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Stress analysis and fracture toughness of notched polyacrylonitrile (PAN)-based and pitch-based single carbon fibers

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A B S T R A C T

The stress analysis and fracture toughness of high tensile strength polyacrylonitrile (PAN)-based (T1000GB, IMS60, and T300), high modulus PAN-based (M60JB), high modulus pitch-based (K13D), and high ductility pitch-based (XN-05) single carbon fibers were investigated using notched specimens and a focused ion beam. The maximum stress of T1000GB, IMS60, T300, M60JB, K13D, and XN-05 single fibers was calculated as 30.5, 29.4, 25.6, 44.3, 68.6, and 15.5 GPa, respectively. The critical stress intensity factor of T1000GB, IMS60, T300, M60JB, K13D, and XN-05 single carbon fibers was calculated as 1.91, 1.82, 1.67, 3.09, 4.84, and 1.02 MPa $m^{1/2}$, respectively. The critical energy release rate of T1000GB, IMS60, T300, M60JB, K13D, and XN-05 single carbon fibers was calculated as 478, 502, 763, 3064, 13266, and 237 J/m^2, respectively. These results indicate that a linear relationship exists between the fracture toughness (critical stress intensity factor and critical energy release rate) and tensile modulus and maximum stress. The results indicate that the maximum stress is an effective parameter for evaluating the fracture toughness of PAN-based and pitch-based single carbon fibers.

1. Introduction

Polyacrylonitrile (PAN)-based and pitch-based carbon fibers are widely used as reinforcements in composite materials owing their high specific strength and specific modulus. These composites are dominant materials in aerospace, automotive, and sporting goods industries [1–5]. The trends in the development of carbon fibers are characterized as follows; high tensile strength, a fairly high strain to failure (approximately 2%) and a high specific modulus with high thermal conductivity. Several high strength (exceeding 5 GPa) PAN-based and high modulus (more than 900 GPa) pitch-based carbon fibers are commercially available at present. However, the mechanical properties of the high strength and high modulus carbon fibers have not yet been characterized well. Recently, Naito et al. characterized the tensile, flexural, and transverse compressive properties and Weibull modulus of high strength PAN-based, high modulus PAN-based, high modulus pitch-based, and high ductility pitch-based single carbon fibers [6–9].

The fracture toughness and strength of single carbon fibers are of great interest and importance, particularly, while studying the fracture behavior of composites [10–14]. However, the measurement of fiber fracture toughness is considered difficult owing to the small fiber diameter. One of the practical ways to estimate fracture toughness is to determine the initial crack size by observing the fracture mirror size of the tensile fracture surface of un-notched specimens [15]. Since mirror size measurement involves considerable error, the estimated fracture toughness values are usually considerably scattered. Accuracy of the measured fracture toughness will be significantly improved if a notch with controlled size and shape is introduced intentionally. Recently, a new measurement technique was proposed to estimate the fracture toughness of single carbon fibers by using a focused ion beam (FIB) [16]. The applicability of the FIB system as a machining tool to create of notches was examined. The FIB is regarded as a useful machining tool to prepare samples for transmission electron microscope observations. High resolution FIB systems have beam diameters <10 nm and achieve micro-machining by scanning the beam

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electronically. In this study, tensile tests were performed on notched single carbon fibers to estimate the stress distribution and fracture toughness.

In this study, tensile tests were conducted on commercially available high strength PAN-based, high modulus PAN-based, high modulus pitch-based, and high ductility pitch-based single carbon fibers with various notch lengths. The stress distribution and fracture toughness of the PAN-based and pitch-based single carbon fibers were evaluated.

2. Experimental

2.1. Materials

The carbon fibers used in the study included high strength PAN-based (T1000GB, IMS60, and T300), high modulus PAN-based (M60JB), high modulus pitch-based (K13D), and high ductility pitch-based (XN-05) carbon fibers. T1000GB, T300, and M60JB PAN-based carbon fibers were supplied by Toray Industries, Inc. IMS60 PAN-based carbon fiber was supplied by Toho Tenax Co., Ltd. K13D pitch-based carbon fiber was supplied by Mitsubishi Chemical Corporation. XN-05 pitch-based carbon fiber was obtained from Nippon Graphite Fiber Corp. Note that when received, all fibers were subjected to commercial surface treatments and sizing (epoxy compatible sizing). The physical properties of the PAN-based and pitch-based carbon fibers are listed in Table 1.

2.2. Specimen preparation

Single carbon fiber specimens were prepared on a stage using a stereoscope. A single carbon fiber was selected from carbon fiber bundles and cut perpendicular to the fiber axis using a razor blade. This fiber was attached to a silicon (Si) substrate using a polyimide tape with silicone adhesive.

The diameter d_f of the single carbon fiber was measured by using a scanning electron microscope (SEM) (FEI, Strata DB235 M) before FIB notching and by using another SEM (FEI, Quanta 200FEG) for fractured surface observation. The measured single fiber diameters are listed in Table 1. All specimens were stored in a desiccator at 20 ± 3 °C and at a relative humidity of $10 \pm 5\%$ prior to testing.

2.3. FIB notching

FIB notching and SEM imaging were performed in a dual beam system (FEI, Strata DB235 M). A notch was introduced in a carbon fiber by using the FIB in a dual beam system (FEI, Strata DB235 M). Notches of various lengths were introduced by scanning the beam position. The FIB column was equipped with a Ga + ion source and operated at 30 kV accelerating voltage with 10 pA (target notch length $\geq$ 150 nm) and 1 pA (target notch length $\leq$ 100 nm) probe current. The notch shapes and dimensions (notch length l_n; notch width w_n; notch-tip radius of curvature ρ_n) of these carbon fibers were examined using the SEM in a dual beam system (FEI, Strata DB235 M) at an operating voltage of 5 kV. The notch length l_n was also measured by using the SEM (FEI, Quanta 200FEG) during fractured surface observation.

2.4. Tensile test

Tensile tests of the single carbon fibers at various notch lengths were performed using a universal testing machine (Shimadzu, Table top type tester EZ-Test) with a load cell of 10 N. The tensile specimen was prepared by fixing the single carbon fiber on a paper holder using an instant cyanoacrylate adhesive, as reported in literature [17,18]. The specimen was set up on the testing machine. The holder was cut into two, before testing. The fracture surfaces of the carbon fibers after the single fiber tensile tests were difficult to observe owing to the extremely small dimensions of the carbon fibers. Therefore, plastic films were placed on both sides of the single carbon fibers and were filled with water to not only avoid secondary damage to the carbon fibers but also to retain the

Table 1
Mechanical and physical properties of the PAN-based and pitch-based carbon fibers.

Fiber	High strength PAN-based			High modulus PAN-based	High modulus pitch-based	High ductility pitch-based
	T1000GB	IMS60	T300	M60JB	K13D	XN-05
	T1000GB-12000-40D	IMS60-24K E30	T300-3000-50A	M60JB-3000-50B	K13D2U	XN-05-30S
Number of single fibers [a] (count)	12000	24000	3000	3000	2000	3000
Yield (Tex) [a] (g/1000m)	485	830	198	103	365	410
Density [a] ρ_f (g/cm^3)	1.8	1.8	1.76	1.93	2.2	1.65
Tensile modulus [a] E_f (GPa) (E, E_{11})	294	285	230	588	935	54
Poisson's ratio [b] ν_f (ν, ν_{12})	0.2	0.2	0.2	0.2	0.2	0.2
Transverse modulus [c] E_{22} (GPa)	8.94	7.79	9.12	3.30	1.44	17.49
In-plane shear modulus [c] G_{12} (GPa)	1.73	1.50	0.37	0.76	0.56	0.28
Tensile strength [d] σ_f (GPa) ($L = 5$ mm)	7.71 (0.88)	6.98 (0.79)	3.95 (0.46)	4.60 (0.56)	4.00 (0.82)	1.34 (0.17)
Modulus of toughness T_m (MJ/m^3) ($L = 5$ mm)	101.2	85.5	34.0	18.0	8.6	16.6
Diameter d_f (μm)	5.05 (0.25)	5.54 (0.31)	7.45 (0.36)	5.10 (0.29)	11.55 (0.71)	9.38 (0.56)
Maximum stress σ_{max} (GPa)	30.53	29.36	25.58	44.27	68.61	15.46
Critical stress intensity factor K_{IC} (MPa m$^{1/2}$)	1.91	1.82	1.67	3.09	4.84	1.02
Critical energy release rate $G_{IC(isotropy)}$ (J/m^2)	9.90	9.34	9.67	12.99	20.08	15.40
Critical energy release rate $G_{IC(othotropy)}$ (J/m^2)	478	502	763	3064	13266	237
Critical notch length $l_{n.crit}$ (nm)	17	21	62	137	568	182

Values in parentheses indicate standard deviations.
[a] Producer's data sheet. T1000GB, T300, and M60JB: Catalog for TORAYCA, Toray Industries, Inc. (Toray), High performance carbon fiber Torayca in Japanese. 2004. IMS60: Catalog for Toho Tenax Fiber, Toho Tenax Co.,Ltd., Properties of fiber. 2008. XN-05: Catalog for GRANOC Yarn, Nippon Graphite Fiber Corp. (NGF), Technical data XNL. K13D: Catalog for DIALEAD, Mitsubishi Chemical Corporation, High performance coal tar pitch carbon fiber. 2009.
[b] Assumed to as 0.2.
[c] Transverse compressive and flexural tests on single carbon fibers from previous investigations [7,9].
[d] Single carbon fiber tensile data (5 mm gage length) from a previous investigation [8].