NOTE

Microporous Polypropylene Fibers Containing Poly(methylsilsesquioxane) Fillers. Interrelation Between Filler Particles and Fibrous Polypropylene Texture

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INTRODUCTION

We reported earlier on microporous polypropylene (PP) hollow fibers containing poly(methylsilsesquioxane) (PMSO) filler, which was prepared by stretching PP microtubes containing the filler.¹ The microporous structure is developed as follows: the PP phase is split at the periphery of the filler particles by stretching, forming minute voids at an early stage, which enlarge to long elliptical micropores by further stretching. Here, the filler particles are dispersed in the resultant fibrous PP texture, and the interrelation between the filler particles and the PP texture is important, especially in relation to practical application. Namely, the filler particles should be stable enough not to fall from the fibrous PP texture in practical uses. Accordingly, we investigated the interrelationship with the aid of scanning electron microscopy.

EXPERIMENTAL

Microporous PP Hollow Fiber

Microporous PP hollow fibers were prepared by stretching a PP microtube containing PMSO filler as reported elsewhere.¹ Here, the particle size and content of the

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filler were 0.3 μm and 65% for PP, respectively, and the stretching ratio was 5.

Scanning Electron Microscopy

The fibrous PP texture was observed with the aid of a scanning electron microscope, JSM-220 from JEOL Ltd. The accelerated voltage and the probe current were 15 kV and 5 mA, respectively. Pretreatment of each sample was carried out by using Au-ion sputtering for 3 min at 1.2 kV and 8–10 mA. The angles of the electron beam and the observation for the test piece (EB angle) were 90° and 30° (Fig. 1).



Figure 1 Observation methods.

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Figure 2 Scanning electron micrographs of inner and outer surfaces. EB angle, 90°.

RESULTS AND DISCUSSION

Figure 2 shows scanning electron micrographs of inner and outer surfaces of a microporous PP hollow fiber containing PMSO filler in the case of the electron beam angle = 90° .

There is a subtle difference between the fibrous PP textures of the inner and outer surfaces. Namely, there are many more unsplit parts of the fibrous PP texture in the outer surface than in the inner surface. Also, the pore size is smaller in the outer surface than in the inner surface. Probably, this comes from the difference of the conditions to prepare the PP microtube between both surfaces: the outer surface is in contact with a hot nozzle and the inner surface of the extruded microtube is faster than that of the inner surface. This means that a skin layer is more easily formed at the outer surface, which is probably less splittable than the inner surface.

Figure 3 shows scanning electron micrographs of the same surfaces shown in Figure 2 except that in this case the electron beam angle was $=30^{\circ}$. It is clear that the filler particles are well trapped in the fibrous PP texture and adhere closely to the PP fibrils. These patterns are quite different from those shown in Figure 2, in which the filler particles seem to fall from the surfaces. Also, the fibrous PP texture contains many more unsplit parts of the PP texture than at the inner surface.

Figure 4 shows scanning electron micrographs of a cross-section of the microporous PP hollow fiber in the cases of electron beam angles = 30° and 90° . The difference in the patterns is rather similar to that of the patterns for the inner surface.

As described above, it can be concluded that the filler particles are well trapped in the fibrous PP texture and adhere closely to both the surfaces, so the filler particles do not fall from the surfaces. In order to confirm this conclusion, a water permeation test was carried out by using a module containing the hollow fibers (effective



Figure 3 Scanning electron micrographs of inner and outer surfaces. EB angle, 30°.



Figure 4 Scanning electron micrographs of cross-section parallel to fiber axis.

surface area, 50 cm²). Water was permeated through the hollow fibers from the outer surface.¹ The initial portion of the permeated water (1 L) was evaporated and it was confirmed that the residue contained no Si component by the fluorescent X-ray method. Accordingly, it can be said that no filler particles fall off in practical use.

REFERENCES

 S. Nagō and Y. Mizutani, J. Appl. Polym. Sci., 53, 1579 (1994).