

# Characterization of Adhesive Layers in Sandwich Composites by Nondestructive Technique

E. Barkanov, E. Skukis, M. Wesolowski, and A. Chate

**Abstract**—New nondestructive technique, namely an inverse technique based on vibration tests, to characterize nonlinear mechanical properties of adhesive layers in sandwich composites is developed. An adhesive layer is described as a viscoelastic isotropic material with storage and loss moduli which are both frequency dependent values in wide frequency range. An optimization based on the planning of experiments and response surface technique to minimize the error functional is applied to decrease considerably the computational expenses. The developed identification technique has been tested on aluminum panels and successfully applied to characterize viscoelastic material properties of 3M damping polymer ISD-112 used as a core material in sandwich panels.

**Keywords**—Adhesive layer, finite element method, inverse technique, sandwich panel, vibration test, viscoelastic material properties.

## I. INTRODUCTION

**D**UE to great importance to define performance, reliability and safety requirements for advanced composite products and services a considerable effort has been devoted to the study of their mechanical material properties and a lot of different methods have been developed in the last three decades. For many orthotropic sheet materials a measurement method based on low frequency vibrations [1] is not only the simplest approach, it is also the only approach, which does not suffer from grave difficulties of principle, when the results are used to make predictions in the same range of frequencies. There are two other general methods might be used - static measurements [2] and ultrasonics [3], but neither is wholly appropriate for characterization of mechanical material properties of advanced composites. The difficulties are most obvious, when it comes to the determination of damping "constants", since these would be expected to be frequency dependent.

Only some researchers [4]-[6] tried to identify the viscoelastic material properties of sandwich composites using an inverse technique based on vibration tests. The forced steady state harmonic vibrations and free vibrations have been utilized in the identification procedure in paper [4] to

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characterize the constitutive parameters of Voigt model and three parameters model of uniaxial viscoelasticity used for a description of viscoelastic materials applied in sandwich beams. These beams consist of a core made of polyvinyl chloride foam and two laminate faces made of glass fiber reinforced polyester. To identify parameters of aluminum honeycomb sandwich panels, an orthotropic Timoshenko beam model has been applied, and the elastic constants and modal damping ratios have been determined in paper [5] minimizing the error between experimental and analytical results. The paper [6] proposes an inverse method based on the flexural resonance frequencies and using the sandwich beam theory for the finite element modeling. It is necessary to note that the examined approaches do not give the possibility to analyze sandwich structures with high damping and to characterize their viscoelastic material properties in wide frequency range, when storage and loss moduli are frequency dependent values.

On this reason the present investigations are focused on the development of new inverse technique based on vibration tests to characterize nonlinear mechanical properties of adhesive layers in different sandwich applications. The developed identification technique has been tested on aluminum panels and applied to characterize viscoelastic material properties of 3M damping polymer ISD-112 used as a core material in sandwich panels with aluminum faces.

## II. INVERSE TECHNIQUE

The basic idea of material identification procedure developed on vibration tests and nondirect optimization methodology is that simple mathematical models (response surfaces) are determined only by the finite element solutions in the reference points of the plan of experiments. The identification parameters for all eigenfrequencies in the examined frequency range are obtained minimizing the error functional, which describes a difference between the measured and numerically calculated parameters of structural response. A significant reduction in calculations of the identification functional is achieved in this case in comparison with the conventional optimization methods, since only one design space is necessary to identify adhesive material properties in the desired frequency range.

The present inverse technique (Fig. 1) consists of experimental set-up, numerical model and material

identification procedure. In the first stage a plan of experiments is produced in dependence on the number of identified parameters and number of experiments. Then finite element analysis is performed in the reference points of experimental design and dynamic parameters of structure are calculated. In the third stage these numerical data are taken to determine simple functions using the response surface methodology. In parallel vibration experiments are carried out with the purpose to determine natural frequencies and corresponding loss factors of viscoelastic sandwich structures. An identification of material properties is performed in the final stage minimizing the error functional between experimental and numerical parameters of structural responses.

*A. Plan of Experiments*

Let us consider a criterion for elaboration of the plan of experiments independent on a mathematical model of the designing object or process [7]. The initial information for development of the plan is number of factors  $n$  and number of experiments  $k$ . The points of experiments in the domain of factors are distributed as regular as possible (Fig. 2). For this reason the following criterion is used

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

where  $l_{ij}$  is a distance between the points having numbers  $i$  and  $j$  ( $i \neq j$ ). Physically it is equal to the minimum of potential energy of repulsive forces for the points with unity mass if the magnitude of these repulsive forces is inversely proportional to the distance between the points.

For each number of factors  $n$  and number of experiments  $k$  it is possible to elaborate a plan of experiments, but it needs much computer time. Therefore each plan of experiment is developed only once and it can be used for various designing

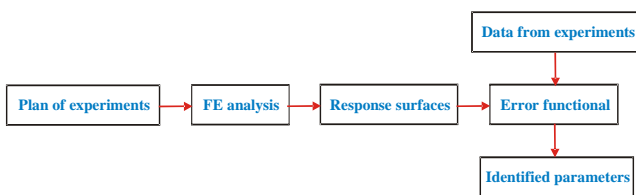


Fig. 1 Inverse procedure

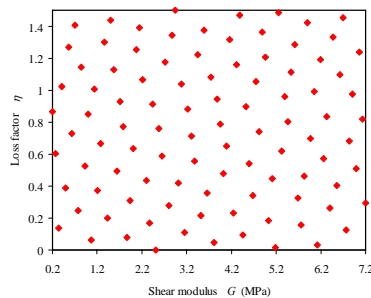


Fig. 2 Plan of experiments:  $n=2, k=98$

cases. The plan of experiments is characterized by the matrix of plan  $B_{ij}$ , when the domain of factors is determined as  $x_j \in [x_j^{\min}, x_j^{\max}]$  and the points of experiments are calculated by the following expression:

$$x_j^{(i)} = x_j^{\min} + \frac{1}{k-1} (x_j^{\max} - x_j^{\min}) (B_{ij} - 1) \quad (2)$$

$i = 1, 2, \dots, k, \quad j = 1, 2, \dots, n$

*B. Finite Element Analysis*

In the present investigations the finite element method is used for the modeling and dynamic analysis of sandwich panels with adhesive core layers.

Finite element modeling is based on the first order shear deformation theory including rotation around the normal. In this case the widely known expressions of displacements have the following form:

$$u = u_0 + z\gamma_x, \quad v = v_0 + z\gamma_y, \quad w = w_0 \quad (3)$$

where  $u_0, v_0, w_0$  are the displacements in a reference plane,  $z$  is the coordinate of the point of interest from a reference plane,  $\gamma_x, \gamma_y$  are the rotations connected with the transverse shear deformations. For sandwiches this hypothesis is applied separately for each layer (Fig. 3). This case corresponds to the broken line model [8] and satisfies to the following displacement continuity conditions between the layers

$$\begin{aligned} u^{(1)} &= u^{(2)} \Big|_{z=z_1}, & u^{(2)} &= u^{(3)} \Big|_{z=z_2} \\ v^{(1)} &= v^{(2)} \Big|_{z=z_1}, & v^{(2)} &= v^{(3)} \Big|_{z=z_2} \\ w^{(1)} &= w^{(2)} \Big|_{z=z_1}, & w^{(2)} &= w^{(3)} \Big|_{z=z_2} \end{aligned} \quad (4)$$

where in the brackets, the numbers of layers are given.

To describe the rheological behaviour of viscoelastic materials, the complex modulus representation [9] is used. Using this model, the constitutive relations will be expressed in the frequency domain as follows

$$\sigma_0 = E^*(\omega)\varepsilon_0 = E(\omega)[1 + i\eta(\omega)]\varepsilon_0, \quad \eta(\omega) = \frac{E''(\omega)}{E'(\omega)} \quad (5)$$

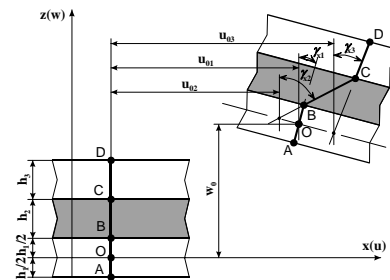


Fig. 3 Kinematic assumptions for a sandwich plate

where  $\sigma_0$  and  $\varepsilon_0$  are an amplitude of the harmonically time dependent stress and strain respectively,  $E^*$  is the complex modulus of elasticity,  $E, E''$  are the real and imaginary parts of the complex modulus of elasticity,  $\eta$  is a loss factor and  $\omega$  is a frequency. It is necessary to note that the storage and loss moduli in this case are frequency dependent values.

In the method of complex eigenvalues damped eigenfrequencies and corresponding loss factors are determined from the free vibration analysis of a structure

$$[\mathbf{K}^*(\omega) - \omega^{*2}\mathbf{M}]\bar{\mathbf{X}}^* = 0 \quad (6)$$

where  $\mathbf{M}$  is the mass matrix of a structure,  $\mathbf{K}^*(\omega) = \mathbf{K}(\omega) + i\mathbf{K}''(\omega)$  is the complex stiffness matrix of a structure,  $\omega^* = \omega + i\omega''$  is the complex eigenfrequency. The real part  $\omega$  represents the damped eigenfrequency of a structure and the imaginary part  $\omega''$  specifies the rate of decay of the dynamic process. The matrix  $\mathbf{K}(\omega)$  is determined using the storage moduli  $E(\omega)$  and  $G(\omega)$ , while  $\mathbf{K}''(\omega)$  is found using the imaginary parts of the complex moduli  $E''(\omega) = \eta_E(\omega)E(\omega)$  and  $G''(\omega) = \eta_G(\omega)G(\omega)$ , where  $\eta_E(\omega)$  and  $\eta_G(\omega)$  are the material loss factors.

Equation (6) can be written as the non-linear generalised eigenvalue problem

$$\mathbf{K}^*(\omega)\bar{\mathbf{X}}^* = \lambda^*\mathbf{M}\bar{\mathbf{X}}^* \quad (7)$$

where  $\lambda^* = \omega^{*2}$  is the complex eigenvalue and  $\bar{\mathbf{X}}^*$  is the complex eigenvector. Solution of (7) starts with a constant frequency ( $\omega = \text{const}$ ). Then at each step the linear generalized eigenvalue problem with  $\mathbf{K}^*(\omega) = \text{const}$  is solved by the Lanczos method [10], which is programmed in a truncated version, where the generalized eigenvalue problem is transformed into a standard eigenvalue problem with a reduced order symmetric three diagonal matrix. Orthogonal projection operations are employed with greater economy and elegance using elementary reflection matrices. An iteration process terminates, when the following condition is satisfied

$$\frac{|\omega_{i+1} - \omega_i|}{\omega_i} * 100\% \leq \xi \quad (8)$$

where  $\xi$  is a desired precision and  $\omega_{i+1}$  is the real part of eigenfrequency of a structure calculated from the linear generalized eigenvalue problem with the storage and loss moduli for the frequency  $\omega_i$ , which was obtained from the same equation in the previous step. The modal loss factors of a structure for each vibration mode are determined by the following relation

$$\eta_n = \frac{\lambda_n''}{\lambda_n} \quad (9)$$

This approach gives the possibility to preserve the frequency dependence of viscoelastic materials and to calculate structures with high damping.

### C. Response Surface Method

In the present approach a form of the equation of regression is unknown previously [7]. There are two requirements for the equation of regression: accuracy and reliability. Accuracy is characterized as a minimum of standard deviation of the table data from the values given by the equation of regression. Increasing a number of terms in the equation of regression it is possible to obtain a complete agreement between the table data and values given by the equation of regression. However it is necessary to note that prediction in intervals between the table points can be not so good. For an improvement of prediction, it is necessary to decrease a distance between the points of experiments by increasing the number of experiments or by decreasing the domain of factors. Reliability of the equation of regression can be characterized by an affirmation that standard deviations for the table points and for any other points are approximately the same. Obviously the reliability is greater for a smaller number of terms in the equation of regression.

The equation of regression can be written in the following form

$$y = \sum_{i=1}^p A_i f_i(x_j) \quad (10)$$

where  $A_i$  are the coefficients of the equation of regression,  $f_i(x_j)$  are the functions from the bank of simple functions  $\theta_1, \theta_2, \dots, \theta_m$  which are assumed as,

$$\theta_m(x_j) = \prod_{i=1}^s x_j^{\xi_{mi}} \quad (11)$$

where  $\xi_{mi}$  is a positive or negative integer including zero. Synthesis of the equation from the bank of simple functions is carried out in two stages: selection of perspective functions from the bank and then step by step elimination of the selected functions.

On the first stage, all variants are tested with the least square method and the function, which leads to a minimum of the sum of deviations, is chosen for each variant. On the second stage, the elimination is carried out using the standard deviation

$$\sigma_0 = \sqrt{\frac{S}{k-p+1}}, \quad \sigma = \sqrt{\frac{1}{k-1} \sum_{i=1}^k \left( y_i - \frac{1}{k} \sum_{j=1}^k y_j \right)^2} \quad (12)$$

or correlation coefficient

$$c = \left( 1 - \frac{\sigma}{\sigma_0} \right) * 100\% \quad (13)$$

where  $k$  is the number of experimental points,  $p$  is the number of selected perspective functions and  $S$  is the minimum sum of deviations. It is more convenient to characterize an accuracy of the equation of regression by the correlation coefficient (Fig. 4). If insignificant functions are eliminated from the equation of regression, a reduction of the correlation coefficient is negligible. If in the equation of regression only significant functions are presented, an elimination of one of them leads to important decrease of the correlation coefficient.

#### D. Experimental Analysis

The experimental set-up used for vibration testing of sandwich panels (Fig. 5) presents the impulse technique, where an excitation is accomplished by PCB impulse hammer with a built in force transducer. The structural response is detected by three accelerometers located on the panel as presented in Fig. 5. Both the input and output signals are converted to the frequency domain by fast Fourier transformation in a signal analyzer and the frequency response functions are created. After that, these frequency response functions are exported to the modal analysis program, where natural frequencies and corresponding loss factors are calculated.

#### E. Error Functional Minimization

The error functional between experimental and numerical parameters of structural responses is written in the case of identification of viscoelastic material properties for each eigenfrequency

$$\Phi_i(x) = \frac{(f_i^{\text{exp}} - f_i^{\text{FEM}})^2}{(f_i^{\text{exp}})^2} + \frac{(\eta_i^{\text{exp}} - \eta_i^{\text{FEM}})^2}{(\eta_i^{\text{exp}})^2} \Rightarrow \min \quad (14)$$

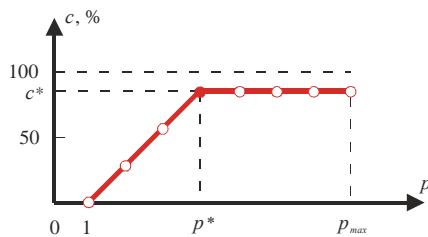


Fig. 4 Diagram of elimination for the correlation coefficient

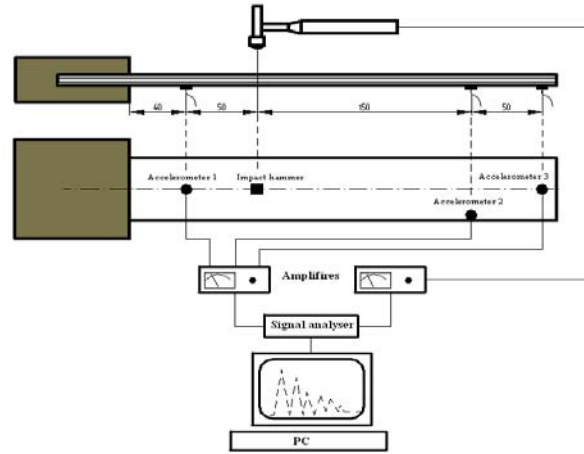


Fig. 5 Experimental set-up

To minimise the error functional, the following constrained nonlinear optimisation problem should be solved

$$\min \Phi(x), H_i(x) \geq 0, G_j(x) = 0 \quad (15)$$

$$i = 1, 2, \dots, I, j = 1, 2, \dots, J$$

where  $I$  and  $J$  are the numbers of inequality and equality constraints. This problem is replaced with the unconstrained minimization problem in which the constraints are taken into account with the penalty functions. New version of random search method [11] is used for a solution of the formulated optimization problem. An application of the curve fitting procedure is required additionally to obtain the frequency dependent viscoelastic material properties of adhesive layers.

### III. IDENTIFICATION EXAMPLES AND RESULTS VERIFICATION

The developed inverse technique is tested on aluminum panels and applied to characterize viscoelastic material properties of 3M damping polymer ISD-112 used as a core material in sandwich panels.

#### A. Testing of Inverse Technique

Testing of the developed inverse technique based on vibration tests has been carried out identifying the material properties of homogeneous aluminum 6082-T6 plate with the following dimension  $a \times b = 0.3 \text{ (m)} \times 0.2 \text{ (m)}$ , thickness  $h = 0.002 \text{ (m)}$  and material density  $\rho = 2700 \text{ (kg/m}^3\text{)}$ . Free-free boundary conditions have been applied and twelve first eigenfrequencies (Table 1) have been measured by the POLYTEC laser vibrometer [12].

To describe the isotropic material properties only two material constants are necessary, modulus of elasticity  $E$  and shear modulus  $G$ . The last is taken instead of Poisson's ratio  $\nu$  to exclude an application of any scaling technique in the identification process. The borders of identified parameters are taken in the present analysis as follows  $E = 60\text{-}80 \text{ (GPa)}$  and  $G = 22\text{-}30 \text{ (GPa)}$ .

The plan of experiments has been produced for 2 design parameters and 38 experiments. Then finite element analysis has been performed in 38 experimental points and 12 first

eigenfrequencies has been determined. Employing these numerical values, the approximating functions (response surfaces) for all eigenfrequencies have been obtained. Minimizing the error functional, the elastic material constants have been identified and presented in Table 2 to compare them with the results of static tension test and technical data presented by producer, where a good correlation is observed. Additionally the identified material properties have been verified comparing the experimentally measured eigenfrequencies with the numerically obtained using the identified elastic constants (Table 1). It is seen from this table that numerical eigenfrequencies are in a good agreement with the experimental results. The difference in terms of residuals is less than 1% in most cases.

### B. Utilization of Inverse Technique

A sandwich beam with the following dimensions: width  $B=0.05$  (m), length  $L=0.3$  (m) and thickness of layers

TABLE I  
DYNAMIC CHARACTERISTICS VERIFICATION FOR ALUMINUM PANEL

Mode $n$	$f_n^{EXP}$ (Hz)	$f_n^{FEM}$ (Hz)	$\Delta$ (%)
1	106	107	0.9
2	117	116	0.9
3	247	249	0.8
4	276	274	0.7
5	313	311	0.6
6	368	369	0.3
7	461	464	0.7
8	535	533	0.4
9	664	661	0.5
10	744	736	1.1
11	789	801	1.5
12	806	801	0.6

TABLE II  
MATERIAL PROPERTIES VERIFICATION FOR ALUMINUM PANEL

Material properties	Identified	Static test	Technical data
$E$ (GPa)	69.0	68.9	68.9
$G$ (GPa)	25.6	26.3	25.9
$\nu$	0.35	0.31	0.33

$h_1=0.0012$  (m),  $h_2=0.0001016$  (m),  $h_3=0.0008$  (m), has been chosen for a characterization of 3M damping polymer ISD-112 used as a core material. The external layers are made from aluminum 2024-T6:  $E=64$  (GPa),  $\nu=0.32$ ,  $\rho=2695$  ( $\text{Ns}^2/\text{m}^4$ ). The clamped boundary conditions are applied from one side of the beam. The structural dynamic characteristics, eigenfrequencies and corresponding loss factors (Table 3), have been obtained from physical vibration experiment by an impulse technique (Fig. 5).

To describe the viscoelastic isotropic material properties only one material parameter is necessary. This is modulus of elasticity  $E^*(\omega) = E(\omega) + iE''(\omega)$ . However in this case it is complex value consisting of storage  $E(\omega)$  and loss  $E''(\omega)$  parts, which are both frequency dependent. As known material parameters, Poisson ratio  $\nu=0.49$  and density  $\rho=1000 \cdot (\text{Ns}^2/\text{m}^4)$  are taken into consideration. The borders of identified

parameters are taken in the present analysis as follows  $G = 0.2 - 7.2$  (MPa) and  $\eta = 0 - 1.5$ .

TABLE III  
DYNAMIC CHARACTERISTICS VERIFICATION FOR SANDWICH PANEL

Mode $n$	$f_n^{EXP}$ (Hz)	$f_n^{FEM}$ (Hz)	$\Delta$ (%)	$\eta_n^{EXP}$	$\eta_n^{FEM}$	$\Delta$ (%)
1	16	16	0	0.12	0.13	8.3
2	100	99	1.0	0.22	0.22	0
3	268	271	1.1	0.26	0.25	3.9
4	496	510	2.8	0.26	0.28	7.7
5	785	808	2.9	0.32	0.31	3.1
6	1180	1166	1.2	0.30	0.32	6.7
7	1570	1588	1.2	0.34	0.33	2.9

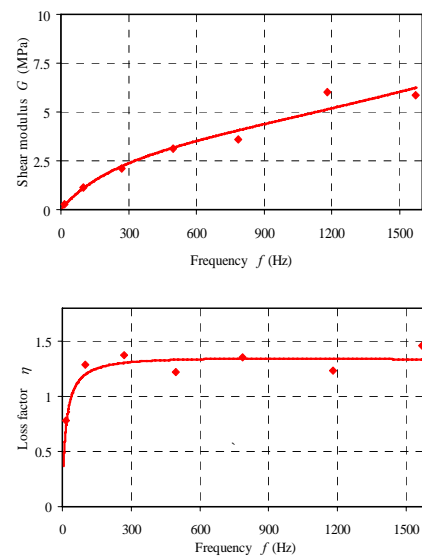


Fig. 6 Identified viscoelastic material properties

The plan of experiments is produced for 2 design parameters and 98 experiments (Fig. 2). Then the finite element analysis is performed in 98 experimental points and seven first dynamic characteristics are determined. Employing these numerical values, the approximating functions (response surfaces) for all eigenfrequencies and corresponding loss factors have been obtained with the correlation coefficients higher than 84%. As an example these approximations with correlation coefficients are given below for the first eigenfrequency and corresponding loss factor:

$$c = 91\%$$

$$f_1 = 18.6 - 0.144/z_1 + 0.0872z_2/z_1^2 \text{ (Hz)}$$

$$\text{where } z_1 = 0.0466E \text{ and } z_2 = 0.0345E''$$

$$c = 93\%$$

$$\eta_1 = -0.211 + 0.216/z_1 + 0.07509z_1 + 0.2527z_2 - 0.06614/(z_1z_2) + 0.01714/z_2^2 - 0.1604z_2/z_1 - 0.116z_1z_2$$

$$\text{where } z_1 = 0.0466E \text{ and } z_2 = 0.5 + 0.345E''$$

Minimizing the error functional (14), material properties of 3M viscoelastic damping polymer ISD-112 are found for each eigenfrequency. These values are presented in Fig. 6 by points. Applying the curve fitting procedure, the following shear modulus and material loss factor as functions on frequency are obtained in the frequency range  $f=5-1600$  (Hz)

$$G=4.759-0.9266/z+2.405z^2 \text{ (MPa)}$$

$$\text{where } z=0.1918+0.0005148f$$

$$\eta=1.385-0.03673z-0.01342/z$$

$$\text{where } z=0.01+0.0006306f$$

These dependencies are presented graphically in Fig. 6 with solid lines and they are used later in the finite element analysis to verify the identified material properties. Table 3 shows a good correlation between experimental and numerical dynamic characteristics.

#### IV. CONCLUSION

New inverse technique operating on vibration tests has been developed to characterize nonlinear mechanical properties of adhesive layers in sandwich composites. The optimization approach based on the planning of experiments and response surface technique to minimize the error functional has been applied in this case to decrease considerably the computational efforts. The present methodology gave the possibility to preserve the frequency dependences for storage and loss moduli of viscoelastic adhesive materials in wide frequency range and to analyze structures with high damping. The developed inverse technique has been tested on aluminum panels and applied to characterize viscoelastic material properties of 3M damping polymer ISD-112 used as a core material in sandwich panels. Good correlation between experimental and numerical results has been observed in time of verification of identified material properties.

It is necessary to note that an examined approach, like any other inverse approach based on vibration tests, has nondestructive character and the same material properties for test specimen and construction due to the same technology used for their production. The present inverse technique due to its universality can be applied to characterize isotropic, orthotropic, elastic or viscoelastic material properties of advanced composites and structures, and viscoelastic material properties obtained in the present investigations can be used easily in other analysis, not presented in the identification procedure, to study the dynamic effects of structures with high damping properties.

#### ACKNOWLEDGMENT

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