

Correlation Between Creep and Dyeability of Fibers

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Synopsis

The dye uptake and creep deformation of commercial and heat-set poly(ethylene terephthalate) and nylon 6 fibers have been studied. On heat-setting, both dye uptake and creep are seen to decrease in the case of PET but show an increase in the case of nylon 6 fibers. This indicates that the rate-controlling factor for both these processes may be the same, and it is suggested that this factor could be sample morphology, in particular, the size and distribution of the amorphous volume in the fiber.

INTRODUCTION

Creep deformation and dye penetration in textile fibers are both diffusion-controlled processes and may therefore be related to segmental mobility in the noncrystalline regions of the fiber. Thus, one can expect some degree of correlation between them, though hardly any direct experimental evidence has been reported which would bring this out.

The structural basis of creep in fibers is not well understood. It is generally believed¹ that increase in crystallinity lowers the creep deformation. It will, however, be shown in this paper that with an increase in crystalline content, creep can either increase or decrease. The structural factors controlling dye penetration are believed to be amorphous content and amorphous orientation.² It has been shown³ that for poly(ethylene terephthalate) these two factors are insufficient to explain the results and that the morphology of the sample, viz., the size and distribution of crystals, should also be taken into account. The simplest model which does this has been proposed by Dumbleton and co-workers.^{4,5} On this model, segmental mobility depends on the average amorphous content per crystal. The higher this quantity, the greater is the segmental mobility and therefore the higher the dye uptake. Prevorsek and his colleagues^{6,7} have correlated dye diffusion with morphology in the case of nylon 6 fibers on a more elaborate level, and dye uptake has been shown to be related to the degree of crystallinity, size of crystallites, orientation of the amorphous phase, etc.

The studies reported in this paper are on poly(ethylene terephthalate) and nylon 6 fibers. In each case, two samples were studied: the first was the commercial fiber which had not been heat-set, and the second was this fiber after being subjected to a suitable heat-setting treatment. The creep and dye uptake curves show similar trends, thus indicating that the structural factors controlling these two processes may be at least partly similar in nature. From the structural data on the present fibers and also on another

TABLE I
Structural Parameters for PET Samples

Sample	Amorphous content, %	Herman's orientation factor for amorphous phase	Amorphous volume per crystal, \AA^3
Parent	80	0.71	16.7×10^4
Heat-set	60	0.47	11.4×10^4

set of fibers on which dye uptake work has already been reported,³ this factor appears to be sample morphology, viz., the amount of amorphous material present and the way it is distributed in the fiber.

STUDIES ON POLY(ETHYLENE TEREPHTHALATE)

Detailed creep and dyeability studies were undertaken on a commercial multifilament PET yarn heat-set at constant length at 188°C for 20 min in a silicone oil bath. The heat-set sample was washed thoroughly in petroleum ether and dried. Parallel studies were made on the parent sample. Both the parent sample and the heat-set sample had approximately the same diameters.

The tensile creep was measured at room temperature and at 130°C. First, the sample was mechanically conditioned at appropriate temperature by subjecting it to repeated loading/unloading cycles till the recovery was complete and reproducible extension was obtained. The mechanically conditioned sample was rigidly clamped at one end and then suspended inside a tube which was maintained at constant temperature. A dead load was applied at the other end, and the extensions at different times between 10 sec and 2 hr were noted. The load was so selected that the strain was less than 3%.

The dye uptake studies were made using an anthraquinone-type disperse dye—Cibacet Torquoise Blue G. The dye bath concentration was 1 g/l., and the dyeing was done at 130°C using a material-to-liquor ratio of 1:1000. The

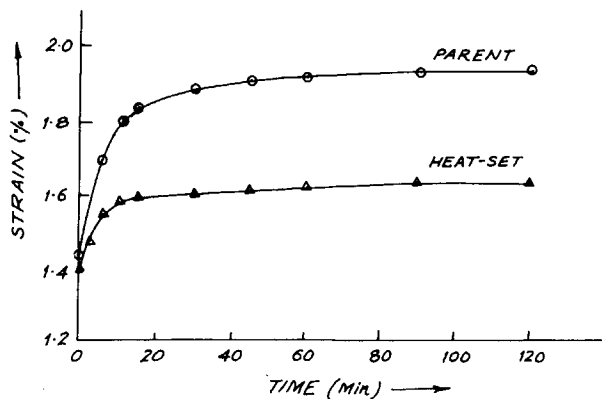


Fig. 1. Creep curves at room temperature for PET fibers (load = 100 g).

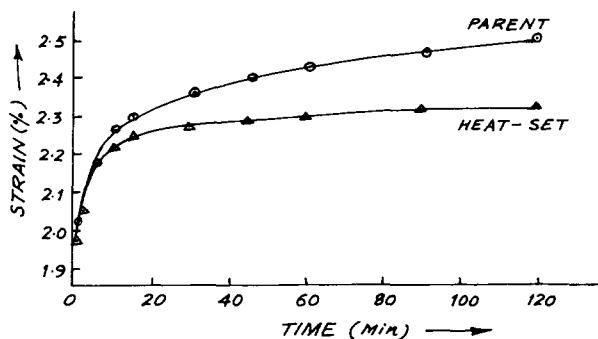


Fig. 2. Creep curves at 130°C for PET fibers (Load = 15 g).

dye uptake for times ranging from 30 min to 8 hr was estimated spectrophotometrically.

The amorphous content and amorphous orientation were determined by methods described elsewhere.⁸ The amorphous volume per crystal was determined using the method of Dumbleton and Murayama.⁴ It is given by $V(1-x)/x$, where V is the volume of a crystallite and $(1-x)$ is the amorphous content. The width of the crystallite normal to (100) plane was determined with the help of wide-angle x-ray diffraction. Then, assuming that the crystallite is a cube, the volume of the crystallite was determined and the amorphous volume per crystal obtained. It may, however, be noted that the measurement of crystal size by low-angle x-ray diffraction may result in values which are different to the ones obtained by wide-angle x-ray diffraction, as observed by Prevorsek et al.⁷ in the case of nylon 6 fibers. These structural parameters are listed in Table I.

The creep data at room temperature and at 130°C are shown in Figures 1 and 2. The dye uptake data are shown in Figure 3. It is interesting to note

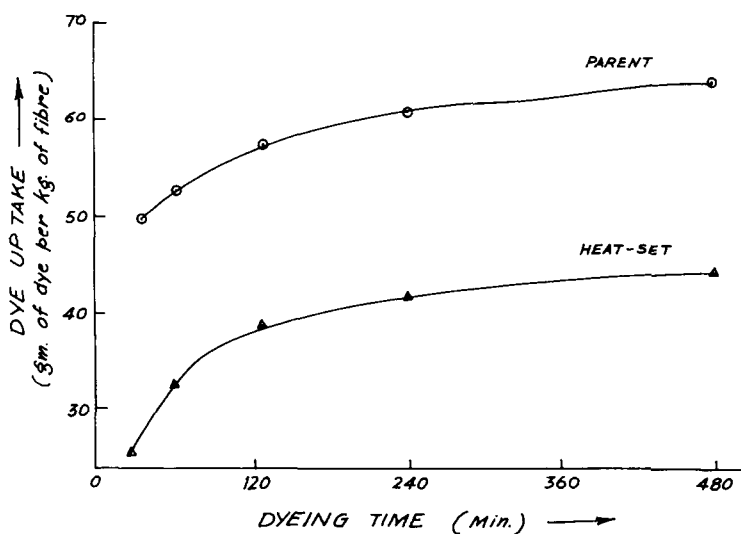


Fig. 3. Dye uptake curves for PET fibers (Dyeing temp. = 130°C).

TABLE II
Structural Parameters for Nylon 6 Fibres

Structural parameter	Creep samples		Dye-uptake samples	
	Parent	Heat-set	Parent	Heat-set
$1/\phi_{1/2}$	0.11	0.09	0.14	0.11
Percentage crystallinity index	7.70	25.70	7.50	33.40
Birefringence	0.055	0.056	0.056	0.054

that both creep deformation and dye uptake values for the parent sample are higher than for the heat-set sample. The parent sample has a relatively higher amorphous content and also higher amorphous orientation. The amorphous volume per crystal for the parent sample is also relatively higher. In another publication,³ the role of structural factors in controlling dye uptake is discussed in detail. It is shown there that dye uptake data cannot be explained on the basis of amorphous content and amorphous orientation alone. It is most satisfactorily explained on the basis of amorphous volume per crystal. Here, too, this parameter is higher for the cold-drawn sample, thus justifying the importance of this factor for dye uptake.

It would be interesting to study creep and dye uptake for samples heat-set at different temperatures and to examine if these follow the same trend. It is intended to undertake this work in the near future. However, it is tempting at this stage to postulate that creep could also possibly be affected considerably by the constraints imposed by the crystallites, and thus sample morphology might turn out to be an important factor in controlling it.

STUDIES ON NYLON 6

The nylon 6 samples for creep and dye uptake studies were prepared by different methods. The creep samples were commercial monofilaments which were heat-set in steam in the relaxed condition. The temperature during steaming was taken from 70°C to 100°C, and the total time for which setting was done was 90 min. For dye uptake studies, multifilament nylon 6 yarn was used. The heat-setting was done by running the yarn over heater plates maintained at 195°C, the contact time between the yarn and the plate being 2.28 sec. The pretension during heat-setting was 0.22 grams per denier.

The creep studies were made at room temperature only. The dyeing was done at 100°C using Cibacet Torquoise Blue G under infinite bath conditions.

Crystallite orientation in terms of reciprocal half-width ($1/\phi_{1/2}$), degree of crystallinity in terms of percentage crystallinity index, and birefringence was measured using methods described elsewhere.⁹ These structural parameters are listed in Table II.

From Table II it is interesting to note that, though the heat treatments for the two sets of samples for creep and dye uptake studies are very different, their effect on structure is essentially similar. For it may be noted that in both cases, on heat-setting of the parent sample (a) crystallite orientation

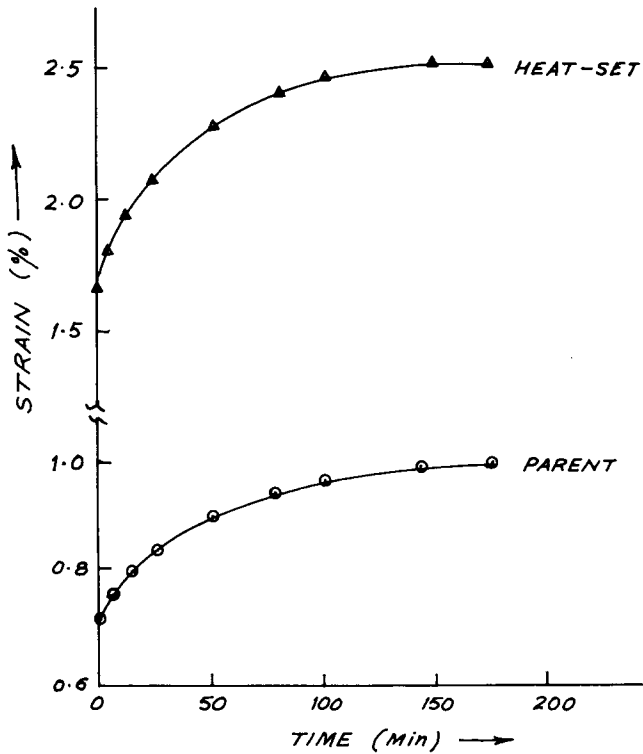


Fig. 4. Creep curves at room-temperature for nylon 6 fibers (Load = 4 g).

shows a slight decrease, (b) degree of crystallinity shows considerable increase, and (c) birefringence does not show any significant change.

The room-temperature creep data are presented in Figure 4, while the dye uptake data are shown in Figure 5. It is interesting to note that the heat-set samples, in spite of increased crystallinity, show higher creep deformation and higher dye uptake.

In the present case, the amorphous orientation and the amorphous volume per crystal could not be calculated because neither the degree of crystallinity

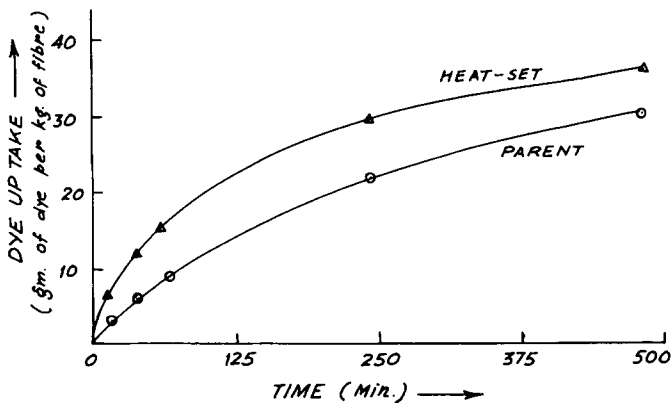


Fig. 5. Dye uptake curves for nylon 6 fibers (Dyeing temp. = 100°C).

nor the crystallite orientation was measured in absolute terms. However, it is generally agreed that heat-setting reduces amorphous orientation. Thus, in nylon 6 fibers, a decrease in amorphous orientation results in an increase in creep and dye uptake, whereas, as seen earlier, in PET the effect is just the opposite.

CONCLUSIONS

It has been shown that (a) increase in crystallinity results in decrease in creep and dye uptake of PET fiber while in the case of nylon 6 fibers the effect is opposite, and (b) decrease in amorphous orientation results in decrease in creep and dye uptake of PET fiber while in the case of nylon 6 fibers the effect is opposite. Since both creep and dye uptake follow the same trend for each set of fibers, the results suggest that the rate-controlling factor for these two processes may be the same. From structural studies, particularly on PET fiber, it is postulated that this factor could be sample morphology, in particular, the size and distribution of the amorphous volume in the fiber.

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