Lamina properties non-destructive characterisation of asymmetric carbon fiber reinforced laminates

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Abstract. A non-destructive technique – numerical-experimental method, based on vibration tests is applied for the characterization of lamina properties of asymmetric carbon fiber reinforced laminates. The main idea of this procedure is the determination of simple mathematical models (response surfaces), using the finite element analysis at the reference points of experimental design. Therefore, a significant reduction of computational efforts can be attained. For the purpose of characterization, two asymmetric carbon fiber laminated specimens were prepared, using the prepreg manual layout technology. To evaluate the elastic material properties obtained, the numerical resonant frequency, determined by using identified material properties, were successfully compared with experimentally measured and high accuracy with average relative error less than 1% was reached.

1. Introduction
Carbon fiber reinforced plastics are widely used now in different lightweight constructions requiring high stiffness-to-weight and strength-to-weight ratios. Unfortunately, the material data provided by manufacturers very often do not contain all necessary information for the analyses [1], design [2] and optimization [3-5] of advanced composite structures. For this reason, the technical data of such materials are estimated in the research laboratories by using conventional fracture methods [6], ultrasonics [7] or inverse technique based on low-frequency vibrations [8-10]. Very often the conventional fracture and ultrasonic methods do not give a desired accuracy of the obtained results or do not allow to find all the required parameters of composite laminates with arbitrary lamina angles. In this case mixed numerical-experimental technique could be successfully applied.

The general idea of the inverse technique based on vibration tests is minimization of the error functional between experimental and numerical parameters of structural responses. [11-13] This approach is especially useful for a characterization of laminated composites since it has a non-destructive character that enables many repeated tests on the same specimen, and provides more flexibility and less restriction in a choice of the test specimen geometry avoiding by this way a damage introduction in the test specimen. Moreover, an influence of experimental and numerical errors on the identified material properties have been intensively investigated [14-16] that allows to speak about high accuracy and reliability of the mixed numerical-experimental technique based on vibration tests.

In most cases, an identification of material properties from vibration tests is carried out using unidirectional or symmetric laminates. The present investigations demonstrate that asymmetric carbon
fiber reinforced laminates could be used also for an accurate characterization of lamina mechanical properties by using the inverse technique based on low-frequency vibrations.

2. Inverse technique
The inverse technique in the present work is realised by using vibration tests and includes physical experiments, numerical modelling and identification procedure, consisting of planning of experiments, response surface methodology and minimization of error functional (Figure 1). First of all, the plan of experiments is created in dependence on the number of experiments and number of identified parameters. Then the finite element code is used for the modelling of reinforced composite laminates. To obtain dynamic parameters, the finite element analysis is applied in the reference points of experimental design. In the next step, the acquired numerical data are used for the determination of approximation functions, using the response surface method. The resonant frequencies are determined from the experimental data of vibration tests. In the final stage, the required parameters of material are identified by minimizing the error functional that describes the residuals between the experimental and numerical data.

2.1. Vibration tests
The vibration testing is fast and low-cost method for an identification of elastic properties. To study the behavior of specimens, one of the common used procedure - experimental modal analysis - is applied. During this analysis, consequent modal parameters, like modal frequency and shape of mode, are determined. The frequency response functions of the system are provided by using the known forces and appropriate output vibrational responses. Usually, the forces are applied at a number of locations and input signals are measured in many points.

2.2. Numerical modelling
In the present inverse procedure, the finite element method is used for the modelling and dynamic analysis of laminated composite specimens. The finite element code ANSYS 19.2 is applied for modelling the composite specimens using the first order shear deformation theory. In this case the displacements can be expressed:

\[ u = u_0 + z \gamma_x, \quad v = v_0 + z \gamma_y, \quad w = w_0 \]  

where \( u_0, v_0, w_0 \) are the displacements in the reference plane, \( z \) is the coordinate of the point of interest in the reference plane, and \( \gamma_x, \gamma_y \) are the rotations according to the transverse shear deformations. Then the following eigenvalue problem is solved:

\[ K \mu = \omega^2 M \mu, \]  

Figure 1. The inverse technique.
where $K$ is the stiffness matrix, $M$ is the mass matrix, $u$ is the displacement vector and $\omega$ is the resonant frequency. The eigenvalue problem was solved by the Lanczos method.

2.3. Material identification procedure

The material identification procedure is based on vibration tests and response surface methodology. The main idea of this procedure is the determination of simple mathematical models (response surfaces) using only the finite element analysis in the reference points of experimental design. Minimizing the error functional, that describes the residuals between the experimentally and the numerically calculated frequencies, the identified parameters are obtained.

2.3.1. Planning of experiments

There are different methods for the planning of experiments, for example, D-Optimal, Central Composite, Factorial, Uniform Design, Latin Hypercube, Orthogonal Array, Minimal MSD, etc. In the present work, the points of experiments are distributed as uniformly as possible and there is a physical relevance to the minimum of potential energy of repulsive forces for a set of points of unit mass. For this reason, the consequent criterion is used:

$$
\Phi = \sum_{i=1}^{k} \sum_{j=i+1}^{k} \frac{1}{l_{ij}^2} \Rightarrow \min
$$

where $l_{ij}$ is the distance between the points with numbers $i$ and $j$ ($i \neq j$). The matrix $B_\eta$ describes the plan of experiment, where the domain of factors and design space is determined as $x_j \in [x_j^{\min}, x_j^{\max}]$. The points of experiments are calculated by a consequent expression:

$$
x_j^{(i)} = x_j^{\min} + \frac{1}{k-1} \left( x_j^{\max} - x_j^{\min} \right) \left( B_\eta - 1 \right),
$$

where $i = 1, 2, \ldots, k$, $j = 1, 2, \ldots, n$. The basic information for the development of the plan is the number of experiments $k$ and number of the design parameters $n$. It is possible to determine a plan of experiment for each $k$ and each $n$, but in that case, it enlarges computational efforts. Thereby, each plan of experiment is determined only once and can be applied for different design situations. The minimal number of experiments is obtained as follows:

$$
k = (n + 1) \cdot (n + 2)
$$

2.3.2. Response Surface Technique

For the approximation of the numerical results, the response surface methodology is used. There are various methods of the approximation that can be applied for the development of the plan of experiments – Polynomial, Rational Functions, Kriging, Splines, Neural Networks etc. Approximations can be completed by the second order polynomials using the least square method:

$$
\bar{F}(x) = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \sum_{j=i}^{k} \beta_{ij} x_i x_j
$$

The error of approximation is determined using the following equations:
\[ \sigma = \sqrt{\frac{\sum_{i=1}^{N} (F(x^i) - \bar{F}(x^i))^2}{N}}; \quad \sigma_0 = \sqrt{\frac{\sum_{i=1}^{N} (F(x^i) - \bar{F}(x^i))^2}{N - L}}, \]

where \( \sigma \) is the squared error, \( \sigma_0 \) is the standard deviation, \( F(x^i) \) and \( \bar{F}(x^i) \) are the values of original and approximating functions in the sample points \( x^i \) of experiments, and \( N \) is the number of sampling points.

2.3.3. Minimization of error functional

In order to identify the required elastic material properties, the error functional, that describes the residuals between the numerical and experimental parameters of structural responses, should be formulated:

\[ \Phi(x) = \sum_{i=1}^{I} \left( \frac{f_i^{\text{EXP}}}{f_i^{\text{EXP}}} - \left( f_i^{\text{FEM}}(x) \right)^2 \right)^2 \Rightarrow \min \]

where \( f_i^{\text{EXP}} \) are experimentally measured frequencies; \( f_i^{\text{FEM}} \) are resonant frequencies of regression equations (determined by the response surface method using numerical calculations); \( I \) is the number of resonant frequencies used in the identification method. A constrained nonlinear optimization problem for the present minimization should be solved:

\[ \min \Phi(x), H_i(x) \geq 0, G_j(x) = 0 \]

\[ i = 1,2,..., I, j = 1,2,..., J, \]

where \( I \) and \( J \) are the numbers of equality and inequality constraints. This minimization problem is replaced by an unconstrained problem; therefore, the constraints are taken into account with the penalty functions. In order to solve the optimization problem, a random search method is used.

3. Samples preparation

While solving the inverse problem of determining the elastic characteristics, multilayer asymmetric carbon fiber samples were manufactured. The specimens consist of four anisotropic layers with the following reinforcement schemes \([0°/90°/0°/90°]\) and \([-45°/45°/-45°/45°]\). Selected thicknesses and reinforcement schemes are applied in the manufacture of power shells in many aircraft structures. Dimensions for both specimens are given in Table 1.

| Dimension parameters of specimens | Specimen 1 \([0°/90°/0°/90°]\) | Specimen 2 \([-45°/45°/-45°/45°]\) |
|-----------------------------------|-----------------|-----------------|
| Mass \( m \), g                  | 22.9            | 25.4            |
| Width \( b \), mm                 | 44.9            | 45.0            |
| Length \( L \), mm                | 400.0           | 400.0           |
| Thickness \( t \), mm             | 0.8             | 0.9             |
| Density \( \rho \), kg/m³         | 1593.5          | 1422.6          |
For manufacturing samples, the prepreg manual layout technology was chosen. The carbon fabric uniformly impregnated with a polymer binder was used. The prepreg technology allows to obtain complex shape monolithic products with minimal tooling. The technology of prepreg molding has the following characteristics: low complexity of laminate laying, clean process, high quality composite material, increased requirements for tooling and support materials (high polymerization temperature). The essence of the method is the following: cutting sheets on a single-sided matrix of the required shape to obtain the required thickness, vacuuming under the polyethylene film, curing in an autoclave at high temperature and pressure. CM-PregF-T27 200/1250 CP0041 45 prepreg carbon fiber was used as the samples material.

The process of samples manufacturing can be divided into the following steps:
1) Prepreg patterns marking;
2) Cutting of the prepreg on the plotter (Figure 2 (a));
3) Laying the cut prepreg on a one-sided matrix of the required shape to obtain the required thickness in accordance with the reinforcement scheme;
4) Strengthening the prepreg over the entire area of adhesion (Figure 2 (c));
5) Laying of auxiliary layers on the prepreg surface: perforated film and absorbent layer (Figure 2 (d));
6) Sealing the mold with a vacuum film and providing a vacuum (Figure 2 (b), (e));
7) Placing the laid prepreg into the autoclave and setting the required curing parameters (Figure 2 (f));
8) Removing the cured laminate from the autoclave and removing the vacuum film after the curing process is complete (Figure 2 (g), (h));
9) Mechanical processing of the cured laminate to obtain the samples of the required size.

Figure 2. Stages of carbon fiber prepreg samples manufacturing by the method of manual layout.
According to the results of the realized technological experiments, the production modes were selected for the method of prepreg manual layout with autoclave molding and sample cutting, which made it possible to manufacture defect-free samples for further testing.

4. Identification of material properties

Two samples of asymmetric carbon fiber reinforced laminates are tested for the identification of following elastic constants:

- two Young’s moduli: $E_1$, $E_2 = E_3$;
- shear moduli: $G_{12} = G_{13}$;
- Poisson’s ratio: $\nu_{12} = \nu_{13}$.

It should be noted that not all of the elastic constants are sensitive to frequencies, so some of them are assumed as fixed values [11]:

- shear modulus: $G_{23} = 3.5$ GPa.
- Poisson’s ratio: $\nu_{23} = 0.3$.

For the vibration tests, the experimental set-up, based on Scanning Laser Vibrometer POLYTEC, is used for the samples with free-free boundary conditions. For this boundary condition, samples are hanged on thin threads, fixed on the top of samples (Figure 3).

The measurements are done at specified scanning points. The signal analyser converts the average values of input and output signals into the frequency domain, using the Fourier transformation. From the response frequency function the structural resonant frequencies and shapes of mode can be easily obtained. After that, the resonant frequencies are calculated by the function transformation to the modal analysis program. During the physical experiments, 9 first resonant frequencies are measured for the specimen 1 and 12 first resonant frequencies – for the specimen 2.

The plan of experiments for the composite specimens is formulated for 4 design parameters and 30 experiments. The limits for the maximum and the minimum values of elastic parameters are given in Table 2.

![Figure 3. Experimental set-up.](image)

The finite element model of samples is built by linear structural shell elements SHELL 181. The element SHELL 181 is a four-node element and it is suitable for the analysis of thin shells. The finite element has six degrees of freedom in each node – 3 displacements in $x$, $y$ and $z$ directions and 3 rotations about $x$, $y$ and $z$ axes. In order to achieve the necessary accuracy for the 16 first frequencies of specimen
1, 125x14 regular finite element mesh is developed, but for the 13 first frequencies of specimen 2 – 107x12 regular mesh.

### Table 2. Boundaries of identified parameters for composite specimens.

| Parameters of identification | Specimen 1 | Specimen 2 |
|------------------------------|------------|------------|
|                              | Minimum value | Maximum value | Minimum value | Maximum value |
| Young’s modulus $E_1$, GPa  | 110        | 145        | 75           | 105           |
| Young’s modulus $E_2 = E_3$, GPa | 12      | 14.5       | 5           | 8            |
| Shear modulus $G_{12} = G_{13}$, GPa | 4.5      | 6         | 2.5         | 4            |
| Poisson’s ratio $\nu_{12} = \nu_{13}$ | 0.2    | 0.4       | 0.2         | 0.4          |

For the approximation and optimization of results, the program EDAOPT was used. As a result of approximation procedure, the equations of regressions were obtained for each resonant frequency. To determine the elastic constants of both specimens, the error functional was minimized. The identified elastic constants for both specimens are presented in Table 3.

### Table 3. Identified elastic constants of composite specimens.

| Dimension parameters of specimens | Specimen 1 | Specimen 2 |
|-----------------------------------|------------|------------|
| Young’s modulus $E_1$, GPa       | 137.0      | 91.8       |
| Young’s modulus $E_2 = E_3$, GPa | 13.6       | 5.75       |
| Shear modulus $G_{12} = G_{13}$, GPa | 5.3    | 3.2        |
| Shear modulus $G_{23}$, GPa – fixed value | 3.5 | 3.5 |
| Poisson’s ratio $\nu_{12} = \nu_{13}$ | 0.33 | 0.34 |
| Poisson’s ratio $\nu_{23}$ – fixed value | 0.30 | 0.30 |

To verify the results, the experimentally measured frequencies were compared to the numerical resonant frequencies, obtained by using the identified material properties. The accuracy of eigenvalues was determined by calculating the relative errors:

$$\Delta_i = \left| \frac{f_i^{\text{FEM}} - f_i^{\text{EXP}}}{f_i^{\text{EXP}}} \right| \cdot 100\%,$$

(10)

The obtained results are presented in Table 4 and it is seen that for both specimens the average relative errors are less than 1%.

### 5. Conclusions

An inverse technique, based on vibration tests and response surface methodology, was successfully applied for an identification of the elastic material properties of asymmetric carbon fiber reinforced composite specimens. The main advantage of the identification procedure, based on non-direct optimization methodology, was the reduction of computational efforts.
Table 4. Frequencies and relative errors for composite specimens.

| Mode | Specimen 1 | Specimen 2 |
|------|------------|------------|
|      | EXP (Hz)   | FEM (Hz)   | Δi (%)     | EXP (Hz)   | FEM (Hz)   | Δi (%)     |
| 1    | 33.250     | 33.146     | 0.31       | 18.125     | 18.149     | 0.13       |
| 2    | 81.875     | 82.024     | 0.18       | 52.250     | 52.099     | 0.29       |
| 3    | 91.750     | 91.383     | 0.40       | 106.875    | 106.717    | 0.15       |
| 4    | -          | 169.878    | -          | 183.750    | 183.795    | 0.02       |
| 5    | 179.375    | 179.191    | 0.10       | 204.500    | 204.483    | 0.01       |
| 6    | -          | 268.810    | -          | 284.000    | 284.354    | 0.12       |
| 7    | 296.625    | 296.308    | 0.11       | 407.875    | 408.764    | 0.22       |
| 8    | -          | 383.332    | -          | 414.000    | 410.411    | 0.87       |
| 9    | 443.000    | 442.815    | 0.04       | 556.375    | 557.063    | 0.12       |
| 10   | -          | 517.288    | -          | 617.500    | 619.223    | 0.28       |
| 11   | 619.250    | 618.673    | 0.08       | 727.500    | 729.118    | 0.22       |
| 12   | 824.250    | 824.298    | 0.01       | -          | 792.879    | -          |
| 13   | -          | 855.398    | -          | 829.625    | 832.352    | 0.33       |
| 14   | 1058.375   | 1059.459   | 0.10       | -          | -          | -          |
| 15   | -          | 1063.677   | -          | -          | -          | -          |
| 16   | -          | 1299.833   | -          | -          | -          | -          |
| AVERAGE | -          | -          | 0.13       | -          | -          | 0.23       |

For the purpose of identification, two asymmetric carbon fiber samples were prepared. To manufacture defect-free samples, the method of prepreg manual layout with autoclave molding and sample cutting, was applied.

The results, obtained during the identification procedure and the dynamic analysis, were sufficiently accurate for both samples as the values of average relative errors were less than 1%. It is necessary to note that out-of-plane material properties could be identified also using the present methodology. However, in this case the thickness of tested samples should be increased (not less than 4 mm).

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