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A NEW APPROACH TO CREATING COMPOSITE MATERIALS ELASTIC PROPERTY DATABASE WITH UNCERTAINTY ESTIMATION USING A COMBINATION OF MECHANICAL AND THERMAL EXPANSION TESTS (*)

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ABSTRACT

In composite structural design, a fundamental requirement is to furnish the designer with a set of elastic constants. For example, to design for a given temperature a laminate consisting of transversely isotropic fiber-reinforced laminae, we need five independent elastic constants of each lamina of interest, namely, E_1, E_2, ν_{12}, G_{12}, and ν_{23}.

At present, there exist seven tests, two of mechanical-lamina, two of thermal-expansion-lamina, and three of thermal-expansion-laminate types, to accomplish this task. It is known in the literature that the mechanical tests are capable of measuring E_1, E_2, and ν_{12}, whereas the two thermal-expansion-lamina tests will measure α_1 and α_2, and the three thermal-expansion-laminate tests yield an over-determined system of three simultaneous equations of the remaining two unknown elastic constants, G_{12} and ν_{23}.

In this paper, we propose a new approach to determining those five elastic constants with uncertainty bounds using the extra information obtainable from an over-determined system. The approach takes advantage of the classical theory of error propagation for which variance formulas were derived to estimate standard deviations of some of our five elastic constants. To illustrate this approach, we apply it to a set of experimental data on PEEK/IM7 unidirectional lamina.

The experiment consists of the following tests: Two tensile tests with four samples of unidirectional specimens to measure E_1, E_2 and ν_{12}; two thermal-expansion-lamina tests for coefficients (α_1 and α_2) each using four $[(0)_{32}]_T$ unidirectional specimens; and three thermal-expansion-laminate tests on four samples of $[(+30/-30)_8]_s$ laminates.

The results of our new approach are compared with those of a similar but more *ad hoc* approach that has appeared in the literature. The potential of applying this new methodology to the creation of a composite material elastic property database with uncertainty estimation and to the reliability analysis of composite structure is discussed.

I. INTRODUCTION

Knowledge of material elastic constants is fundamental for any structural analysis and design. Transversely isotropic unidirectional fiber reinforced composites are fully characterized by five independent elastic constants. Melo and Radford [1] has shown that three of these independent constants (E_1, E_2 and ν_{12}) are relatively easily, and accurately, measured using tensile tests. However, as shown in five ASTM standards on composite materials [2,3,4,5,6], the measurements of the other constants (G_{12}, and ν_{23}), particularly in the through-thickness direction (ν_{23}), involve more complicated techniques, apparatus and specimen preparation. Consequently, experimental data regarding these constants are often limited.

Relationships between mechanical properties and thermal expansion coefficients of composites have been described in the literature. Equations relating the elastic constants of a lamina and the coefficients of thermal expansion of the laminate have been developed (see, e.g., Melo and Radford [7]). This method allows the computation of any three of the five independent elastic constants, assuming that the other two can be experimentally measured from mechanical tests. However, the procedure and its equations presented by Melo and Radford [1] involve only average values and are therefore incomplete. A new approach allowing the creation of a composite material elastic properties database including uncertainty estimation is of great interest for a reliability analysis of composite structures.

II. STATEMENT OF THE PROBLEM

Unidirectional fiber reinforced composites are generally considered transversely isotropic materials. The experimental determination of all five independent elastic constants that completely characterize these materials using mechanical testing is challenging. Yet, information regarding this set of constants is fundamental for any structural design and analysis involving composite laminates composed by unidirectional fiber reinforced plies.

Out of the five independent elastic constants of each lamina, namely, E_1, E_2, ν_{12}, G_{12}, and ν_{23}, while three (E_1, E_2 and ν_{12}) can be directly determined from tensile tests, the determination of the remaining two constants involve complicated techniques and specimen preparation and may become a technical challenge.

Therefore, approaches able to determine all five elastic constants of transversely isotropic materials are of great interest.

III. A NEW APPROACH

The conventions used in this work are shown in Fig. 1. The coordinates (1; 2; 3) refer to the local coordinate system for a single layer or lamina, which corresponds to the fiber direction, the in-plane direction transverse to the fiber, and the through-thickness (lamination) direction, respectively. The coordinates (x; y; z) correspond to the global or laminate directions.

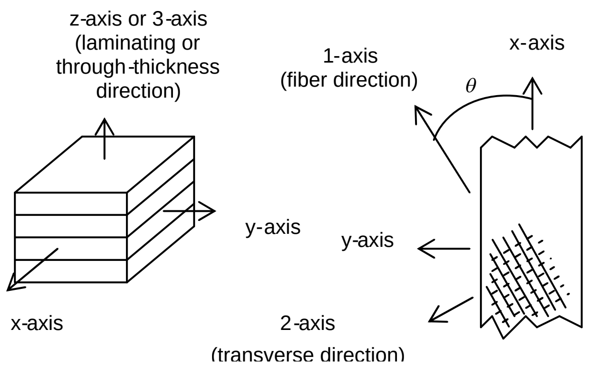

Figure 1. Definition of local or lamina reference axis (1-2-3) and global or laminate reference axis (x-y-z) [7].

"The general relationships which relate the coefficients of thermal expansion (CTEs) of a $[(+\theta/-\theta)_n]_s$ laminate and the laminae elastic constants have been developed by Melo and Radford [7], described in details by Tsai [15], and discussed by McCartney and Kelly [16]. It is well-known that the CTEs of a unidirectional composite lamina are not related to its elastic constants. However, in a multidirectional laminate, the difference in thermal expansion between the longitudinal and transverse directions gives rise to stresses among plies. The CTEs of a laminate can therefore depend on the CTSs of the laminae and their elastic constants as shown in Appendix A, Equation 4.33 of the book by Tsai [15]. For a transversely isotropic material, where plane (2; 3) coincides with the plane of isotropy, the mathematical expressions relating the coefficients of thermal expansion of a $[(+30/-30)_n]_s$ laminate and the laminae elastic constants are as follows [1, 15]:"

$$E_2 = \frac{E_1\left(4\alpha_1 - 3\alpha_x(30) - \alpha_y(30)\right)}{4\nu_{12}\left(\alpha_x(30) + \alpha_y(30) - \alpha_1 - \alpha_2\right) + \alpha_x(30) + 3\alpha_y(30) - 4\alpha_2} \tag{1}$$

$$G_{12} = \frac{E_1\left(4\alpha_1 - 3\alpha_x(30) - \alpha_y(30)\right)}{6\left(\nu_{12}+1\right)\left(\alpha_x(30) - \alpha_y(30)\right)} \tag{2}$$

$$\nu_{23} = \frac{\nu_{12}\left(4\alpha_1 + 4\alpha_2 - 3\alpha_x(30) - \alpha_y(30) - 4\alpha_z(30)\right) + 4\alpha_2 - 4\alpha_z(30)}{4\nu_{12}\left(\alpha_x(30) + \alpha_y(30) - \alpha_1 - \alpha_2\right) + \alpha_x(30) + 3\alpha_y(30) - 4\alpha_2} \tag{3}$$

Where E_1, E_2 and ν_{12} are experimentally measured elastic constants; $\alpha_x(30)$, $\alpha_y(30)$ and $\alpha_z(30)$ are the CTEs for the $[(+30/-30)_n]_s$ laminate in *x*, *y*, and *z* directions, respectively; α_1 and α_2 are the CTEs for the unidirectional lamina in the 1 and 2 (or 3) directions, respectively. Since these equations are also dependent on α_1 and α_2, CTE measurements of unidirectional laminates in directions 1 and 2 are necessary to determine the laminae elastic constants.

These equations (1 to 3) have been used to calculate three of the five independent elastic constants (E_2, G_{12}, and ν_{23}) based on the other two (E_1 and ν_{12}) and the CTEs for the unidirectional lamina and for the $[(+30/-30)_n]_s$ laminate [1,7]. In other words, the average elastic modulus E_2, the average shear modulus G_{12} and the average through-thickness Poisson's ratio, ν_{23}, were determined based on the average values of E_1, ν_{12} and a set of average values of laminate CTE's. No attempt was made to estimate the standard deviations of those parameters, nor the 95 % confidence intervals that are critically needed in a reliability analysis of composite structures.

The new approach proposed in this paper takes advantage of the classical theory of error propagation and apply the variance formulas developed by Ku [8] to equations (1), (2) and (3), using experimentally measured elastic constants, namely E_1, E_2 and ν_{12}, and a set of CTE data of laminates, to determine three elastic constants of a transversely isotropic lamina, namely, E_2, G_{12}, and ν_{23} with uncertainty bounds. This approach also takes advantage of the extra information obtained from the over-determined system, *i.e.*, an experimental value of E_2, with sample mean and standard deviation, and a calculated one, with average based on Equation (1) and an estimated standard deviation using Ku's variance formulas [8].

IV. AN EXAMPLE APPLICATION

To illustrate this new approach, we choose to present a numerical example based on the determination of the elastic properties of PEEK/IM7 unidirectional prepreg lamina. Tensile tests were conducted in unidirectional specimens for the determination of E_1, E_2 and ν_{12}. CTE measurements were also conducted in unidirectional specimens for the determination of α_1 and α_2 and in $[(+30/-30)_8]_s$ laminates for the determination of α_x, α_y and α_z.

Material and Specimen Preparation

The material tested was APC-2/IM7, PEEK/carbon unidirectional prepreg tape, manufactured by Cytec Engineered Materials, with a quoted resin content of 32% and fiber areal weight of 145g/m^2. Two 150x200 mm unidirectional laminates were processed for the preparation of the tensile coupons. A laminate with fibers oriented at 0° with respect to the largest tool dimension was used to fabricate specimens for the determination of E_1 and ν_{12}. The other laminate was manufactured with fibers at 90^o with respect to the largest tool dimension and used to fabricate specimens for the determination of E_2. Eight prepreg layers were used for the 0° laminate and sixteen layers for the 90° laminate. In addition to the two unidirectional laminates for the tensile tests, two 64x178 mm laminates, a 32 ply $[(+30/\ 30)_8]_s$ and a 32 ply unidirectional $[(0)_{32}]_T$, were produced for the CTE specimens fabrication.

The laminates were processed in a hot press using an aluminum tool at a temperature of 395°C for 45 minutes, under an applied pressure of 690 KPa (100 psi). First, the lay-ups were heated at a rate of 5°C/min and the pressure was gradually applied after the temperature reached 340°C. After the processing was completed, the laminates were cooled, under pressure, to room temperature, at a rate of 3 to 5°C/min. The specimens were cut from the processed laminates using a diamond saw. The final dimensions of the tensile tests specimens were nominally 15x200x1 mm, for the 0°, and 25x175x2 mm, for the 90° specimens. The CTE specimen dimensions were nominally 5x5x5 mm. After cutting, these specimens were fixtured to grind and polish all faces, square and flat, using a grinding/polishing custom-made fixture [1].

Testing

The tensile tests were carried out in an ATS Series 910 universal testing machine, at 20°C and using a constant cross head displacement rate of 1.0 mm/min. Strain gage rosettes (EA 13 125RA 120 from Measurements Group, Inc.) were used for the strain measurements. The coefficients of thermal expansion (CTE's) were measured over the temperature range of 10°C to 30°C using a Thermo Mechanical Analyzer with a quartz probe and stem (Seiko 5200 TMA/SS 120C). All CTE measurements were performed using a heating rate of 1.0°C/min and a constant applied load of 20 g.

Results

The results of the mechanical tests in unidirectional coupons are presented in Table 1. In Table 2, the results for the CTE measurements in unidirectional laminates are presented. The difference between α_2 and α_3, is only about 1%. This small difference is considered to satisfy the condition of $\alpha_2 = \alpha_3$, as implied by transverse isotropy. For the computation of the elastic constants, α_2 is used. The measured CTE data of $[(+30/\ 30)_8]_s$ specimens in three perpendicular directions are presented in Table 3. All data presented in Tables 1 to 3 were averaged from four different specimens for each of the measured property.

Table 1. Tensile test measured elastic constants.

Elastic constant	$\bar{x}$	s_{n-1}
E_1 *(GPa)*	164.4	3.0
E_2 *(GPa)*	9.9	0.2
ν_{12}	0.34	0.01

where: $\bar{x}$ = sample mean (average); s_{n-1} = sample standard deviation.

Table 2. TMA measured CTE data of unidirectional specimens.

CTE (μm/m/ºC)	$\bar{x}$	s_{n-1}
α_1	-0.545	0.331
α_2	26.51	0.59
α_3	26.80	0.53

where: $\bar{x}$ = sample mean (average); s_{n-1} = sample standard deviation.

Table 3. TMA measured CTE data of $[(+30/\ 30)_8]_s$ specimens.

CTE (μm/m/ºC)	$\bar{x}$	s_{n-1}
$\alpha_x(30)$	-2.82	0.61
$\alpha_y(30)$	13.46	0.43
$\alpha_z(30)$	34.82	0.47

where: $\bar{x}$ = sample mean (average); s_{n-1} = sample standard deviation.

The CTE measurements presented in Tables 2 and 3 indicate a relatively large standard deviation for α_1 and $\alpha_x(30)$, where the thermally induced displacements are very small.

The approximate formulas for calculating variance (var) of sum, quotient, and product of two algebraic quantities have appeared in the literature since the 1960s (see, e.g., Ku [8]), and have been applied to the study of reliability of engineering materials such as metals [9, 10], and ceramics [11, 12]. In Ku's 1966 paper, a total of 14 formulas are listed, of which three are used in this paper. For completeness, those three formulas are reproduced in Table 4.

Table 4. Three variance formulas based on the theory of error propagation (after Ku [8], or Appendix A of Fong, et al, [9]).

#	*Function form*	*Approx. formula for variance*
1	$w = A\bar{x} + B\bar{y}$	$\mathrm{var}(w) = A^2 \cdot \mathrm{var}(x) + B^2 \cdot \mathrm{var}(y)$
2	$w = \bar{x} / \bar{y}$	$\mathrm{var}(w) = \dfrac{\mathrm{var}(x)}{(\bar{y})^2} + \dfrac{\mathrm{var}(y) \cdot (\bar{x})^2}{(\bar{y})^4}$
3	$w = \bar{x} \cdot \bar{y}$	$\mathrm{var}(w) = (\bar{y})^2 \cdot \mathrm{var}(x) + (\bar{x})^2 \cdot \mathrm{var}(y)$

where: $\bar{x}$ = sample mean of x; $\bar{y}$ = sample mean of y; s_{n-1} = sample standard deviation; var. = square of std. dev. $(s_{n-1})^2$.

<u>Note:</u> Formulas in Table 4 are strongly valid for Coefficient of Variation ($c.v. = \dfrac{s_{n-1}}{\bar{x}}$) of each quantity less than 0.10; approximately valid for c.v. of each quantity not greater than 0.20; and not valid for c.v. greater than 0.20.

The experimental data from tables 1, 2 and 3 with the square of the mean values and variance are listed in Table 5.

Table 5. Measured parameters of APC-2/IM7.

Elastic constant	$\bar{x}$	$\bar{x}^2$	s_{n-1}	*var(x)*
E_1 *(GPa)*	164.4	$2.703 \cdot 10^4$	3.0	9.00
ν_{12}	0.34	$1.156 \cdot 10^{-1}$	0.01	$1.0 \cdot 10^{-4}$
α_1	-0.545	$2.25 \cdot 10^{-2}$	0.331 (*)	$1.095 \cdot 10^{-1}$
α_2	26.51	$7.028 \cdot 10^2$	0.59	$3.481 \cdot 10^{-1}$
$\alpha_x(30)$	-2.82	7.952	0.61 (*)	$3.721 \cdot 10^{-1}$
$\alpha_y(30)$	13.46	$1.812 \cdot 10^2$	0.43	$1.849 \cdot 10^{-1}$
$\alpha_z(30)$	34.82	$1.212 \cdot 10^3$	0.47	$2.209 \cdot 10^{-1}$

(*) Coefficient of variation greater than 0.20, thus making Ku's formulas (Table 4) invalid [8]. However, Ku's formulas are used in this paper to illustrate the methodology in cases where the coefficient of variation of some quantities may exceed 0.20.

By substitution of the data from Table 5, the estimated mean of E_2 can be calculated using Equation (1) according to the procedure shown in Table 6:

Table 6. Parameters for estimated mean of E_2.

Parameter	*Parameter*^{2}
$w_1 = 4\alpha_1 - 3\alpha_x(30) - \alpha_y(30) = -7.18$	$5.1552 \cdot 10$
$w_2 = E_1 \cdot w_1 = -1.1804 \cdot 10^3$	$1.3933 \cdot 10^6$
$w_3 = \alpha_x(30) + 3\alpha_y(30) - 4\alpha_2 = -6.848 \cdot 10$	$4.6895 \cdot 10^3$
$w_4 = \alpha_x(30) + \alpha_y(30) - \alpha_1 - \alpha_2 = -1.5325 \cdot 10$	$2.3486 \cdot 10^2$
$w_5 = \nu_{12} \cdot w_4 = -5.2105$	$2.7149 \cdot 10^2$
$w_6 = 4 \cdot w_5 + w_3 = -8.9322 \cdot 10$	$7.9784 \cdot 10^3$

Thus, the estimated mean of E_2 is given by

$$E_2 = \frac{w_2}{w_6} = \frac{-1.1804 \cdot 10^3}{-8.9322 \cdot 10^1} = 13.215\, GPa$$

The estimated standard deviation of E_2 can be calculated using the formulas shown in Table 4. The results are shown in Table 7.

Table 7. Parameters for estimated standard deviation of E_2.

Parameter	*var*
$\mathrm{var}(w_1) = 16\,\mathrm{var}(\alpha_1) + 9\,\mathrm{var}(\alpha_x(30)) + \mathrm{var}(\alpha_y(30))$	5.286776
$\mathrm{var}(w_2) = w_1^2 \cdot \mathrm{var}(E_1) + E_1^2 \cdot \mathrm{var}(w_1)$	$1.4335 \cdot 10^5$
$\mathrm{var}(w_3) = \mathrm{var}(\alpha_x(30)) + 9\,\mathrm{var}(\alpha_y(30)) + 16(\alpha_2)$	7.6058
$\mathrm{var}(w_4) = \mathrm{var}(\alpha_x(30)) + \mathrm{var}(\alpha_y(30)) + \mathrm{var}(\alpha_1) + \mathrm{var}(\alpha_2)$	1.01466
$\mathrm{var}(w_5) = w_4^2 \cdot \mathrm{var}(\nu_{12}) + \nu_{12}^2 \cdot \mathrm{var}(w_4)$	$1.4078 \cdot 10^{-1}$
$\mathrm{var}(w_6) = 16\,\mathrm{var}(w_5) + \mathrm{var}(w_3)$	9.85830
$\mathrm{var}(E_2) = \dfrac{\mathrm{var}(w_2)}{w_6^2} + \dfrac{\mathrm{var}(w_6) \cdot w_2^2}{w_6^4}$	$1.8183 \cdot 10^1$

Therefore, the estimated standard deviation of E_2 is 4.264 GPa. Based on this standard deviation, the coefficient of variation of E_2 is c.v. = 0.323 (high).

Thus, while the experimental sample mean of E_2 is 9.9 GPa with a sample standard deviation of 0.2 GPa (Table 1), the estimated mean of E_2 equals 13.215 GPa with a sample standard deviation of 4.264 GPa.

A two-sided t-test [13. 14] is conducted to determine if the estimated value of E_2 is acceptable with 95 % confidence as compared with the experimental data. In this case, the sample size was (n = 4) and the number of degrees of freedom is (nu = n – 1 = 3). The "null hypothesis" is that E_2 = 9.9 GPa.

A two-sided *t*-test begins with calculating the *t*-statistic given by the following equation:

$$t = \frac{13.215 - 9.90}{\left(\frac{4.264}{\sqrt{4}}\right)} = \frac{3.315}{2.132} = 1.555$$

From a table of *t*-values (see, e.g., [13, 14]), $t\,(\alpha/2, \text{n-1}) = t\,(0.025, 3) = 3.183$ is obtained. Since 1.555 is not greater than 3.183, the null hypothesis is not rejected, *i.e.*, the estimated mean of E_2 is statistically valid with a 95 % confidence when compared with the experimentally measured sample mean of E_2.

Similar procedure can be used to calculate the estimated mean and standard deviations of G_{12} and ν_{12}. The measured elastic constants, E_1 and ν_{12}, and the CTE's, (α_1 and α_2) and ($\alpha_x(30)$, $\alpha_y(30)$ and $\alpha_z(30)$), are substituted into equations (2), and (3). Based on these values, the estimated mean of G_{12} is 9.018 GPa with a sample standard deviation of 2.923 GPa. For ν_{12}, the estimated mean is 0.499 with a sample standard deviation of 0.039.

The coefficients of variation are c.v. = 0.324 (high) for G_{12} and c.v. = 0.10 (good) for ν_{12}.

In summary, the procedure described yielded all five elastic constants for the transversely isotropic lamina with uncertainty estimation. The results are presented in Table 8.

Table 8. Elastic constants of APC-2/IM7.

Elastic constant	$\bar{x}$	s_{n-1}
E_1 *(GPa)*	164.4	3.0
ν_{12}	0.34	0.01
E_2 *(GPa)*	13.215	4.264
G_{12} *(GPa)*	9.018	2.923
ν_{23}	0.499	0.039

where: $\bar{x}$ = sample mean (average); s_{n-1} = sample standard deviation.

V. SIGNIFICANCE OF RESULTS

In a previous work, the relationships presented in equations (1), (2) and (3) were used in the three dimensional elastic characterization of a PEEK/IM7 unidirectional prepreg lamina [1]. The elastic modulus E_2, the shear modulus G_{12} and the through-thickness Poisson's ratio, ν_{23}, were determined based on known values of E_1, ν_{12} and a set of laminate CTE's. In this previous work, uncertainty bounds were not included.

With the new approach presented in this work, all three measured elastic constants are used for the determination of the complete set of five elastic constants with uncertainty bounds. Using the extra information obtained from the over-determined system, a two-sided t-test is conducted to determine if the estimated value of the redundant parameter is acceptable within a given confidence as compared with the experimental data.

VI. CONCLUDING REMARKS

Information including only average values of the material elastic constants is incomplete and thus should not be used for structural analysis and design. The methodology presented in this study allows using a combination of CTE measurements and standard tensile tests, for the determination of the three-dimensional elastic constants of a transversely isotropic lamina with uncertainty bounds. The approach was used in the three-dimensional elastic characterization of a PEEK/IM7 unidirectional prepreg, resulting in the determination of all the independent constants with uncertainty bounds. A t-test was conducted to compare the estimated mean of one of the elastic parameters with the experimentally measured sample mean and the estimated mean was found to be statistically valid.

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