Temperature/Humidity Effects on the Strength of Graphite/Epoxy Laminates

R. M. Verette*
Northrop Corporation, Hawthorne, Calif.

In this paper, the influence of temperature and humidity on the strength and modulus of graphite/epoxy is examined. The mechanical properties at several temperature/humidity conditions are compared to baseline room-temperature dry conditions. A technique for using a high-temperature/high-humidity soak prior to test to accelerate attaining a "wet" condition is discussed. Stresses and strains at failure are presented and reduced to a B-basis. Data for unidirectional laminates are given for tension, compression, and shear both parallel and transverse to the fiber direction. Three multidirectional laminates typical of aircraft construction are also examined.

Introduction

IT has been recognized for some time that once flight loads and temperature profiles are defined for an aircraft system, materials used in the construction of the aircraft must be such that they possess sufficiently high strength at temperatures encountered for safe and durable aircraft operation. More recently, the effects of moisture and its influence on advanced composite properties have come to be appreciated. Since life requirements for aircraft, as specified in MIL-STD-1530, range from 15 years for fighters to 25 years for bombers, tankers, cargo, and trainer vehicles, it is clear that absorption of moisture must be considered in the assessment of laminate strength. This is particularly cogent in view of the uncertain status of sealants, paints, and other moisture-inhibiting agents at the present time. The moisture equilibrium level attained is a function of the relative humidity, with high-humidity environments permitting more moisture absorption than less humid environments. Since one cannot dictate usage over an entire aircraft fleet lifetime, and since it is necessary to maintain a state of "operational readiness," one can conservatively assume conditions corresponding to saturation at 100% relative humidity. Any real life case can be expected to be less severe.

This paper presents data on the rate of moisture absorption and the saturation levels attained for a currently used graphite/epoxy system, AS/3501-5 prepregged by Hercules, Inc. The influence of humidity and temperature on failure stresses and strains and on modulus is then examined. In reviewing the data to be presented, one should keep in mind that, just as the realization that moisture effects must be considered in the use of composites is a rather recent development, the formulation of specific systems to optimize saturated strength has only recently been initiated. Thus, it is not particularly surprising to find that saturated strengths of existing prepregs can be substantially lower than corresponding dry strenghts. These results are outlined in detail in the following paragraphs.

Absorption/Desorption Studies

Accelerated Moisture Conditioning

As noted above, graphite/epoxy laminates, when exposed to a given environment, will absorb moisture until a state of equilibrium is reached with respect to the relative humidity of the environment. Thus, an aircraft fabricated from composite materials may be expected to absorb moisture until equilibrium is reached for the geographical location involved, unless the aircraft is retired prior to reaching equilibrium. Even though the rate of absorption may be such that some time is required before saturation occurs, eventually an equilibrium position will be attained, and this would occur in many cases before the aircraft is retired from service.

In order to assess the effects of moisture on laboratory specimens made of composites, a number of techniques have been used to accelerate moisture pickup and thus compress the time required to attain a wet condition. Several methods have been described.[1-4] These include immersion in room-temperature water for prescribed periods of time, prolonged immersion in room-temperature water until saturation is attained, immersion in boiling water for prescribed periods, immersion in elevated-temperature water for prescribed periods, exposure to high-humidity environments at elevated temperature for prescribed periods, and exposure to high-humidity high-temperature environments until saturation occurs. In reviewing the several methods available, those that employ the highest humidity, highest temperature environments will result in the greatest weight gain for a given period of time, and thus a saturation condition can be reached in a minimum period of time under these conditions.

The data reported in this paper were obtained by continued immersion in 180°F deionized water until saturation, as measured by reaching an equilibrium weight gain condition, was reached. Thus, the specimens tested gained the maximum weight due to moisture that they were capable of acquiring, regardless of exposure time. The specimens were immersed in 180°F water as against being contained in a 180°F, 95% relative-humidity environment strictly as a matter of convenience since a) the two methods provide approximately equivalent moisture-gain rates and equilibrium levels; b) the two methods appear to yield laminates having approximately equivalent wet/dry strength comparisons; and c) the epoxy molecular bonds are exceptionally strong relative to those found in other plastics, and thus the probability of chemical attack by 180°F water or other response not experienced or exhibited by an identical specimen in a high-humidity 180°F environment is remote.

Processing Effects

In this paper, four cure-cycle/postcure combinations are examined to assess the influence of processing on weight gain and saturated properties. The essential features of the four processing cycles are: Cycle A – one stage cure, 30 min at 350°F and 100 psi with no postcure; Cycle B – one stage cure, 30 min at 350°F and 100 psi, followed by a 4-hr postcure at 370°F; Cycle C – two-stage cure, 1 hr at 225°F and 100 psi,

Presented as Paper 75-1011 at the AIAA 1975 Aircraft Systems and Technology Meeting, Los Angeles, Calif., Aug. 4-7, 1975; submitted Aug. 11, 1975; revision received June 1, 1976.

Index categories: Aircraft Structural Materials; Materials, Properties of; Structural Composite Materials (including Coatings).

*Manager, Structural Mechanics Research Department, Aircraft Division. Member AIAA.

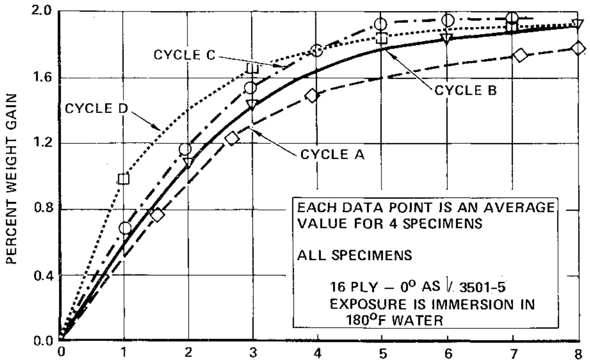

Fig. 1 Influence of cure cycle on moisture absorption.

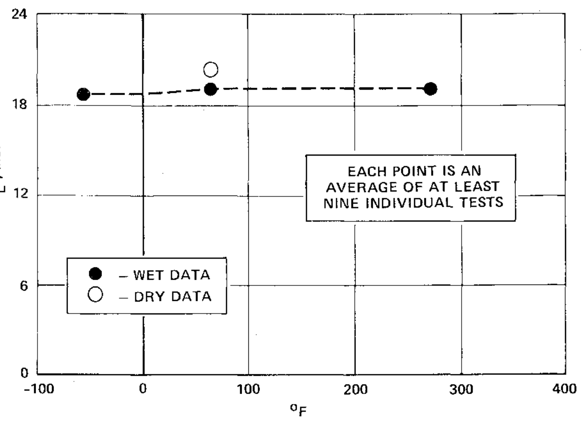

Fig. 2 Effect of temperature and humidity on longitudinal tensile elastic modulus.

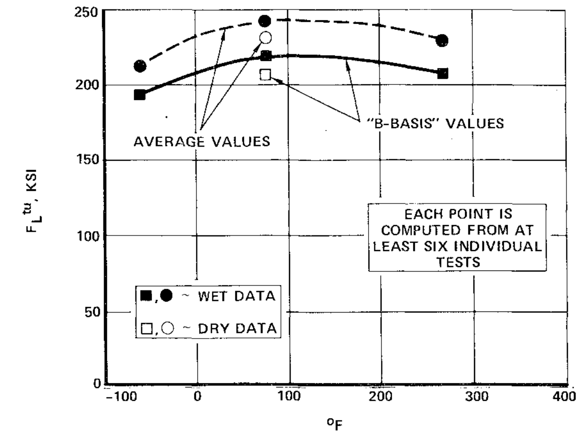

Fig. 3 Effect of temperature and humidity on longitudinal tension stress at failure.

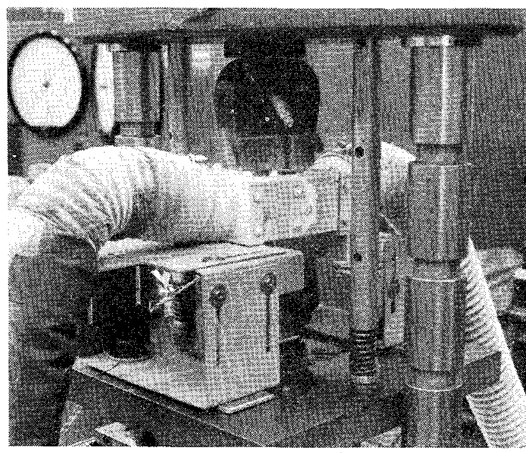

Fig. 4 Low-temperature test setup.

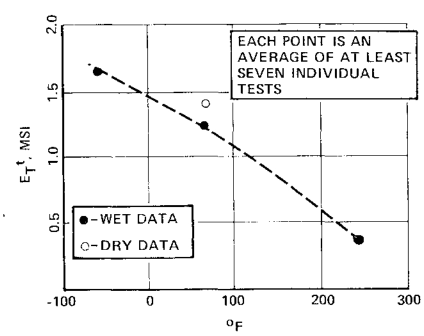

Fig. 5 Effect of temperature and humidity on transverse tensile elastic modulus.

followed by 1 hr at 350°F and 100 psi, with no postcure; Cycle D-two-stage cure, 1 hr at 225°F and 100 psi, followed by 1 hr at 350°F and 100 psi, followed by a 4-hr postcure at 370°F. Figure 1 shows the weight gain experienced by specimens cured using the four cycles. All date shown in Fig. 1 were obtained from 16-ply AS/3501-5 graphite/epoxy laminates immersed continuously in 180°F water. Note that for the cases illustrated, saturation level ($1.9 \pm 0.1\%$ weight gain) is essentially independent of processing employed. Also, rate of moisture pickup is nearly independent of specific cure cycle. The saturation level shown ($1.9 \pm 0.1\%$) has been reconfirmed repeatedly and is independent of lay-up pattern and number of plies in the laminate.

Unidirectional Data

Using the 180°F water immersion method to provide accelerated moisture conditioning to reach saturation ($1.9 \pm 0.1\%$ weight gain), a series of tests were conducted using AS/3501-5 graphite/epoxy. Unless otherwise stated, all data reported below were obtained using specimens cured according to cure Cycle A referred to above.

Longitudinal Tension

Figures 2 and 3 show the effects of temperature and humidity on the modulus and strength of 0° tension specimens. Data were collected at four temperature/humidity conditions: room temperature dry, room temperature wet, −67°F wet, and 265°F wet. Specimens utilized were straight-sided coupons, 8 in. long by 0.5 in. wide with tapered fiberglass loading tabs providing a 3-in. gage section. Heating was provided by a baffled hot-air enclosure and cooling was provided by a cold-gas enclosure (see Fig. 4). Dwell time at temperature prior to test was 10 min. Temperatures were measured by thermocouples. The thermocouples indicated a uniform-temperature state throughout the gage area. Strains were measured using strain gages. AE-10/15 adhesive was used to bond the strain gages in place for the −67°F and room-temperature tests. The strain gaging was followed by soaking to recapture moisture desorbed during strain-gage cure. Elevated temperature tests used Loctite Hot Strength 306 adhesive, which cures during heat up and dwell at temperature prior to test. It is apparent from Figs. 2 and 3 that neither longitudinal tensile modulus nor tensile strength is dramatically affected by humidity or temperature. In all cases considered, similar failure modes were observed. At room temperature, modulus is lowered slightly and failure stress increased slightly due to the presence of moisture. The B-basis values shown in Fig. 3 were obtained by estimating Weibull scale and shape parameters from the sample data and then using a confidence level of 95% to set the population mean along with a probability of survival of 90%.

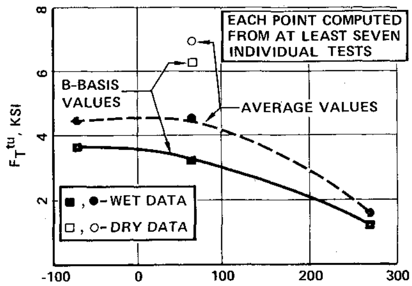

Fig. 6 Effect of temperature and humidity on transverse tensile stress at failure.

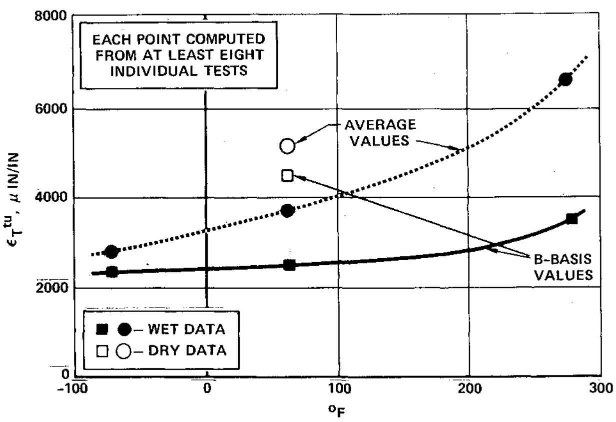

Fig. 7 Transverse tension failure strain data.

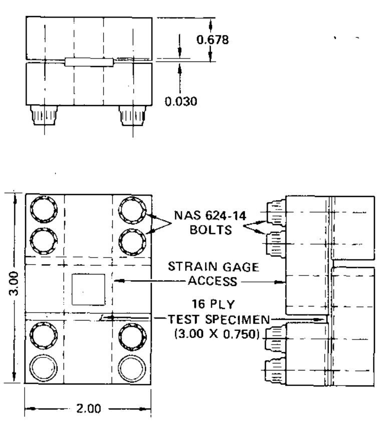

Fig. 8 Compression test fixture.

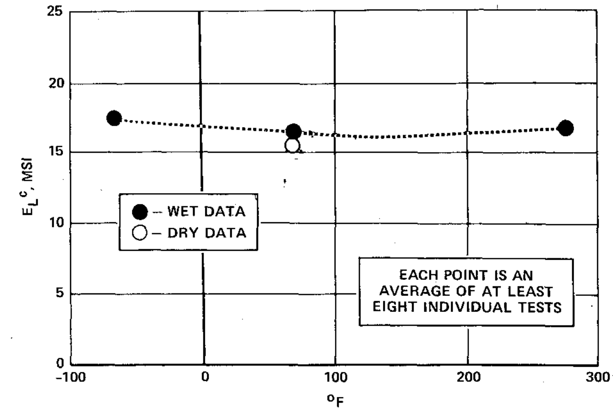

Fig. 9 Effect of temperature and humidity on longitudinal compression elastic modulus.

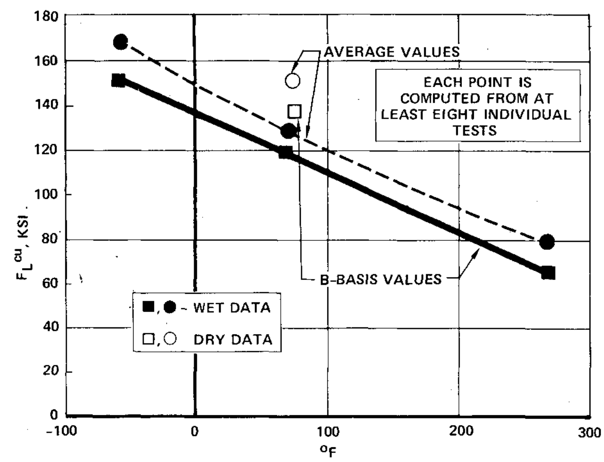

Fig. 10 Effect of temperature and humidity on longitudinal compression stress at failure.

Although the data of Figs. 2 and 3 above were obtained from material cured using cure cycle A (30 min at 350°F and 100 psi), numerous replicated tests on specimens cured using cure cycles B, C, and D mentioned above yielded substantially the same results, indicating no effect of particular cure cycle on longitudinal tensile properties.

Transverse Tension

Using 16-ply untabbed straight-sided specimens 8 in. long by 1 in. wide and material cured according to cycle A above, the data shown in Figs. 5 and 6 were obtained. In contrast with the fiber-dominated failure characteristics described above in the case of longitudinal tension, the data of Figs. 5 and 6 show pronounced temperature and humidity effects.

Failures at −67°F wet (−67W), room temperature dry (RTD), and RTW were all straight across with minimal fiber splitting. At 265 W, the failed specimens often remained in one piece, with the two halves connected by a mesh of graphite fibers. Figure 7 shows failure strain data. Failure criteria are often expressed in terms of strain, and the transverse tensile strain is frequently a critical design parameter.

Longitudinal Compression

Compression data were generated using the Northrop-developed fixture shown in Fig. 8. This fixture is employed in conjunction with a 16-ply 3 × ¾-in. untabbed coupon, and it provides support along both the machined and nonmachined surfaces to preclude undesired specimen buckling. Results are shown in Figs. 9 and 10 for specimens cured according to cure cycle A. Heating is accomplished by heating the fixture of Fig. 8 with quartz lamp heaters. The specimen is then heated conductively through the fixture. Thermocouples have indicated a condition of uniform temperature throughout the test coupon. The longitudinal compressive modulus is quite insensitive to temperature and humidity as was found to be the case for the longitudinal tensile modulus. The longitudinal

Table 1 Unidirectional static 0° compressive strength, 16 ply AS/3501-5 graphite/epoxy

Cure cycle[a]	RTD ksi	RTW ksi	265W ksi
A	152.4	129.7	79.6
B	155.0	119.4	72.8
C	155.8	114.1	76.5
D	151.0		79.6

[a] A, 30 min at 350°F and 100 psi, no postcure; B, 30 min at 350°F and 100 psi, 4-hr postcure at 370°F; C, 1 hr at 225°F and 100 psi, followed by 1 hr at 350°F and 100 psi, no postcure; D, 1 hr at 225°F and 100 psi, followed by 1 hr at 350°F and 100 psi, 4-hr postcure 370°F.

Table 2 Multidirectional data-wet/dry comparisions AS/3501-5 graphite epoxy

Test type	Laminate	Temperature humidity condition	Number of plies	Number of replicates	Specimen width	Average failure stress ksi
0° tension	25% 0°	RTD	16	3	1.00	73.7
	25% 90°	RTW	16	10	1.00	86.2
	50% ±45°					
	62½% 0°					
	12½% 90°	RTD	16	3	1.00	144.2
	25% ±45°	RTW	16	10	1.00	138.3
	25% 0°	RTD	16	3	1.00	82.4
	75% ±45°	RTW	16	10	1.00	78.7

compressive strength at 265W is about 50% of the RTD value, whereas the −67W value typically exceeds the RTD value.

Various processing alterations do not appear to materially affect the RTD, RTW, and 265W longitudinal compression strengths, as shown in Table 1. These data are all from high-quality material and all are averages of at least three individual tests. The data scatter associated with the several cure cycles at 265W is shown to be roughly similar in Fig. 11.

Transverse Compression

Transverse compression tests also use 3 × ¾-in. coupons and the fixture shown in Fig. 8. Results for specimens cured using cure cycle A are shown in Figs. 12 and 13. Note that the transverse compression modulus data are quite similar to the transverse tensile modulus data. Strong reduction of strength at saturated elevated temperature conditions is quite apparent. At 265W, pronounced local bulging of specimens occurred at failure.

In-Plane Shear

In-plane shear tests were conducted using 16-ply 0° rail shear specimens. Results are given in Figs. 14 and 15. Failure modes were rather consistently at or near the rail boundaries or at the specimen centerline.

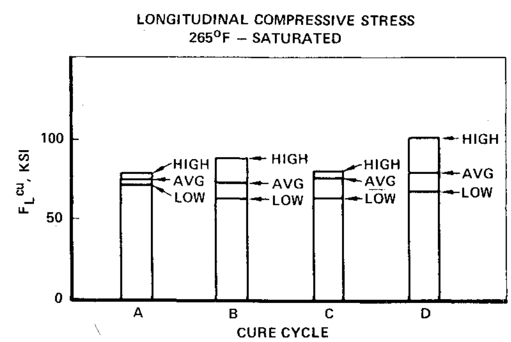

Fig. 11 265W longitudinal compressive strength of AS/3501-5 as a function of cure cycle.

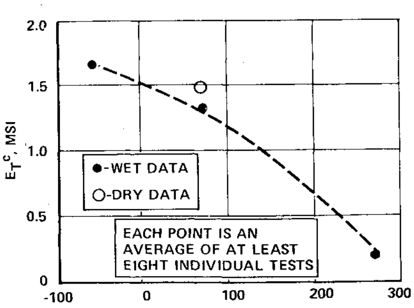

Fig. 12 Effect of temperature and humidity on transverse compression elastic modulus.

Multidirectional Data

Three multidirectional laminates regarded as typical of those encountered in aircraft applications have been studied. The particular laminates examined include a "π/4 laminate," consisting of equal percentages of 0°, 90°, +45°, and −45° plies; a laminate consisting of 62.5% 0°s, 12.5% 90°s, and 25% ±45° plies; and a laminate containing 25% 0° plies and 75% ±45° plies. Although numerous tests have been conducted on these multidirectional laminates, only those that

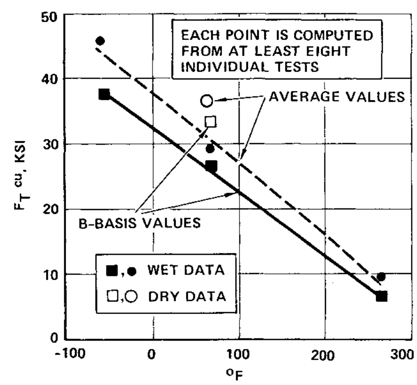

Fig. 13 Effect of temperature and humidity on transverse compression stress at failure.

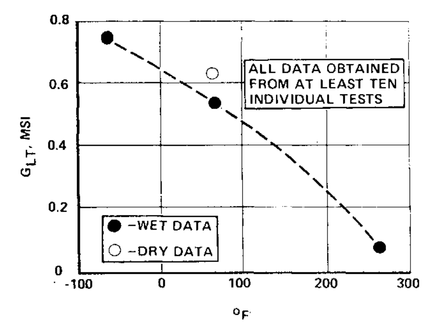

Fig. 14 Effect of temperature and humidity on in-plane shear modulus.

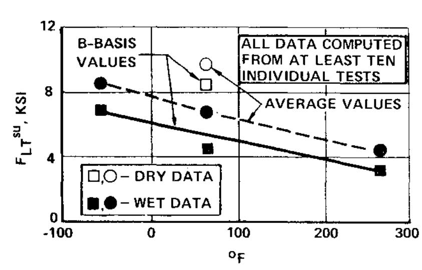

Fig. 15 Effect of temperature and humidity on in-plane shear strength.

provide clear-cut wet/dry comparisons will be mentioned here. Table 2 provides data comparisons for the three multidirectional laminates. This table allows wet/dry comparisons for 0° tension loading to be made. In this instance, the failures are fiber-dominated, and one would not expect, based on the unidirectional data, that significant strength reductions would be observed in going from a dry to a wet condition. The 0° tension data of Table 2 indicate mild strength changes as a result of moisture, a result consistent with the undirectional data. The $\pi/4$ 0° tension data of Table 2 are from two different programs, and hence the slight differences in material quality encountered could explain the larger-than-expected strength enhancement in going from dry to wet conditions.

Conclusions and Recommendations

The effects of temperature and humidity on composites have been examined in this paper. Weight gain due to absorbed moisture, and modulus and strength changes as a result of certain moisture/temperature combinations have been presented. The data obtained suggest that fiber-dominated failures are relatively unaffected by the absence or presence of moisture, whereas matrix-dominated properties are adversely affected, especially at elevated temperatures, and also at room temperature.

In order to clarify the picture, a number of activities should be pursued. First, more data are needed on the effects of high-humidity and high-temperature environments on multidirectional laminates common to aircraft use. Loading conditions other than uniaxial tension should be employed so that one can establish whether or not moisture-related strength reduction observed in unidirectional laminates continues to be observed, and to what degree it exists, in multidirectional laminates. Also, the influence of temperature/humidity combinations on behavior of laminates in structural applications should be studied in detail. Possible strength reduction due to decreased interlaminar properties should be investigated and possible decreases in the strength of bonded assemblies by virtue of moisture should be quantified. Second, the question of whether alterations in processing, e.g., employing postcures of various temperatures and durations are beneficial or detrimental in increasing saturated strength should be resolved. The advantages to postcuring may be real or illusory. Third, emerging material systems, some of which have had high moisture resistance as one of the key requirements in their formulations, should be surveyed and tested to establish high-confidence data for future use. Fourth, basic material studies should be encouraged, aimed at developing new, higher wet strength composites. Finally, work on sealants and moisture-inhibiting agents should be continued. Although the basic thrust of this paper has been on the influence of temperature and humidity on static strength, the influence of these environments on residual strength after fatigue exposure is also of obvious interest and should be investigated.

Acknowledgments

This work was performed under Contract F33615-74-C-5182. The author wishes to acknowledge the assistance given by E. Demuts who served as the Air Force Technical Monitor on this program. In addition, some of the data were generated under Northrop IRAD funds.

References

[1] "Plastics: Methods of Testing," Federal Test Method Standard No. 406, Oct. 1961.

[2] Scow, A. L., "Advanced Development of Not-Critical-to-Flight-Safety Advanced Composite Aircraft Structures," Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, AFML-TR-74-38, March 1974.

[3] "Plastics for Aerospace Vehicles," MIL-HDBK-17A, Jan. 1971.

[4] Hofer, K. E., Rao, N., and Larsen, D., "Development of Engineering Data on the Mechanical and Physical Properties of Advanced Composite Materials," Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, AFML-TR-72-205, Feb. 1974.

U.S. POSTAL SERVICE

STATEMENT OF OWNERSHIP, MANAGEMENT AND CIRCULATION

(Required by 39 U.S.C. 3685)

1. TITLE OF PUBLICATION: JOURNAL OF AIRCRAFT
2. DATE OF FILING: OCT 1 1976
3. FREQUENCY OF ISSUE: MONTHLY
A. NO. OF ISSUES PUBLISHED ANNUALLY: 12
B. ANNUAL SUBSCRIPTION PRICE: $7
4. LOCATION OF KNOWN OFFICE OF PUBLICATION *(Street, City, County, State and ZIP Code) (Not printers)*: 1290 AVE OF AMERICAS, NEW YORK NY 10019
5. LOCATION OF THE HEADQUARTERS OR GENERAL BUSINESS OFFICES OF THE PUBLISHERS *(Not printers)*: (see no. 4)
6. NAMES AND COMPLETE ADDRESSES OF PUBLISHER, EDITOR, AND MANAGING EDITOR

PUBLISHER *(Name and Address)*: AIAA (see no. 4)

EDITOR *(Name and Address)*: ALLEN E. FUHS (see no. 4)

MANAGING EDITOR *(Name and Address)*: MARK MANDELBAUM (see no. 4)

7. OWNER *(If owned by a corporation, its name and address must be stated and also immediately thereunder the names and addresses of stockholders owning or holding 1 percent or more of total amount of stock. If not owned by a corporation, the names and addresses of the individual owners must be given. If owned by a partnership or other unincorporated firm, its name and address, as well as that of each individual must be given.)*

NAME	ADDRESS
AMERICAN INSTITUTE OF AERONAUTICS & ASTRONAUTICS	1290 AVE OF THE AMERICAS NEW YORK NY 10019

8. KNOWN BONDHOLDERS, MORTGAGEES, AND OTHER SECURITY HOLDERS OWNING OR HOLDING 1 PERCENT OR MORE OF TOTAL AMOUNT OF BONDS, MORTGAGES OR OTHER SECURITIES *(If there are none, so state)*

NAME	ADDRESS
NONE	

9. FOR COMPLETION BY NONPROFIT ORGANIZATIONS AUTHORIZED TO MAIL AT SPECIAL RATES *(Section 132 122, PSM)*
The purpose, function, and nonprofit status of this organization and the exempt status for Federal income tax purposes *(Check one)*

[X] HAVE NOT CHANGED DURING PRECEDING 12 MONTHS
[ ] HAVE CHANGED DURING PRECEDING 12 MONTHS
(If changed, publisher must submit explanation of change with this statement.)

10. EXTENT AND NATURE OF CIRCULATION	AVERAGE NO. COPIES EACH ISSUE DURING PRECEDING 12 MONTHS	ACTUAL NO. COPIES OF SINGLE ISSUE PUBLISHED NEAREST TO FILING DATE
A. TOTAL NO. COPIES PRINTED *(Net Press Run)*	4,617	4,700
B. PAID CIRCULATION 1. SALES THROUGH DEALERS AND CARRIERS, STREET VENDORS AND COUNTER SALES	—	—
2. MAIL SUBSCRIPTIONS	4,194	4,241
C. TOTAL PAID CIRCULATION *(Sum of 10B1 and 10B2)*	4,194	4,241
D. FREE DISTRIBUTION BY MAIL, CARRIER OR OTHER MEANS SAMPLES, COMPLIMENTARY, AND OTHER FREE COPIES	25	25
E. TOTAL DISTRIBUTION *(Sum of C and D)*	4,219	4,266
F. COPIES NOT DISTRIBUTED 1. OFFICE USE, LEFT OVER, UNACCOUNTED, SPOILED AFTER PRINTING	398	434
2. RETURNS FROM NEWS AGENTS	—	—
G. TOTAL *(Sum of E, F1 and 2—should equal net press run shown in A)*	4,617	4,700

11. I certify that the statements made by me above are correct and complete.
SIGNATURE AND TITLE OF EDITOR, PUBLISHER, BUSINESS MANAGER, OR OWNER: NELSON W. FRIEDMAN, DIR, MGT SYS