COMPARISON OF TWO MODAL PROCEDURES TO DETERMINE MECHANICAL PROPERTIES OF NANOCOMPOSITE LAMINATED STRUCTURE

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Abstract. *In this work a Finite Element model of a laminate plate is used to approximate the modal natural frequencies to experimental results by an optimization procedure. Using the experimental frequencies, the tensile and shear modulus of a woven orthotropic composite plate were estimated by inverse method. The plates used in the experiment are made of S2-glass/epoxy with 16 layers manufactured by vacuum assisted wet lay-up. The nanocomposite plate has been obtained by adding 0%, 1%, 2%, 5% and 10% nanoclays in weight into epoxy matrix. A Finite Element model of the composite plate coupled with a gradient method were applied to optimize the model and gotten the mechanical properties from experimental data. Two different modal procedure were employed the laser anemometry and hammer test. The modal analysis was made in both results with the objective to determinate the structural mode shape to compare the frequencies and modal properties for different nanoclay composite plates.*

Keywords:*vibration methods, material identification, nanocomposites, finite elements, inverse methods*

1. INTRODUCTION

There is a numbers of researchers who studied the effect of nanoparticles (organically modified montmorillonite - Cloisite 30B) into epoxy systems. One aspect of the nanocomposite structure is the damage reduction due to impact loadings. Among researchers who studied the effect of nanoparticles, Yasmin *et al.* (2003) and Isik *et al.* (2003) found also an increase in the elastic modulus and toughness Isik *et al.* (2003).

A more comprehensive study on clay-epoxy nanocomposites was performed by Haque and Shamsuzzoha (2003), since they evaluated both mechanical and thermal properties. Their main conclusions were that thermo-mechanical properties mostly increase at low clay loadings (1-2% in weight) but decrease at higher clay loadings ($\geq$ 5% in weight). In addition, the uses of nanoclays also decrease the coefficient of thermal expansion (CTE). They also observed a degradation of properties at higher clay loadings. This phenomenon can be due to the phase-separated structures and defects in cross-linked structures.

The objective of this paper is to study the nanoparticle influence into the plate vibration behavior and to determine the material properties using optimization procedure. In order to analyze only the nanoparticle influence, all manufacturing parameters are kept fixed but the nanoclay concentration. The elastic properities play a relevant role in the vibration behavior. The inverse method uses the measured vibration data of samples to estimate the material elastic properties. The modal analysis was made with the objective to determine the structural mode shape to compare the frequencies and modal properties for different nanoclay composite plates.

A commercial code, Ansys, which offers the function of design optimization, is employed to model the woven laminate orthotropic model. The optimization allows to approximate the theoretical laminate model modal results to experimental ones and by this way determining the elastic properties, it is a mixed numerical and experimental method.

2. MATERIAL AND TESTING PROCEDURES

The nanocomposite prepared for this work is a S2-glass/epoxy-clay. The epoxy formulation is based on two parts, part A (diglycidyl ether of bisphenol A) and part B - hardener aliphatic amine- (triethylenetetramine). The nanoclay particles used in this study are organically modified montmorillonite in a platelet form, while the S-2glass fiber has a plain-weave woven fabric configuration with density of 180 g/m^2 from Texiglass. The S2-glass/epoxy-nanoclay composite is a laminate plate with 16 layers and 65% fiber volume fraction. The nanocomposite synthesis followed the methodology proposed by Ávila et al. Ávila *et al.* (2006) and Ávila and Duarte (2006). In order to be able to investigate the nanoparticles influence into vibration analysis and in its mechanical properties, samples with 0%, 1%, 2%, 5% and 10% of nanoclay with respect to the matrix mass were employed. All plates were rectangular and had the same dimensions 136mm x 116mm x 2.4mm.

2.1 Modal Analysis

The vibration analyses were performed to determine the mode shapes and natural frequencies, mode shape amplitude and the damping coefficient. The first vibration testing were performed using a Laser Doppler Vibrometer, laser model OFV-303.8 and controller model OFV 3001 S from Politec, a Hewlett Packard data acquisition system model 35670A, a mini-shaker, a shaker power amplifier and force transducer from PCB. The test plates were hanged by a fine nylon wire and excited by a random signal (white noise). A piezoelectric force transducer was used as the reference for the force bonded to the plate and linked to the stinger/shaker exciter, which transform the amplified electrical signal in force. There was only one force excitation point at the same position for all plates. The velocity of the plate surface was measured using a grid of 35 points by the laser Doppler vibrometer. The data acquisition system processed the signal response of the measurement point generating the Mobility (velocity/force) Function Response Frequency (FRF), for each point of the plate.

A modal analysis program has done the mode shape identification from the 35 FRF for each plate. This modal analysis program is based on polynomial interpolation and employs Chebyshev Orthogonal Polynomials method, Arruda *et al.* (1996).

A second modal technique was also employed. In this second experiment the tests were performed with same plates and on the same grid points. But in this case the system was excited by a hammer impact in each point of the grid, the response, rms acceleration, is measured by the micro-accelerometer in a fixed point, the same position for all plates. The accelerometer is PCB type 352A71 and it has a mass of 0.64 grams, the excitation hammer is PCB model 086C05.

The usual procedure is to use the hammer to excite a fixed point on the structure and the impulse response of plate by moving the accelerometer on each point of the grid. But this usual procedure did not produce good data, the shape mode and frequencies has showed to be very sensitive to the accelerometer mass and its position in spite of the accelerometer mass being very small. This mass sensitivity do not allow the modal analysis program identify more than the first two modes and the procedure was changed to fix the accelerometer and excite the structure in each grid point. The modal analysis program employed to analyse the results was the same as the previous case.

3. RESULTS AND DATA ANALYSIS

The elastic properties was determined by inverse method. This method is a coupling of gradient method and a Finite Element Method of the composite plate to find the mechanical properties using experimental frequencies. The optimization is a zero-order approach method, offered as a tool of the FE commercial code, and followed the Hu and Wang (2009) procedure. The state variables ξ_n are related to difference between FE, f_n^{FE}, and experimental modal nth frequency f_n for each one of the nanocomposite sample:

$$\xi_n = \frac{f_n^{FE} - f_n}{f_n} 100 \tag{1}$$

The cost function F is defined as:

$$F(E_{xy}, G_{xy}, \nu_{xy}) = \sum_{n=1}^{k} \xi^2 \tag{2}$$

Where the mechanical properties are E_{xy} the Tensile Modulus, G_{xy} the Shear Modulus and ν_{xy} the Poisson's ratio.

3.1 Laser Doppler Vibrometer Method

On Gagneja and Gibson, Gagneja *et al.* (2001), there are theoretical discussion and experimental results about length to thickness ratios on experimental vibrations measurements of composite structures. For a refined theory on laminate plate models, its implementation in Finite Element and a discussion about the more efficient method a good reference is still the classical book Ochoa and Reddy (1992).

In both methods, Laser Doppler and hammer, the Finite Element routine employed shell elements to model the plates, the shell element was type $shell181$. This element is a Ansys product that has four nodes and six degrees of freedom in each node and it accepts multi-layers allowing to model laminate structures. The $shell181$ element is based on Mindlin-Reissner theory which is usualy called first order shear deformation theory. The FE model also employed the $mass21$ element to consider the effect of mass and rotary inertia of the force transducer bonded to the plate. The structure was modeled by 320 shell elements in a regular quadrilateral mesh and by one concentrated mass element.

There is no difference between the experimental shape modes for 1%, 2%, 5% and 10% nanoclay composite and those of the reference resin plate, Figure 1, the main difference is on the frequency mode. But in this procedure there are unusual shape modes that are plotted in Fig. 2 and Fig. 3.

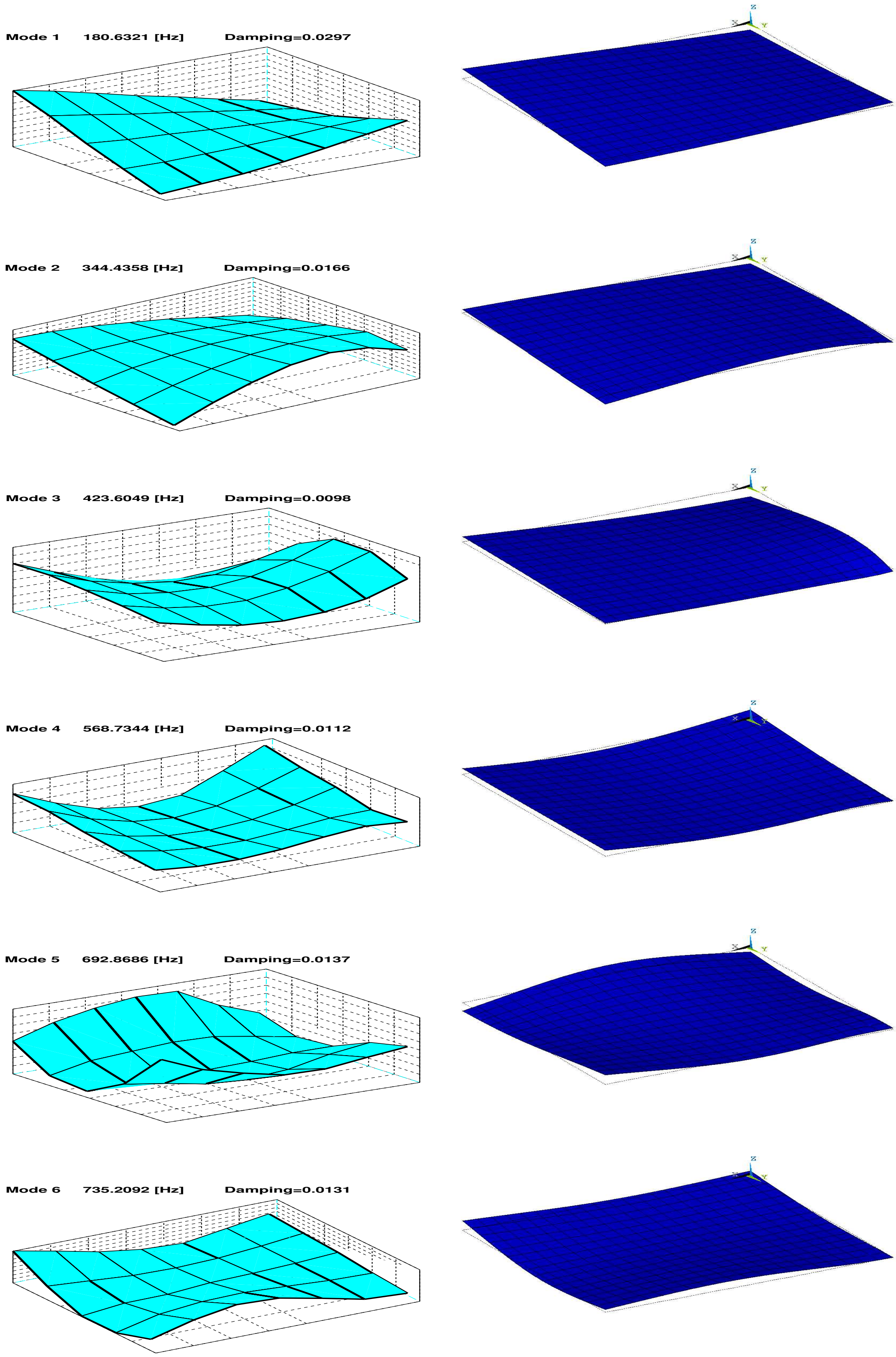

Figure 1. Mode shapes for Resin plate. At the left side the experimental shape modes at right the same modes at the end of the inverse approximation method.

On Table 1 there is a summary of the modal Doppler experiments, the natural frequencies and the associated damping coefficients for each experimental mode in the appearing frequency range. For the 2%, 5% and 10% nanoclay composite plates the analysis has identified 'spurious modes' and this modes and its analysis are detached. On Tab 1 this identified 'spurious modes' are labeled by a $sm*$ on the right upper side of the mode frequency.

Table 1. Matrix composite content and Modal Properties.

Composite Matrix	Mass [gr]	Modal Properties	Shape Modes 1^{th}	2^{th}	3^{th}	4^{th}	5^{th}	6^{th}	7^{th}
Pure Epoxy	66.37	Nat. Frequency [Hz]	180.6	344.4	423.6	568.7	692.9	735.2	
		Damping Coef.	0.0297	0.0166	0.0098	0.0112	0.0137	0.0131	
Nanoclay 1% weight	65.27	Nat. Frequency[Hz]	184.9	347.4	424.6	550.1	683.4	728.2	
		Damping Coef.	0.0209	0.0247	0.0088	0.0136	0.0137	0.0138	
Nanoclay 2% weight	66.92	Nat. Frequency [Hz]	173.6	361.3	424.0	578.0	655.3^{sm*}	716.1	783.0
		Damping Coef.	0.023	0.0248	0.0116	0.0173	0.0052	0.0134	0.0166
Nanoclay 5% weight	67.46	Nat. Frequency [Hz]	202.3	336.0^{sm*}	430.1	494.6	645.0	747.6	821.5
		Damping Coef.	0.0372	0.0445	0.0162	0.0233	0.0134	0.0011	0.0169
Nanoclay 10% weight	67.89	Nat. Frequency [Hz]	196	329.8^{sm*}	425.5	477.6	610.3	702.8	764.1
		Damping Coef.	0.0335	0.0412	0.0266	0.0163	0.0133	0.0135	0.0153

For the reference Resin and 1% Nanocomposite plate the shape modes does not show any special behavior and the mechanical properties are obtained by direct application of the inverse method. The 2% nanocomposite plate showed a singular situation. The 4^{th} and 5^{th} has resembling shape modes at different frequencies, Fig 2. This behavior does not make sense theoretically or experimentally and it was not observed in the previous cases.

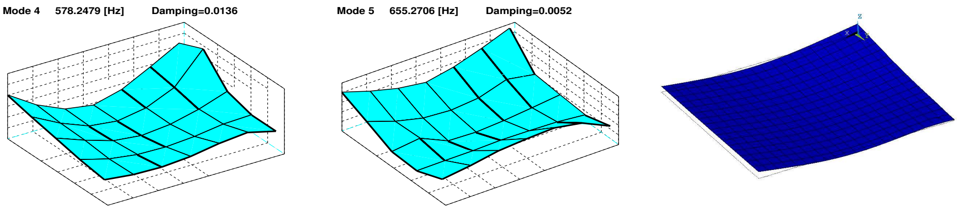

Figure 2. Shapes modes for 2% nanocomposite plate. At the left side the experimental 4^{th} mode (578.0 Hz), at center the experimental spurious 5^{th} mode (655.3 Hz) and at right the final optimized FE shape mode.

The finite element simulation showed a coupling mode between the plate and the force transducer at a low frequency, below the first shape mode natural frequency, for all plate simulations. Those low frequency mode seemed as rigid body mode. This FE low frequency mode offered a answer for the similar modes, it was a coupling mode between the experimental apparatus and the elastic plate.

The coupling mode between the test plate and the force transducer are named 'spurious mode', but in reality it is not spurious, it is not a experimental or an analysis error. This mode is termed as spurious because it is merging with the natural vibration set frequencies of the plate and disturbing the analysis.

Due to complication of modeling the contacts and connections, there is not implemented a numerical model to deal whit the dynamical coupling between the plate and the force tranducer, elastic stinger and shaker. So the dynamical coupling appeared something like a low frequency rigid body mode. The nanocomposite mass in the matrix changed the elastic properties of the plates and the coupling frequency mode has moved inside the range of the experimental plate natural frequencies as the nanoclay mass and mechanical properties changes for each plate matrix composition.

The problem is to determine what mode is a plate mode and what is not. The solution presented here is to find the real mode by comparing the error between the experimental mode frequencies and the frequencies of the optimized or minimized model.

The minimization was not done for all frequencies in a unique process. A new mode frequency was included in the previous set, the minimization was performed and elastic properties found. The latest elastic properties are used as initial value in a new minimization process where the next mode frequency will be included in this update set. This procedure will be called iteration. The latest iteration is performed using a small tolerance for the cost function F, Eq. 2, for all cases $F < |10^{-2}|$.

Table 2. Frequency mode error ξ_n in each iteration. Frequency for the 4^{th} mode 578.0 Hz

iteration	ξ_1	ξ_2	ξ_3	ξ_4	ξ_5	ξ_6	$\sum \xi_n$
1^{th}	0.01	0	0	0	0	0	0.01
2^{th}	0.04	0.04	0	0	0	0	0.08
3^{th}	0.99	2.08	2.22	0	0	0	5.28
4^{th}	0.98	2.10	2.20	0.79	0	0	6.08
5^{th}	1.03	2.02	2.28	0.67	2.30	0	8.29
6^{th}	1.03	2.03	2.27	0.69	2.28	3.92	12.2

Table 2 and Tab. 3 show the frequency error for each mode set in the minimization procedure for the 2% content nanoclay plate. Table 2 presents the error ξ for each mode frequency n in each iteration, in this case the fourth mode frequency is supposed to be the 578.0 Hz, the similar mode shape with 655.3 Hz frequency is removed from the frequency set, so there are six actual modes. Table 3 presents the same error evolution considering the fifth experimental mode frequency (655.3 Hz) as the actual fourth mode frequency and the mode with 578.0 Hz discarded from this analyses.

Table 3. Frequency mode error ξ_n in each iteration. Frequency for the 4^{th} mode 655.3 Hz

iteration	ξ_1	ξ_2	ξ_3	ξ_4	ξ_5	ξ_6	$\sum \xi_n$
1^{th}	0.01	0	0	0	0	0	0.01
2^{th}	0.04	0.04	0	0	0	0	0.08
3^{th}	0.99	2.08	2.22	0	0	0	5.28
4^{th}	1.01	7.52	2.06	2.73	0	0	13.3
5^{th}	1.03	2.42	1.53	8.84	1.67	0	15.5
6^{th}	0.99	2.46	2.10	9.53	2.11	4.14	21.3

The Tab. 3 shows highest error while Tab. 2 shows the lowest errors ξ for the fourth mode and for the sum of errors. So the fourth mode with frequency 578.0 Hz was used to determine the elastic properties showed on Tab. 7. The fifth shape mode, with frequency of 655.3 Hz, was considered a coupling mode between the experimental apparatus and the elastic plate for this 2% content nanoclay plate and named 'spurious mode'.

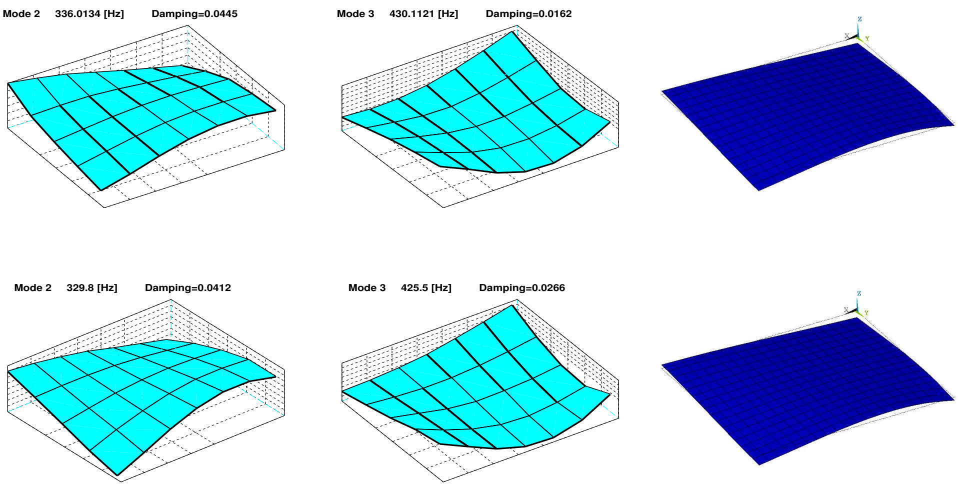

Figure 3. On upper line the Shapes modes for 5%, 10% nanoclay plate on the bottom line. On the left column the experimental spurious modes (336.0 Hz) for 5% nanocomposite and below (329.8 Hz) for 10% nanocomposite. At center column the experimental best fit for the 2^{th} mode 430.1 Hz for 5% and 425.5 Hz for 10% nanocomposite, both out of phase by 180°. At right the final optimized FE shape mode.

As it was showed on Tab. 1 the same behavior is observed for plates with 5% and 10% of nanoparticles content. On Tab. 4 are presented the final iteration for both plates with the best frequency set. For the 5% nanoclay content plate

Table 4. Frequency mode error ξ_n at the end of best set minimization process, for 5% and 10% plates

plate content	ξ_1	ξ_2	ξ_3	ξ_4	ξ_5	ξ_6	$\sum \xi_n$
Nanoclay 5%	1.02	5.14	6.68	2.10	2.54	6.34	23.8
Nanoclay 10%	1.00	8.53	8.04	2.09	3.42	6.69	29.8

modes with 336.0 Hz and 430.1 Hz have a similar shape mode, and the experimental third mode, 430 Hz, has fitted better at the end of optimization process. For the 10% nanoparticle plate, the second and third modes are similar also, and the third mode, with 425.5 Hz, has been the best approximation too. On Tab. 7 there are the final properties for the best best set minimization process as presented on Tab. 2, Tab. 3 and Tab. 4.

3.2 Hammer Excitation Method

This method is described in Section (2.1), and the excitation is performed by a hammer impact in each point of the grid and response measured in a fixed point by a mini-accelerometer. The Finite Element model employed was the same of the previous case. The only difference is that the plate structure was modeled by 1190 shell elements plus a concentrated mass element. This regular quadrilateral mesh was used to match the mass element position, accelerometer mass, with a node on the mesh. The accelerometer position was chose to provide the best signal for all excitation points.

Table 5. Matrix composite content and Modal Properties for Hammer Excitation.

Epoxy Matrix	Mass [gr]	Modal Properties	Shape Modes 1^{th}	2^{th}	3^{th}	4^{th}	5^{th}	6^{th}
Pure Epoxy	66.37	Nat. Frequency [Hz]	245.9	492.1	655.7	702.1	816.4	1295
		Damping Coef.	0.0271	0.0077	0.0083	0.0108	0.0135	0.0154
Nanoclay 1% weight	65.27	Nat. Frequency[Hz]	250.5	469.9	646.2	692.4	813.4	1294
		Damping Coef.	0.0209	0.0069	0.0074	0.0109	0.0114	0.0091
Nanoclay 2% weight	66.92	Nat. Frequency [Hz]	260.0	487.0	668.6	722.6	848.5	-
		Damping Coef.	0.0201	0.0069	0.0069	0.0100	0.0104	-
Nanoclay 5% weight	67.46	Nat. Frequency [Hz]	268.7	503.4	675.7	743.5	861.2	-
		Damping Coef.	0.0230	0.0073	0.0081	0.0119	0.0120	-
Nanoclay 10% weight	67.89	Nat. Frequency [Hz]	260.8	500.6	673.1	726.3	847.5	-
		Damping Coef.	0.0242	0.0077	0.0073	0.0132	0.0118	-

The modal properties on Tab. 5 presents several differences as compared with Tab. 1. The main difference are on natural frequencies, that is because the force transducer used on Doppler Vibrometer test has a big mass, so the experimental assembly has a big mass and low natural frequencies.

The damping coefficient changed with the frequencies and resin matrix contents in both experiments. Damping factor is sensitive to frequency for the great majority of materials. As the excitation force and technique to implement it is different in each experiment and the frequencies are quit different in each mode so it is difficult to compare the damping results.

But for the hammer method, the damping coefficient has the same numerical order of magnitude for each mode independently of material in this experimental procedure. For the Doppler Vibrometer method the damping coefficient presents a dispersion without a trend.

Table 6. Frequency mode error ξ_n at the end of best set minimization process, for 5% and 10% plates

plate content	ξ_1	ξ_2	ξ_3	ξ_4	ξ_5	ξ_6	$\sum \xi_n^2$
Resin	0.43	1.20	1.26	1.14	1.03	0.35	5.70
Nanoclay 1%	0.02	0.48	0.13	0.09	0.10	0.53	0.55
Nanoclay 2%	0.39	0.11	0.43	0.72	0.02	-	0.87
Nanoclay 5%	0.12	1.22	1.26	1.27	0.73	-	5.24
Nanoclay 10%	0.07	0.76	0.55	0.16	0.42	-	1.08

Table 6 presents the final individual error ξ_n for each mode n at the final minimization process that approximate the FE model mode frequencies to experimental natural frequencies by changing the mechanical properties, the dependent variables, planar Elastic Modulus E_{xy}[GPa], shear Modulus G_{xy}[GPa] and Poisson coefficient ν_{xy}.

On Tab. 7 there are the mechanical properties for the Doppler Vibrometer, Method (1), and for Hammer Method, method (2). In this Tab. 7 the Shear Modulus shows good agreement between the two methods. There are a rising for the Shear Modulus with nanoclay mass until 5% content and a decreasing tendency for the highest content. Except for 2% nanocomposite content that, in both experiment, showed a different behavior, the difference between the results for the two methods is less than 6.5% for the Shear Modulus.

On Tab. 7 the Elastic Modulus showed a noticeable difference comparing both methods. The Elastic Modulus in the Doppler Method (1) presents a continuous and remarkable improvement with the nanoclay content while in the Hammer Method (2) there is a 4.3% improvement from resin to 1% nanoclay and almost the same value is maintained for the other specimens. The 2% nanocomposite plate shows a Little decrease in in the Hammer method.

Table 7. Elastic Properties, Doppler Vibrometer Method$^{(1)}$ and Hammer Method$^{(2)}$

Composition	E_{xy}[GPa]$^{(1)}$	E_{xy}[GPa]$^{(2)}$	G_{xy}[GPa]$^{(1)}$	G_{xy}[GPa]$^{(2)}$	$\nu_{xy}{}^{(1)}$	$\nu_{xy}{}^{(2)}$
Resin	27.3	23.3	4.2	4.2	0.10425E-07	0.182E-03
Nanoclay 1%	28.3	24.3	5.1	4.8	0.10212E-03	0.133
Nanoclay 2%	31.9	23.6	3.8	4.7	0.75339E-03	0.078
Nanoclay 5%	36.1	24.7	5.3	5.0	0.13571E-03	0.044
Nanoclay 10%	35.7	24.6	5.0	4.7	0.27087E-03	0.104

For both methods the maximum Elastic Modulus value occurs for 5% nanocomposite plate, and for this specimen the difference between the methods reach its maximum too. This behavior can be attributed to be the presence of the 'spurious modes' in the Doppler Method and the analysis made without considering them. For two plates where the Doppler method does not show the 'spurious modes' the Resin and 1% Nanoclay plates the difference between the methods is constant and under 17%.

But the biggest difference between the methods is on the Poisson Coefficient. This property has very low value when compared with Shear or Elastic Modulus, and it presents some numerical precision problems when it is evaluated. From Tab. 7 it is obvious that the data from the Doppler Method is far from usual values. The results from Hammer Method are more close to expected values.

The Poisson Ratio is very sensitive to aspect ratio between width and length of the plate, and it is related to coupled deformation in two orthogonal directions, Lauwagie *et al.* (2003). This means that coupled modes presenting bending in both orthogonal directions, torsional-bending modes, are the best shape modes to identify the Poisson Coefficient. The three last shape modes, on Fig. 4 are the best to identify the Poisson Ratio when they are well defined, Lauwagie *et al.* (2010). The Poisson Coefficient is related to a well identified experimental coupled shape modes and good consistency between them.

The Figure 4 presents the six shape modes for 1% nanoclay plate, for highest content plate only the first five shape modes were identified Tab. 5. On Fig. 1 for the Doppler Method all shape modes seem to be deformed by the presence of sensor mass and have a not well defined coupled bending mode.

4. CLOSING REMARKS

The results summarised on Tab. 7 have suggested that the behavior of the shear modulus are linked to the improvement of the shock resistance with the nanoclay content matrix plate. This tendency was previously associated to damping coefficient increase but in this work the high damping coefficient was linked to 'spurious modes'.

The Hammer Method seems to be more reliable due to lower errors between experimental frequencies and those obtained from the Finite Element model. The results obtained by this technique has showed a little growth in the Elasticity Modulus value with the content of nano-particles in the matrix of the composite. A standard tensile test is the the next step to confirm the results.

The result for the laser vibrometer measurements showed a more significant increase in Elasticity Modulus, but the presence of 'spurious modes' in the range of natural frequencies of the plate showed that the Finite Element model does not accurately describe the phenomenon. As the result, this Method presented the greatest errors.

The pure resin and 1% nano content plates have not presented spurious modes inside the of experimental frequencies range, but they have showed 17% difference in the Elasticity Modulus value with those obtained with the Hammer procedure. To make the model more realistic probably 'spurious modes' need to be included in the finite element model analysis even if this mode is out of the frequency range of interest.

The Poisson's ratio measurement proved to be a difficult task because it requires torsion-bending modes, these modes are usually difficult to be obtained. In the method that employs laser Doppler vibrometer the transducer mass has distorted these modes and the results were very poor.

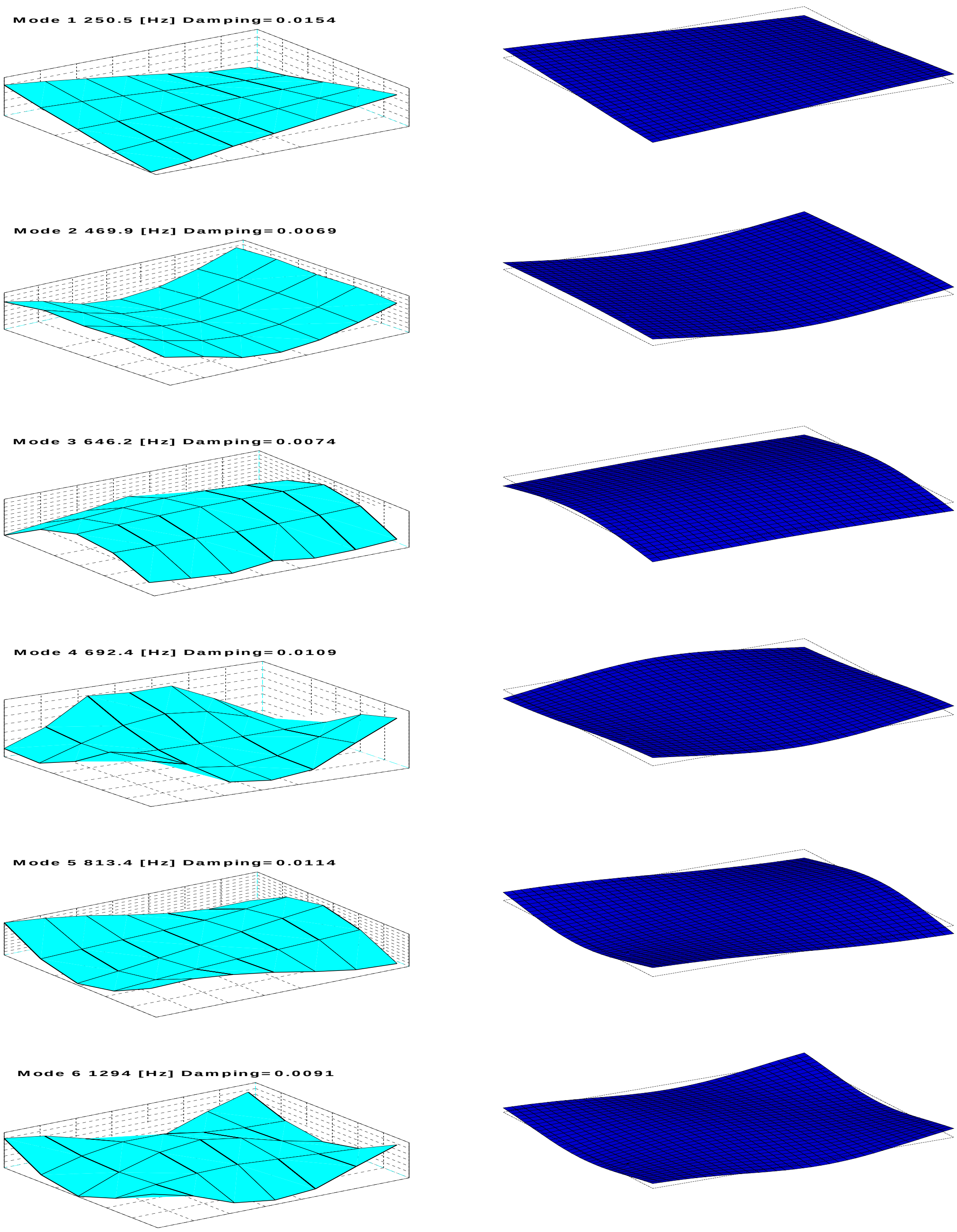

Figure 4. Shape modes for 1% nanocomposite plate, hammer method. At the left column the experimental shape modes at right the FE mode at the end of optimization process.

The Shear Modulus was less sensitive to the observed problems for Elastic Modulus and Poisson's ratio experimental values. The Shear Modulus presents close values for both methods with the dispersion of values within the expected. This mechanical property is linked to the first shape mode, a torsion mode, that is well defined and of easy measurement and identification. This robustness of the Shear Modulus measurements may be an important aspect for choosing mechanical properties for monitoring production or structural health of components in special conditions.

The inverse method minimizing a finite element model showed to be powerful, fast and very flexible method of analysis. In both measurement methods there are problems with force or acceleration transducers mass and, in both cases, mass and rotational inertia were considered in FE model. With mechanical excitation the Shaker Doppler Vibrometer is less dependent on the operator's skills and less sensitive to environmental conditions making it ideal for operating in confined environments such as in temperature controlled chambers. However, the presence of 'spurious modes' or coupling modes between the transducer and the plate brought difficulties in the analysis and inaccuracy in the results. There is also the problem of excessive distortion mode, that can be see comparing mode 3 in Fig. 1 with the same mode on Fig. 4, which resulted in unreliable values for Poisson's ratio.

5. ACKNOWLEDGEMENTS

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7. Responsibility notice

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