CARBON FIBERS FROM ALTERNATIVE PRECURSORS

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Introduction

Carbon fibers have excellent mechanical properties that, combined with their light weight, make them attractive for structural applications, ranging from aircraft structures to sporting goods. However, their high cost limit their use to specialty products [1]. High volume markets such as the automotive industry can be reached only if the fiber cost drops below $5/lb. Such a decrease would require fundamental changes in the precursor architecture coupled with novel fiber processing techniques. The present work investigates the possibility of using a melt spinnable PAN polymer as precursor for carbon fibers and proposes a new route for fiber stabilization.

Experimental

Materials

Fibers were obtained from two types of PAN precursors: a wet spun fiber, from a Mitsubishi polymer (control), and a melt spun fiber (experimental), from a polymer synthesized at the Virginia Tech and spun at Clemson University. Typical commercial PAN precursors are copolymers of acrylonitrile (AN) and methylacrylate (MA), with a nominal ratio of 94:6, whereas the experimental fiber had a AN/MA ratio of 85:15.

Fiber treatment

The experimental fibers were melt processed. Therefore, the fibers cannot be heat stabilized, as they melt before reaching the temperature of the exothermic reaction. Fiber crosslinking was attempted by UV irradiation. Two independent sets of control fibers were heat treated and UV irradiated for crosslinking. Carbonization was performed by heating the fibers up to 1500 °C at 10 °C/min, and holding them for 30 min at this temperature, under an argon purge. The conditions used for fiber stabilization are presented in Table 1.

Results and Discussion

FTIR microscopy was used to evaluate the extent of fiber stabilization. For the heat treated control fibers a decrease in nitrile concentration was observed, indicating the occurrence of cyclization; the spectrum exhibits broadening of the peaks in the 3000-3600 cm^{-1} and fingerprint regions, and this is consistent with crosslinking. For the UV irradiated control fibers, broadening of the peaks was detected for both polymers, but no decrease in nitrile groups was found. However, both types of fibers did not dissolve in DMSO after UV exposure, suggesting that the polymer was crosslinked.

The tensile properties of the fibers, before and after stabilization, and after carbonization, are presented in Table 2. A considerable decrease in fiber strain-to-failure is observed after UV irradiation. The relative decrease, however, is less than that observed for thermally stabilized fibers. As a consequence, the later present a brittle fracture, as can be observed in Figure 1 c). The control fibers that were carbonized after UV irradiation show some tendency to stick together (Figure 1 d)), and their mechanical properties are much lower than those obtained for the thermally stabilized carbon fibers. The experimental fibers partially melted during carbonization forming a fibrous aggregate, as shown in Figure 1 h).

Concluding Remarks

Fibers were produced from a melt processable PAN-based precursor. Both wet and melt spun fibers crosslinked when exposed to UV radiation. Suitable stabilization conditions were not yet achieved. The UV stabilized, wet-spun fibers could be carbonized, but their mechanical properties are not comparable to those for a fully stabilized (heat treated) fiber. Optimization of the stabilization process is under study. For both types of fibers, application of tension during UV exposure will be attempted. For the Experimental fibers, a post-drawing operation will be included to induce orientation, before UV irradiation.

References

1. Fitzer E. Carbon fibers-present state and future expectations. In Figueiredo JL, Bernardo CA, Baker RTK and Hüttinger KJ, editors. Carbon Fibers Filaments and Composites, New York: Springer-Verlag, 1989: 3-41.

Acknowledgments

This research is funded by the U.S. Department of Energy, under the contract no. 4500011036.

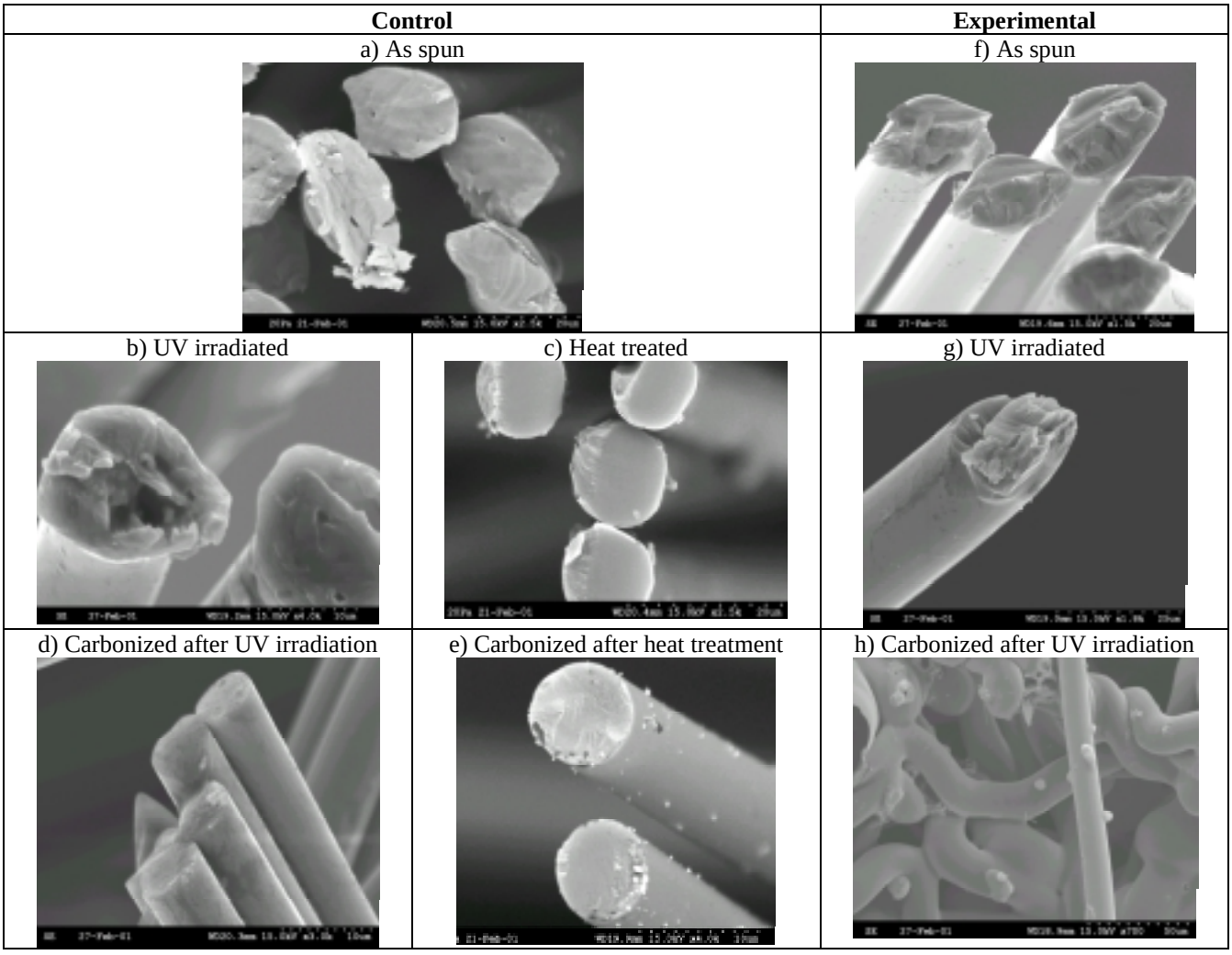

Figure 1. SEM micrographs of the control (AN/MA 94/60) and experimental (AN/MA 85/15) fibers as spun, after stabilization and after carbonization.

Table 1. Heat treatment and irradiation conditions used for fiber stabilization.

Fiber	Heat treatment	UV irradiation
Control	240 °C in air for 2 hours, tension	Exposed for 6 and 12 hours
Experimental	Unable to stabilize	Exposed for 24 hours

Table 2. Tensile properties of the fibers, obtained from single filament tensile testing, before and after stabilization, and after carbonization

	Diameter (μm)	Yield Strength (MPa)	Maximum Strength (MPa)	Modulus (GPa)	Strain-to-failure (%)
Control					
As spun	13.6 ± 0.9	116 ± 16	471 ± 66	5.0 ± 0.8	>68
Heat treated	12.5 ± 0.6	156 ± 20	308 ± 40	11.9 ± 1.5	8.8 ± 1.9
UV irradiated	13.4 ± 0.6	103 ± 10	185 ± 16	4.4 ± 0.5	16.5 ± 4.4
Carbonized after heat stabilization	8.6 ± 0.2	-	2200 ± 380	317 ± 9	0.68 ± 0.10
Carbonized after UV irradiation (6.5 h)	5.1 ± 0.2	-	523 ± 147	55 ± 9	1.0 ± 0.2
Carbonized after UV irradiation (12 h)	5.6 ± 0.5	-	761 ± 301	61 ± 25	1.4 ± 0.7
Experimental					
As spun	20.7 ± 1.3	120 ± 44	357 ± 72	3.9 ± 1.8	49 ± 10
UV irradiated	26 ± 9	85 ± 8	101 ± 22	3.2 ± 0.6	14 ± 12
Carbonized after UV irradiation*	-	-	-	-	-