

CARBON FIBERS FROM A PARTIALLY ORIENTED POLYVINYL ALCOHOL FIBER

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INTRODUCTION

Attempts were made to produce carbon fibers from polyvinyl alcohol (PVA) way back in 1960s by Shindo et al. [1]. In that process PVA fiber was heated in air for 16 hours to make it infusible so that the stabilized fiber could be heated to higher temperatures to produce carbon or graphite fibers. During heating, the PVA was dehydrated and oxidized with the possible formation of crosslinks making the fiber infusible [2]. The stabilized fibers were carbonized to 1000 °C and heated upto 2500 °C for graphitization. The carbon yield was only 32 % by weight compared to the theoretical yield of 54.5%

In the above process, there was very high shrinkage of fibers during the conversion step and the properties were poor. However, further studies where stabilization conditions were altered showed significant improvement in tensile properties [3]. It has been well known that the carbon fiber properties depend on the precursor properties. In this case, a PVA fiber produced from high molecular weight polymer by the gel spinning process that has very good mechanical properties is used to determine the effect of stabilization and carbonization.

EXPERIMENTAL

The fiber used in this study was an experimental fiber supplied by Allied Fibers. Heat treatment of the fibers was carried out using a tubular furnace with a ceramic tube [4]. Glass end caps cover the two sides of the ceramic tube. Gases to create the required environment in the heater were fed from both sides of the tube through the end caps. The heater temperature was controlled by an Omega CN2000 temperature controller. The precursor fiber loops were held by Kevlar leader at both ends and tensioned by hanging weights. They were then heated for varying lengths of time at different temperatures. The temperatures were raised in steps starting from 200 °C to an appropriate temperature to get stabilized fiber and upto 1175°C for carbonization. Whereas for stabilization air or ammonia/air environments were used, carbonization was done in an inert environment. Samples

treated under different conditions were collected and analyzed for their chemical and physical structure.

The precursor as well as the fibers treated under different conditions were characterized using thermal analyses (such as DSC and TGA), scanning electron microscopy, and physical properties. Thermal analyses were done using the Mettler thermal analysis system at heating rates of 20°C per min with either nitrogen or air as the purge gas.

RESULTS AND DISCUSSION

The DSC scans shown in Figure 1 show the effect of heat treatment on the PVA fibers. The precursor shows a distinct melting endotherm starting at 210 °C. On heat treatment of the fiber the DSC endotherm slowly disappears. The sample heated in air shows a very small endotherm. But the endotherm has completely disappeared for the sample heated in air/ammonia for the same time. Additional experiments showed that the endotherm disappears in much shorter heating time in the ammonia/air atmosphere indicating the faster reaction kinetics in the presence of ammonia.

The effect of stabilization is evident from the TGA data (Figure 2) as well. Whereas the precursor fiber lost all its mass by 500 °C, the samples heat treated in air showed weight retention of more than 25% even after 800 °C. Mass retention was much higher for samples heated for same time in ammonia/air. This was an evidence that the fiber could be carbonized. The result also indicated that unless the reactions are accelerated, it will take long time to get a stable fiber as observed by Shindo *et al.* It is important to note that the fibers treated in the presence of ammonia were shiny black colored, whereas samples heated in air alone were still turning to black color. The fibers that appear to be well stabilized will be carbonized and characterized.

CONCLUSION

The study showed that PVA fibers can be stabilized much faster using the ammonia/air mixture. Thus stabilized fiber could be carbonized with a reasonable yield. Where as the study has shown that a high molecular weight PVA fiber which was partially oriented did not shrink much during the conversion process, the conversion process and the properties of the fiber have to be thoroughly investigated.

REFERENCES

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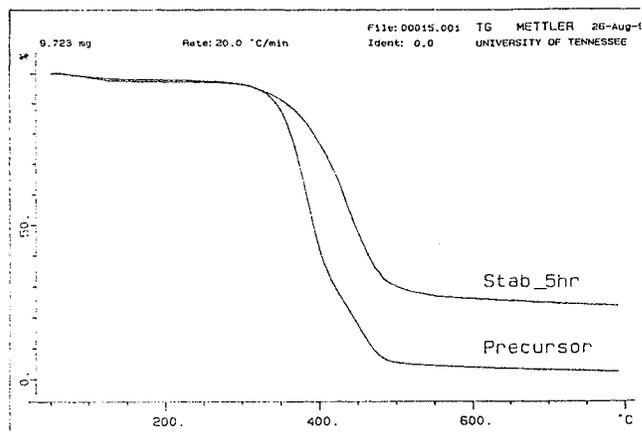


Figure 2. TGA of PVA Fiber Stabilized under Different Conditions (20 °C /min).

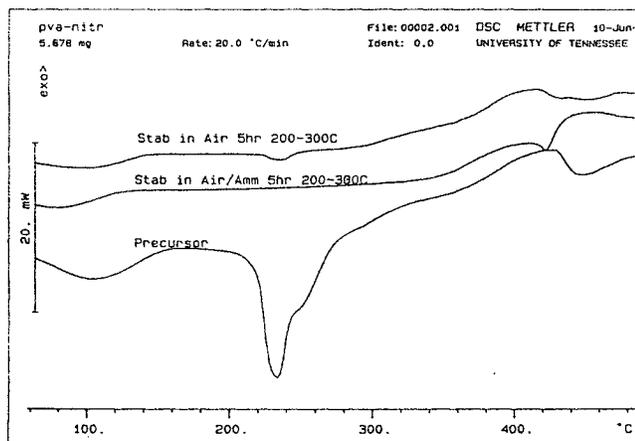


Figure 1. DSC of PVA Fiber Heated under Different Conditions (20 °C /min).