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An experimental-numerical methodology for the non-destructive assessment of the dynamic elastic properties of adhesives

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Abstract. In the last years, lightweight design has become a priority in many industrial sectors, like as the aerospace and the automotive industry, mainly due to the strict regulations in terms of gas emission and pollution. Together with lightweight materials, the use of adhesives to join different parts permits to significantly reduce the weight of mechanical assemblies. For a proper design of the joints, the mechanical properties of adhesives should be correctly experimentally assessed. However, the experimental assessment of the adhesive mechanical properties can be complex, since they can be hardly estimated from traditional experimental tests on lap-joint or butt-joint specimens. The development of an experimental procedure for the assessment of the adhesive properties is therefore of interest. In the present paper, a methodology for the assessment of the dynamic elastic properties of adhesives, i.e., Young's modulus and the loss factor, is proposed. The procedure is based on the Impulse Excitation Technique and Finite Element Analyses (FEA). An automated routine has been written to assess the elastic properties by minimizing the difference between the frequency response obtained experimentally and through FEA. The proposed methodology has been experimentally validated to estimate the mechanical properties of an epoxy adhesive for automotive applications.

1. Introduction

In the last years, lightweight design has become a priority in many industrial sectors, like as the aerospace and the automotive industry, mainly due to the strict regulations in terms of gas emission and pollution. Indeed, by reducing the mass of an aircraft or a vehicle it is possible to limit the fuel consumption and consequently the gas emission. For example, a 10% reduction of vehicle weight can result in a 6% - 8% of fuel saving, according to [1], and a 20% aircraft weight-saving permits to improve the fuel efficiency by about 10% - 12% [2]. Indeed, the International Civil Aviation Organization aims halving the aviation emissions by 2050 [2]. For these reasons, the use of lightweight advanced materials (e.g., composite materials [3,4]), characterized by high specific strength, and of design procedures and production processes allowing for maximizing the component strength while minimizing its mass (topology optimization and additive manufacturing) has significantly increased. Moreover, the choice of the appropriate joining technique between parts in mechanical assemblies, like as bonded joints, can significantly contribute to a further weight reduction. For example, adhesives, besides having a mass significantly smaller than traditional fasteners [5], permit to easily join lightweight materials (composite and metallic material) which can be hardly joined with traditional techniques [5], without affecting the structural integrity of the mechanical assembly [6]. In addition, adhesives can be easily employed with a limited number of operations and ensure a homogeneous stress distribution [6] within the joint, differently from traditional joining techniques (e.g., a threaded hole generates a stress concentration which is generally critical for parts used in structural applications). For these reasons, the use of adhesive has become fundamental in applications where lightweight design is a primary objective.

For a proper design of the adhesive joint with analytical methodologies or through Finite Element Analyses (FEA), the mechanical properties of the adhesive should be reliably assessed [7]. In some applications, moreover, adhesives are subjected to vibration loads [8-10], which could lead to the weakening of the adhesive and, eventually, to its failure. In this case, not only the quasi-static mechanical properties of the adhesive should be known for the design, but also its damping properties which would help designing the joint. In general, the experimental assessment of the adhesive mechanical properties can be complex, since they can be hardly estimated from traditional experimental tests on lap-joint or butt-joint specimens, but only through tests on specimens made of the tested adhesive. The development of non-destructive methodologies allowing to easily measure the adhesive properties are of interest and can simplify the design procedure of the joint. Among these, techniques based on resonance frequencies and modal analysis have been widely employed for the assessment of the dynamic elastic properties. For example, in [11, 12] the Young modulus and the damping properties are assessed by measuring the longitudinal resonance frequency of a bar and the resonance frequency of the same bar cut into two parts bonded together to form the adhesive butt-joint. The elastic properties are assessed by considering analytical approximated solutions which are valid under reported assumptions and conditions: these techniques are mainly suitable to detect damaged specimens in a group of specimens subjected to a particular treatment [12, 13]. In [13], this approach is extended to measure the Young modulus, the shear modulus and the Poisson ratio: an electro-mechanic vibrator is used to excite the specimen and the parameter estimation is based on analytical solutions. Even in this case, there are some limitations concerning for example the adherend lengths and the adhesive thickness, affecting the accuracy of the estimated elastic moduli. In [14, 15], the dynamic elastic properties are experimentally assessed through the “Direct Model Updating Method”. The proposed methodology involves FEA and the measurement of the bending frequency with an accelerometer. The model of the specimen is more complex than that of a butt joint and must take into account also the mass of accelerometer, which could affect the parameter estimation. Differently from the other techniques, the procedure developed in [14, 15] permits to assess the dynamic properties in bending.

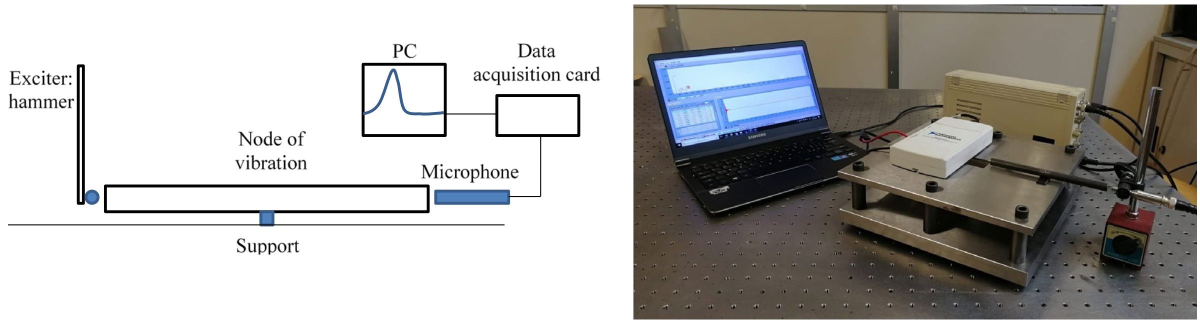
In the present paper, a methodology for the assessment of the dynamic Young’s modulus and of the loss factor in adhesives is proposed. Differently from the above mentioned literature references, the procedure is based on the Impulse Excitation Technique (IET, [16]) and FEA. In particular, the elastic properties of the adhesive are assessed by measuring the longitudinal resonance frequency of an adhesive butt-joint obtained with the investigated adhesive and through an optimization process aiming at minimizing the difference between the experimental resonance frequency response and the numerical frequency response obtained through FEA. The experimental setup for measuring the longitudinal resonance frequency is quite simple and the dynamic elastic properties are estimated through an automated procedure. The proposed methodology has been validated to estimate the elastic properties of an epoxy adhesive used for automotive applications. The potentialities and the factors affecting the accuracy of the proposed methods have been finally discussed.

2. Estimation of the dynamic elastic properties

In this Section, the procedure developed for the assessment of the dynamic elastic properties of adhesives is described. In Section 2.1, the IET is described, whereas in Section 2.2 the proposed experimental-numerical methodology is analyzed.

2.1. Impulse Excitation Technique: first longitudinal resonance frequency

The procedure is developed to non-destructively assess the dynamic elastic properties of the adhesive. It is based on the IET. This technique is generally used for assessing the elastic moduli of isotropic material with prescribed geometry. The International Standard [16] prescribes the geometry of the specimens for assessing the longitudinal modulus for isotropic materials. In this work, the IET is exploited to measure Young's modulus and the loss factor of adhesives by considering the first longitudinal resonance frequency of a butt-joint made with the investigated adhesive. Fig. 1a) and Fig. 1b) schematically show the working principle of the IET for assessing the first longitudinal resonance frequency and the experimental configuration used at Politecnico di Torino, respectively.



(a) (b)

Figure 1. Impulse Excitation Technique for assessing the longitudinal resonance frequency: a) schematic standard configuration; b) IET used a Politecnico di Torino.

As shown in Fig. 1, the investigated bar is supported at the half length, which corresponds to the node of vibration for the first resonance frequency. The specimen is hit at one free end and a microphone is placed at the other free end, where the displacement amplitude reaches its maximum, in order to obtain a high signal to noise ratio. The signal measured by the microphone is properly amplified and acquired with a data acquisition card. Then, it is analyzed with a software based on the Fourier Transform to assess the frequency response. Given the first resonance frequency, the Young' modulus for an isotropic material can be assessed according to [16].

In this work, the bar is replaced by a specimen obtained by bonding together two bars with the investigated adhesive. The geometry of the specimen is reported in Figure 2. The hammer is made by a flexible bar with a sphere used for hitting the specimen, in order to obtain a clean excitation signal. The system for measuring the pressure wave is composed by a microphone (PCB 130E20), a signal amplifier produced by PCB and a data acquisition card (National Instruments, NI 6210) with a maximum sampling frequency of 250 kHz. The software Buzz-o-sonic ® [17] and a program developed by the Authors in the LabView environment are used for computing Fourier Transform and assessing the resonance frequency.

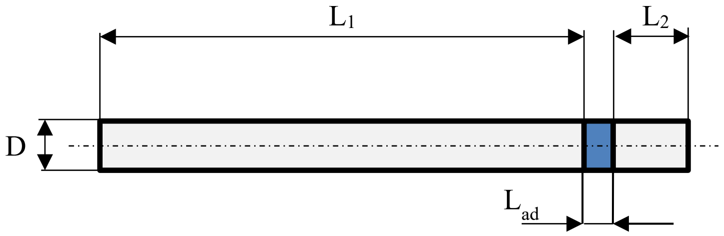


Figure 2. Representation of the butt-joint specimen used for the assessment of the adhesive properties.

According to Fig. 2, the two bars are bonded together in a butt-joint configuration (L_1 and L_2 are the lengths of the adherends, L_{ad} is the adhesive thickness and D is the diameter of the bars). For the application of the proposed procedure, the lengths L_1 and L_2 should be chosen so that the first longitudinal resonance mode can be easily excited with the configuration shown in Fig. 1. In general, the ratio between the length of the whole bar (in this case the length $L_{tot} = L_1 + L_2 + L_{ad}$) and the diameter of the bar, D, should be large enough (at least larger than 5) to properly excite the first longitudinal mode. In the literature [13], the analytical models used for the estimation of the elastic properties are obtained by considering a length L_1 close to L_2.

2.2. Adhesive elastic properties: iterative process

Fig. 3 shows the flow chart of the proposed methodology.

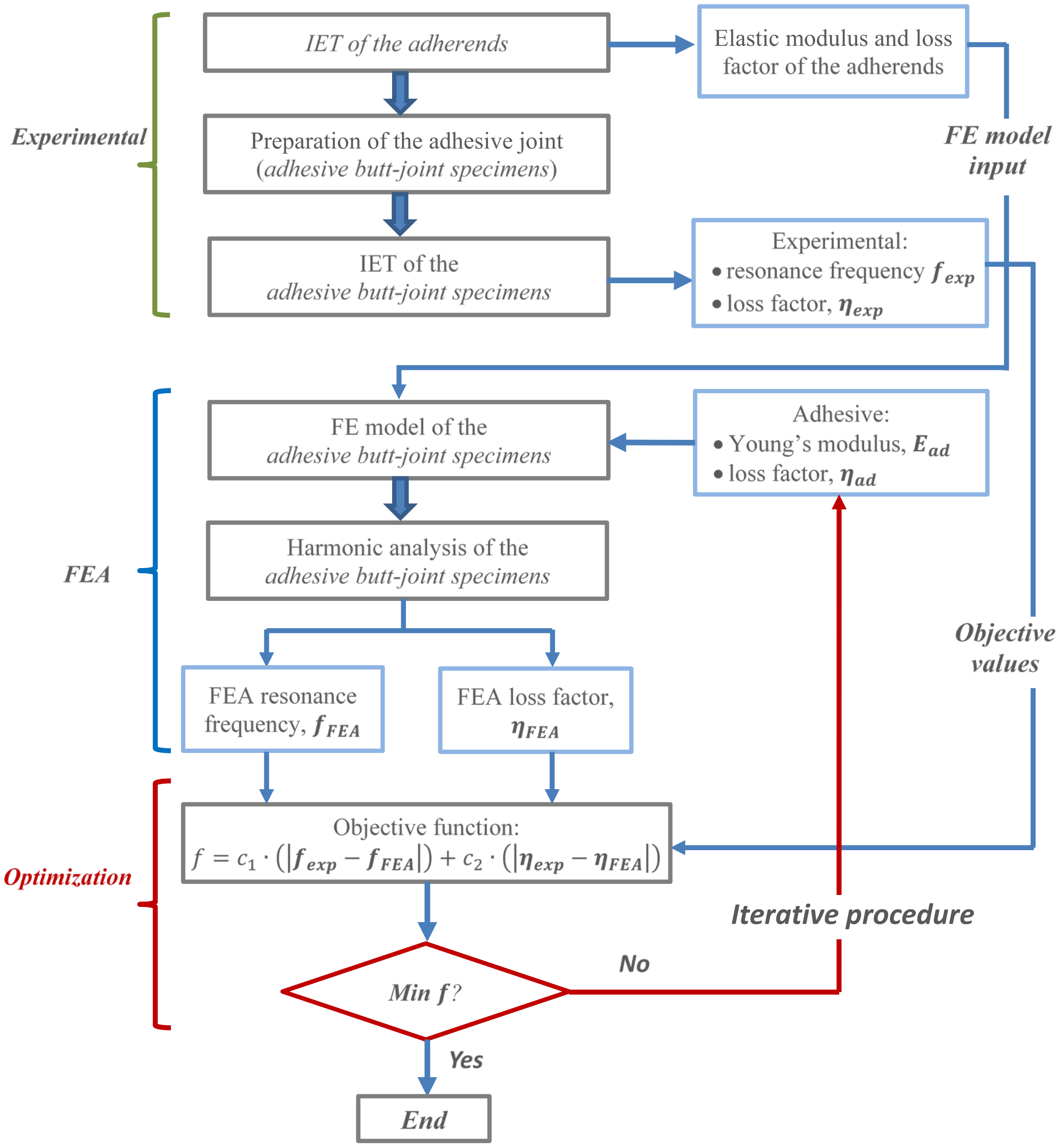


Figure 3. Flow chart of the experimental – numerical procedure for assessing the elastic properties of adhesives.

According to Fig. 3, the first step involves the experimental assessment of the elastic properties of the adherends. In particular, a first IET test is carried to assess the first longitudinal resonance frequency and, accordingly, the Young's modulus and the loss factor of the adherends. These two parameters, together with the density, represent the necessary input for the model of the adhesive butt-joint specimen. Thereafter, the two adherends are bonded together with the investigated adhesive to form the adhesive butt-joint specimen. A second IET test is carried out to assess the experimental resonance frequency, f_{exp}, and the loss factor, η_{exp}, of the adhesive butt-joint specimen. The loss factor is computed by applying the *half-power bandwidth method* [18]. A FE model of the adhesive butt-joint specimen is thereafter created by considering the mechanical properties of the adherends estimated at the beginning of the procedure. On the other hand, the mechanical properties of the adhesive, i.e., the Young's modulus E_{ad}, and the loss factor η_{ad}, represent the variables of the optimization process. A first initial guess of these two parameters is reasonably represented by literature values. A Harmonic analysis is thereafter carried out to assess the frequency response, allowing to compute the first resonance frequency and the loss factor of the adhesive butt-joint specimen, f_{FEA} and η_{FEA} respectively. These two parameters, together with f_{exp}, and η_{exp} are involved in the optimization function, f, defined according to Eq. (1), as the weighted sum (through the coefficients c_1 and c_2) of the absolute difference between the f_{exp} and f_{FEA} and η_{exp} and η_{FEA}:

$$f = c_1 \cdot \left(\left| f_{exp} - f_{FEA} \right| \right) + c_2 \cdot \left(\left| \eta_{exp} - \eta_{FEA} \right| \right) \tag{1}$$

The optimization process aims at finding the minimum of the function reported in Eq. (1), with the parameters E_{ad} and η_{ad} iteratively varied in order to minimize the objective function f. The procedure ends when the minimum of the function is found, and the convergence criterion depends on the selected optimization method.

The proposed procedure permits therefore to non-destructively assess the elastic properties of the investigated adhesive. From an experimental point of view, only two IET tests should be carried out to assess the first longitudinal frequency. The finite element model of the adhesive butt-joint specimen can be created with commercial software and the f_{FEA} and η_{FEA} values should be exported in an optimization tool to minimize the objective function.

3. Experimental validation

In this Section, the proposed procedure is experimentally validated. In Section 3.1, the proposed procedure is applied to assess the elastic properties of an epoxy resin used for automotive applications. In Section 3.2 the estimated values are experimentally validated through tensile tests and a second IET test. In Section 3.3 the validity of the proposed methodology and the influence of the parameters involved in the optimization process are finally discussed.

3.1. Epoxy resin Betaforce 4600G: algorithm implementation

The procedure has been experimentally applied to assess the elastic dynamic properties of the epoxy adhesive Betaforce 4600G (supplied by Dow Automotive) used for automotive applications. The Betaforce 4600G is a mono-component adhesive loaded with glass spheres to keep the adhesive thickness close to 200 μm, which maximises its strength.

Two bars in Ti6Al4V alloy with diameter D equal to 14.6 mm were used for the adherends. The length of adherend 1, L_1, was equal to 114.2 mm, whereas the length of adherend 2, L_2, was equal to 7.7 mm, according to Fig. 2. The length L_1 was significantly larger than the length L_2 since the butt-joint specimen was initially designed for being tested under Very-High-Cycle Fatigue (VHCF). For more details on the choice of the length L_1 and L_2, the reader is referred to [19].

The first step of the proposed procedure (Fig. 3) involves the assessment of the dynamic elastic properties of the Ti6Al4V adherend, according to [16]. The adherend 1 was used for the IET test: since both adherend 1 and adherend 2 were cut from the same bar, it was reasonably assumed that the elastic properties of adherend 1 and adherend 2 were the same. The bar, supported at the half length, was hit with the hammer. The signal, acquired with a microphone located at the other free end, was analysed by the software Buzz-o-sonic®, which provides the frequency response. From the first longitudinal frequency, Young's modulus of 107.5 GPa and a loss factor of $2.92 \cdot 10^{-4}$ were assessed for the Ti6Al4V bars.

Following the scheme in Fig. 3, the adhesive butt-joint specimen was thereafter manufactured according to the indications of the supplier. In particular, after a careful cleaning of the surfaces to be bonded, the adhesive was applied at a temperature of 60° C with a nozzle. The adhesive was finally cured at 180 °C for 30 minutes [19]. A second IET test was performed on the adhesive butt-joint specimen: a longitudinal resonance frequency f_{exp} in the range [20176-20180] Hz and a loss factor η_{exp} in the range [3.49-3.59] $\cdot 10^{-4}$ were experimentally found. Fig. 4 shows the frequency response of the adhesive butt-joint specimen. The first longitudinal resonance frequency is clearly visible even for a bar obtained with a butt-joint. In Fig. 4, in the ordinate axis the acquired amplitude is normalized by the largest amplitude in correspondence of the resonance frequency ("Normalized amplitude").

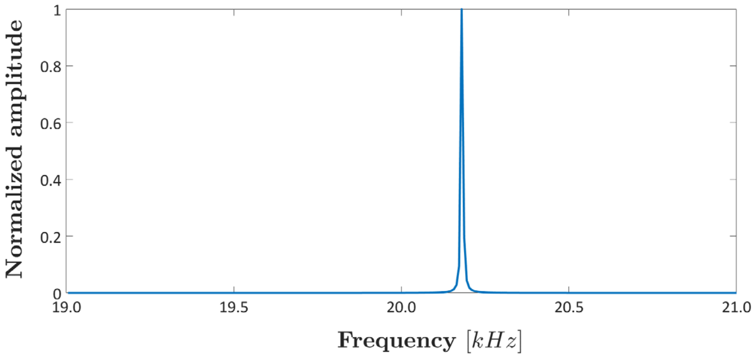


Figure 4. Frequency response of the adhesive butt-joint specimen.

Given f_{exp} and η_{exp}, the elastic parameters were thereafter estimated by minimizing the objective function. The procedure was implemented in the Matlab. In particular, a Matlab routine was written to iteratively launch the harmonic analysis and vary the optimization variables, i.e., the Young's modulus and the loss factor of the adhesive. The function f was minimized by using an algorithm based on the *Interior-point method*. The coefficients c_1 and c_2 in Eq. (1) were set equal to 1 and 10^5, respectively, in order to avoid that the frequency difference had a major weight in the optimization process. The FEA was run with the commercial software Ansys. Plane axisymmetric elements were used and the adhesive butt-joint specimen was loaded with a harmonic force at one hand with the loading frequency varying in the range [20.1-20.2] kHz, to simulate the IET test. A mesh refining was considered to properly model the thin adhesive layer [20]. However, since a linear elastic behavior of the materials is considered and it is not necessary to assess the stress within the adhesive layer, a coarse mesh permits to properly simulate the IET test and to reduce the testing time. Final values for E_{ad} in the range [2.9-3.3] GPa and for the loss factor η_{ad} in the range [0.0190-0.0210] were experimentally found. Fig. 5 shows an example of the convergence plot for the function f : the abscissa axis reports the iteration number, whereas the ordinate axis the value of the function f . At the last iteration, the difference between the experimental and the numerical values was smaller than 0.5% for both the resonance frequency and the loss factor, thus confirming the validity of the proposed procedure.

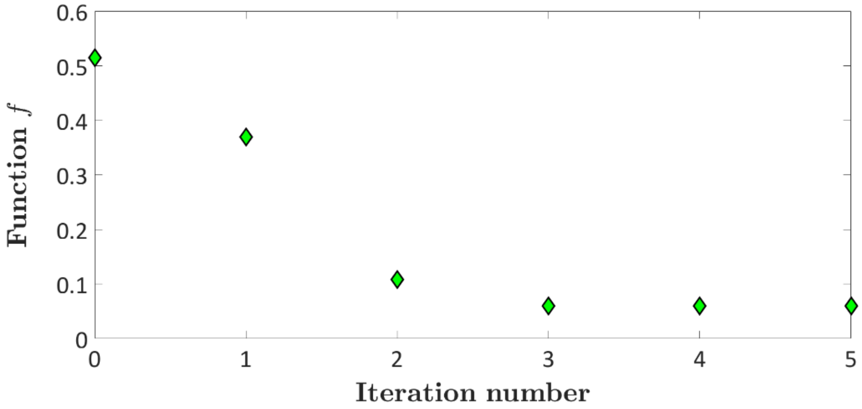


Figure 5. Convergence plot: Function *f* value with respect to the iteration number.

3.2. *Experimental validation: tensile tests and IET*

The estimated elastic properties were finally experimentally validated. A tensile test was carried out on an adhesive butt-joint specimen obtained by bonding two steel bars with an equal length of 60 mm and a diameter of 10 mm. The tensile test was carried out by using a servo-hydraulic testing machine (Instron 8801), with a crosshead displacement of 1 mm/min. The force-displacement curve was acquired and used for the validation of Young's modulus. The experimental curve was compared with the force-displacement curve obtained through FEA. An FEA of the adhesive butt-joint specimen used for the tensile test was run and a transient nonlinear finite element analysis was performed. Both the adherends and the adhesive were modeled with eight-nodes solid elements with a fully integrated formulation. The mechanical properties of the adherends were experimentally assessed through IET, whereas the adhesive properties were those estimated through the procedure proposed in Section 2. In particular, a median value for the Young Modulus (3.1 GPa) was considered. Both materials, steel and epoxy resin, were modeled as perfectly elastic and the commercial software LS-DYNA was used. Simulations were performed by clamping the extremity of one adherend (imposed nodal displacement equal to 0) and applying a prescribed motion law to the other end. In particular, according to Niutta et al. [21], an initial ramp up to a constant velocity corresponding to the experimental crosshead displacement was considered.

Fig. 6 compares the simulated and experimental force-displacement curves in the range [0-600] N, since above 600 N a specimen slippage was observed during the experimental tests.

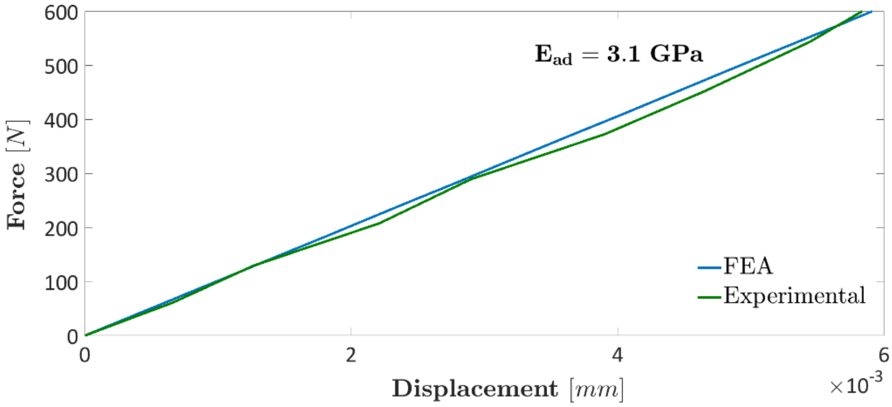


Figure 6. Force-displacement curve for the adhesive butt-joint specimen: experimental and numerical curve.

According to Fig. 6, the simulated curve perfectly overlaps the experimental curve.

The Young's modulus and, especially the loss factor, were further validated. A second IET test was carried on the adhesive butt-joint specimen with the same geometry used for the tensile test. The experimental resonance frequency was found to be in the range [20174-20180] Hz and the loss factor η in the range $[6.22\text{-}6.48]\cdot10^{-3}$. Thereafter, the resonance frequency and the loss factor were assessed through FEA. It was found that with a Young modulus of 2.9 GPa and a loss factor equal to 0.02 (both in the range estimated in Section 3.1), the first longitudinal resonance frequency of the adhesive butt-joint specimen was equal to 20178 Hz and the loss factor was equal to $6.3\cdot10^{-3}$, within the measured range. This second validation confirms that the set of optimized parameters is close to the actual elastic properties. The estimated parameters, therefore, permit to properly assess the dynamic elastic properties of the adhesive and can be adopted for the simulation of the response of adhesive joints.

3.3. Discussion

It is worth noting that the proposed methodology depends on the initial values for the parameter to be optimized. These parameters should be reasonably chosen from literature value, thus permitting to speed up the optimization process and to properly estimate the actual parameters. If literature data are not available, multiple optimizations can be performed, running a first preliminary analysis and then using the optimized parameters as initial parameters for a second optimization.

Moreover, differently from [19], where E_{ad} and η_{ad} were estimated sequentially without an iterative procedure, with the proposed methodology they are estimated together. In general, the resonance frequency varies with the loss factor: if the variation of the resonance frequency with the loss factor is negligible, a sequential scheme could provide a very good approximation of the real parameters. Otherwise, the concurrent optimization scheme proposed in this work is the only solution to properly estimate the elastic properties.

It must be also pointed out that the adhesive thickness significantly affects the parameter estimation and, therefore, should be accurately measured. If this length cannot be accurately measured, this methodology still provides good estimates, which are slightly affected by the uncertainty in the adhesive thickness.

Moreover, for the experimental assessment of the loss factor, the support at half of the specimen length (Fig. 1a) should be properly chosen. It should as thin as possible, otherwise, it could affect the experimental measurement. For this reason, different solutions have been tried in order to obtain a range of variation of the measured η_{exp} in a limited range among two subsequent measures.

Furthermore, differently from the techniques proposed in the literature [11, 12], which are based on the experimental assessment of the resonance frequency of butt-joints and on approximated analytical solutions, the proposed methodology has no limitations, provided that the eigenfrequency can be properly measured and a clear peak for the first longitudinal mode can be assessed. The adherend length can be reduced: for example, for a diameter D (Fig. 2) equal to 14.6 mm, a length of 120 mm is sufficient, which is significantly smaller than the length adopted in [13] and equal to 350 mm. Finally, the use of a simple hammer to hit the specimen in place of an electro-mechanic vibrator [13], and the use of a microphone for acquiring the pressure wave in place of the accelerometer in [14, 15], strongly simplifies the experimental setup.

4. Conclusions

In the present paper, a methodology for the assessment of the dynamic elastic properties of adhesives is proposed. The procedure is based on the Impulse Excitation Technique (IET) and Finite Element Analyses (FEA): the elastic properties of the adhesive were assessed by measuring the longitudinal resonance frequency of an adhesive butt-joint obtained with the investigated adhesive and through an optimization process aiming at minimizing the difference between the experimental resonance frequency response and the resonance frequency obtained through FEA.

The proposed procedure was applied to estimate the dynamic Young's modulus and the loss factor of an epoxy resin used in automotive applications. The estimated values were experimentally validated through a tensile test and a second IET test with limited differences. The experimental validation proved the effectiveness of the proposed methodology for assessing the dynamic properties of adhesives, with a relatively easy experimental configuration and simple FEA.

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