Effects of Heat Treatment on Tensile Properties of Diacetate Fibers

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ABSTRACT: Diacetate filaments were heat-treated (without tension or with tension) under dry-heat or wet-heat environment, respectively. The effects of temperature, time, and tension on tensile properties of diacetate fibers after heat treatments were discussed. The results show that diacetate fibers present no obvious improvement on its tensile properties after dry-heat treatments without tension. It was also found that during dry heat treatments with tension, the increase in tensile properties of fibers mainly depends on temperature and tension. Moreover, being dry-heat treated with tension instant after wet-heat treatment without tension, diacetate fibers exhibit a higher improvement on its tensile properties comparing with dry heat method with tension. The shrinking measurement for the fibers indicates different supermolecular structures were developed in the fiber before treatment and after treatment, which leads to the different extent in the improvement of tensile properties for the fibers. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 787–791, 2006

Key words: diacetate fibers; tensile properties; heat treatment; heat shrinking

INTRODUCTION

It is well known that cellulose from either cotton linters or wood pulp commercially used as starting materials for cellulose acetate is highly crystalline. However, the regular structure order of cellulose gradually disappears during the normal process of esterification. The basic reason is that the acetylation process followed by partial hydrolysis of the triacetate to a secondary acetate destroys the fine structure of the original cellulose.[1] As a result, diacetate fibers are of low crystallinity and low molecular orientation. In 1949, Work[2] studied the x-ray diffractions of cellulose acetate fibers and pointed out that because of the steric effect of random arrangement of side groups—acetyls group and hydroxyl group—along the molecule chain, the crystallinity of diacetate fibers was not increased even after annealing or stretching the yarn (with solvents such as dioxane-water), but the orientation of the already existing crystalline region was increased to some extent after annealing or stretching. He also pointed out that swelling agents, such as mixture of dioxane and water, provided the molecular chains more freedom and greater mobility, and therefore, molecular orientation of diacetate fibers was improved further after stretching the fibers with such solvents. After drawn, the tenacity of diacetate fibers increased while the elongation at break of the fibers decreased, but no detailed data have been reported. In addition, as early as in 1936, Henry Dreyfus[3] published a patent on manufacturing high-tenacity acetate fibers by high ratio stretching (drawn at least to 200% of its original length) and subsequent saponification process. However, this treatment is little practical for clothing acetate fibers. Another literature also described similar works.[4]

Methods for improving the strength of fibers usually involve increasing the molecular orientation, increasing the degree of crystallinity, improving the molecular tautness distribution, or some combination of these factors.[5,6] In this article, diacetate filaments were treated (without tension or with tension) under dry-heat and wet-heat conditions, the effects of temperature, time, and tension on tensile properties of fibers were discussed.

EXPERIMENTAL

Materials

Diacetate filaments of 173.3dtex/33F used in the experiments were produced by Nantong Cellulose Fibers Company Limited (China).

Dry-heat treatments

Diacetate filaments were dry-heated in an oven with tension or without tension at different temperatures

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TABLE I
Dry-Heat Treatment with Tension or without Tension

Time (min)	Without tension (°C)	With tension (°C)	
		20 g	50 g
15	100	100	100
	120	120	120
	140	140	140
	160	160	160
	180	180	180

for different time. The dry-heat treatments are summarized in Table I.

After heat treated in the dry air, the fibers were taken out from the oven and cooled for 5–10 min at room temperature with corresponding tension respectively, to maintain the heat treatment effects.

Wet heat treatments

The wet heat treatments of diacetate filaments were carried out as follows: diacetate filaments were treated without tension at temperature of 100°C and relative humidity of 90% for 30 min in WGDSH7015 oven, followed by dry-heat treatments as stated in Table II. The other steps were same as dry-heat treatments.

Tensile tests

Tensile properties tests of fibers were performed on a XQ-1 fiber tensile tester, using a gauge length of 20 mm and an extension rate of 20 mm/min.

Heat shrinking measurement

Samples: (1) untreated; (2) treated: being dry heated with a tension of 50 g at 160°C for 15 min.

The original length of each sample is 20 cm. Heat shrinking conditions without tension are shown in Table III.

The purpose of this measurement is to understand the effect of the heat treatments on the fiber structure.

TABLE II
Dry-Heat Treatment With Tension after Wet-heat Treatment

Tension(g)	Time (min)	Temperature (°C)
50	15	100
		120
		140
		150
		160

TABLE III
Heat Shrinking Conditions without Tension

Time (min)	Temperature (°C)
30	100
	120
	140
	160
	180

RESULTS AND DISCUSSION

Dry-heat treatment

Figures 1 and 2 are the stress–strain curves for the fibers after being dry heated with tension and without tension. It is seen that the tensile properties of diacetate fibers changed little after dry heat treatments without tension at different temperatures when compared with that of untreated fibers, while the tensile properties of diacetate fibers after being dry heated with tension show great difference from that of untreated fibers.

It can be seen from Figure 1 that, after dry heat treatment without tension, the stress–strain curves of treated fibers for different temperature are similar in shape and close to each other, below that of untreated fibers. Treated fibers show a more distinct yield behavior and their ductility increased with an increase of treatment temperature. It is also found that all curves for both treated and untreated fibers have an obvious yield point at their small deformation region.

Figure 2 shows the stress–strain curves for diacetate fibers heat treated with tension of 50 g. The shapes of three curves of 1, 2, and 3 are similar to curve of 0 for untreated fibers, each of them having a distinct yield point and the curve for higher temperatures above those for lower temperatures. This shows that the tensile properties of diacetate fibers changed more at a

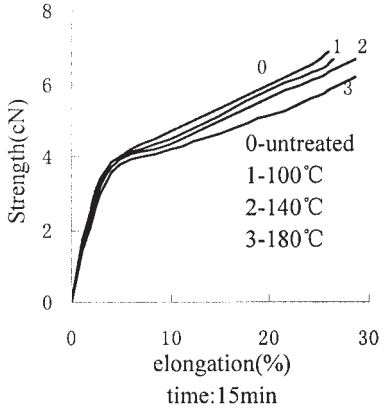

Figure 1 Strength-elongation curves for heat-treated fibers without tension.

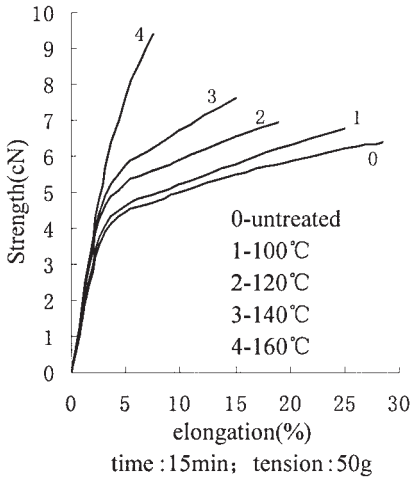

Figure 2 Strength-elongation curves for heat-treated fibers with tension.

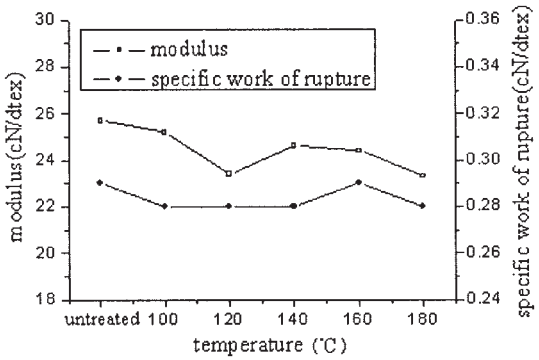

Figure 4 Modulus and specific work of rupture versus temperature (heat treatment without tension).

higher temperature. But the curve of 4 is quite different from the other 4 curves, having no yield point, which indicates that the tensile properties of diacetate fibers dry heated with tension of 50 g at 160°C changed significantly.

Again, Figures 3 and 4 show that the tensile properties of diacetate fibers changed little after dry heat treatments without tension at different temperatures compared with that of untreated fibers. The tenacity and modulus of treated fibers were slightly decreased while the elongation at break of fibers was a bit higher than that of untreated fibers. During the heat treatment without tension, fibers shrank lengthwise, and a higher temperature led to a higher shrinkage. In this way, the internal stress in the fibers was released with the decreasing of molecular orientation, which resulted from the motion of molecular chains in amorphous regions, and thus the elongation at break of heat treated fibers was increased. Additionally, because of the motion of molecular chains in amorphous regions, some smaller crystals might be disturbed while other larger crystals were formed so that the crystallinity and crystal size of fibers were increased and the tenacity and modulus of fibers were enhanced. However, the effect of decrease in molecular orientation on the tenacity and modulus of fibers took the leading place and therefore the tenacity and modulus of treated fibers were decreased.

Figures 5 and 6 represent that, in the case of a tension of 20 g, the tenacity and modulus of dry heated diacetate fibers were increased with an increase in the treatment temperature, while the elongation at break and specific work of rupture of fibers were decreased with an increase in the treatment temperature. Compared with untreated fibers, the increase in the tenacity of the fibers treated at 180°C is still less than 10%. This shows that the tension of 20 g is not sufficient to improve the tensile properties for diacetate fibers under the used temperature conditions. However, diacetate fibers might soften at temperature of 190–205°C[1] and will be damaged when temperature is too high.

In the case of heat treatment with a tension of 50 g (as shown in Figs. 7 and 8), the tenacity and modulus of treated fibers were increased obviously, especially at temperature higher than 140°C, and the elongation

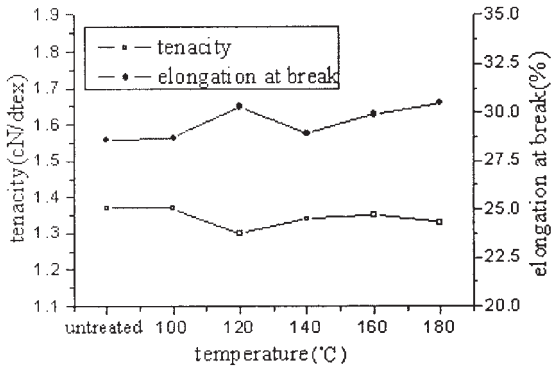

Figure 3 Tenacity and elongation at break versus temperature (heat treatment without tension).

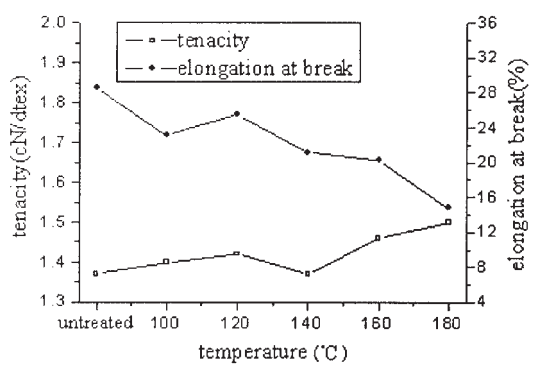

Figure 5 Tenacity and elongation at break versus temperature (dry heat treatment with tension, 20 g).

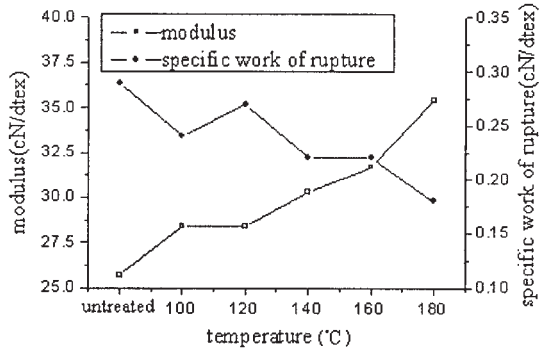

Figure 6 Modulus and specific work of rupture versus temperature (dry heat treatment with tension, 20 g).

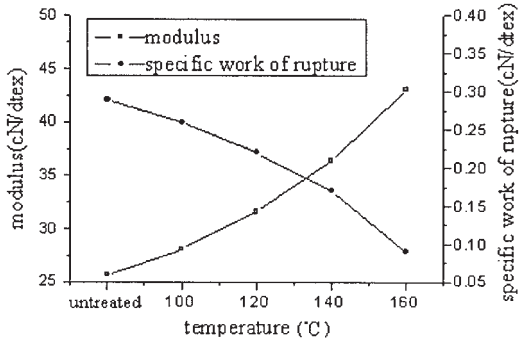

Figure 8 Modulus and specific work of rupture versus temperature (dry heat treatment with tension, 50 g).

at break and specific work of rupture of fibers were decreased with the rising of temperature. At temperature of 160°C, the tenacity and modulus of fibers were increased about 35 and 65% respectively, comparing with untreated fibers. Diacetate filaments broke at temperature of 180°C.

In the process of heat treatment with tension, the molecular chains is apt to orient in the direction of applied force, i.e., direction of fiber axis, which also accelerate the re-crystallization in the fibers. The higher the temperature and the greater the applied force, the higher the molecular chain orientation and more re-crystallization, resulting in higher tenacity and modulus for the fibers.

To investigate the influence of heat treatment time on the tensile properties of fibers, diacetate filaments were heated with tension of 50 g for 15, 30, 45, 60 min at temperature of 160°C. From Table IV, it can be observed that the tenacity and modulus of fibers were increased to some extent with increasing of treatment time, while the elongation at break and specific work of rupture of fibers were decreased.

Wet-heat treatments

It can be seen from Figures 9 and 10 that the tensile properties of fibers after wet heat treatment were improved, and it is clear that the wet-heat treatment is better than the dry-heat treatment for improving the tensile properties. The tenacity of wet heated fibers with tension of 50 g at temperature of 150°C is 35% higher than that of untreated fibers, equal to the increase rate obtained in the dry heat treatment with same tension at temperature of 160°C. Moreover, diacetate filaments broke within 15 min when temperature reached 160°C. The reason for the better improvement in the tensile properties of fibers after wet-heat treatment should be attributed to the presence of water molecules in the fiber, which weakens the intermolecular interaction of fiber molecules and thereby accelerates the rearrangement and orientation of fiber molecules along the applied force.

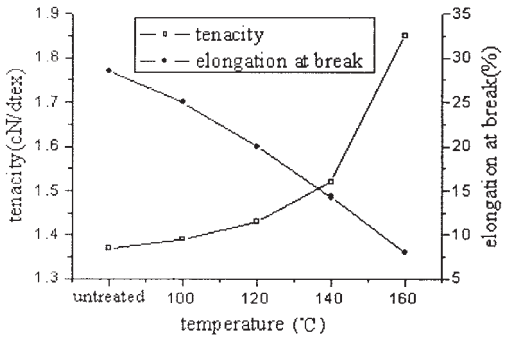

Figure 7 Tenacity and elongation at break versus temperature (dry heat treatment with tension, 50 g).

Heat shrinking

As shown in Figure 11, the heat shrinkage of treated fibers is less than that of untreated fibers when temperature is below 160°C, and the shrinkage of the fibers for both treated and untreated samples were increased with the increasing of temperature. During heat treatment from 100 to 180°C, the shrinkage change of untreated fibers is comparatively slow. While treated fibers were more stable than untreated fibers when temperature is below 160°C, its heat shrinkage was lower than that of untreated fibers. The heat shrinkage of treated fibers at 160°C is same as that

TABLE IV
Influence of Heat Treatment Time on the Tensile Properties of Fibers

Time (min)	Tenacity (cN/dtex)	Elongation at Break (%)	Modulus (cN/dtex)	Specific work of rupture (cN/dtex)
15	1.81	8.5	42	0.1
30	1.83	8.4	43.4	0.1
45	1.85	7.8	43.8	0.09
60	1.87	7.5	44.3	0.09

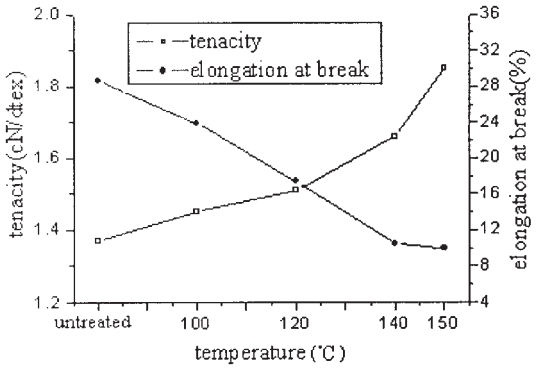

Figure 9 Tenacity and elongation at break versus temperature (wet heat treatment with tension, 50 g).

of untreated fibers; however, its shrinkage changing became fast and no more stable when temperature was above 160°C.

The shrinking behavior of the fibers demonstrates that different supermolecular structures were developed in the fiber before treatment and after treatment, which leads to the different extent in the improvement of tensile properties for the fibers.

CONCLUSIONS

From the results of our study, we can conclude that

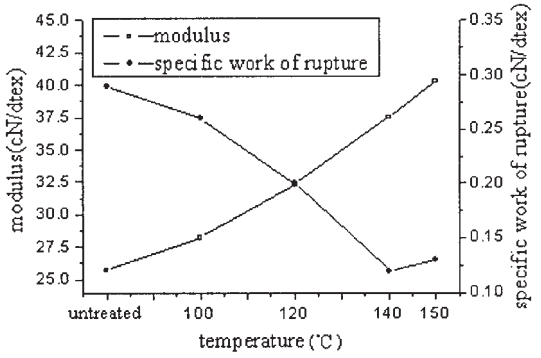

Figure 10 Modulus and specific work of rupture versus temperature (wet-heat treatment with tension, 50 g).

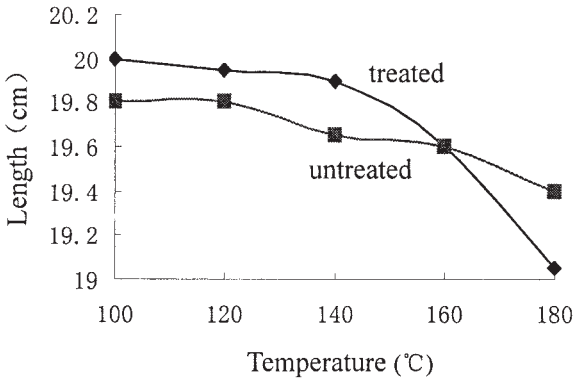

Figure 11 Heat shrinking.

1. The tensile properties of diacetate fibers change little after dry heat treatments without tension at different temperatures on comparing with that of untreated fibers, while the tensile properties of diacetate fibers after being dry heated with tension show great difference from that of untreated fibers.
2. The improvement in tensile properties of fibers with wet-heat treatment is better than that of the fibers without wet-heat treatment.
3. The shrinking behavior of the fibers demonstrates different supermolecular structures were developed in the fiber before treatment and after treatment, which leads to the different extent in the improvement of tensile properties for the fibers.

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