Oxidative stabilization of acrylic fibres: Part 4 Moisture sensitivity

Stabilized acrylic fibres are very hygroscopic. Dried fibres rapidly equilibrate under ambient conditions with a gain in moisture of about 8 wt % and concurrently elongate about 1.5%.

A number of accounts in the literature on producing carbon fibres allude to the moisture sensitivity of stablized acrylic fibres (see, e.g., [1] and [2]). Indeed, Kinoshita [3], uses the moisture regain at 25° C, 81% r.h. as a measure of the extent of stabilization: fibres with less than 5% regain are understabilized, those with more than 15% regain are overstabilized. But to date no detailed account of moisture regain has been presented. This paper reports such data and directs attention to some of the important consequences of the pronounced moisture sensitivity of these materials.

The equilibrium moisture regains (under ambient conditions) of four acrylic fibres heat treated in air at 255° C for various times while wrapped about a glass mandrel are shown in Fig. 1. Virgin acrylic fibres are seen to absorb 0.5 to 1.0% by weight of water, in agreement with previously published data. Well-stabilized fibres absorb 8% or more by weight. The most remarkable feature about Fig. 1 is the similarity in the shape of these curves to those of oxygen uptake [4, 5].

The dynamic moisture regain of well-stabilized Monsanto acrylic and Courtelle is shown in Fig. 2 (c.f. [5] for details on fibres). The data points were determined as follows: (1) Fibres were heat treated at 255° C in air while wrapped about a glass mandrel; (2) after 230 min of heat treatment time, the fibres were removed from the furnace

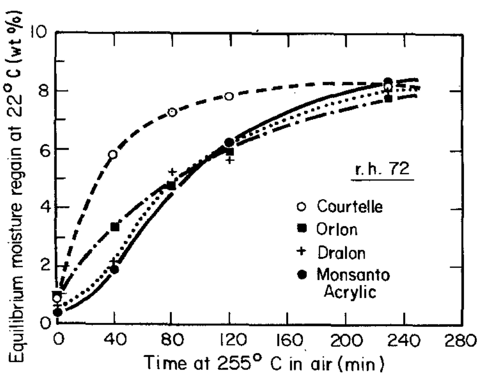

Figure 1 Equilibrium moisture regain at 22° C (wt %).

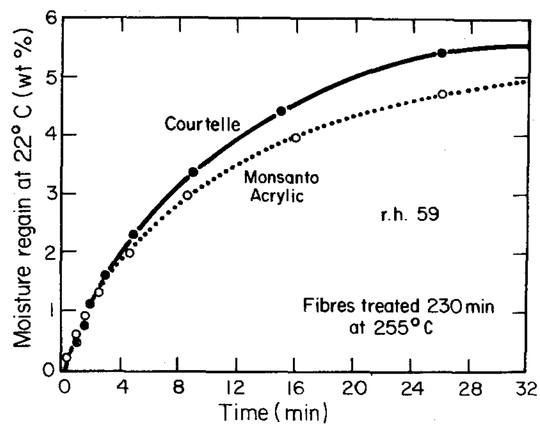

Figure 2 Moisture regain at 22° C (wt %).

and separated from the mandrel; (3) the fibres were reheated to about 150° C for several hours, a treatment which has been found to eliminate all but tenaciously bound water; and (4) the fibres were removed from the drying oven and immediately placed on a Mettler balance where weight was monitored as a function of elapsed time.

The data indicate that the uptake of moisture occurs with similar kinetics in stabilized material, independent of the precursor. Within just a few minutes of exposure to the ambient (in excess of 50% r.h.), the fibres absorb a considerable amount of water. This behaviour can be of major importance, for example, when analysing the fibres for oxygen.

Fig. 3 shows the effect of moisture content on the measured length of the stabilized fibre. The designation "Gas ON" indicates the time when the relative humidity of the environment around the fibres was changed from ambient to essentially zero by flowing dry forming gas (5% hydrogen; the balance, nitrogen) around the fibre. The designation "Gas OFF" indicates the time when the gas was shut off and ambient air was allowed to diffuse back into the vicinity of the fibre tow. The length changes were measured directly with a cathetometer; the fibres were subjected to a stress of 5 to 12 × 10^{-3} g denier^{-1} (0.5 to 1.2 MPa) during the experiment.

Clearly, the uptake of moisture is accompanied by expansion of the fibres, as is the case with cotton, wool and nylon at temperatures as high as 160° C. The data indicate the occurrence of another phenomenon which may have important

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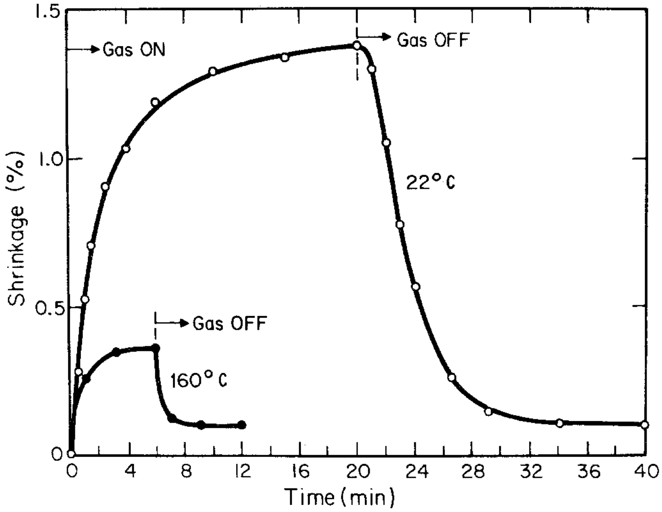

Figure 3 Effect of relative humidity on isothermal length of stabilized Monsanto acrylic at 22° and 160° C.

consequences in processing. As the temperature of a stabilized fibre is cycled about 90 to 100° C, the length of the fibre changes dramatically. Hence when a fibre is cooled from the reaction temperature, it expands in length. On a molecular level, the process may be envisioned as follows: polar groups which bind molecular segments in the polymer are solvated by the water, and Van der Waal's bonds are formed between the polymer and the water. In this way the polymer is allowed to relax or swell, leading to fibre extension. Any model of the stabilized material must, therefore, have extensive secondary bonding.

In summary, the strong secondary forces between molecular segments in stabilized acrylic fibres make the material very hygroscopic. Upon exposure to moisture the stabilized acrylic fibres rapidly equilibrate with a gain in moisture of about 8 wt% and concurrently elongate about 1.5%. As they exist in the laboratory, the stabilized fibres can therefore be regarded as plasticized material.

Acknowledgements

Financial support for the present work was provided by the Office of Naval Research. This support is gratefully acknowledged.

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Received 13 February and accepted 23 February 1979

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Au thin films with nm-sized pores made from Au–Ge eutectic alloy films

Microporous membranes and filters have been developed for use in various fields, e.g. medicine, drug manufacture, air pollution, and food industries. These devices are made from metals, ceramics, and polymeric materials. Desorbo and Cline [1] developed porous metal filters produced by selective etching of the rod phase of a directionally solidified eutectic alloy and a two-stage replica process used to produce porous thin films.

