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Test Method

Dynamic studies of polypropylene nonwovens in environmental scanning electron microscope

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Abstract

Environmental scanning electron microscopy (ESEM) provides new tools to examine the dynamic behaviour of various materials under different conditions. The dynamic experiments of water wetting, oil sorption and loading deformation of polypropylene (PP) nonwovens in the ESEM were studied in this paper. Water wetting tests were performed by controlling the temperature of the specimens and chamber pressure in favour of water condensation at 100% relative humidity. The wetting by oil was made using a micro-injector to add oil droplets onto specimens being observed. The ESEM observations revealed the contrast in the wetting behaviour of the PP nonwovens towards water and oil. Tensile testing experiments were performed in the ESEM using a tensile stage. The dynamic studies gave new insight into microscopic behaviour of PP nonwovens.

Keywords: Polypropylene; Dynamic; ESEM; Wetting; Tensile; Nonwoven

1. Introduction

The nonwoven industry is one of the fastest growing industries in the world [1]. Technological innovation and commercial development have been driving the industry into a sophisticated and diverse market with versatile products for a wide spectrum of applications in many industries, such as agricultural, automotive, building and construction, medical and hygiene, packaging, protective clothing, sportswear, transport, defence, leisure and safety. One of the most important factors fuelling the growth of nonwovens is the development and application of man-made fibres, dominated by polyolefins [2].

For these increasing applications, nonwoven products are specially engineered to create the structure that gives the product its characteristic properties. The characterization of a nonwoven material under varying or dynamic conditions is of importance in understanding how the particular structure of the material is engineered, and, therefore, how it relates to the properties of the product. Microscopy technology has provided the tools for the observation, analysis, and explanation of phenomena occurring at a micrometer scale of textile materials [3]. Scanning electron microscopes (SEMs) have long been important instruments in

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these studies. However, the high vacuum and the imaging process in SEMs impose special requirements for specimen preparation. Specimens that are not naturally conductive must be coated with a thin layer of a conductive material to bleed off any charge on the specimens imposed by the incident electron beam. One major disadvantage of the SEM is that it is normally not possible to examine wet specimens or dynamic processes of specimens in a wet state. Environmental scanning electron microscopy (ESEM) is a newer development in microscope technology, which is specifically suited to dynamic experimentation on the micron scale [4].

The Philips XL30 ESEM was used for the dynamic experiments of water wetting, oil sorption and tensile deformation of polypropylene (PP) nonwovens in this study.

2. Experimental

2.1. Materials

The materials used in this study were needle punched nonwovens made of PP staple fibre and melt blown nonwovens extruded from PP polymers. The details are listed in Table 1.

2.2. ESEM

ESEM represents several important advances in scanning electron microscopy as it is able to image uncoated and hydrated samples by means of a differential pumping system and a gaseous secondary electron detector (GSED).

A differential pumping system [5] allows the entire ESEM specimen chamber to be maintained at a specific gas pressure of up to 20 Torr, whilst it enables the electron gun and upper parts of the column to be held at high vacuum (10^{-6} to 10^{-7} Torr). Pressure limiting apertures allow the electron beam to pass through, but minimise the leakage of gases between zones pumped at different rates.

The ESEM has a unique GSED [6], which is based on the principle of gas ionization. When the primary electron beam hits the specimen, it causes the specimen to emit secondary electrons. The electrons are attracted to the positively charged detector electrode. As they travel through the gaseous environment, the electrons collide with the gas molecules in the chamber resulting in emission of more electrons and ionization of the gas molecules. This increase in the amount of electrons effectively amplifies the original secondary electron signal. The positively charged gas ions are attracted to the negatively biased specimen and offset charging effects. Therefore, an ESEM is able to examine wet, oily and outgassing samples without coating.

ESEM is specifically suited to dynamic experimentation on the micron scale and below. ESEM technology allows for dynamic experiments at a range of pressures, temperatures and under a variety of gases/fluids. Some accessories can also be added into an ESEM to expand its observation capacity. A micro-injector can be installed in the specimen chamber for applying liquids during examination. A specimen heating system is available and a mechanical testing stage can also be placed in the ESEM chamber. Dynamic experiments can be recorded on a video for further analysis using image software.

2.3. Dynamic experiments

2.3.1. Wetting by water

In the ESEM, specimens can be hydrated or dehydrated by controlling the temperature of the specimens and chamber pressure in favour of water condensation or evaporation at different relative humidity [7,8].

In water wetting experiments, the specimen was pre-cooled to approximately 4–6 °C in a fridge

Table 1
Polypropylene nonwovens

Material	PP stable fibre	PP resins
Process	Carded and needle punching (NP)	Melt blown (MB)
Weight (g/m^2)	300	300
Fibre (dtex)	3	Variable from 0.1 to 1
Finish	No	No