1-2005

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Journal of Testing and Evaluation, Jan. 2005, Vol. 33, No. 1
Paper ID JTE12521
Available online at: www.astm.org

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Results of an Interlaboratory Study of the ASTM Standard Test Method for Tensile Properties of Polymer Matrix Composites D 3039

ABSTRACT: An investigation was conducted on the ASTM Standard Test Method for Tensile Properties of Polymer Matrix Composites (D 3039). This investigation consisted of both preliminary testing and an interlaboratory test program. Information generated from preliminary testing was used to determine the effects of various parameters and to optimize the interlaboratory test plan and test protocol. The interlaboratory study portion of this investigation was conducted on six composite material systems in a variety of lay-up configurations. The number of participating labs ranged from five to nine depending on the material type. Precision statistics were determined for the ASTM D 3039 standard from the data generated by the interlaboratory testing in accordance with the ASTM Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method (E 691).

KEYWORDS: polymer matrix composites, tensile testing, modulus, strength, failure strain, precision statistics, repeatability, reproducibility, interlaboratory testing

Introduction

The ASTM Standard Test Method for Tensile Properties of Polymer Matrix Composites (D 3039) is a commonly used ASTM composite material test method. This test method provides for the determination of a material's tensile strength and modulus and is rather straightforward in comparison with other composite material test methods. The ASTM D 3039 standard also provides the basis for several ASTM standards, including the ASTM Standard Test Method for Tension Fatigue of Polymer Matrix Composites (D 3479), the ASTM Standard Test Method for Open Hole Tensile Strength of Polymer Matrix Composite Laminates (D 5766), and the ASTM Standard Test Method for In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a $\pm 45^\circ$ Laminate (D 3518).

This investigation consisted of both a preliminary test program and an interlaboratory test program. The purpose of the preliminary testing was to assess the ruggedness of the ASTM D 3039 test method. In this preliminary testing, the effects of various testing and material parameters on the tensile strength of composite materials were examined. The parameters investigated included: (1) coupon interface design (tabbing procedures), (2) material form and fiber orientation, and (3) reinforcing fiber type. Another objective of the preliminary testing was to determine the effect of having the coupons prepared at one laboratory versus multiple laboratories. Additionally, the effect of strain-rate on the tensile properties of various composite material systems was investigated. This was done to address a concern associated with testing strain-rate sensitive materials at the strain-rate limits suggested by the time-to-failure (1–10 min) prescribed in the ASTM D 3039 standard. Data generated from the preliminary testing were used to optimize the interlaboratory test plan.

The purpose of the interlaboratory test program was to obtain measures of precision including repeatability and reproducibility for several commonly used composite materials systems using the ASTM D 3039 test method. This information has been incorporated into the precision and bias section of the ASTM D 3039 standard. Since this standard is based on the English system of units, both the preliminary testing and the interlaboratory study were conducted using the English system of units. Therefore, data presented throughout this report are in English units with the SI equivalent provided in parenthesis.

Experimental Program

Preliminary Testing

Preliminary testing was conducted prior to the interlaboratory test program to provide information regarding the effects of various material, specimen preparation, and testing parameters on the tensile performance of composite materials. Another objective of the preliminary investigation was to determine the effect of strain-rate on the tensile properties of composite materials. The test matrix used for the preliminary testing is provided in Tables 1 and 2. To determine the effect of testing and material parameters on the tensile performance of composite materials, three fiber orientations—glass/epoxy unitape, carbon/toughened epoxy

Manuscript received Jan. 16, 2004; accepted for publication Aug. 3, 2004; published Jan. 2005.

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TABLE 1—*Summary of testing parameters used for preliminary testing to assess the effects of material and testing parameters.*

Materials	Laminate Configurations	Tab Types
S2 Glass/Epoxy Tape	90_n	none
IM-7/Toughened Epoxy Tape	0_n	90° (square)
K 49 Aramid/Epoxy Fabric	$[90/0]_{ns}$	7° (beveled)

TABLE 2—*Summary of testing parameters used for preliminary testing to assess the effects of strain-rate on S2 glass/epoxy tape with 90° square tabs.*

Test Speed, s to Failure	Laminate Configuration	Replicates
6	90_n	5
6	0_n	5
60	90_n	5
60	0_n	5
600	90_n	5
600	0_n	5
6000	90_n	5
6000	0_n	5

unitape, and aramid/epoxy fabric—were tested at three U.S. laboratories, using 7° beveled tabs, square tabs (90° bevel), and no tabs. All of the test coupons were machined by a single test laboratory. To evaluate the effects of strain-rate on the strength and modulus data generated via the ASTM D 3039 test method, one material known to be strain-rate sensitive was tested at test speeds ranging from 6 to 6000 s to failure, which includes the failure envelope of 60 to 600 s to failure prescribed by the ASTM D 3039 standard. This material was an S2 glass/epoxy tape. The tensile tests were conducted in accordance with the ASTM D 3039 standard at room temperature ambient conditions. The material was tested at four speeds and two stacking sequence configurations. Square tabs were used for all of the coupons.

Interlaboratory Study

To get an accurate measure of the precision of the ASTM D 3039 test method over a wide range of composite material systems, six different materials were included in the interlaboratory test program. These materials represent commonly used composite material systems as well as materials that pose a significant challenge when testing. Material forms studied in this investigation included both unidirectional tapes and fabrics in a variety of fiber orientations. Materials, forms, and orientations are summarized in the final test matrix (Table 3). Table 4 provides a list of the laboratories that participated in this program.

All materials were donated in panel form. Panel quality was checked using ultrasonic inspection prior to the panel being machined into test coupons. Initial inspection of the glass/polypropylene material indicated the panels were badly warped, contained multiple resin rich and resin dry areas, and were discolored. Because of this, it was decided by the committee that the data generated for this material would not be included in the interlaboratory study.

Three laboratories machined the test coupons. However, all coupons of a particular material were machined by a single laboratory. Coupons were machined in accordance with the ASTM Standard Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation (D 5687) and specimen preparation protocol using the specimen geometry prescribed in the ASTM D 3039 standard. Prepared test coupons were distributed randomly to the ten participating test laboratories. Each test lab was provided with a minimum of six test coupons per test condition (material/lay-up). Each laboratory was provided with test coupons, specimen data sheets, test protocol, the current ASTM D 3039 standard, and general instructions for measuring, testing, and data reporting. Laboratory participants were asked to provide the tensile strength, tensile chord modulus, and failure strain for each test specimen. Additionally, they were requested to record the specimen failure mode using the codes provided in the ASTM D 3039 standard.

Statistical Analysis

Statistical analysis of the data generated through the interlaboratory test program was done in accordance with ASTM Standard E 691-92. This method uses a one-way analysis of variance technique to estimate the precision statistics for each material. In the ASTM Standard E 691-92, precision is defined as a measure of the degree of agreement among test results. The precision of a test method is measured by both the repeatability (r) and the reproducibility (R). Repeatability provides a measure of the variability between the independent test results whereas reproducibility provides a measure of the between-laboratory variability of the test results obtained at different laboratories. A complete definition of these and other statistical values is provided in the ASTM Standard E 691-92.

A preliminary statistical analysis was conducted on the raw interlaboratory test data to generate consistency statistics, h and k. The h-value provided an indication of the between-laboratory variability and the k-value provided an indication of the within-laboratory variability. In addition to providing some insight into the overall variability of the test method, the consistency statistics also served as a guide for identifying suspicious data points. The suspicious data points were examined for clerical and/or procedural errors by the program managers, steering committee, and participating laboratories. Following the guidelines provided in the ASTM Standard E 691-92 for eliminating data, a final decision was made as to the treatment of the raw data set. The suspicious cells were voted on by means of a ballot. The statistical analysis was conducted for

TABLE 3—*Materials and specimen configurations used for the interlaboratory test program.*

Material	Laminate Configuration	Tab Type	Number of Participating US Test Labs	Material Designation Letter
IM-6/3501-6 unitape (Hercules)	90_n	90° (square)	10	A
	0_n	7° (beveled)	10	B
	$[90/0]_{ns}$	none	10	C
‘E’ Glass/Polypropylene (ISO)	90_n	none	5	D
	0_n	90° (square)	5	E
Glass/Epoxy Fabric (Ciba Composites)	Warp Aligned	none	10	F
Graphite/Toughened Epoxy Fabric (Ciba Composites)	Warp Aligned	none	10	G

TABLE 4—*Laboratories that participated in the interlaboratory test program.*

Laboratory	Point of Contact
Ciba Composites	Bill Allen
Delsen Test Labs	John Moylan
E.I. DuPont de Nemours & Co	Marilyn Wardle, Ph. D.
Hercules Corporation	Jim Ferrel and R. Han
Hexcel Corporation	Rosanna Falabella, Ph. D.
Intec Composites	Rod Wishart
Lockheed Martin Orlando	Richard Fields
L. J. Broutman	Gregory Skaper
Metcut Research Associates	Ray Rawlinson
NPL (ISO)	Graham Sims, Ph.D.
3M Aerospace	Jeff Kittleson
University of Dayton Research Institute	Allan Crasto, Ph.D.

TABLE 5—*Data censored from interlaboratory study.*

Material—Lab—Data Point	Action
Strength Data	
A-4-2	Remove outlier, data not consistent with data obtained from that or other labs, specimen not fully gripped.
E-All	Remove from study, majority of labs indicated poor quality coupons due to warping during cure of tab adhesive.
F-8-All	Remove since data not consistent with that obtained from other labs and unacceptable failure modes.
G-8-All	Remove since data not consistent with that obtained from other labs and unacceptable failure modes.
Modulus Data	
C-1-All	Remove since data not consistent with that obtained from other labs, test lab suspects problem with instrumentation.
E-All	Remove from study, majority of labs indicated poor quality coupons due to warping during cure of tab adhesive.
F-1-All	Remove since data not consistent with that obtained from other labs, test lab suspects problem with instrumentation.
Failure Strain	
C-1-All	Remove since lab suspects problem with strain measurement instrumentation.
E-All	Remove from study, majority of labs indicated poor quality coupons due to warping during cure of tab adhesive.
F-1-All	Remove since lab suspects problem with strain measurement instrumentation.
G-8-All	Remove since data not consistent with that obtained from other labs, unacceptable failure modes.

the modified data set. A close examination of the modified data set as well as tested coupons resulted in further censorship of the data. A summary of the data eliminated from this study as well as the rationale supporting this censorship is provided in Table 5. Precision statistics were generated on the final data set. Data for all materials except material B (90_n lay-up) were normalized with respect to an average thickness as an approximation of normalizing with respect to fiber volume by assuming reasonable fiber aerial weight tolerances.

Results

Preliminary Testing

A summary of the data generated from the preliminary testing is provided in Tables 6 and 7. These data suggest the aramid fabric composite to be essentially insensitive to the tabbing procedures. The majority of the zero degree carbon/epoxy and glass/epoxy tape coupons failed in an "exploded" type failure mode. This type of failure mode made it difficult to distinguish any but the most severe testing problems. The remaining configurations of these materials were found to fail in varying degrees of acceptable failure modes. It was noted through the preliminary testing that the failure mode codes prescribed in ASTM D 3039 require further education and explanation. Additionally, it was noted that two new failure mode codes should be considered. These failure modes include grip shearout and gage/grip rebound, which were identified as gage failures in the results presented above.

The data from this portion of the investigation also suggest that the untabbed test coupons performed well on coupons that did not have a unidirectional laminate configuration. Laboratories that used coarsely serrated grips noted that the untabbed $[90/0]_{ns}$ glass epoxy coupons experienced grip failures. For the IM-7 Carbon/Toughened Epoxy Tape, it was noted that a ductile tab adhesive provided superior tab performance, more acceptable failure modes, and higher failure strengths. In general, however, the data obtained from coupons having bonded tabs were variable, implying that a different adhesive should be used for different testing conditions. An attempt to try to optimize the adhesive for the different test conditions would complicate large scale testing. An alternative solution to this problem would be to avoid the use of tabs where possible. Neither the square nor beveled tabs were found to perform as well as the untabbed coupons for the $[90/0]_{ns}$ carbon/epoxy material. The glass/epoxy and the aramid/epoxy materials both appeared to be insensitive to tab type.

The investigation conducted to evaluate the effect of strain-rate on strength and modulus at the limits of the ASTM D 3039 recommended time-to-failure range indicated that there was relatively little difference in the test data obtained at the strain-rates investigated. For the 0_n configuration, the strength data appeared to increase slightly with increasing rate (decreasing time-to-failure). No consistent trend was noted for the modulus data of the 0_n configuration or for the strength or modulus data of the 90_n configuration.

ASTM D 3039 Interlaboratory Study

Precision statistics generated for the final data set are provided in Tables 8–10. The precision statistics were used to write a precision statement that has been incorporated into the ASTM D 3039 standard. From these tables it can be seen that the 90_n lay-up of the IM6/3501-6 material had the highest interlaboratory (reproducibility) and intralaboratory (repeatability) coefficient of variation (CV) for strength, modulus, and failure strain. This suggests that the 90_n configuration may not be optimum for generating tensile data of matrix-dominated lay-ups. Since only one material provided valid data for this configuration, confirmation of this observation would require additional testing of 90_n laminates for other materials.

As expected, the strain data were found to have slightly higher variability than that noted for the other properties measured. Interlaboratory variability of the strain data is most likely due to differences in strain measurement techniques. Intralaboratory variability in the strain data might be attributed to problems associated with strain measuring techniques such as premature gage failure, or might indicate an inherent variability in the materials' strain response.

Variability of the modulus data was found to be the lowest of the measured properties. This finding suggests that the definition of modulus provided in the ASTM D 3039 standard is clear and

TABLE 6—*Summary of D 3039 preliminary testing study to determine the effects of material and testing parameters on tensile performance.*

Configuration	Tab Type	Failure Mode	Strength, ksi (MPa)	CV, %	Lab
Material: IM-7 Carbon/Toughened Epoxy Tape					
0_n	None	Exp	361 (2489)	4.0	A
	90° (square)	Exp	327 (2255)	5.2	C
	7° (beveled)[a]	...	...	...	B
$[90/0]_{ns}$	None	Gage	201 (1386)	1.9	C
	90° (square)	Gage	194 (1338)	2.4	B
	7° (beveled)	Gage	194 (1338)	2.5	A
90_n	None	Gage	11 (74)	1.8	B
	90° (square)	Grip	10 (67)	3.4	A
	7° (beveled)	Grip	9 (59)	4.7	C
Material: Kevlar 49 Aramid/Epoxy Fabric					
0_n	90° (square)	Gage	66.0 (455)	3.4	A
	7° (beveled)[a]	Gage	65.0 (448)	3.4	C
$[90/0]_{ns}$	None	Gage	64.5 (445)	4.2	A
	90° (square)	Gage	65.5 (452)	3.1	C
	7° (beveled)	Gage	65.2 (450)	3.5	B
90_n	None	Gage	72.2 (498)	3.2	C
	90° (square)	Gage	66.9 (461)	7.9	B
	7° (beveled)	Gage	70.7 (487)	2.8	A
Material: S2 Glass/Epoxy Tape					
0_n	None	Exp/split	208 (1434)	5.0	C
	90° (square)	Exp	238 (1641)	1.5	B
	7° (beveled)[a]	Exp	250 (1724)	2.1	A
$[90/0]_{ns}$	None	Grip/del/sh	109 (752)	1.5	B
	90° (square)	Exp/del	129 (889)	3.8	A
	7° (beveled)	Exp/del	121 (834)	2.9	C
90_n	None	Grip	6.0 (41)	17.4	A
	90° (square)	Gage	5.9 (41)	40.5	C
	7° (beveled)	Gage	4.7 (32)	53.7	B

[a] Three different sets of replicates were tested for this case by laboratory B

Set	Failure Mode	Strength, ksi (MPa)	CV, %
Trial 1	Exp/grip	357 (2462)	5.0
Trial 2	Exp/grip	376 (2593)	2.9
Ductile Adhesive	Exp	399 (2751)	3.2

CV = Coefficient of Variation.

TABLE 7—*Summary of D 3039 preliminary testing study to determine the effect of strain-rate on the performance of glass/epoxy tape.*

Configuration	Time-to-Failure, s	Strength, ksi (MPa)	CV, %	Modulus, Msi (MPa)	CV, %
0_n	6000	210 (1448)	4.2	6.67 (45990)	0.9
	600	228 (1572)	5.1	6.61 (45576)	1.9
	60	251 (1731)	8.1	6.73 (46403)	4.1
	6	254 (1751)	2.3	6.76 (46610)	2.8
90_n	6000	6.4 (44)	8.7	1.68 (11584)	2.5
	600	6.1 (42)	17.3	1.75 (12066)	6.2
	60	6.4 (44)	10.7	1.69 (11653)	7.4
	6	6.8 (47)	14.9	1.77 (12204)	7.2

TABLE 8—*Precision statistics generated for the censored and normalized strength data.*

Mat	$\bar{x}$,ksi (MPa)	$s_{\bar{x}}$,ksi (MPa)	s_r,ksi (MPa)	$S_r/\bar{x}$, %	S_R, ksi (MPa)	$S_R/\bar{x}$, %	r, ksi (MPa)	R,ksi (MPa)	$r/\bar{x}$, %	$R/\bar{x}$, %
A	342.69 (2363)	8.49 (58.54)	10.68 (73.64)	3.12	12.78 (88.12)	3.73	29.9 (206)	35.78 (247)	8.73	10.44
B	8.52 (59)	0.52 (3.59)	0.85 (5.86)	9.94	0.92 (6.34)	10.84	2.37 (16)	2.59 (18)	27.84	30.34
C	156.37 (1078)	3.84 (26.48)	10.85 (74.81)	6.94	10.85 (74.81)	6.94	30.37 (209)	30.37 (209)	19.42	19.42
F	66.18 (456)	3.20 (22.06)	1.52 (10.48)	2.3	3.48 (23.99)	5.26	4.25 (29)	9.74 (67)	6.43	14.71
G	121.52 (838)	1.59 (10.96)	3.92 (27.03)	3.23	3.92 (27.03)	3.23	10.98 (76)	10.98 (76)	9.04	9.04

TABLE 9—*Precision statistics generated for the censored and normalized modulus data.*

Mat	$\bar{x}$, Msi (MPa)	$s_{\bar{x}}$, Msi (MPa)	s_r, Msi (MPa)	$S_r/\bar{x}$, %	S_R, ksi (MPa)	$S_R/\bar{x}$, %	r, Msi (MPa)	R, Msi (MPa)	$r/\bar{x}$, %	$R/\bar{x}$, %
A	23.57 (162515)	0.65 (4482)	0.63 (4344)	2.69	0.86 (5930)	3.66	1.77 (12204)	2.42 (16686)	7.52	10.26
B	1.30 (8964)	0.05 (345)	0.04 (276)	3.12	0.06 (414)	4.57	0.11 (758)	0.17 (1172)	8.74	12.79
C	12.38 (85360)	0.29 (2000)	0.37 (2551)	2.98	0.44 (3034)	3.54	1.03 (7102)	1.23 (8481)	8.34	9.92
F	3.95 (27235)	0.08 (552)	0.04 (276)	1.01	0.09 (621)	2.28	0.11 (758)	0.25 (1724)	2.81	6.37
G	9.47 (65296)	0.16 (1103)	0.12 (827)	1.29	0.20 (1379)	2.06	0.34 (2344)	0.55 (3792)	3.62	5.78

TABLE 10—*Precision statistics generated for the censored and normalized failure strain data.*

Mat	$\bar{x}$, %	$s_{\bar{x}}$, %	s_r,%	$S_r/\bar{x}$, %	S_R,%	$S_R/\bar{x}$, %	r, %	R, %	$r/\bar{x}$, %	$R/\bar{x}$, %
A	1.36	0.0576	0.0673	4.95	0.0836	6.15	0.19	0.23	13.86	17.22
B	0.66	0.0435	0.0823	12.47	0.0859	13.02	0.23	0.24	34.92	36.44
C	1.22	0.0292	0.0640	5.25	0.0643	5.27	0.18	0.18	14.69	14.76
F	2.04	0.1531	0.0650	3.19	0.1639	8.03	0.18	0.46	8.92	22.5
G	1.27	0.0288	0.0486	3.83	0.0525	4.13	0.14	0.15	10.71	11.57

concise, since the value of modulus for composite materials can vary significantly with engineering interpretation.

It was noted through the interlaboratory testing program as well as the preliminary testing that the failure mode codes prescribed in the ASTM D 3039 standard require further explanation. Inconsistency was noted in the reported failure modes. Further analysis of the returned coupons needs to be conducted in order to determine whether the inconsistency in failure modes is the result of a difference in the laboratories' interpretation of the definition of the identified failure modes, or whether the difference in failure modes is real and due to a difference in testing techniques. Additionally, it was noted that two new failure mode codes should be considered. These failure modes include grip shearout and gage/grip rebound.

Conclusions

Results of the preliminary investigation suggest that the optimum tab geometry for the test coupons is dependent upon both the material and fiber orientation. Additionally, it appears that the aramid fabric is insensitive to the parameters studied and that the $[90/0]_{ns}$ configuration provided superior performance over the other configurations investigated. The use of a ductile adhesive for adhering tabs onto the test coupons was found to provide superior tab performance and higher specimen failure strengths. Little difference in strength was noted for the strain-rate sensitive material tested at either extreme of the time-to-failure limit indicated in the ASTM D 3039 standard.

Proposed precision statistics were generated for the materials investigated in the interlaboratory testing program for the ASTM D 3039 test method. From these statistics, the strain data were found to have the highest variability of the properties measured for a majority of the materials tested. Variability of the modulus data was found to be the lowest of the measured properties suggesting that the definition of modulus provided in the ASTM D 3039 standard is clear and concise. The method used to measure strain does not appear to have an effect on the recorded value of strain or modulus. A large inconsistency in noted failure modes indicates a need for optimizing the failure mode descriptions provided in the ASTM D 3039 standard.