STRUCTURE INVESTIGATIONS FOR PyC SAMPLES FABRICATED AT SEVERAL FLUIDIZED BED AXIAL POSITIONS USING X-RAY DIFFRACTION

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Introduction

Silicon carbide alloyed isotropic pyrolytic carbons (PyC) which are deposited by means of chemical vapor deposition (CVD) in a bed of fluidized particles are the principle material used in the manufacture of mechanical heart valves. In an effort to explore the coating environment within the CVD reactor, a series of samples were produced at specific reactor locations.

As described elsewhere in these proceedings [1], the modulus, hardness, and density of these samples were measured. This work will present an examination of the microstructure of these samples using x-ray diffraction (XRD).

Since these PyC have a turbostratic microstructure they were by determining the out of plane coherence length L_C and the lattice spacing d from their x-ray diffraction profiles.

Experimental

To simplify the analysis of the XRD data, the samples examined were unalloyed. These samples are R1(run2) and R2(run1) from reference 1. The samples labeled R1 were taken at various heights along the coater's central axis and R2 samples were taken at a various heights along a fixed radial position near the coater's wall. From details of the sample preparation, the reader is referred to this reference.

The XRD was performed on a Siemens GADDS system equipped with an area detector. All data was collected using a copper K_α source (λ=1.5406Å) and a 0.3 mm pinhole collimator. All data was corrected for instrument broadening which was determined to be 0.22 degrees using a LaB_6 standard.

The out of plane coherence length sometimes referred to as crystallite domain size was calculated using the Scherrer equation:

$$L_C = \frac{0.89\lambda}{B\cos\theta}$$

where B is the broadening of the (002) diffraction line measured at half its maximum intensity, λ is x-ray wavelength, and θ is the position of the (002) diffraction line. The lattice spacing was determined by applying the Bragg equation

$$\lambda = 2d\sin\theta .$$

Results and Discussion

The calculated L_C and d-spacings are summarized in Tables 1 and 2 for samples R1(run2) and R2(run1), respectively. Figures 1 and 2 show a graphical comparison of these results

Figure 1 shows a decrease in L_C as a function of increasing z-position for samples taken along the central axis R1 while the opposite trend is clearly evident for the samples taken near the coater wall R2. Figure 2 shows very little change in the d-spacing for the R1 samples, but for the R2 samples the d-spacing increases with increasing z-position.

The number of coherently diffracting graphitic basal planes can be calculated by dividing L_C by d. The results are shown in Tables 1 and 2. The number of planes decreases with increasing z-position for the samples taken from the center axis of the coater R1. Along the wall of the reactor R2, the number of planes increase with increasing z-position.

Conclusion

The out of plane coherence length and lattice spacings were calculated from unalloyed PyC samples taken at various coater positions. From these calculations the number of coherently diffracting basal graphitic planes were calculated.

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References

1. Hofmann, G., et al in these proceedings.

Table 1

z (inches)	L_C (Å)	d (Å)	Layers
1.0	40	3.47	12
1.5	43	3.49	12
2.0	49	3.45	14
2.5	44	3.49	13
3.0	44	3.49	13
3.5	32	3.51	9
4.0	33	3.51	9
4.5	24	3.53	7
5.0	25	3.53	7

Table 2

z (inches)	L_C (Å)	d (Å)	Layers
4.25	45	3.46	13
4.75	48	3.45	14
5.25	49	3.45	14
5.75	51	3.45	15
6.25	51	3.46	15
6.75	60	3.45	17
7.25	67	3.44	18
7.75	63	3.44	19

Figure 1: L_c for Samples R1(run2) and R2(run1)

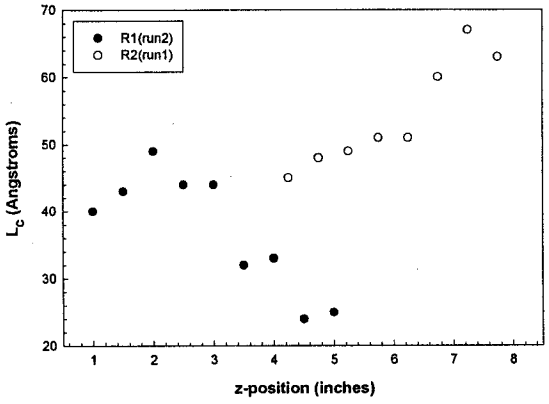

Figure 2: d-Spacing for Samples R1(run2) and R2(run1)