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In-situ X-ray study of the deformation mechanisms of non-woven polypropylene

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ABSTRACT

Non-wovens are of emerging research and industrial importance due to the characteristic high surface area, high porosity, high damage tolerance, and low cost. However strength and toughness are difficult to design for due to the structural irregularity and multitude of deformation mechanisms. Here we investigate the mechanical properties and deformation mechanisms of polypropylene non-wovens as a function of areal weight. Mechanical characterization showed that higher areal weight corresponds to equivalent stiffness and greater strength per weight compared to lower areal weight. Two x-ray studies were carried out with synchrotron radiation during uniaxial tensile testing: micro-computed tomography (3D microstructure) and x-ray diffraction (fiber orientation). These techniques combined to reveal that in compact non-wovens inter-fiber bonds provide strong constraints on the network structure and limit fiber rotation until the bonds fail. Conversely, in sparse non-wovens localized fibers align, plastically deform, and eventually fracture. These comprehensive combined mechanical response and microstructural data sets will serve to build microstructure-based constitutive models in future work.

1. Introduction

Non-wovens are fibrous materials manufactured without a weaving or knitting process. A typical non-woven consists of a set of fibers oriented according to a manufacturing process determined statistical distribution. Due to versatile manufacturing methods, high porosity and high impact absorption capabilities, non-woven materials constitute a rapidly growing portion of the textile industry, with demonstrated applications in a variety of fields including energy, water, ballistic protection, tissue engineering and medical disposables (Ahmed et al., 2015; Huang et al., 2003; Liu et al., 2013; Russell, 2006; Yoon et al., 2008). Non-woven mechanics are complex and still poorly understood due to the random nature of fiber distributions and their evolution under straining (Picu, 2011). A better understanding of the relationship between microstructure and macroscale mechanical behavior will improve and accelerate the non-woven design process.

The relationship of non-woven elasticity to microstructure and constituent material has been established with increasing fidelity over the last 60 years. In pioneering work conducted by Cox (Cox, 2002), the elastic properties of a fiber network consisting of long, straight, non-interacting fibers were established to depend on the initial fiber orientation, fiber cross-sectional area, and intrinsic fiber elastic modulus. This framework was later extended to 3D fiber orientation distributions (Narter et al., 1999). Later studies revealed that initial fiber curvature and fiber bending can both significantly influence elastic network properties (Pai et al., 2011a; 2011b; Shahsavari and Picu, 2012; Wilhelm and Frey, 2003), decreasing stiffness and increasing in-plane Poisson's ratio. Sparse networks with high fiber length to diameter aspect ratios tend toward bending-dominated rather than stretching-dominated elasticity (Shahsavari and Picu, 2012; Wilhelm and Frey, 2003).

The relationship of non-woven mechanical properties to microstructure and constituent material under large deformation remains challenging to establish due to the combination of fiber deformation, fiber orientation, and fiber-fiber contact change. Material strength depends on both specimen size and boundary conditions with long range correlations among stresses in the fibers (Hatami-Marbini and Picu, 2009; Kulachenko and Uesaka, 2012). Prior work on thermally-bonded polypropylene non-wovens demonstrated that bond damage leads to extensive fiber realignment. The speed of the realignment process results in

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strain rate dependent failure modes (Jubera et al., 2014; Ridruejo et al., 2011). In order to properly describe the large transverse strain observed in high aspect ratio non-wovens in uniaxial tensile tests, fiber elastic-plastic bending needed to be considered (Silberstein et al., 2012). Bond damage is the main damage mechanism in paper-like materials (Isaksson et al., 2006; 2004). Bronkhorst (Bronkhorst, 2003) studied the elastic-plastic behavior of a 2D fiber network and found that neglecting connectivity through the thickness caused a significant disparity with experiment results.

One promising avenue to understanding non-woven mechanics is to characterize microstructure change during deformation. Micro-computed tomography (µCT) generates a 3D image of a material and has been successfully applied to a variety of non-wovens such as paper (Isaksson et al., 2012), gas diffusion layers (Tötzke et al., 2014), and needle punched (Jeon et al., 2014) and point bonded non-wovens (Demirci et al., 2011). Changes in structural parameters, including pore size, fiber orientation and contact efficiency, have been computed. X-ray diffraction (XRD) experiments on fiber networks with crystalline fibers probe for information such as orientation distribution changes. The anisotropic fiber realignment behavior of a needle punched polyethylene non-woven was revealed through XRD in a recent work by Martínez-Hergueta et al. (2015).

The aim of this paper is to elucidate the deformation mechanisms of a series polypropylene (PP) non-wovens and explore the effects of areal weight on mechanical behavior. Through the application of synchrotron radiation, non-woven microstructure changes under uniaxial tensile loading were imaged. Structural information (number of bonds, volume fraction and fiber orientation distribution) was extracted to quantitatively describe damage progression and the fiber realignment process. Specifically, µCT was used to visualize the deformed material configurations at strains spanning elastic, pre-, and post-yield regions. It was observed that low areal weight non-woven had more damage than high areal weight counterparts at same strain. Moreover, XRD experiments were used to continuously track local fiber orientation change in both monotonic tensile and stress relaxation tests. The kinematic constraints imposed by bonds were apparent through the limited fiber realignment in high areal weight non-wovens at small strains. The experimental results also indicated whether fiber bending was a significant deformation mode for each of the different areal weight non-wovens.

2. Experimental

2.1. Materials

The Dupont Typar geotextile series of non-wovens used in this investigation are composed of PP fibers with diameters ranging in size from 40 µm to 60 µm. The fibers are created by extrusion and stretched to induce a semi-crystalline structure. These fibers are randomly distributed and bonded into a sheet by applying simultaneous heat and pressure Han. The manufacturing process results in both isolated fibers and small fiber bundles within the material (Ridruejo et al., 2011). The three investigated non-wovens, SF20, SF32 and SF65, are differentiated by thickness, areal weight, and the fiber-filled volume fraction of the material (Table 1). All three quantities increase following the sequence: SF20, SF32, SF65. These materials essentially consist of layers of fibers, however most fibers do cross between layers over relatively short distances. SF20, SF32, and SF65 have approximately 7, 9, and 12 layers of fibers respectively.

Table 1
Material properties of the investigated non-wovens provided by the manufacturer Han, where $v_f = a_w/(t\rho_{fiber})$ and $\rho_{fiber} = 946$ kg/m^3.

Material Type	Thickness t (mm)	Areal weight a_w (g/m^2)	Volume fraction v_f
SF20	0.35	68	0.205
SF32	0.43	110	0.270
SF65	0.59	220	0.394

2.2. Characterization of mechanical behavior

A Zwick/Roell Z010 testing system was used to characterize the mechanical properties of the non-wovens and single PP fibers under uniaxial tension. The load was recorded using a 10 kN and a 20 N load cell for non-woven and fiber tests, respectively. Five specimens of each non-woven type were tested to capture specimen dependent variation. Rectangular non-woven specimens 45 mm in length and 9 mm in width were tested with the gauge length set to 27 mm. This gauge dimension was chosen to achieve uniaxial tension while keeping the width small enough to image with the µCT setup (explained in Section 2.3.1). A limited set of experiments were also performed on larger and smaller specimens with results provided in the supplementary materials. The thickness of each specimen was measured at three different locations with a micrometer to determine an average initial cross-sectional area. To create single fiber specimens, fibers were extracted from non-woven SF20 by tweezers and then glued on a cardboard template, spanning across a 15 mm cut-out region on the cardboard. The single fiber diameter was measured at nine locations under an optical microscope prior to testing. Force and crosshead displacement were converted to engineering stress and engineering strain by dividing by the initial cross-sectional area and the initial gauge length respectively. Specimens were elongated at a constant strain rate of 0.00588 s^{-1} until failure to determine the Young's modulus E, strength σ_f and failure strain ϵ_f. Cyclic tests were carried out at a strain rate of 0.00588 s^{-1} for loading, unloading, and reloading portions; strains at the start of the unloading steps range from 0.02 to 0.8. The unloading modulus was calculated from the first 0.02 strain of each unload. To investigate relaxation behavior, another set of specimens were stretched at a constant strain rate of 0.00588 s^{-1} to an engineering strain of 0.1 and held at constant strain for 10 min. A stretched exponential function (Williams and Watts, 1970) was fit to the normalized stress relaxation curve to obtain a characteristic relaxation time.

2.3. Synchrotron X-ray studies

X-ray studies were performed at the F3 beamline at the Cornell High Energy Synchrotron Source (CHESS). A Si(111) double crystals monochromator was used to tune the incident X-ray beam energy to 10 keV and 8 keV for the µCT and XRD experiments, respectively. The resulting energy bandwidth was on the order of 10^4. For the µCT imaging, the second Si(111) crystal was replaced with 5° miscut Si(111) crystal to increase the size of the incident beamspot on the specimen and allow for a larger imaging region. A custom-built dual actuator load frame (see supplementary material) was used at the F3 beamline for in-situ deformation throughout the X-ray experiments. The same 27 mm by 9 mm gauge section was used for all X-ray experiments as for the Zwick mechanical testing.

2.3.1. Micro-computed tomography

Changes in the 3D microstructure of the non-woven specimens, induced by deformation, were monitored using in-situ µCT imag-