

CONVERSION OF A CO-POLYIMIDE FIBER INTO CARBON FIBER

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INTRODUCTION

Polyimide films have been carbonized and graphitized [1, 2] and are attractive as precursors because they carbonize with minimum change in shape. Polyimide structure is interesting because of simple release of non-carbon atoms without any remarkable disturbance in orientation during carbonization. The carbonized films can be converted to graphite with high crystallinity and orientation by high temperature treatment.

While several different polyimides are available as polymers or films, fiber formation from them has been very limited. Only a couple of the copolyimides have been spun into fibers, and one of them is the P84 which is a semi-commercial fiber produced by the Austrian company Lenzing AG [3]. Because of their excellent thermal and chemical properties, Lenzing P84 fibers are used in applications such as high temperature filtration, braided packings and protective clothing [4]. This fiber does not melt and has a high glass transition temperature ($T_g=315^\circ\text{C}$) [4]. Thermal characterization of this fiber indicated that it is possible to obtain carbon fiber from this precursor [5].

EXPERIMENTAL

The fiber used in this study was supplied by Lenzing USA. Heat treatment of the fibers was carried out using a tubular furnace with a ceramic tube. 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 temperature of the heater was controlled by an Omega CN2000 programmable temperature controller. The polyimide fiber loops held by Kevlar leader at both ends and tensioned by hanging weights were heated for varying lengths of time at different temperatures. The temperatures were raised in steps from 300°C till 560°C for stabilization and upto 1175°C for carbonization. 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), elemental analysis, 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. Elemental analysis was done at Atlantic Microlab, Atlanta, GA.

RESULTS AND DISCUSSION

The effect of heat treatment on the fibers can be seen by the DSC scans shown in Figure 1. P84 is a thermally stable fiber with no melting point and a very high glass transition temperature of around 315°C . In the vicinity of the reported glass transition temperature there seems to be an onset of reactions. Fibers treated at higher temperatures show an increase in the inflection point as well as reduction in the exothermic heat evolved which indicates an increase in thermal stability of the fiber. Also, on heat treatment, the golden colored starting material turns into black with intermediate colors in between depending on the time and temperature of the treatment.

The elemental composition of the fibers did not change significantly with initial heat treatment (Table 1). The carbon content was about 70 % for the precursor and it remained the same for all treated fibers. For samples treated in air, the oxygen content was slightly higher (by a couple of per cent) with a simultaneous decrease in hydrogen content compared to that of the samples heated in nitrogen. Fiber treated at 1100°C was about 90% carbon.

The TGA studies (Figure 2) show only a marginal change in weight retention of the heat treated samples compared to that of the precursor. The weight loss of the fiber treated to temperatures close to that of carbonization was really small. On treatment to temperatures close to or above 1000°C , the fiber had a very high carbon content with trace quantities of other elements indicating that it was essentially carbon fiber. The carbonized fiber showed weight loss around 100°C , due to the moisture present. Beyond that temperature, the loss in weight was less than five per cent. The SEM photograph of carbon fiber (Figure 3) shows a smooth surface and the trilobal cross section that comes from the precursor. However, on heat treatment the fibers became brittle, which is not a

desirable characteristic. This aspect needs to be investigated further.

CONCLUSION

The DSC scans of the polyimide fibers done in air and nitrogen showed the differences in reactivity with a sharper exotherm in air. Heating under carefully selected range of temperatures in the presence of air results in stabilized fibers as indicated by negligible exotherms. However, on heating in air the samples became stable and they could be successfully carbonized. Among the two environments investigated, air was more effective than nitrogen in getting a more stable fiber.

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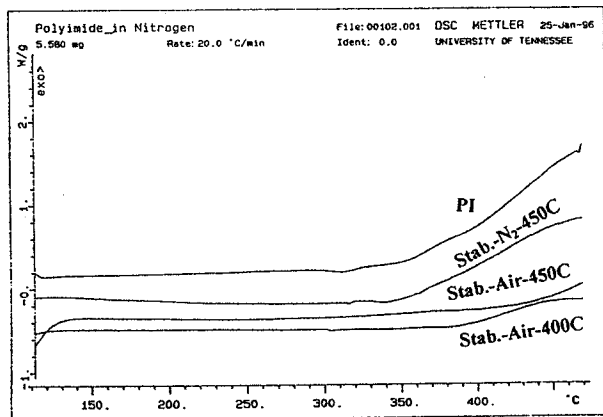


Figure 1. DSC of P84 Fiber Heated under Different Conditions (20°C/min).

Table 1. Elemental Composition

	C	H	N	O
Precursor	70.4	3.3	6.7	19.6
Stab. (N ₂)	71.3	3.2	6.6	18.9
Stab. (Air)	70.2	3.0	6.7	20.1
Carbonized (1000°C)	90.0	0.5	3.1	6.4

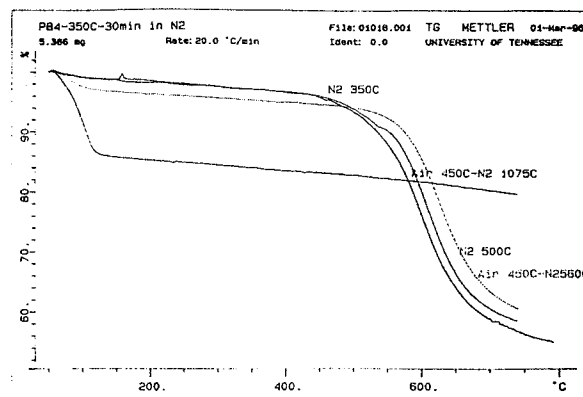


Figure 2. TGA of P84 Fiber Stabilized under Different Conditions (20°C/min).



Figure 3. SEM Photograph of Carbonized Fiber.