ON-LINE MEASUREMENTS DURING MELT SPINNING OF AR MESOPHASE PITCH

C. Lin, A. A. Ogale and D. D. Edie
Department of Chemical Engineering
Center for Advanced Engineering Fibers and Films
Clemson University, Clemson, SC 29634

Introduction

Mesophase pitch-based carbon fibers are one of the most promising materials for future structural and thermal-management applications because of their superior mechanical, electrical, and thermal properties [1-2]. These properties are directly related to the degree of preferred orientation of the graphene basal planes with respect to the fiber axis [3]. Thus, optimizing orientation and microstructure during fiber spinning is critical for improving specific fiber qualities while lowering processing costs. Mathematical modeling is a desirable way to study the complex process of fiber spinning; however, on-line experimental data are needed for validating model predictions and thus improving the model. Due to the weak and brittle nature of pitch fibers, on-line experimental data are not available in the literature. The objective of this work was to develop techniques for on-line measurements during melt spinning of AR mesophase pitch, including fiber temperature and diameter along the spinline.

Experimental

The synthetic naphthalene-based (AR) mesophase pitch used in this work was provided by Mitsubishi Gas Chemical Company. Before use, the pitch was dried overnight in a vacuum oven set at 150 ^{o}C.

Fiber spinning was carried out in a specially-designed capillary die (D=0.4 mm and L=1.2 mm) fitted in an Instron capillary rheometer (Model 2512-202, Instron Inc.). The spinning temperature was controlled by the furnace temperature; there was about a 40 ^{o}C difference between the furnace temperature and that at the die exit, since the die stuck out of the furnace about 3 mm. The fiber draw-down ratio (DDR) was varied by changing the take-up speed while maintaining a constant plunger speed.

Fiber surface temperatures along the spinline were measured using an IR camera (Model TH5104, Mikron Instrument Co., Inc.). Thermal images were taken and stored in a memory card, then processed using a thermal image processing software.

Because of the vibration of filaments during spinning, diameters measured from photography were not accurate. Therefore, a "frozen filament" approach was developed in which the winder was stopped suddenly and the extrudate was quenched and recovered from the spinline. Fiber diameters were then measured under a microscope with the aid of a Microcode indexer.

Results and Discussion

Figure 1 displays the fiber temperature profiles for two different DDRs at the same spinning temperature of 265 ^{o}C. The results indicate that the effect of DDR on the fiber temperature profiles was negligible. The higher DDR produces fibers with finer diameters that cool at a faster rate. However, this faster cooling is balanced by the shorter time that they were exposed to the ambient air. As a result, the fiber temperature profiles are not affected much by DDR. In both cases, the major temperature drop occurred within 10 mm of the die exit.

Figure 2 shows the fiber temperature profiles for two different spinning temperatures at a constant DDR of 12.1. As expected, the spinning temperature significantly affected the temperature profiles. A 10-20 ^{o}C downshift in temperature profile is observed for the case of the lower spinning temperature. Also, in both Figures 1 and 2, a shoulder is observed in the temperature profiles at a position between 3 and 10 mm of the die exit. This phenomenon was unexpected since AR mesophase pitch does not display any significant ΔH_{cryst} upon solidifying.

Figure 3 displays the fiber diameter profiles for two different DDRs at the same spinning temperature of 265 ^{o}C. Within 5 mm of the die exit, the diameter profiles were almost identical, but they gradually diverged as the fiber was drawn to different extents. Comparison of the final diameters reveals that the fiber produced at DDR=12.1 was about 37% thicker than that at DDR=26.1. A calculation based on the conservation of mass would indicate a 47% difference in fiber diameter for these two DDRs. Figure 4 shows the fiber diameter profiles for two different spinning temperatures at a constant DDR of 12.1. The diameter profiles were not affected much by the spinning temperature. An 8% difference in their final diameters was observed.

Conclusions

On-line measurements of temperature during melt spinning of AR mesophase pitch indicates that the DDR hardly affected the fiber temperature profiles, while the spinning temperature significantly affected the temperature profiles. Diameter measurements show that the spinning temperature did not affect much the diameter profiles, but the DDR affected greatly the diameter profiles The results can be used for model validation and simulation studies.

Acknowledgements

This work was supported by the ERC Program of the National Science Foundation under Award Number EEC-9731680.

References

1. Edie DD and Diefendorf RJ, "Carbon Fiber Manufacturing" in Carbon-Carbon Materials and Composites, J. D. Buckley and D. D. Edie, Eds., Noyes Publications, Park Ridge, NJ, 1993.
2. Edie DD and Mchugh JJ, "High Performance Carbon Fibers" in Carbon Materials for Advanced Technologies, T. D. Burchell, Ed., Elsevier Science Ltd., Oxford, UK, 1999.
3. Savage G, Carbon-Carbon composites, Chapman & Hall, London, 1993.

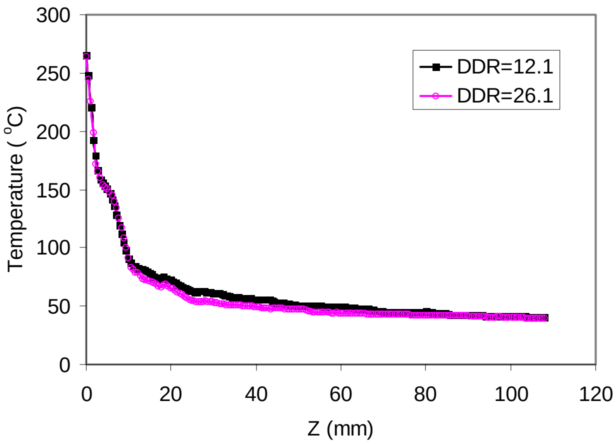

Figure 1. Effect of DDR on the fiber surface temperature profiles. Spinning temperature was 265 ^{o}C.

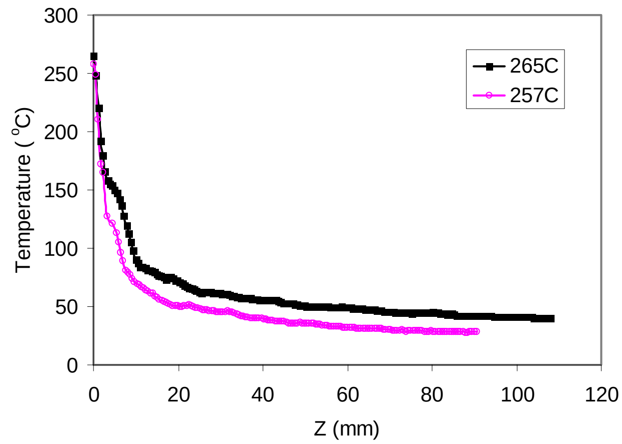

Figure 2. Effect of spinning temperature on the fiber surface temperature profiles. DDR was 12.1.

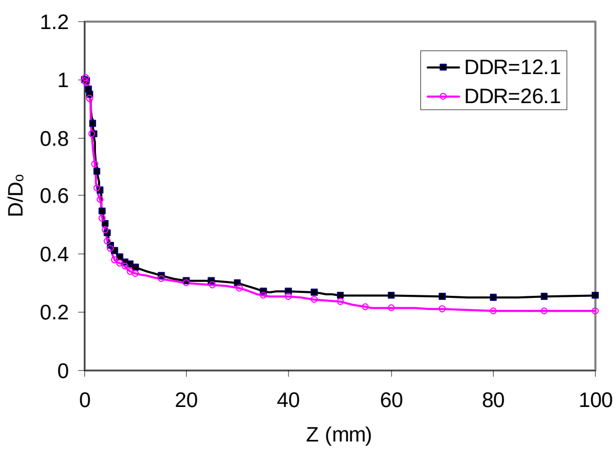

Figure 3. Effect of DDR on the fiber diameter profiles. Spinning temperature was 265 ^{o}C.

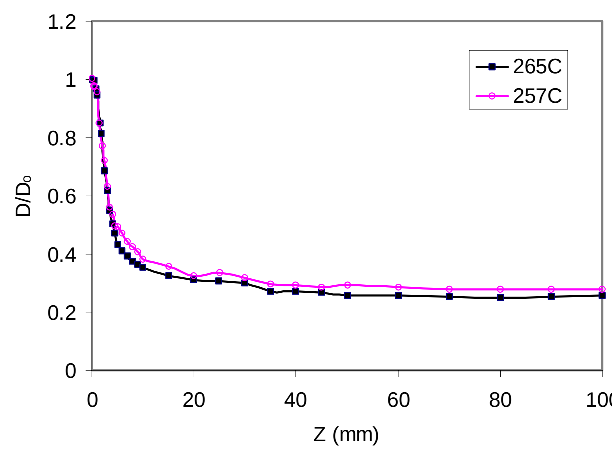

Figure 4. Effect of spinning temperature on the fiber diameter profiles. DDR was 12.1.