IN-24-CR

142710

P-20

PROGRESS REPORT
NAG-1-706

1 OCTOBER 1987 - 15 APRIL 1988

ABSTRACT

Differences in the adhesion of three carbon fibers (Hercules AS1 and AS4, and Hysol-Grafil XAS) to polycarbonate (PC) have been shown to correlate with the adsorptivity of PC on the three fiber types. The adsorptivity (energy of adsorption) was determine using retention time liquid chromatography and the adhesion was measure using the single embedded filament tensile test. A correlation was also found between adhesion strength and the O/N surface element ratio using XPS analysis. The chemical details for these correlations have not yet been determined.

A study of filament fracture statistics has been initiated using single and multiple embedded filament tensile tests. Filament fracture has been measured as a function of strain and for different interfiber distances. Preliminary results indicate that fiber fracture is a discontinuous function of increasing strain and may in fact occur at discrete strain intervals. Fiber-fiber interaction effects on fiber fracture have been found for interfiber distances of up to two to three fiber diameters.

Submitted by:

W. D. Bascom
Materials Science and Engineering Department
University of Utah
Salt Lake City, UT 84112

(NASA-CR-182890) SURFACE AND INTERFACIAL PROPERTIES OF CARBON FIBERS (Utah Univ.) 20 p CSCL 11D

N88-22951

Unclas
G3/24 0142710

SURFACE AND INTERFACIAL PROPERTIES OF CARBON FIBERS

NAG -1-706

TASK I

ADHESION TO THERMOPLASTICS

OBJECTIVE

DETERMINATION OF THE EFFECT OF CARBON FIBER SURFACE CHEMICAL AND SURFACE MECHANICAL PROPERTIES ON COMPOSITE PERFORMANCE; NOTABLY **DELAMINATION**

BACKGROUND

THREE COMMERCIAL CARBON FIBERS DIFFER IN THEIR ADHESION TO THERMOPLASTIC POLYMERS (POLYCARBONATE,ULTEM,PEEK,ETC)

AS1 (HERCULES) -----	LOW ADHESION
AS4 (HERCULES) -----	LOW ADHESION
XAS (HYSOL-GRAFIL) -	STRONG ADHESION

USUAL REASONS FOR DIFFERENCES IN ADHESION HAVE BEEN ELIMINATED:

- WEAK BOUNDARY LAYERS
- SURFACE ROUGHNESS
- WETTABILITY

APPROACH

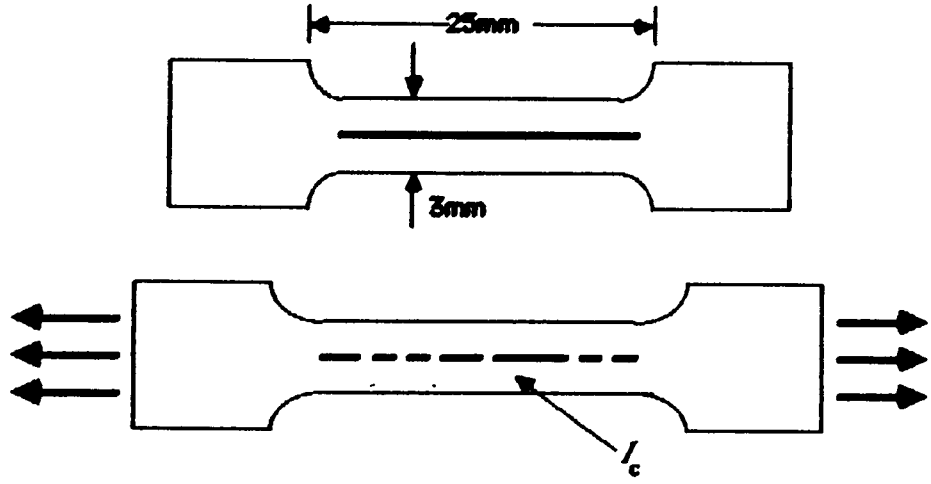
TEST FOR FIBER/MATRIX ADHESION

- SINGLE EMBEDDED FILAMENT TEST

DETERMINATION OF FIBER SURFACE PROPERTIES

- XPS
- ACID-BASE CHARACTER FROM INVERSE GAS PHASE CHROMATOGRAPHY
- RETENTION TIME CHROMATOGRAPHY

THE SINGLE EMBEDDED FILAMENT TEST



- the critical length (l_c) is related to the interphase shear strength by,

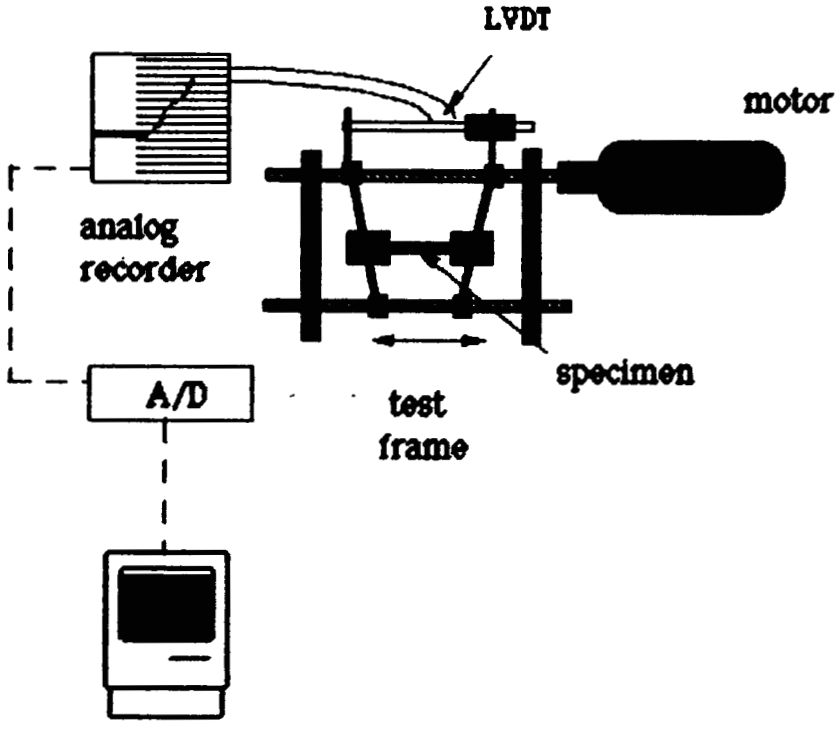
$$\tau_c = \frac{\sigma_c d}{2 l_c}$$

τ_c = interphase shear strength
σ_c = fiber strength
d = fiber diameter
l_c = fiber critical length

so that;

$$\tau_c = f\left(\frac{1}{l_c}\right)$$

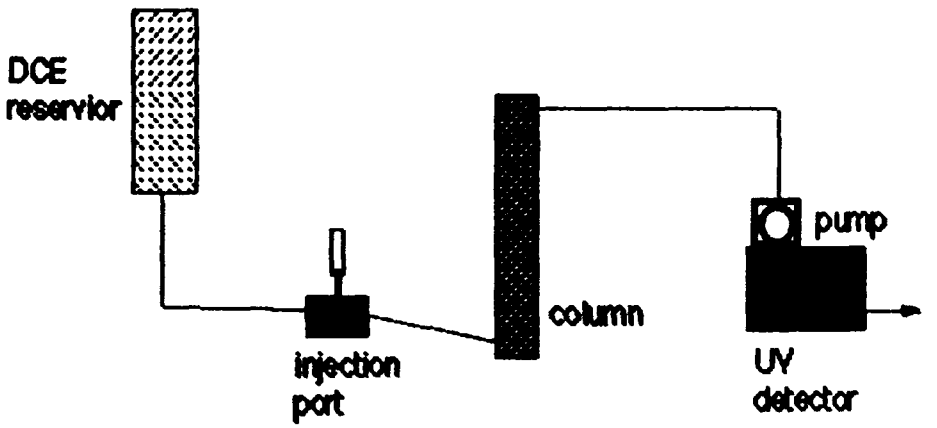
other factors being equal



APPARATUS FOR TENSILE TESTING OF EMBEDDED SINGLE FILAMENT SPECIMENS

- Motor Driven
- Strain Readout (LVDT)
- Computer Data Processing (to be installed)

RETENTION TIME LIQUID CHROMATOGRAPHY



Dichloroethane (DCE) Solution of Polycarbonate is Injected and the Retention Time Taken at the Absorption Maximum.
UV detector set at 265nm

RESULTS

ADHESION

- CRITICAL LENGTH DATA INDICATE DIFFERENT BOND STRENGTHS TO THERMOPLASTICS. FOR EXAMPLE POLYCARBONATE *VS* EPOXY
- THE THREE FIBERS HAVE ESSENTIALLY THE SAME BOND STRENGTH TO THE EPOXY BUT DIFFER IN THEIR ADHESION TO POLYCARBONATE

Critical Length Data

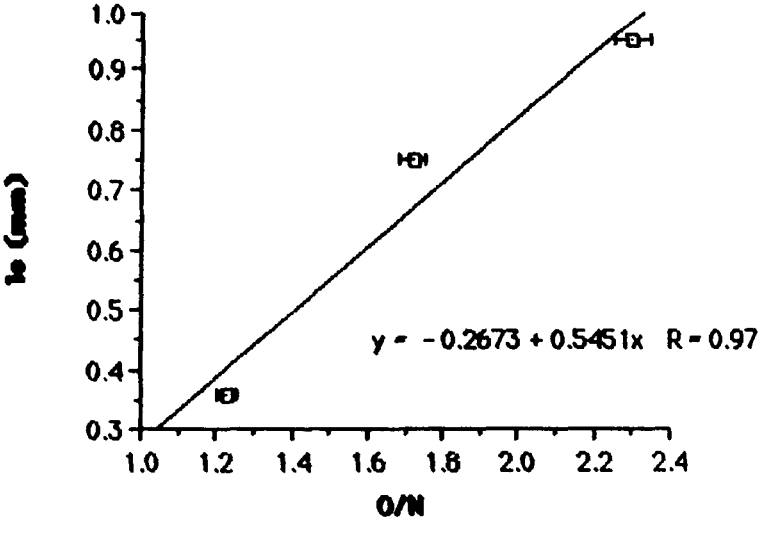
Matrix	Critical Lengths (mm)		
	AS1	AS4	XAS
epoxy (828/m-PDA)	0.30	0.38	0.21
polycarbonate	0.95	0.74	0.36

SURFACE ANALYSIS USING XPS

Surface Composition of Carbon Fibers From XPS Analysis

Carbon Fiber	Elemental Composition, %					
	C	O	N	Na	S	Cl
AS1	79.6	12.2	5.3	2.3	0.4	0.2
AS4	89.3	6.7	3.9	-	-	-
XAS	82.7	9.5	7.7	-	-	-

- NO CORRELATION BETWEEN ADHESION (I_c)AND ELEMENTAL COMPOSITION, HOWEVER:-
  - AN APPARENT CORRELATION WITH OXYGEN/NITROGEN (O/N) RATIO



ACID-BASE CHARACTER

- DETERMINED USING INVERSE GAS PHASE CHROMATOGRAPHY (PROF. TOM WARD, VIRGINIA TECH)
- AS4 MORE ACIDIC THAN XAS
  - AS1 TESTS IN PROGRESS
- BOTH FIBERS HAVE SIMILAR NONPOLAR CHARACTER

ACID -BASE ANALYSIS

probe molecule	character	I_{sp}(mJ/m^2)	
		AS4	XAS
$CHCl_3$	acidic	-	21.9
CCl_4	acidic	12.8	11.6
CH_3COCH_3	amphoteric	149	87.2
THF	basic	150	92.7
		$\gamma\ s^D$ (mJ/m^2)	
n-alkanes	nonpolar	40.0	39.3

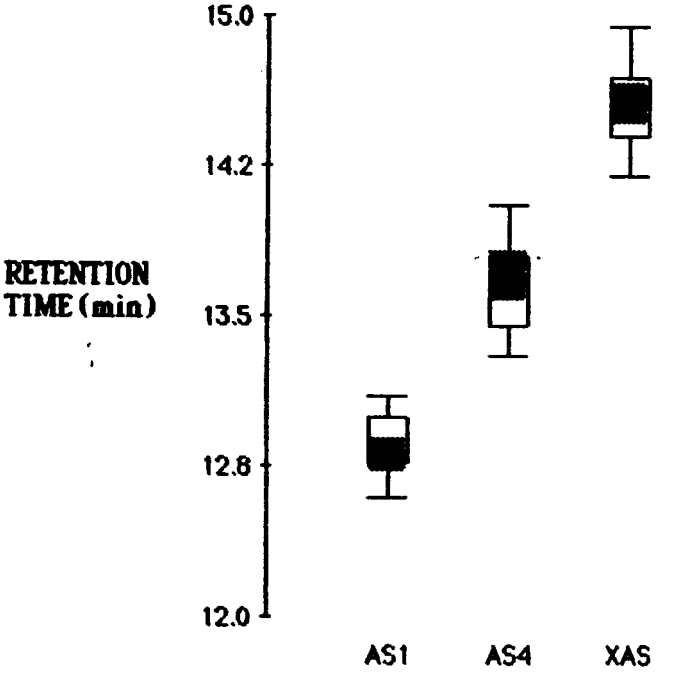
I_{sp} - free energy of interaction
$\gamma\ s^D$ - nonpolar surface energy

RETENTION TIME CHROMATOGRAPHY

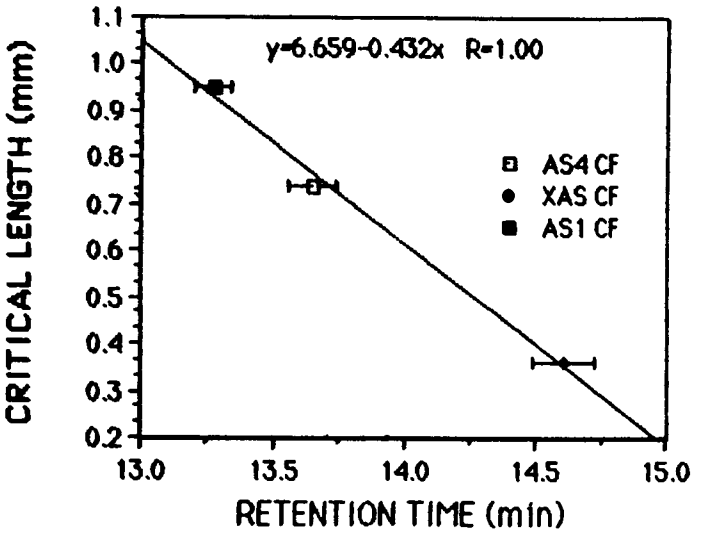
- SIGNIFICANT DIFFERENCE BETWEEN THREE FIBERS

- ABSORPTIVITY OF POLYCARBONATE :

XAS > AS4 > AS1



•STRONG CORRELATION BETWEEN RETENTION TIME AND ADHESION



CONCLUSIONS

- CARBON FIBER ADHESION TO POLYCARBONATE LINKED TO ADSORPTIVITY (ENERGY OF ADSORPTION).
- APPARENT CONNECTION WITH THE O/N RATIO OF SURFACE CHEMICAL SPECIE
- RETENTION TIME LIQUID CHROMATOGRAPHY A SENSITIVE TECHNIQUE FOR MEASURING POLYMER/FIBER CHEMICAL INTERACTIONS (ENERGY OF ADSORPTION)

FUTURE WORK

- LIQUID CHROMATOGRAPHY OF POLYMER ADSORPTION ON CARBON FIBER
  - RETENTION TIME *VS* CONCENTRATION ⇒ ADSORPTION ENERGY
  - REFURBISH CHROMATOGRAPH SUPPLIED BY NASA
  - TEST POLYAMIDEIMIDE (ULTEM), POLYPHENYLENE OXIDE AND POLYSULFONE
- RE-EXAMINE XPS DATA
  - DECONVOLUTE NITROGEN AND OXYGEN PEAKS FOR POSSIBLE DIFFERENCES BETWEEN FIBERS
- DETERMINE ACID-BASE CHARACTER OF FIBERS
  - AS1 DATA FROM VIRGINIA TECH
  - AN INVERSE GAS PHASE CHROMATOGRAPH WILL BE IN PLACE WITHIN 6 MONTHS (CENTER FOR BIOPOLYMER INTERFACES, UNIVERSITY OF UTAH)

- SURFACE MODIFICATION BY PLASMA TREATMENT
  - FACILITY AT BYU; DR. BRENT STRONG
  - CHANGE OXYGEN/NITROGEN RATIO
    - TREAT WITH O_2, NH_2, NO, CO_2, ARGON
    - MEASURE ADHESION
    - DETERMINE SURFACE CHEMICAL COMPOSITION USING XPS
- INTRODUCE OTHER FIBERS INTO PROGRAM, e.g. IM7

TASK II

FIBER FAILURE STATISTICS

OBJECTIVE

USE EMBEDDED FILAMENT TEST TO DETERMINE "IN-SITU" FIBER FAILURE STATISTICS AND FAILURE MICROMECHANICS UNDER LONGITUDINAL LOAD.

APPROACH

- EXPERIMENTAL TECHNIQUES HAVE BEEN DEVELOPED TO FABRICATE "MICROCOMPOSITE" SPECIMENS OF 1 TO 5 FILAMENTS
  - FILAMENTS ARE COPLANER AND THE INTERFIBER DISTANCE IS ADJUSTABLE
- EMBEDDED FILAMENT TEST TECHNIQUE OFFERS A VERY SIMPLE AND UNIQUE OPPORTUNITY TO DETERMINE;
  - STATISTICAL LAWS GOVERNING FIBER FRACTURE WITHIN A MATRIX POLYMER

THEORIES OF COMPOSITE LONGITUDINAL FAILURE AND OTHER FAILURE MODES ASSUME WEIBULL STATISTICS BASED ON SINGLE FILAMENT TESTING IN AIR

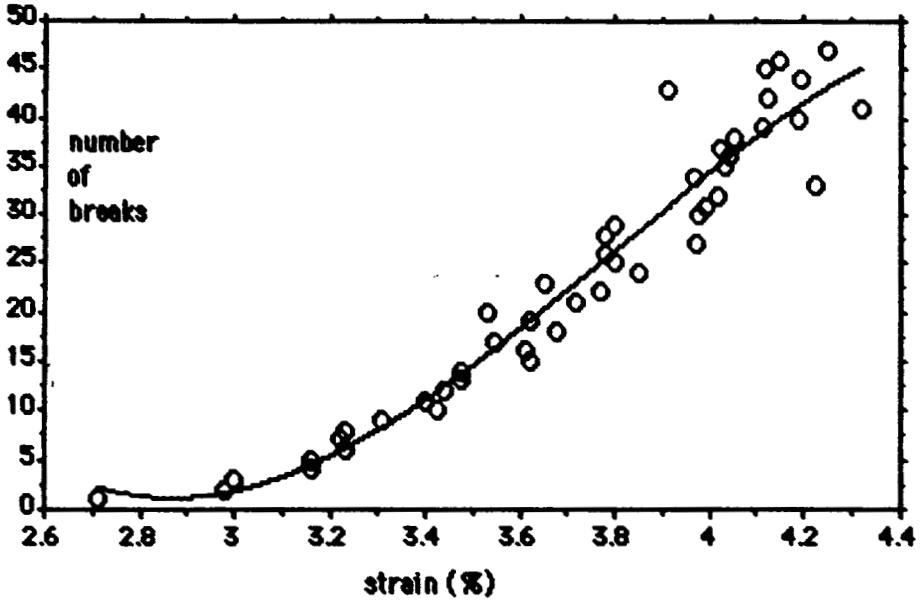
  - FIBER FAILURE STATISTICS AS A FUNCTION OF STRAIN
  - EFFECT OF ADJACENT FIBERS ON FAILURE STATISTICS AND STRESS DISTRIBUTION AT ADJACENT FIBER BREAKS (FROM STRESS BIREFRINGENCE PATTERNS)

RESULTS

•FIBER BREAKS WITH INCREASING STRAIN

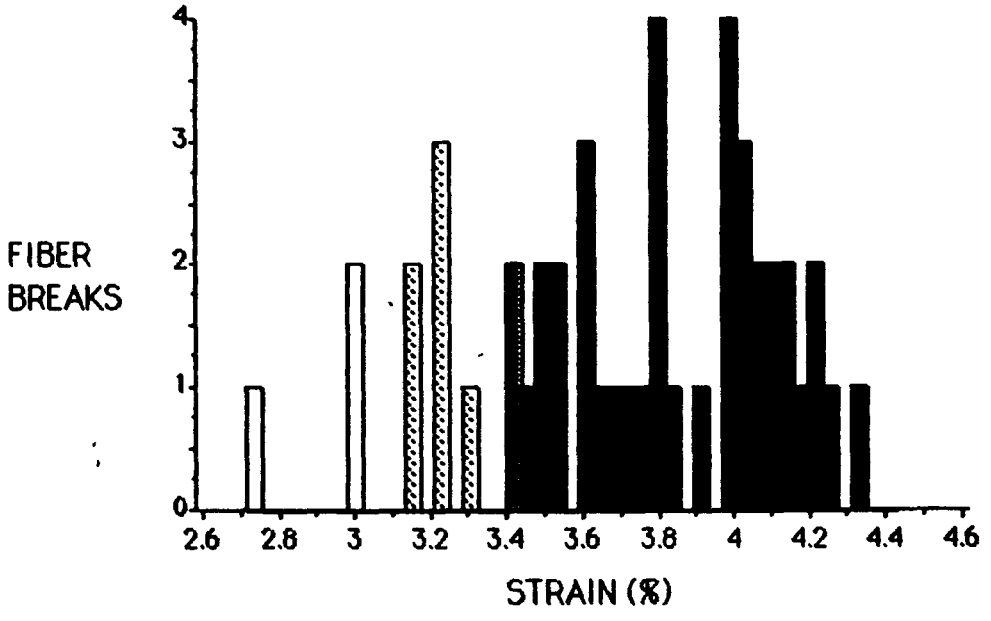
•• SINGLE FILAMENT OF AS4 IN EPOXY

•• COMBINED DATA FOR THREE SPECIMENS



CUMULATIVE FIBER BREAKS WITH INCREASING STRAIN

•• FILAMENT FRACTURE IS A DISCONTINUOUS FUNCTION OF STRAIN: BREAKS OCCUR WITHIN THREE OR POSSIBLY FOUR REGIONS OF STRAIN



FREQUENCY DISTRIBUTION OF FIBER BREAKS

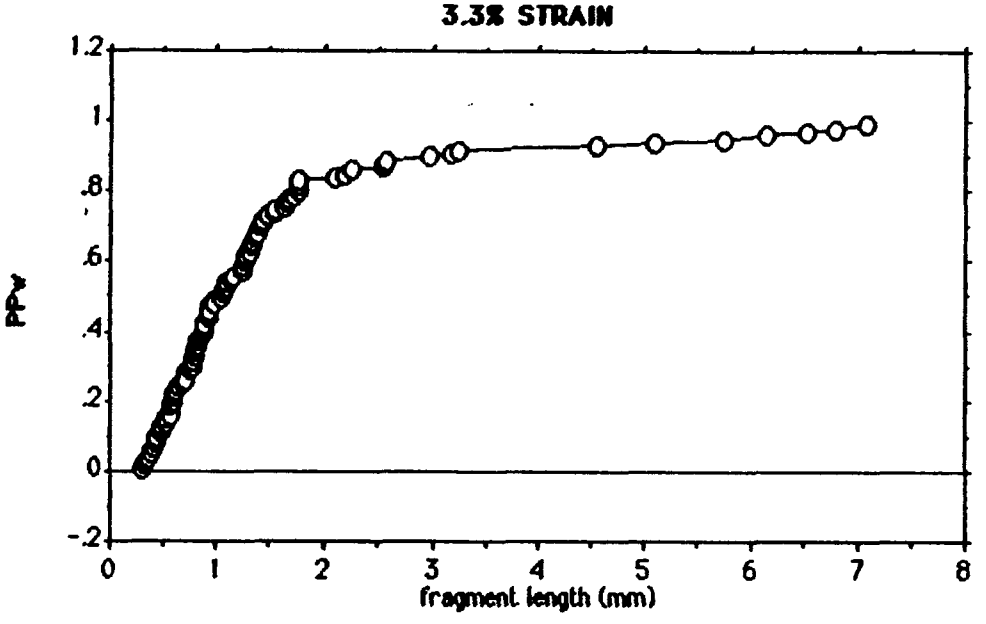
• FILAMENT FRACTURE STRAIN (3-4%) EXCEEDS LAMINATE FAILURE STRAIN (1-2%)

•STATISTICAL DISTRIBUTION OF FRAGMENT LENGTHS WITH INCREASING STRAIN

•• AT <u>LOW STRAIN</u> LEVELS FRAGMENT LENGTHS EXHIBIT A BIMODAL <u>NORMAL</u> DISTRIBUTION

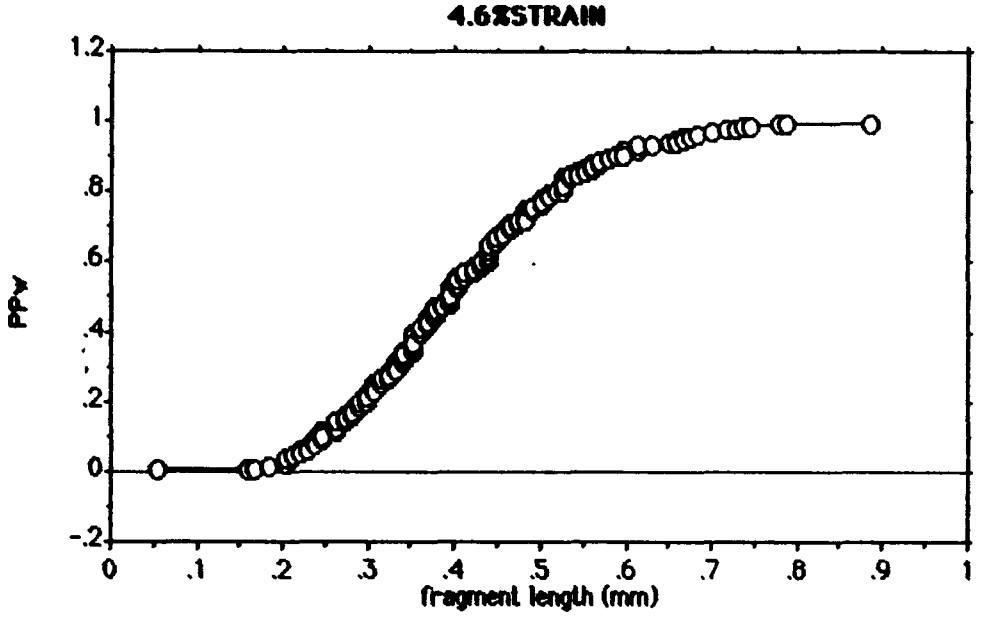
Test Data for "Normality"

Two Linear Regions



• AT HIGHER STRAIN LEVELS FRAGMENT LENGTHS DO NOT FIT NORMAL DISTRIBUTION

•• BETTER FIT BY A TWO-PARAMETER WEIBULL DISTRIBUTION



• EFFECT OF ADJACENT FIBERS ON FILAMENT FRACTURE STATISTICS (AS4-epoxy)

•• TWO FIBERS

•• THREE INTERFIBER DISTANCES

a) < 1 fiber diameter

b) 1-2 fiber diameters

c) 4-5 fiber diameters

•• SIX STRAIN LEVELS; 3.0, 3.3, 3.6, 4.0, 4.3,4.6 %

• DATA COLLECTED AND BEING ANALYZED

•• Adjacent(< d separation) Fiber Breaks vs Interfiber Distance

STRAIN (%)	INTERFIBER DISTANCE		
	<1 diameter	1-2 diameters	4-5 diameters
3.0	1	0	1
3.3	9	2	3
3.6	21	8	6
4.0	27	15	9
4.3	27	17	9
4.6	32	20	9

• PRELIMINARY RESULTS:

•• INTERFIBER EFFECTS DECAY WITH INCREASING FIBER SEPARATION. NO INTERACTION AT DISTANCES > 4 DIAMETERS

•• INTERACTIONS "SATURATE" AT ABOUT 4% STRAIN

CONCLUSIONS

- FIBER FAILURE STATISTICS CHANGE WITH INCREASINGS STRAIN
  - APPROXIMATE NORMAL DISTRIBUTION AT LOW STRAINS (< 3%)
  - APPROXIMATE WEIBULL DISTRIBUTION AT HIGH STRAINS (>4%)
- FIBER-FIBER INTERACTIONS DECREASE WITH INTERFIBER DISTANCE

FUTURE WORK

- REFINE STATISTICAL ANALYSIS OF FILAMENT FRACTURE VS TENSILE STRAIN
- ANALYZE "TWO-FIBER" DATA
  - FRAGMENT SIZE DISTRIBUTION RELATIVE TO SINGLE FILAMENT

    Strain Level
    Interfiber Distance
- DETERMINE FILAMENT FRACTURE STATISTICS FOR THREE AND FIVE ADJACENT FIBERS
  - INTERFIBER DISTANCE
  - STRAIN LEVEL
- REPEAT EXPERIMENTS FOR OTHER FIBER TYPES(e.g., IM7, HMS)
- REPEAT EXPERIMENTS FOR LOW FIBER/MATRIX ADHESION (e.g., AS4-polycarbonate)