the fabrics with isotropic connectivity, Figs. 7.12(a) and (b) and Figs. 7.12(e) and (f), while anisotropic connectivity led to a faster wave propagation along TD, Figs. 7.12(c) and (d) and 7.12(g) and (h). Overall, the effect of the isotropic or anisotropic fiber connectivity on the energy absorption capability can be assessed by comparing the contour plots in the first and the second row (for affine deformation) or in the third and fourth row (non-affine deformation). In both cases, the differences are minimum although fabrics with an anisotropic fiber connectivity (second and fourth row) seemed to present slightly higher stresses and strains than those with isotropic fiber connectivity (first and third row) and thus provide a marginal increase in energy dissipation. These results are in agreement with the ballistic curves in Fig. 7.11, which show a marginal improvement in the ballistic performance for anisotropic fabrics, which is of the same order as the scatter in the simulations due to the variability in the pull-out strength of each set of fibers.

On the contrary, changes in affinity led to large differences in the ballistic response and nonwoven fabrics which present non-affine deformation possess better ballistic properties both in terms of the ballistic limit and of the residual velocity after penetration (Fig. 7.11). The nonwoven fabrics with non-affine deformation (third and fourth rows in Fig. 7.12) showed much higher strains and similar stresses than those that underwent affine deformation (first and second rows in Fig. 7.12) and were able to absorb much more energy during the impact. These results are in perfect agreement with the effect of non-affine deformation in the mechanical response under in-plane deformation, see Chapter 6.

Figure 7.12: Contour plot of the maximum principal logarithmic strain, ϵ_I , and of the Von Mises stress, σ_{VM} , in the virtual fabrics impacted at 300 m/s 125 μ s after the impact. (a) and (b) Nonwoven fabric with affine deformation and isotropic connectivity. (c) and (d) Nonwoven fabric with affine deformation and anisotropic connectivity. (e) and (f) Nonwoven fabric with non-affine deformation and isotropic connectivity. (g) and (h) Nonwoven fabric with non-affine deformation and anisotropic connectivity.

7.3 Concluding remarks

The outstanding predictive capacity of the physically-based micromechanical model for mechanically-entangled nonwovens fiber networks was demonstrated. The model was able to capture the ballistic limit of the as-received and pre-deformed samples along different orientations (TD and MD). Moreover, the anisotropy of the nonwoven as well as the localization of damage around the impact point were adequately predicted. Nevertheless, the model tended to underestimate the residual velocity of the projectile above the ballistic limit because final penetration took place in these cases by fiber disentanglement, pull-out and fracture due to thermal softening (instead of fiber disentanglement and pull-out from the fabric) and this latter mechanism was not explicitly introduced in the model.

The physically-based constitutive model provided information about the strain and stress fields in the fabric during impact as well as about the evolution of the fiber orientation and the localization of damage. Energy was dissipated by the tensile deformation of the fabric around the impact point in an elliptical region whose boundaries were controlled by the wave propagation with different velocities along TD and MD. Final penetration of the fabric around the ballistic limit occurred by the disentanglement of the fiber from the fabric network around the impact point. Pre-deformation of the fabric around TD or MD led to a reduction in the ballistic performance, which was much more noticeable in the former case. The numerical simulations showed that pre-deformation along TD consumed a large fraction of the energy dissipation capacity of the fabric and reduced the efficiency of the fabric against ballistic impact. However, these effects were ameliorated in the case of pre-deformation along MD because deformation was non-affine and the active fiber fraction was smaller in this orientation. Thus, much larger pre-deformation along MD was necessary to exhaust the energy absorption capability of the nonwoven fabric.

The constitutive model was finally used to explore the influence of non-affine deformation and of the anisotropy in the fiber connectivity on the impact response. It was found that the presence of non-affine deformation enhanced the energy absorption capability of the nonwoven because the deformability increased while the load bearing capability did not change. The anisotropy in the fiber connectivity did not modify, however, the ballistic response of the material. This information was very useful to guide the microstructural design of nonwoven fabrics with improved properties.

CHAPTER 8

Conclusions and future work

8.1 Conclusions

The investigations carried out in this thesis have led to the following conclusions:

- A multiscale characterization strategy (which included macromechanical and micromechanical tests in quasi-static, dynamic and ballistic regimes) was designed and carried out to ascertain the deformation and failure micromechanisms of mechanically-entangled nonwoven fabrics. Various novel experimental techniques (such as pull-out tests of single fibers and fiber bundles, 2D wide angle X-ray diffraction and split-Hopkinson bar tests for soft materials) were successfully used for first time for the characterization of nonwoven fabrics.
- The activation sequence of the deformation micromechanisms was assessed and the failure micromechanisms were quantified. The initial deformation was transferred to the nonwoven fabric through the random and isotropic network of knots created by needlepunching, leading to the formation of an active fiber network. Uncurling and stretching of the active fibers was followed by fiber sliding and pull-out from the entanglement points. Most of the strength and energy dissipation was provided by the extraction of the active fibers from the knots and final fracture occurred by the total disentanglement of the fiber network in a given section at which the macroscopic deformation was localized. However, although the initial fiber orientation distribution

8.1 Conclusions

was isotropic, the mechanical properties (in terms of stiffness, strength and energy absorption) were highly anisotropic. Pull-out tests of multiple fibers different orientations showed that structure of the knots connected more fibers in the transverse direction as compared with the machine direction. The better fiber interconnection along the transverse direction led to a denser active fiber skeleton, enhancing the mechanical response. In terms of affinity, fabrics deformed along the transverse direction essentially displayed affine deformation –i.e. the macroscopic strain was directly transferred to the fibers by the surrounding fabric–, while fabrics deformed along the machine direction underwent non-affine deformation, and most of the macroscopic strain was not transferred to the fibers. The same deformation mechanisms were found under quasi-static and dynamic loading conditions, regardless of the strain rate.

- A physically-based constitutive model was developed to simulate the mechanical response of needle-punched nonwoven fabrics, based upon the experimental observations of the deformation and failure micromechanisms. The model accounted for the effects of non-affine deformation, anisotropic connectivity induced by the entanglement points, fiber uncurling and re-orientation as well as fiber disentanglement and pull-out from the knots. The model provided the constitutive response for a mesodomain of the fabric corresponding to the volume associated to a finite element and was divided in two blocks. The first one was the network model which established the relationship between the macroscopic deformation gradient and the microscopic response obtained by integrating the response of the fibers in the mesodomain. The second one was the fiber model, which took into account the deformation features of each set of fibers in the mesodomain, including non-affinity, uncurling, pull-out and disentanglement. As far as possible, a clear physical meaning was given to the model parameters, so they can be identified by means of independent tests. The numerical simulations based on the model were in very good agreement with the experimental results of in-plane and ballistic mechanical response of the fabrics in terms of the macroscopic mechanical response and of the micromechanisms of deformation. Furthermore, it provided additional information about the influence of the microstructural features (fiber orientation, anisotropic fiber connectivity, affinity) on the mechanical performance of mechanically-entangled nonwoven fabrics.

8.2 Future work

The following topics for further investigation have been identified:

8.2.1 Experiments

- A deeper understanding of the actual fiber disentanglement process in the needle-points could be obtained in nonwoven fabrics with lower areal density in combination with more sophisticated experimental techniques such as *in situ* mechanical tests in an scanning electron microscope and/or an X-ray tomography scanner. This information will shed light in the physical processes responsible for the differences in fiber connectivity and affinity along the transverse and machine directions.
- A new design of the Split Hopkinson tensile bar is required for the characterization of the whole stress-strain curve in needle-punched nonwoven fabrics with very large failure strains. Higher pressure of the chamber combined with higher length and section of the bars should be enough to achieve the failure strain in one single pulse.
- Further studies on the evolution of the Poisson's coefficient in needle-punched nonwoven fabrics are required during in-plane deformation. The large strain gradients in the transverse direction found during in-plane tensile deformation did not agree with the plane strain theory and the Saint-Venant's principle. The differences may be due to out-of-plane buckling and wrinkling, but these mechanisms should be analyzed in more detail.

8.2.2 Modeling

- Implementation of a correct wave propagation algorithm that takes into account the differences in the wave propagation (scattering and dissipation) between a continuous medium and the actual network structure of the nonwoven fabric.
- Extension of the constitutive model to other nonwoven fabrics (engineering scaffolds, geotextiles, biological tissues, etc.) which also present complex deformation mechanisms, including anisotropic connective, non-affine deformation, etc.

APPENDIX A

Novel Split-Hopkinson tensile bar

To overcome the limitations of conventional Split-Hopkinson bars to accomplish the dynamic testing of the needle-punched nonwoven fabric (Chapter 4), a novel Split-Hopkinson Tensile Bar (SHTB) apparatus was designed. Main characteristics and theoretical background was exposed in previous Section 2.2.2 together with the state-of-art focused on the dynamic characterization of soft materials.

The main requirements of the novel SHTB were set as:

1. High sensitivity of the output bar to register the mechanical response of soft materials
2. High deformation of the specimen to reach the strain to failure with the minimum number of crossing pulses
3. Very long stress pulse (1 ms of duration) to achieve the equilibrium conditions
4. High initial sample size to characterize the fiber network response and avoid the influence of the material constituents

In order to achieve the proposed statements, important modifications of all the bars (loading, input and output) were carried and a whole description is included below. Minimum representative sample size of the specimen was also determined by means of quasi-static tensile tests.

Figure A.1: Configurations for the tensile Split-Hopkinson Bar apparatus. (a) Classic. (b) Non-conventional (Gerlach et al., 2012).

Modification of the loading bar

In a conventional SHTB system, see Fig. A.1(a), the striker tube sits around the loading bar and is accelerated by compressed air, moving along the input bar and hitting the impact flange, which generates the desired tensile stress pulse in the loading bar. The loading bar remains unsupported along the entire length between the impact flange and the beginning of the striker tube. This means that the stress wave travels through a sagging bar, resulting in low signal quality.

In the present experimental set-up the loading bar is surrounded by a striker with a U shaped cross-section, without being in contact with it, see Fig. A.1(b). The loading bar is supported along its entire length and thus prevented from bending. The striker is supported along its circumference through brass railings, and can freely slide along the length of the loading bar to strike the impact flange. The impact flange is supported through a circular bearing to prevent movement out from the longitudinal direction. A pulling rod is mounted above the striker bar connected to a hook. The piston sits within a low-pressure chamber towards the impact flange and, due to its relatively large cross section, it can be accelerated using low pressure compressed air. The piston pulls the pulling rod, which, via the hook accelerates the U shaped striker towards the impact flange generating the stress pulse. The SHTB is reloaded by manually moving the projectile (and thus also pulling the rod including the piston) back towards the input bar.

Figure A.2: Correlation between experimental and numerical strains in the SHTB configuration manufactured. (a) Pressure load *vs* time introduced in the system (b) Comparison between the experimental and numerical response of the gauges of the input bar.

For the current experiments a titanium loading bar of 3.6 m length and 20 mm diameter was used, surrounded by a striker with a U shaped cross-section of 35 mm diameter and 2.5 m length manufactured in Titanium as well. For further details it can be consulted the reference Gerlach et al. (2012).

Modification of the input and output bars

The design of the input and output bars was carried out comparing different configurations by numerical simulation. A Python script was implemented in the commercial software Abaqus/Explicit which generated different finite element models (FEM) of the Split-Hopkinson bar system modifying geometries, materials, pulses, etc. until finding the optimal solution. Numerical simulations have been carried out within the framework of dynamic deformations and large displacements and rotations.

The constitutive model developed in the previous Chapter 6 was used for the simulation of the mechanical behaviour of the nonwoven as a first approximation. The nonwoven fabric was discretized with M3D4R membrane elements, with a single integration point through the thickness. Regular mesh of $1 \times 1 \text{ mm}^2$ square elements was selected with a thickness of 1.5

mm. The rest of the components of the SHTB system (grips, input and output bar) were discretized with C3D8R solid elements with enhanced hourglass control of sizes varying from $1 \times 1 \text{ mm}^2$ to $5 \times 5 \text{ mm}^2$. The clamping system was also included in the simulation to analyze the impedance mismatch. Metallic material properties were described by a linear elastic constitutive model. The threads and the specimen attachments were simulated by tie constraints, and a general contact property with friction coefficient 0.2 was included involving the rest of the pieces. The bars were allowed to move just in the longitudinal direction and the load was introduced by a pressure pulse of magnitude 0.11 GPa in the input bar with a trapezoidal shape, see Fig. A.2(a), causing a similar response in both gauges of the input bar during the first pulse, see Fig. A.2(b).

Solid rods and hollow tubes for several materials (Copper, Steel, Magnesium, Aluminium and Titanium) were virtually compared, and for the given pulse, Titanium or high strength Aluminium hollow tubes resulted in the best configuration in terms of sensitivity and displacement. A scheme of the chosen experimental set-up is shown in Fig. A.3(a). Aluminium 7075-T6 alloy hollow tubes of 50.8 mm in outer diameter, 1.651 mm thickness and 2.7 m length were used, see Fig. A.4(a). Input bar was instrumented with two strain gauges at the distances 2.124 and 0.405 m from the specimen, and the output bar was instrumented with one strain gauge at the distance 0.472 m from the specimen.

A special conical clamping system for the testing of planar specimens based on previous designs of clamping grips for dry woven fabrics (Koh et al., 2010; Tan et al., 2008; Chocron et al., 1997) was manufactured, see Fig. A.3(b). The maximum admissible width of the samples was 35 mm. The pieces of the clamps were all manufactured in Aluminium except the threads which were manufactured in brass to avoid the welding of the pieces during the loading process, see Fig. A.4(b). A thread adaptor manufacture in steel was included to attach the loading and input bar, and bars were supported by 14 Aluminium and 14 teflon pieces. Planar samples were chosen to obtain the strain field by a 2D Digital Image Correlation DIC method.

Characterization of the minimum representative sample size

In order to accomplish the dynamic tests with the smallest admissible specimen, the minimum representative size of the nonwoven was determined. This parameter is critical

Figure A.3: New Split-Hopkinson tensile bar design. (a) General scheme composed by two hollow input and output bars. b) Detail of the conical grips for planar samples.

Figure A.4: Novel design of a Split-Hopkinson tensile bar. (a) Specimen between bars. (b) Detail of the interior pieces of the clamping system.

as the sample size determines the maximum strain and strain rates reachable during the tests. As a result, the smaller the specimen gauge length, the higher the strain and strain rate.

The influence of the gauge length in the mechanical properties was studied by means of quasi-static tensile tests. Samples were deformed following the procedure described in Section 2.2.2. Stress-strain curves for different gauge lengths are shown in Fig. A.5. Samples with gauge length lower than 30 mm presented a higher strength and stiffness

Figure A.5: Stress-strain curves for different specimen gauge length. Similar mechanical behaviour is found for samples with a gauge length higher than 30 mm.

as a consequence of a significant percentage of fiber breakage, which was identified due to little sudden drops of the force, leading to a non-smooth stress-strain curve at the onset of damage. Hence, samples of 40 mm gauge length were selected as the minimum representative sample size required for a dynamic characterization.

APPENDIX B

Continuum damage model for fiber pull-out

Fiber pull-out and disentanglement in the constitutive model, Chapter 6, was included using the Lemaitre continuum damage model (Lemaitre, 1996). The stiffness of the fiber is modified in terms of the damage variable d which controls the evolution of damage during an arbitrary loading path.

$$\sigma_f = (1 - d)\sigma_{trial} \quad (\text{B.1})$$

The onset and propagation of damage is controlled by the damage activation function F , which is expressed as

$$F = \phi - r \quad (\text{B.2})$$

where ϕ is the loading function, which depends on the applied stretch λ and is given by

$$\phi = \frac{K_{po}\eta(\beta_0)^3(\lambda - 1)^3}{\sigma_{po}} \quad (\text{B.3})$$

and r is the damage threshold, an internal variable of the model which is initially equal to 1. While $F < 0$, the fiber response is inelastic. Damage develops when $F = 0$ and $\dot{\phi} > 0$ and the evolution of damage is controlled by the consistency condition that establishes

$$\dot{F} = \dot{\phi} - \dot{r} = 0 \Rightarrow \dot{r} = \dot{\phi} \quad (\text{B.4})$$

Figure B.1: Differences between the computed softening by Equations B.6 and B.7. For a given value of $\phi > 1$, the Equation B.6 which follows a linear law leads to a higher softening (higher value of the damage variable d) than the Equation B.7 which follows a power law.

The evolution of the damage threshold r is linked to the damage variable d in equation (6.18) according to an exponential law given by

$$d = 1 - \frac{1}{r} \exp [A(1 - r)] \quad (\text{B.5})$$

where A is a numerical parameter that controls the energy dissipated during fracture. In order to ensure that the energy dissipated during fracture is independent on the size of the finite elements used in the discretization, A is a function of the characteristic length of the finite element, l_{ch} (which is equal to the diagonal length in the case of square elements) Bazant and Planas (1997). Formulation for linear-elastic materials is given by the expression:

$$A = \frac{2l_{ch}\sigma_{trial}^2}{2E_{po}G - l_{ch}\sigma_{trial}^2} \quad (\text{B.6})$$

being E_{po} the equivalent secant modulus and G the total amount of energy dissipated. However, applying equation (B.6) for non-linear elastic materials leads to wrong results, with an excessive softening during damage, see Fig. B.1. The softening parameter is finally

computed by the expression:

$$A = \frac{2\sqrt{l_{ch}^3}\sigma_{po}^2}{8\sqrt{K_{po}G^3} - \sqrt{l_{ch}^3}\sigma_{po}^2} \quad (\text{B.7})$$

where G is the energy dissipated by fiber pull-out according to equation (6.23).

A damage variable D was defined for each finite element (Gauss point), as the average damage of all the fiber sets in the element. Elements with $D > 0.99$ were removed from the simulations to avoid excessive element distortion.

APPENDIX C

Mesh objectivity

The effectiveness of the mesh regularization through the parameter A (presented in Appendix B) was checked by carrying out simulations of the quasi-static tensile tests presented in Chapter 4 along TD and MD orientations with different finite element discretizations. Three initial element sizes l (1, 2 and 4 mm) were used and the corresponding nominal stress - engineering strain curves are depicted in Figs. C.1 and C.2 for the samples deformed along TD and MD, respectively, together with the contour plot of the damage variable D at the onset of localization. The differences in terms of the nominal stress - strain curves were very small and the damage patterns were also equivalent, although the specific section at which damage developed depended on the discretization. It should be noticed that the computational cost of the simulations with the smallest elements was very large due to the larger number of elements and to the reduction in the time step. Moreover, finer meshes were more prone to give numerical problems due to excessive element distortion.

Figure C.1: Effect of element size l on numerical simulations along TD. (a) Nominal stress *vs.* engineering strain curve for different element sizes. (b) Contour plot of the damage variable D at 65% of deformation with $l = 4$ mm. (b) *Idem.* (c) $l = 2$ mm. (d) $l = 1$ mm.

Figure C.2: Effect of element size l on numerical simulations along MD. (a) Nominal stress *vs.* engineering strain curve for different element sizes. (b) Contour plot of the damage variable D at 120% of deformation with $l = 4$ mm. (b) *Idem.* (c) $l = 2$ mm. (d) $l = 1$ mm.

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