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〈研究論文(學術)〉

산소 저온 플라즈마 처리한 PET 직물의 열처리 온도가 감량에 미치는 영향

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Effect of Preheat-Treatment Temperature on Weight Loss of Poly(Ethylene Terephthalate) Fiber by Low-Temperature Oxygen Plasma Treatment

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요 약－열처리 온도를 각각 다르게 한 Poly(ethylene terephthalate)에 반응기 내의 처리 조건이 서로 다른 두 가지 플라즈마 장치를 사용하여 산소 저온 플라즈마 처리를 했다. 열처리 온도가 플라즈마 감량에 미치는 영향은 열처리 온도가 증가함에 따라 결정화도도 증가하지만 감량율은, 열처리 온도가 160℃까지는 감소하다가 160℃ 이상에서는 증가하는 경향을 보이며, 분산염료에 의한 Poly(ethylene terephthlate) 섬유의 염색특성과 유사한 거동을 나타냈다.

Abstract－Effect of low temperature oxygen plasma treatment on the weight loss of poly (ethylene terephthalate) fiber heat-treated at various temperatures was studied using two kinds of plasma apparatus. Investigation was done on the basis of the increased crystallinity up to about 160 ℃, above this temperature weight loss increased significantly with the increased crystallinity in spite of crystallinity increased according to the increased heat-set temperature. The weight loss showed a minimum at about 160 ℃ just like in dyeing of poly(ethylene terephthalate) fiber with disperse dye.

Introduction

Low temperature plasma is effective to modify surface characteristics of fiber and polymeric materials. It is well known that the treatment causes implantation, crossliking, polymerization and etching. Many studies have been already done with regard to wetting of the film and fabric[1], adhesion of film[2], weight loss by plasma treatment[3,4] and dyeing property of wool[5].

In this paper, the effect of crystallinity of poly (ethylene terephthalate) fibers heat-set at various temperature on the weight loss by low temperature oxygen plasma was studied using two kinds

of low temperature plasma apparatus.

Experiment

Materials

Poly(ethylene terephthalate)(PET) yarn of 75 denier composed of 36 filaments produced by Japan Ester Ltd. Co. was used as a material.

Heat-setting

The yarn was would on a stainless steel frame and then heat-treated at 120, 140, 160, 180, 200 and 220 ℃ for 10 min. After the treatment the fibers were washed with toluene, acetone and distilled water and finally dried in air at 60 ℃.

Apparatus for low-temperature plasma treatment

Two types of plasma apparatus were used, one is Samco Basic Plasma Kit BP-1 and the other is Yamato Plasma Reactor PR-501A. Low temperature oxygen plasma by glow discharge was generated inductively at a radio frequency of 13.56 MHz. Plasma treatment by BP-1 was done at a power of 20 W and at pressure of 0.1 Torr for 3 to 7 min. On the other hand the treatment by PR-501A was carried out at a power of 300 W and at pressure of 1 Torr for 3 to 7 min. After the treatments the fibers were dried on P_2O_5 for 72 hrs. and then average was obtained.

Crystallinity

The densities of PET fibers before and after plasma treatment were measured at 25 ℃ with density gradient tube of toluene and tetrachloromethane. Crystallinity of the fiber calculated according to the following formula :

$$1/d = X_c/d_c + (1-X_c)/d_a$$

where Xc is crystallinity of the fiber , d is density of the fiber measured and dc and da are the densities of the crystalline and amorphous PET fiber with values of 1.455 and 1.335 g/cm^3 respectively.

Results and Discussion

Weight loss of the heat-set PET fiber by low temperature plasma treatments using Samco Basic Plasma Kit BP-1 and Yamato Plasma Reactor PR-501A is shown in Fig.1 and 2. The weight

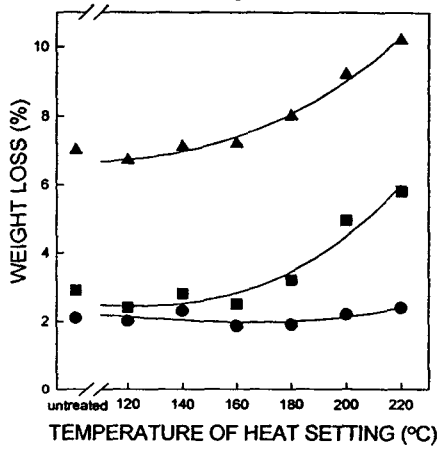

Fig. 1. Effect of preheat-treatment on weight loss of PET fiber by Samco Basic Plasma Kit BP-1, ●, 3 min ; ■, 5 min ; ▲, 7 min.

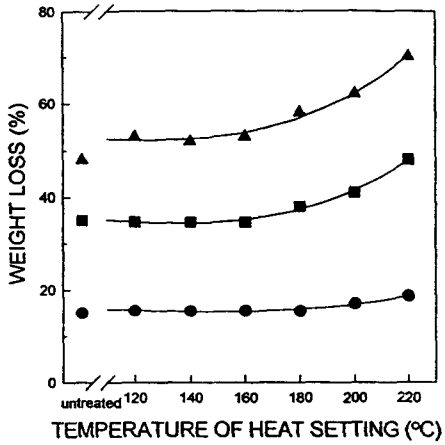

Fig. 2. Effect of preheat-treatment on weight loss of PET fiber by Yamato Plasma Reactor PR-501A, ●, 3 min ; ■, 5 min ; ▲, 7 min.

losses by both apparatus are very different. The treatment by PR-501A showed significantly higher weight loss than that of BP-1 because of high power and gas pressure of former apparatus.

The crystallinities of the PET fibers heat-set at 120 to 220 ℃ are shown in Table 1. They increased with the increased heat-setting temperatures. The crystallinities of the fibers treated by BP-1 have almost no change compared with those of the controls, whereas the treatment by PR-501A showed higher crystallinity than those by the controls. It is clear that etching by PR-501A proceeds at high temperature and leads to high weight loss and increase of the crystallinity.

Table 1. Changes of crystallinity of PET fiber treated with low temperature oxygen plasma

Temperature of heat setting	Before plasma	Crystallinity, % After plasma by BP-1	After plasma by PR-501A
Untreated	33.3	33.6	52.7
120℃	40.4	41.2	54.2
140℃	43.8	44.2	54.2
160℃	47.1	48.5	54.2
180℃	51.3	51.3	54.2
200℃	53.6	53.1	56.5
220℃	59.3	59.3	60.7

The curves of the weight loss by both plasma apparatus showed a minimum at about 160 ℃ independent of time of the plasma treatment and plasma apparatus. The weight loss decreased slightly up to about 160 ℃ with the increase of crystallinities, whereas above the heat-setting temperature of 160 ℃ the weight loss increased greatly with the increased crystallinity. In dyeing of heat-set PET fibers with disperse dye exhaustion decrease to about 160 ℃ and then increase with the increased crystallinity above the temperature(6). These behaviors are well reflected the fine structure of amorphous region that are related not only to the dyeing in water but also to the weight loss by plasma etching in dry process.

From these results it seems likely that fine structure of the surface and inside of the fiber similarly correspond to diffusion of dye into fiber, weight loss by plasma etching and chemical reactivity such as weight loss by alkali hydrolysis[7] aminolysis[8]. Thus, it is suggested that amorphous regions in the fiber surface are attacked first in plasma etching and fine structure of the fiber surface, especially fine structure of amorphous region, plays an important role for weight loss by the plasma etching.

Conclusions

Results from two kinds of plasma apparatus are as follows ;

Crystallinites of the PET fibers heat-set at 120 to 220 increase with the heat-setting temperatures.

The weight loss of PET fibers are increased crystallinity up to about 160 ℃ and increase significantly with the increased crystallinity above this temperature.

These behavior are similar to dyeing property of heat-set PET fiber with disperse dye.

The plasma apparatus PR-501A is the effect of heat-setting to PET fiber during plasma treatments at 120 to 180 ℃.

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