DENSITY CHANGES IN CARBON FIBERS, INDUCED BY HOT STRETCHING

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INTRODUCTION

Substantial efforts over the years have been devoted to improving the mechanical properties of carbon fibers from a variety of source materials (such as PAN, rayon, pitch etc.) [1,2]. Commercially, PAN is now the most important precursor, and various studies have indicated that high temperature treatment (over 2500°C) can be used to increase the modulus, although unfortunately this is generally accompanied by a reduction in tensile strength [3,4]. More recent data have suggested that if, during the heat treatment, a load is applied to the fibers, then simultaneous increases in modulus and strength can be achieved [5]. Structural studies have linked the substantial improvements in modulus with significant increases in preferred orientation and the apparent crystallite stacking height of the carbon rafts, although no three dimensional crystallinity is observed [6].

On the other hand, there is relatively little information available concerning the density changes that occur during this type of processing. Some early work by Shindo [7] indicated that density of PAN-based fibers increased with heat treatment temperature in the absence of loading, and he put this down to crystallite growth. He reported density variations in the range of ~1950-2030kg.m^{-3} following heating at 2500°C and 3000°C respectively. A later study by Bacon [8] on graphitised rayon-based fibers suggested that the induced strain was the most significant factor affecting the density. A wide variation of densities was found, ranging from ~1320kg.m^{-3} for low modulus graphitised fibers to ~1936kg.m^{-3} for fibers with a Young's modulus of ~690GPa.

EXPERIMENTAL

An experimental PAN-based 3000 filament tow supplied by Courtaulds was the starting material for all the experiments reported. The tow had been heat treated to ~1300°C during manufacture, and fibers had a nominal diameter of 7μm, modulus ~180GPa and strength ~3.9GPa, as measured by single filament testing.

For the main series of experiments, twelve experimental tows were produced by stretching at four different temperatures (2700-3000°C) with three loads (5, 43 and 85MPa) for a time of 5 minutes. In order to test the time dependence, a second series of ten tows was made using a fixed stretching stress (79MPa), two temperatures (2600°C and 2800°C) and five different dwell times (2-30minutes).

Densities of the fibers were determined by the flotation technique in a mixture of tetrabromoethane and carbon tetrachloride. Mechanical properties were measured for at least eight fibers of each type, of length 27mm, using a specially designed apparatus for single fiber testing[5]. X-ray diffraction patterns of individual filaments were recorded using the synchrotron radiation source at EPSRC Daresbury Laboratory. The preferred orientation parameter (Z) was taken to be the full width at half maximum height of an azimuthal scan through the (00.2) reflection and the stacking height (L_c) was determined from a radial scan through the (00.2) [6].

RESULTS AND DISCUSSION

It may be seen from Figure 1 that increasing either the processing temperature or the stretching stress increases the density (c.f. an untreated figure of 1739kg.m^{-3}). In particular, at the highest temperatures (>~2850°C) and loads, as plastic deformation takes place, large increases in density occur. On the other hand, the time for which fibers are at the high temperature is much less important (Figure 2). Large density changes occur in the first few minutes but after ~5-10 minutes the densities show very little further increase. The best fit curves suggest an approximately logarithmic increase in density with dwell time.

Young's modulus also increases with the severity of the processing conditions, but it is not directly dependent on the density, since similar modulus values are achieved with quite different treatments and densities (Figure 3). On the other hand, a much closer relationship is apparent between the density and the microstructural parameters, Z and L_c, as shown in Figures 4 and 5 respectively.

CONCLUSIONS

The stretching of carbon fibers at high temperatures increases the density significantly. This density is strongly

dependent on stretching temperature and stress, but is less sensitive to the dwell time. It is also closely related to the microstructural parameters such as preferred orientation and apparent crystallite size.

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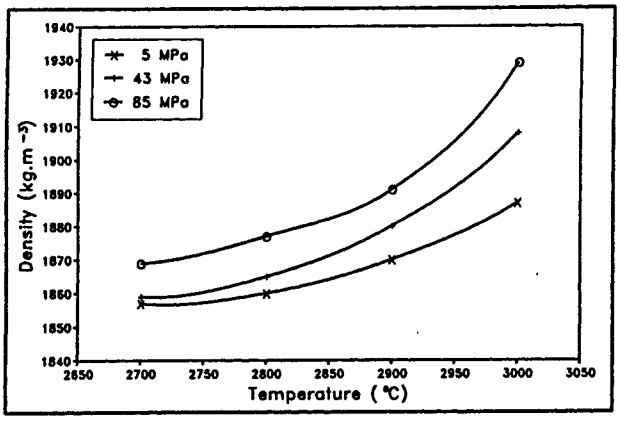

Figure 1 Fiber Density as a function of Processing Temperature

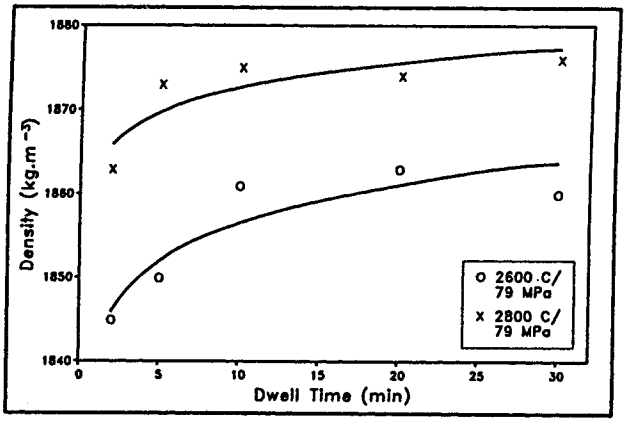

Figure 2 Fiber Density as a function of Processing Dwell Time

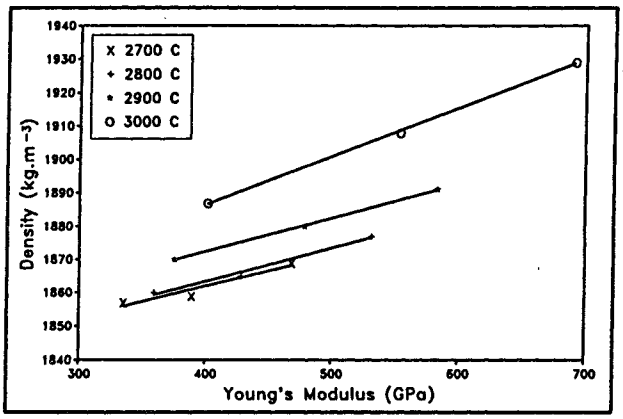

Figure 3 Fiber Density as a function of Young's Modulus

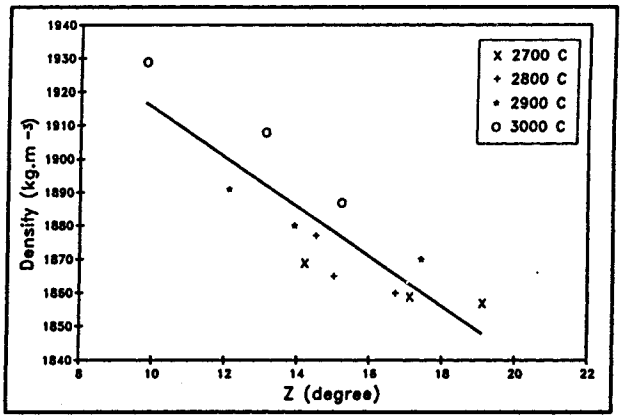

Figure 4 Fiber Density as a function of Preferred Orientation (Z)

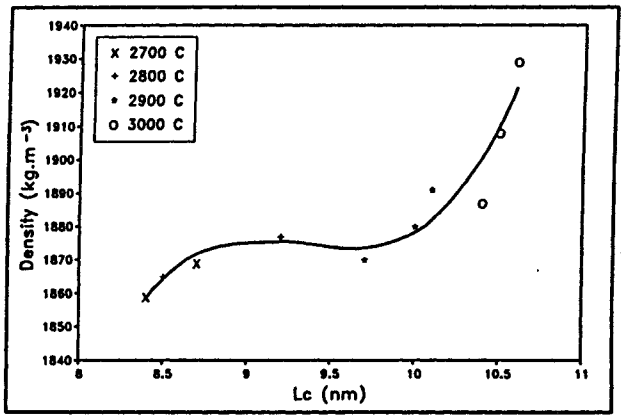

Figure 5 Fiber Density as a function of 'Apparent Crystallite Size' (L_C)