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Investigation of Damage in Composites Using Nondestructive Nonlinear Acoustic Spectroscopy

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Abstract The presented experimental work describes the nondestructive damage examination of polymer-matrix composites using acoustic methods under the consideration of nonlinear effects. The aim is to analyze these nonlinear effects in order to provide a quantification of the nonlinear acoustic transmission which is related to the damage state and its severity in the composite material. The first objective was to study the effectiveness of the distortion evaluation method and its related parameter: the "Total Difference Frequency Distortion" *(TDFD)* parameter. The *TDFD* was utilized as a new damage indicator to quantify the progressive damage state in composite materials. The *TDFD* method had initially been proposed to characterize the distortion of audio amplifiers. A custom-made setup was developed that imposes acoustic signals to the structure. The samples' vibrations were afterwards analyzed by a laser vibrometer and further spectrum evaluations. The developed method was applied to two composite materials, both reinforced with taffeta woven glass-fibers, but having different thermoset polymer matrix, i.e. vinylester and epoxy. The damage was introduced in the specimen by tensile tests with a stepwise increase of the tension loading. It was observed that damage influences the intensity of nonlinear intermodulation after having introduced two harmonic and constant signals of different and randomly chosen frequencies in the specimen. The nonlinear intermodulation was then quantified by computing the *TDFD* parameter. In the specific case of epoxy based composites, high frequency peaks were noted for the high tensile loading levels only. The *TDFD* parameter was then modified in order to take into account this effect. For both studied composites, the modified *TDFD* parameter increases with the damage accumulation caused by the applied stepwise tensile loading.

Keywords Polymer-matrix composites · Damage detection · Nondestructive testing · Nonlinear acoustics · Intermodulation

Introduction

Composite materials are currently widely used in industry. One reason is their compliant adjustability to prerequisite mechanical characteristics; another reason is their lightweight properties, which appeal to lucrative automotive and aerospace construction applications.

In this paper, the focus is on laminated polymer matrix composites reinforced by woven glass fibers. These composite materials exhibit complex damage mechanisms including delamination, fiber breakage, and interface debonding which all affecting the overall mechanical response. To detect and quantify such damage in a structure, appropriate testing techniques are needed. In quality control and inspection, specific nondestructive testing is required to verify the integrity of the examined material. The range of nondestructive testing techniques is wide and covers for instance radiographic, infrared and electromagnetic characterization. Furthermore, some testing techniques are based on ultrasonic and acoustic methods, which are also known as appropriate and suitable damage characterization techniques in composites [1]. We will focus on the latter testing techniques, and in particular on the application of nonlinear acoustic methods which stand in contrast to linear methods like, for example, the classical C-scan or the polar-scan described in reference [2, 3].

Indeed, besides the linear ultrasonic evaluation of materials, such as the well-established A-, B- and C-scan, there is another approach to evaluate the damage using nonlinear elastic wave spectroscopy methods. This technique is based on the fact that a material behaves much more nonlinearly in the presence of damage, than in the undamaged state [4] during dynamic loading. In particular a damaged material shows an increased nonlinear transmission behavior [5]. This can be explained by the formation of contact surfaces with increasing damage and degradation. The dependency between nonlinear acoustic behavior and contacts has been treated by Jiao et al. [6]. Qualitatively, one can state that the nonlinear response of the material increases with increasing damage state [4]. Consequently, the level of the acoustic nonlinearity is an indicator of damage severity. Measuring and analyzing these nonlinear effects is the aim of "Nonlinear Elastic Wave Spectroscopy" (NEWS). Its main advantage is its higher sensitivity for the detection of damage in comparison to classical linear methods [7] such as those based on bulk wave velocity measurements. Furthermore, it is more appropriate to heterogeneous materials such as composites where in particular the crack size is comparable to the wavelength of the ultrasonic waves [8]. Nonlinear acoustic or ultrasonic methods have also been successfully applied to other materials like concrete [9] and metals [10]. Indeed it has even been shown by Li et al. [10] that nonlinear acoustic methods reveal material property improvement as a consequence of heat treatments. The nonlinear spectroscopy method can also be carried out using a combination of a low frequency vibration and an acoustic signal as excitation; in this case it is called a nonlinear vibro-acoustic method [11].

Two main types of NEWS methods are described in the literature [4, 7, 8]: resonance based methods and non-resonance based methods. The present paper focuses specifically on the second type of nonlinear ultrasonic spectroscopy methods, namely the non-resonance methods that analyze the modulation spectrum. Consider two harmonic waves having two different frequencies f_1 and f_2 ($f_1 < f_2$) in a damaged material: the amplitude of the high-frequency wave will be modulated by the low-frequency wave due to nonlinear transmission behavior. It is worth noting that f_1 and f_2 are commonly chosen among the eigen frequencies of the undamaged sample [8, 12]. As a consequence, the created spectrum manifests a nonlinear intermodulation response by the generation of new frequencies such as sidebands ($f_2 \pm nf_1$; $n = 2, 3, \ldots$) and higher harmonics (nf_1; $n = 1, 2, 3, \ldots$). This is actually a hysteretic nonlinear effect that increases with damage evolution because the damage operates as a multiplier and nonlinear mixer of the excitation frequencies. Figure 1 schematically depicts this effect [8].

In the current work, a factor quantifying the nonlinear transmission behavior of composite samples is adopted, originally from the field of sound system equipment, to nondestructive evaluation. The latter is defined as the "Total Difference Frequency Distortion" (*TDFD*), which is a meaningful quantity known from the technology of audio amplifiers and their nonlinear behavior [13]. On a broader level, the range of standardized methods for characterizing distortion in the audio field are extended in this work to the specific field of nondestructive evaluation of composite materials.

The main purpose of the present work is hence to analyze and to quantify the nonlinear effects of the nonlinear acoustic transmission and to relate them to the damage state evolution and its severity in the composite material. A modified *TDFD* method was proposed and applied to characterize the damage accumulation of two composite materials. A specific MATLAB routine was developed to assess the ability of this nondestructive damage investigation to detect and to quantify the damage evolution in composite materials.

In what follows, research and results are presented in subsequent sections. The research section is devoted to the description of the two investigated composites which have two different matrix systems, epoxy matrix and vinylester matrix, reinforced with woven glass fabric. The experimental procedure to induce increasing levels of damage via tensile tests is consequently described in more detail. Our in-house designed non-linear acoustic set-up and the related MATLAB evaluation routine developed to estimate the damage level, are described. The results of the nonlinear elastic wave spectroscopy are exposed and discussed in the third section. In the specific case of the glass epoxy matrix based composite specimen, the appearance of high frequency peaks is noted for the severest damaged samples (loaded in tension from 250 MPa to 431 MPa). A modification of the *TDFD* parameter was introduced for measurement processing, related to these specific frequencies. Finally, the paper ends with some concluding remarks and an outlook on further experimental work and industrial applications.

Materials Description and Experimental Procedure

Here the experimental procedure is presented and the two studied composite materials are described. Both composites used for this research are reinforced with the same woven glass fiber fabric but have a different thermoset polymer matrix. All samples have been kept at room temperature and ambient relative humidity long enough in order to guarantee the same initial conditions. Before the tests, all samples were verified as 'undamaged' using ultrasonic C-scans in transmission. A 5 MHz transducer was considered for all those tests, which corresponds to a wavelength of 0.7 mm. Therefore, only samples that did not show defects bigger than 0.35 mm were considered in this study.

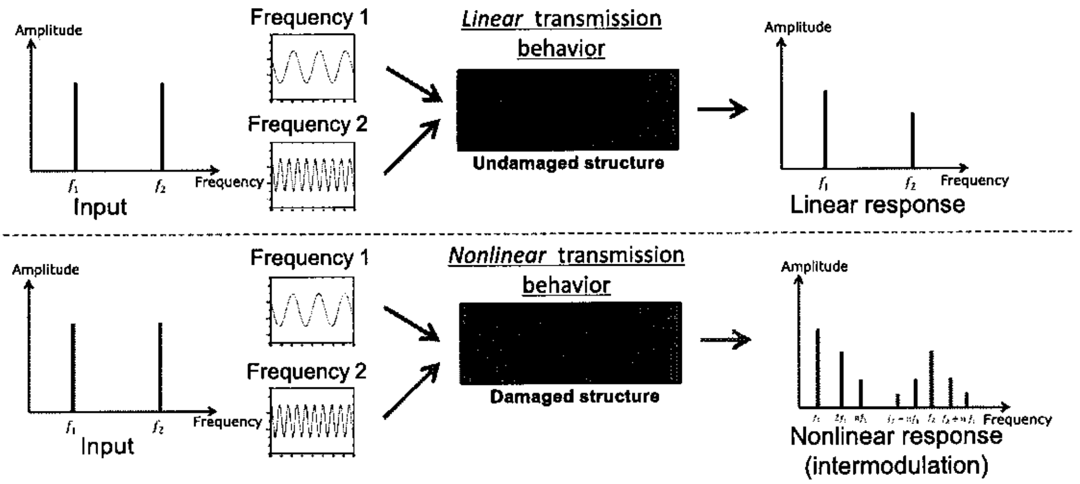


Fig. 1 Linear and nonlinear modulation effect. Top: Excitation frequencies and response of an undamaged sample with linear transmission behavior. Bottom: Excitation frequencies and response of a damaged sample with nonlinear transmission behavior

Preliminary tensile tests have been performed at room temperature to determine a representative stress/strain curve for both studied composite materials. These samples were loaded in tension until failure at a constant strain rate of 10^{-4} s^{-1}. All the tensile tests mentioned in this paper were performed on a tensile test machine "Z050" manufactured by *Zwick Roell Gruppe* in compliance with the standard ISO 5893. The strain was measured using an extensometer from *Epsilon Technology corp* (Model: 3542-025 M-010-ST), clips on the samples. A camera and the image processing software *ImageJ* were used to verify the alignment of the composite samples in the tensile test machine.

The average values of Young's modulus, the strain to failure and the stress to failure were already known to us from previous experimental investigations. Five different samples were used for each material and the representative stress/strain curves plotted in Fig. 3 are the ones matching those known averaged values.

For all the interrupted tensile tests performed to induce damage, the procedure is described as follows. The samples have first been loaded at a constant strain rate of 10^{-4} s^{-1} until the predefined stress level was achieved. The applied loading was maintained for 4 s, and then the specimen was unloaded at a constant strain rate of 10^{-4} s^{-1}. For each stress level, the tensile test was repeated a sufficient number of times until the stress/strain curve matched the previously discussed master stress/strain curve. The sample was subsequently selected for the nondestructive evaluation method.

Materials and Samples

Woven glass vinylester composite

First, samples made of glass fiber reinforced vinylester are considered. The woven fabric is a taffeta weave with orientations 0° and 90° and a fiber weight ratio of 73 %. The samples are produced by a resin infusion process and cut by a water jet cutter. The longitudinal Young's modulus of this material is 20 GPa and the longitudinal strength to failure is about 431 MPa. The specimen geometry is detailed in Fig. 2 (top) and the samples are cut parallel to the 0° orientation. Four samples were prepared, by application of tensile tests that are interrupted at predefined stress levels: 100, 200, 300 and 400 MPa. This allows introducing progressive damage in the samples. Additionally, a fifth reference sample is left in the initial undamaged state. The different states of tension, and as a consequence the different states of damage, are illustrated by means of circles on the stress/strain curve in Fig. 3.

Woven glass epoxy composite

The second set of samples is made of glass fiber reinforced epoxy with a taffeta weave tissue and 53 % fiber mass fraction. The specimen's geometry is detailed in Fig. 2 (bottom) and again the samples are cut parallel to the 0° orientation. A water jet cutter has been used to cut the sample. The longitudinal Young's modulus is 22 GPa and the longitudinal strength to failure is about 436 MPa. Ten samples were prepared by tensile tests to introduce progressive damage level by a series of tensile tests that are interrupted at predefined stress levels: 50, 100, 150, 200, 250, 300, 350, 400, 417 and 431 MPa. An eleventh reference sample is maintained in the initial state. The different states of tension are illustrated by crosses on the stress/strain curve in Fig. 3.

Custom-Made Experimental Setup

Here the experimental setup, the deployed instruments and their settings are described in detail.

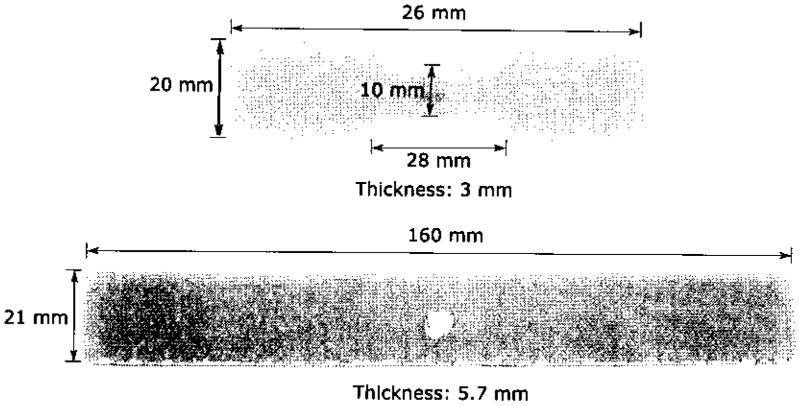


Fig. 2 Studied composite samples with given dimensions. Top: Woven glass vinylester composite sample; Bottom: Woven glass epoxy composite sample with silver-colored spot at the center to improve reflection

The two excitation signals are generated by the sound card of a common PC using a free signal generator, *Daqarta* (version 7.60.01), developed by *Interstellar Research*. The frequencies are randomly chosen, with the difference between the two frequencies smaller than the lower chosen frequency as is required by the DIN standard [14]. The generated signals are transmitted to the sound signal amplifier over the speaker output of the PC. The used audio power amplifier is that of a speaker system *Home Arena 5.1* from *TerraTec Electronic GmbH*. The amplified sound-signals are then transformed into vibrations using the piezo elements of two common piezoelectric loudspeakers (model *XTC PT1*) that are not fixed to the ground as to reduce coupling with the environment. The loudspeakers are opened to place the two sample's extremities in direct contact with the two piezo elements (see Fig. 4). The dimension of each contact zone is approximately 1.6 cm^2. To improve contact, an ultrasonic coupling gel is used. The sample's vibrations are surveyed by the sensor of a laser vibrometer *Fiber Vibrometer OFV-551* and the *Vibrometer Controller OfV-5000* both manufactured by *Polytec GmbH*. To improve the signal, the reflection of the laser is enhanced by silver color paint (see Fig. 2). The laser emitter is positioned orthogonal at a distance of 12 cm above the central point of the sample and is manually focused. The vibrometer's output is connected to a second amplifier, namely the *2.45.70 A* model manufactured by *Nucletudes*. This amplifier sends the vibration information to the oscilloscope, which transforms the signal to its frequency spectrum using the Fast Fourier Transformation (FFT) with a rectangular window function (window time width: 0.001 s). The transformation products are then continuously averaged over 100 sweeps using the relative "weight" of each sweep equal to 1/100. The complete experimental setup is shown in Fig. 5.

Spectrum Evaluation Routine and Signal Treatment

To evaluate the given frequency spectrum of the samples' vibration response, an evaluation routine has been written in MATLAB, described further. The aim of this routine is to compute the *TDFD* according to reference [13]. It provides a measure for the intensity of the intermodulation:

$$TDFD = \frac{\sqrt{\sum_i A^2_{i,\text{intermodulation}}}}{A_{excitation\ 1} + A_{excitation\ 2}} \tag{1}$$

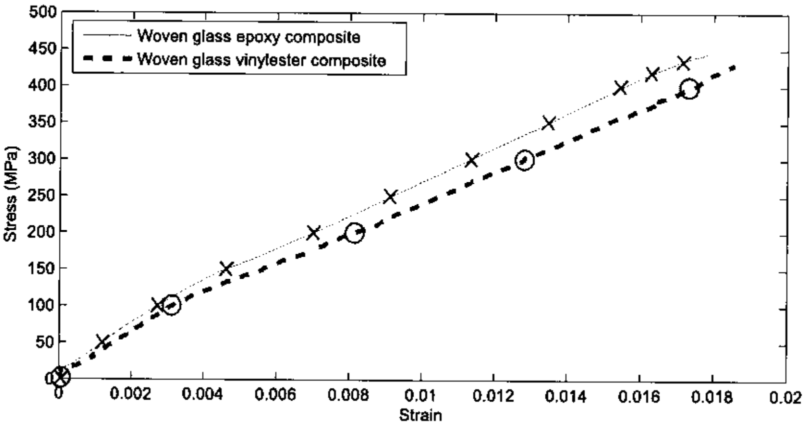


Fig. 3 Representative stress-strain curves of the two investigated materials; the solid curve represents the woven glass epoxy composite and the dashed curve represents the woven glass vinylester composite; the crosses and circles indicate the different states of tension applied in this work to damage the samples

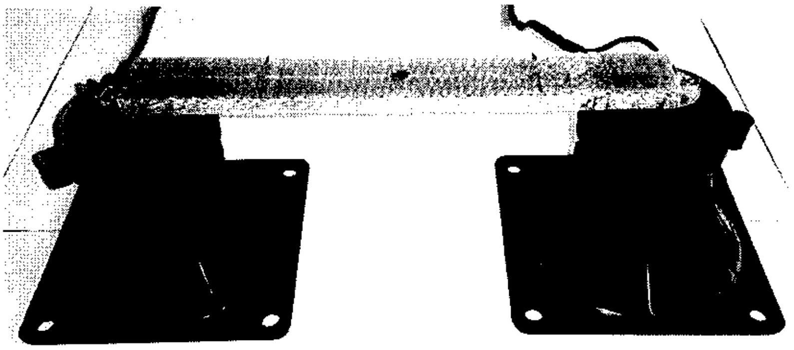


Fig. 4 The two piezoelectric actuators (*opened loudspeakers*) with a sample in place

where $A_{i,\text{intermodulation}}$ are the amplitudes of the intermodulation products and $A_{excitation\ 1}$ and $A_{excitation\ 2}$ respectively are the amplitudes of the two excitation frequencies in the response's spectrum. Intermodulation is defined as the creation of new frequencies when two or more different frequencies are treated by a system with a nonlinear transfer function.

Note that the distortion measure is well-established in the field of sound system equipment, and is used to evaluate unwanted distortion behavior of amplifiers. For this reason, the introduction of this parameter in the field of nondestructive testing by nonlinear acoustics is suitable. Indeed, the distortion measurement method of audio amplifiers is comparable to the nonlinear transmission behavior of damaged composites. Both cases consider the nonlinear frequency response of a system tested with two signals of two different frequencies. Before the *TDFD* can be calculated, the spectrum must be modified as described below. For example, the spectrum is exposed to a damping effect that must be eliminated before calculation of the introduced parameter.

First, the spectrum is shifted upward by adding the amplitude (in dBm) corresponding to the highest frequency to all values of the spectrum. This ensures positive values for all frequencies. The result of this processing on the first sample of woven glass vinylester composite is shown in Fig. 6.

It is clearly visible that an underlying "background function" shifts the peaks to a higher or lower power depending on the frequency. This effect can be explained by damping: waves at higher frequencies are more damped than

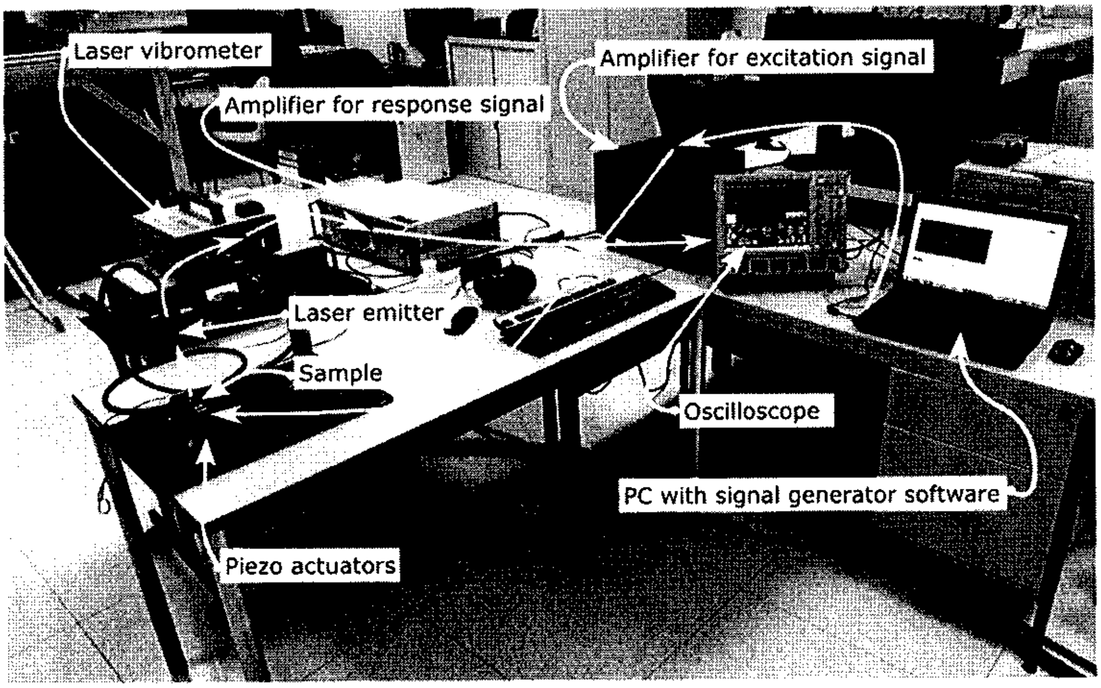
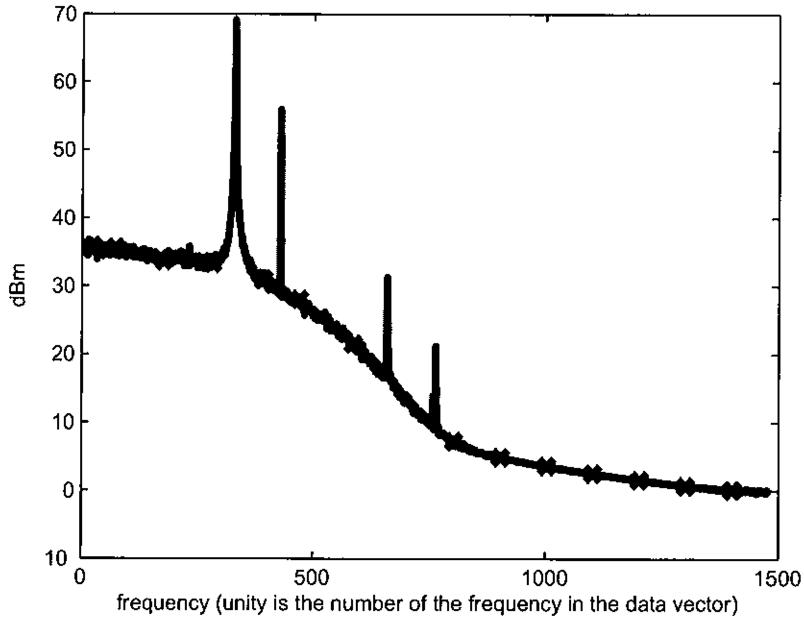


Fig. 5 The complete experimental setup. Components are labeled and signal paths are illustrated by arrows

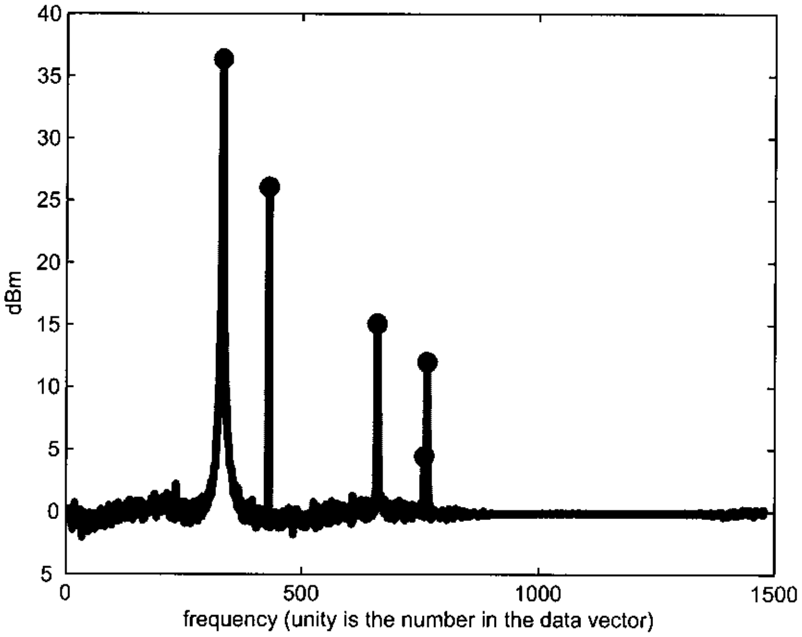
Fig. 6 Spectrum of the undamaged woven glass vinylester composite sample excited by 10 kHz and 13 kHz, shifted to positive values and plotted markers of the background function



waves at lower frequencies. As a consequence, waves of higher frequencies are positioned in a lower power range. These waves are more damped because they have to cover a longer distance (relative to their wavelength) in the material since they are oscillating faster. To subtract this background function, the following steps are applied to the spectra:

1. Manually identify points on the spectrum that represent the background function but are clearly not peaks of the spectrum (see Fig. 6).
2. Calculate a spline that represents the points marked in the previous step.
3. Subtract the spline from the spectrum to subtract the background function.
4. Use the "PeakFinder"-MATLAB-function [15] to find the peaks of the spectrum and to store their location and amplitude (see Fig. 7).
5. Add the squares of all peak amplitudes that are not the peaks of the two excitation frequencies but that are products of their intermodulation $\sum_i A_{i,\text{intermodulation}}^2$.

Fig. 7 Spectrum of the undamaged woven glass vinylester composite sample excited by 10 kHz and 13 kHz, shifted to positive values. The background function is subtracted and the peaks are marked



6. Add the amplitudes of the excitation frequencies $A_{excitation\ 1} + A_{excitation\ 2}$.
7. Calculate the *TDFD* according to equation (1) to get a measure for the intensity of the intermodulation.d

It is worth mentioning that measurements were done 3 times on each sample, and the same *TDFD*'s values were obtained for the three measurements.

Experimental Results and Discussion: Nonlinear Wave Modulation Spectroscopy (NWMS)

As described above, the NWMS method should exhibit an evolution of the intermodulation products with increasing damage. As an example, the resulting spectra of the tests executed on the woven glass vinylester composite samples are shown in Fig. 8. It is a clear observation that the number of peaks increases with increasing damage. For the undamaged sample, there are only two peaks beside the peaks resulting from the excitation, which indicates a nearly linear transmission behavior. The nonlinear transmission behavior increases with increasing tensile stress. This behavior is visible in the appearance of peaks in sidebands and higher harmonics.

The subsequent paragraphs describe the results of the Nonlinear Wave Modulation Spectroscopy method for the two different materials.

Results for Woven Glass Vinylester Composite

The method is applied to the woven glass vinylester composite samples using the excitation frequencies of 10 kHz and 13 kHz. The resulting dependence between damage and intermodulation (as a consequence of nonlinear transmission behavior) is shown in Fig. 9. The chart traces the *TDFD* over the tensile stress that has been used to damage the corresponding sample for five different cases. To support the interpretation, the quadratic regression of these points is also determined and plotted. It is clearly visible that the intermodulation increases with increasing damage, from *TDFD* = 0.3176 in the case of the undamaged sample to *TDFD* = 0.7014 in the case of the maximal damaged sample.

Results for Woven Glass Epoxy Composite

The method is also applied to the woven glass epoxy composite samples using excitation frequencies of 11 kHz and 14 kHz. The resulting chart, shown in Fig. 10, includes a cubic regression to support the estimation of the general behavior. The chart traces the *TDFD* over the tensile stress used to damage the corresponding sample for 11 different cases.

After the increased of the damage parameter from *TDFD* = 0.3557 to *TDFD* = 0.7910 (for the samples loaded at 150 MPa), the parameter is subjected to oscillations; and even decreases slightly with increasing damage when considering the cubic regression. The parameter's value of the maximum damaged sample is finally positioned at TDFD =0.5444.

This oscillation and reduction could be explained by large cracks or open delaminations in the composite material. In fact, delaminations that are completely open are sources of nonlinearities in their boundary zones; this is due to the introduction of a contact surface as explained by [16]. The oscillating behavior is consequently justifiable as follows: First, small defects are created that increase the nonlinearity because of the creation of new contact areas. However, when cracks and delaminations begin to open and to coalesce, the level of nonlinearity decreases partially because of contact zone reduction. Second, new small cracks are produced that increase the nonlinearity again until they also become too open to produce these nonlinearities. This phenomenon has been mentioned in [16] and [17] where a distortion factor has been recorded during a high cycle fatigue test of carbon fiber reinforced plastic.

Employing a slightly different spectrum evaluation leads to fewer oscillations and a clearer increase of the parameter with increasing damage. Only in the case of highly damaged samples (from 250 MPa to almost the failure strength), peaks occurred far away from the excitation frequencies in the woven glass epoxy samples. These peaks appear in the range of 4 kHz to 5 kHz and are at a distance of 2 kHz from the other main peaks located between 0 kHz and 2 kHz. They are marked in Fig. 11 and appear to be of critical interest to characterize increased damage and therefore can be taken into account by attributing a higher weight in the numerical analysis. This is achieved as follows: their amplitude is cubed, unlike the other peaks which are only squared, and those values are added to the sum of all peaks' amplitudes. Hence the formula changes to:

$$TDFD_{alt} = \frac{\sqrt{\sum_i A^2_{i.\mathrm{intermodulation}<4\mathrm{kHz}} + \sum_k A^3_{k.\mathrm{intermodulation}>4\mathrm{kHz}}}}{A_{\mathrm{excitation}\ 1} + A_{\mathrm{excitation}\ 2}} \tag{2}$$

The resulting mutual dependence of damage and intermodulation (as a consequence of the nonlinear transmission behavior) is shown in Fig. 12. The chart traces the "Alternative Total Difference Frequency Distortion" ($TDFD_{alt}$) over the tensile stress that has been used to damage the corresponding sample for 11 different cases. To support the interpretation, the quadratic regression of these points is also plotted. Note that the intermodulation is generally increasing with increasing

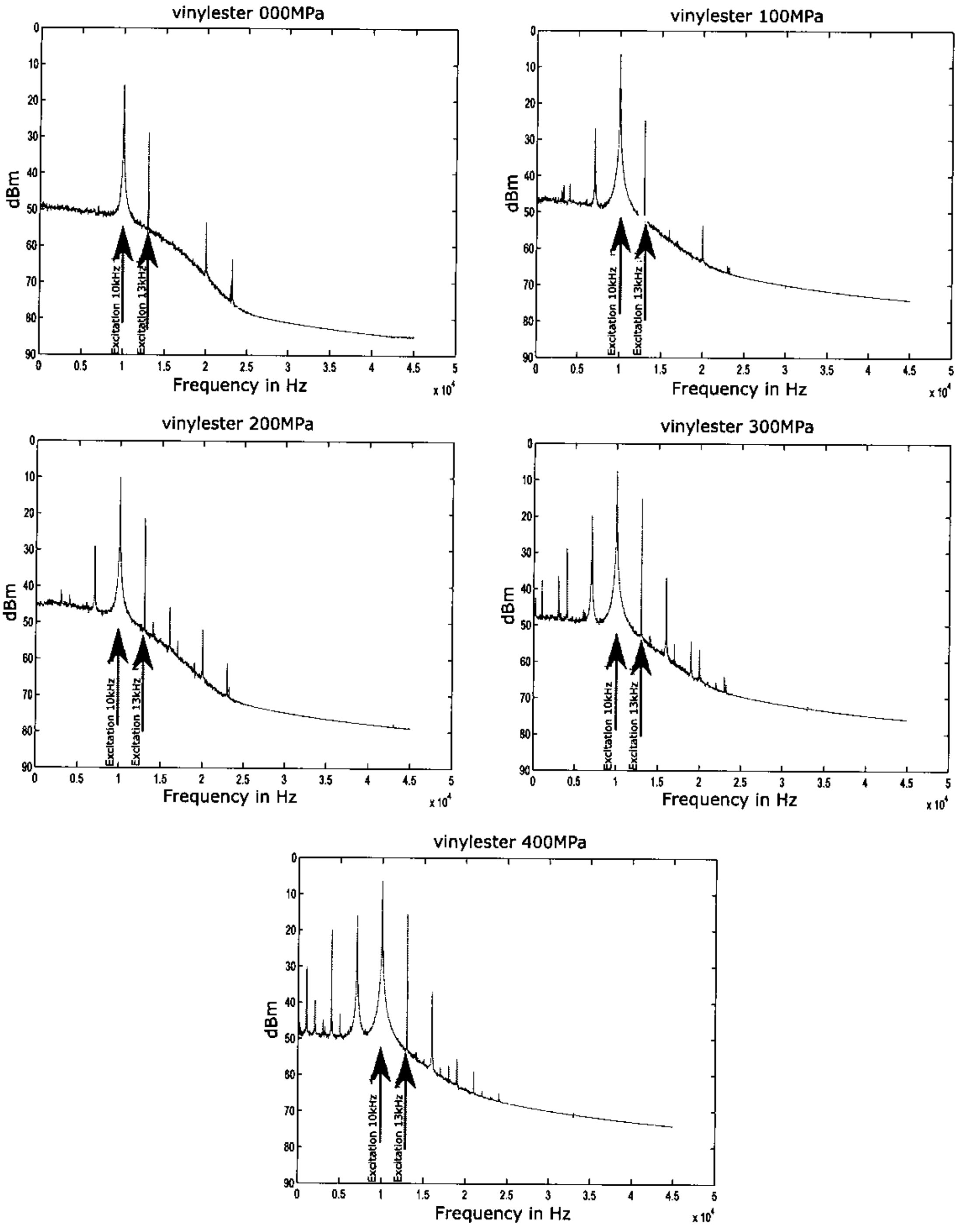


Fig. 8 Evolution of the intermodulation products with increasing damage for the woven glass vinylester composite samples excited by frequencies of 10 kHz and 13 kHz. The damage state is indicated by the corresponding applied tensile stress

damage (from $TDFD_{alt}$ = 0.3578 in the case of the undamaged to $TDFD_{alt}$ = 1.1820 with the maximum damaged sample). Considering the measured points, an oscillatory evolution of the damage parameter is still observable between some of the tested composite samples (see also Fig. 12). The reduction of the nonlinear transmission behavior is obvious between for the 150–200 MPa and 350–400 MPa tensile stresses that were imposed to damage the concerned samples. The average reduction of the parameter is $\Delta TDFD_{alt} = 0.13 \pm 0.03$.

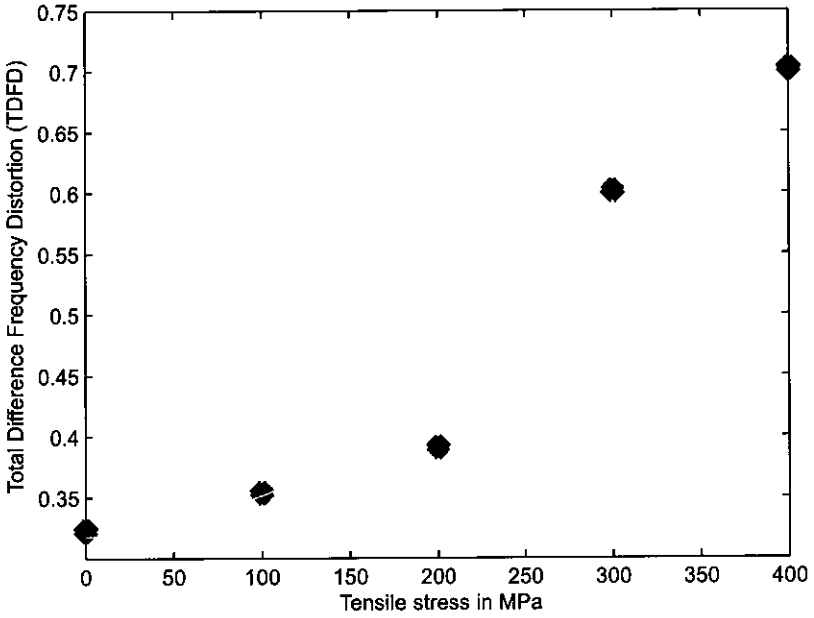


Fig. 9 Measure of the nonlinear transmission behavior of the woven glass vinylester composite samples under excitation of 10 kHz and 13 kHz

Concluding Remarks

The presented work is based on the current, yet limited literature of nondestructive examination of composites using nonlinear acoustic methods. The dependence of the global nonlinear transmission behavior, for two different materials, on the damage evolution was investigated to verify and advance these methods. A new experimental setup as well as a new evaluation routine has been developed. The introduction of intermodulation measure, from the field of sound system, has then been made possible. It has been shown that a strong dependency exists between the nonlinear transmission behavior of the samples and the damage state introduced by prior interrupted tensile tests performed at defined stress levels. Consequently, the method turns out to be an appropriate instrument to nondestructively evaluate the damage state of composite samples, at least for the geometries and materials used in the presented work under laboratory conditions. The method exhibits a strict increase of nonlinearity with an increase in damage for samples made of woven glass vinylester. In the case of woven glass epoxy composite material, an oscillation has been observed. This oscillation is of sufficient

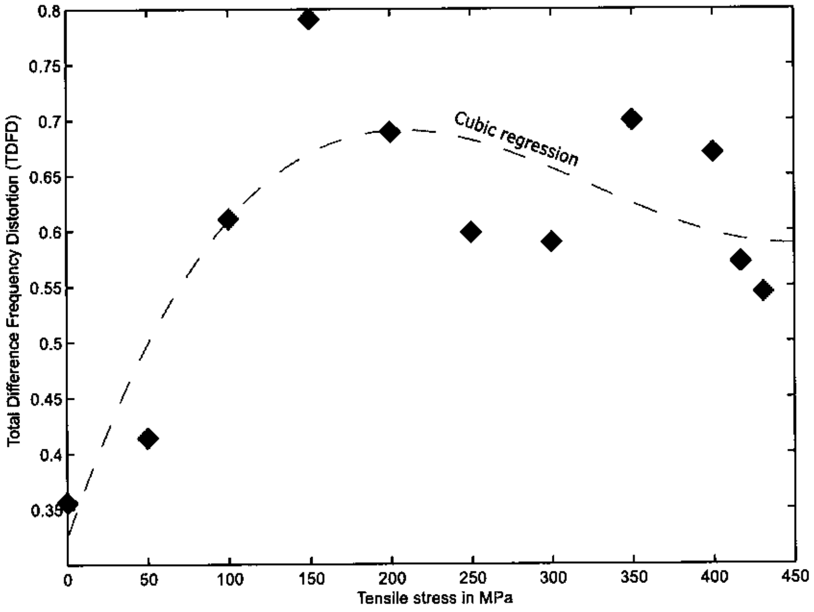


Fig. 10 Standard measure of the nonlinear transmission behavior of the woven glass epoxy composite samples under an 11 kHz and 14 kHz excitation and the cubic regression plot (*dashed line*)

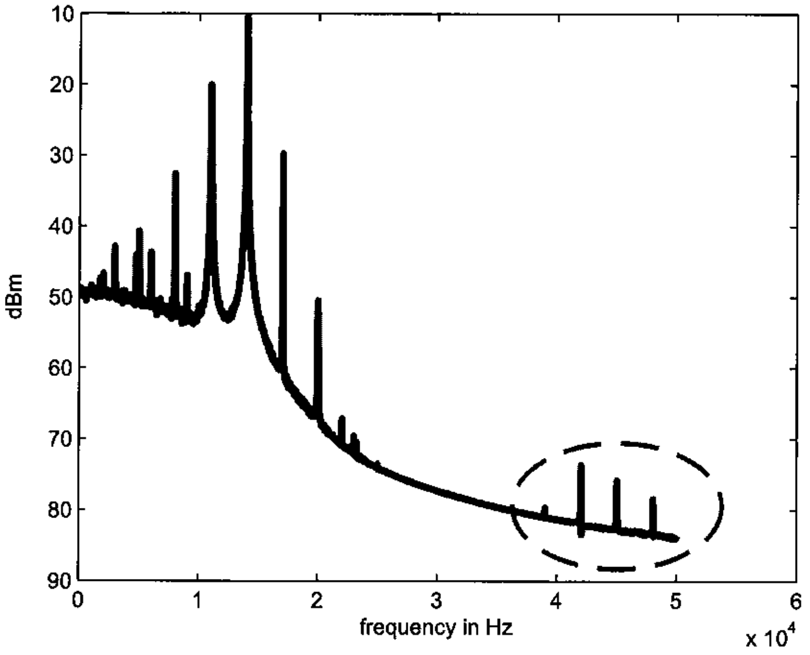


Fig. 11 Spectrum of the woven glass epoxy composite sample damaged by a tensile stress of 250 MPa and excited by frequencies of 11 kHz and 14 kHz

interest to justify further research to verify the extent to which this research explains the observed phenomenon, namely being caused by a varying amount of contact surface in relation to the damage state level. Finally, it has been stated that the Nonlinear Wave Modulation Spectroscopy and the *TDFD* provide an effective damage investigation. In addition, it is worth noticing that tensile tests along the fiber's axis were considered in the present paper. Yet, as Pandita et al. [18], among others, show that this specific loading configuration leads to low damage levels in woven composite materials, compared to off axis tensile solicitations. Therefore, the present nonlinear acoustic method is sensitive enough to detect low damage levels.

The method's current state is not yet industrially applicable, but it could be improved for the testing of industrial structures in the future. Perhaps it will be possible to excite whole automotive structures by piezo elements or contactless by high-power loudspeakers. These structures could hang in free air to reduce environmental disturbance. The measurement of defect regions and even their location could afterwards perhaps be

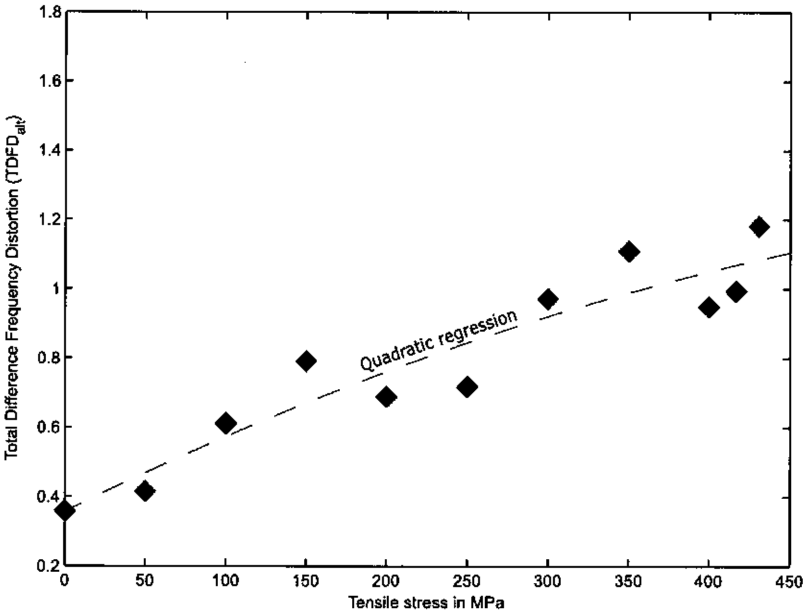


Fig. 12 Modified measure of the nonlinear transmission behavior of the woven glass epoxy composite samples under excitation of 11 kHz and 14 kHz and the quadratic regression plot (*dashed line*)

investigated by a 3D laser vibrometer: Knowledge of the spectral response on each surface point could be used to achieve this aim.

In future projects the research method explained here will be used in combination with imaging techniques for nondestructive evaluation (NDE) such as ultrasonic C-scan and electromagnetic Terahertz. Also the research shows that this specific technique, used in the audio field, appears to effectively detect damage in materials; more signal processing techniques from this field may be evaluated in the future, such as for instance a combination of wavelet transformed *TDFD*. Now, that the present nonlinear acoustic method is proven to be effective, some additional tests are necessary in order for the method to be fully reliable. Various mechanical solicitations must be tested on multiple composite materials, in addition to the tensile tests along the axis of the fiber considered here, to assess the efficiency of this NDE method. Moreover, it would be interesting to survey if any dependency between the samples' geometry and the acoustic response can be demonstrated. The outcome would be helpful for optimizing a design of the sample geometry suitable for the developed investigation method.

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