Contributions to real-time monitoring of polymer composites processing using mechanical impedance analysis

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Abstract: This article proposes a sensor concept based on mechanical impedance analysis, allowing real-time measurement of the viscoelastic properties of thermosetting polymer resins and composites with such a matrix, and determining the composite material processing degree during treatment.

Key-Words: polymer composites, mechanical impedance, sensor, real time monitoring.

1. Introduction
1.1. The mechanical sensor

For a dynamic system, ratios between quantities characterizing the excitation and system’s response are used to describe its vibratory properties. Since these reports cover a harmonic vibration regime, they are expressed by complex quantities, each ratio is actually defined by two quantities: an amplitude ratio and phase difference.[4],[5],[7].

To get good results in interpreting the relation between the output signal and the properties of the studied material we have used the principle of mechanical impedance, such as impedance expressed as a ratio of mechanical forces applied to the system and its speed.

\[ Z = \frac{F}{v} = \frac{F}{x} \] (1)

The system studied in this paper was equated with a linear dynamic system (fig.1.).

**Fig. 1. Validation of the studied system to a linear dynamic system.**

To simplify calculations, for all the experiments, the excitation force exerted on the structure and resultant movements (acceleration) were measured on the same surface. Also, for all analysis the following approximations were made:

- the whole resin - fiber treating assembly can be represented as a linear dynamic system;
- the vibrating probe used to monitor the composite is moving between two layers of resin;
- the thickness of the layer on each side of the probe, between it and the reinforcement fibers was considered to be constant;[1],[2].
- the mass of resin surrounding the probe is very small and can be ignored.

Given these approximations, the motion equation of the system is:

\[ m\ddot{x} + c\dot{x} + kx = F_e e^{i\omega t} \] (2)

where: \( m \) is the of the system’s mass; \( c \) - viscous damping coefficient; \( k \) - spring constant; \( x \) – displacement; \( F_e e^{i\omega t} \) – system’s harmonic excitation force.

**Fig. 2. The system’s equated electric circuit.**

The fundamental equation of the equated electrical circuit (Fig. 2.) composed of inductance \( L \), resistance \( R \), and the capacity \( C \), connected in series is:

\[ L\ddot{q} + R\dot{q} + \frac{1}{C}q = u(t) \] (3)

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It has been established the following correspondence between the parameters involved in the two equations:
- the displacement \( x \) is analogous to the electric charge \( q \);
- the speed \( \dot{x} \) is analogous to the current intensity \( I \);
- the exciter force \( F_0 e^{j\omega t} \), is analogous to the terminal voltage \( u(t) \);
- the mass \( m \) is analogous to \( L \);
- the damping coefficient \( c \) is analogous to \( R \);
- the spring constant \( k \) is analogous to \( 1/C \).

The mechanical impedance \( Z \) of this system is:
\[
Z = i\left( m\omega - \frac{k}{m} \right) + c \quad (4)
\]
and the electric one:
\[
Z = i\left( L\omega - \frac{1}{C\omega} \right) + R \quad (5)
\]

For the system in Figure 2 spring and damper heads have the same speed. Also the sum of forces from the three elements equals the exciter force, namely:
\[
F = \dot{x}(Z_k + Z_m + Z_c) \quad (6)
\]
The total impedance at the force application point is:
\[
Z_p = \frac{F}{\dot{x}} = Z_k + Z_m + Z_c = -\frac{i k}{\omega} + i\omega m + c = c + i\left(\omega m - \frac{k}{\omega}\right) \quad (7)
\]

2. Modeling the frequency response function

For determining the dynamic characteristics of the studied composite material the modeling of the transfer function was necessary, which gives the relationship between output signal resulting from the movement of the vibrating probe in the composite material under the action of an exciting force produced by the electrodynamic exciter and material properties. The purpose is to define the dependence „response – excitement”, under the action of the known disturbing source. In this case, the request was made with an electrodynamic vibrator and the response was measured by the vibration sensors.[1],[2].

The real structure, which has an infinite number of degrees of freedom, has been replaced by the mathematical model of the linear dynamic system which has a finite number of degrees of freedom, with constant parameters. For each resonance the frequency was determined, the shape of the vibration mode and corresponding damping factor (which cannot be evaluated analytically by any method), generalized mass and generalized spring constant. [3],[4],[9].

The sistem’s response depends on the characteristics of excitation as well as on its dynamic properties. The studied system was considered to be a linear system, where the input, \( x(t) \) is the system’s excitation and the output, \( y(t) \) is the answer. The system’s frequency response function \( H(\omega) \), is:
\[
H(\omega) = \frac{Y(\omega)}{X(\omega)} \quad (8)
\]
where: \( X(\omega) \) and \( Y(\omega) \) are Fourier transforms of the input and output quantities calculated with:
\[
X(\omega) = \int_{-\infty}^{+\infty} x(t)e^{-j\omega t} dt \quad (9)
\]
\[
Y(\omega) = \int_{-\infty}^{+\infty} y(t)e^{-j\omega t} dt \quad (10)
\]

By doing the following replacements in the system’s equation of motion (2):
\[
p^2 = \frac{k}{m}, \quad c = \zeta c, \quad c_c = 2mp \quad (11)
\]
it becomes:
\[
\ddot{x} + 2p\zeta\dot{x} + p^2 x = \frac{F_0}{m} \sin \omega t \quad (12)
\]
The equation’s result (2) is a harmonic form displacement:
\[
x(t) = X_0 \sin(\omega t - \theta) \quad (13)
\]
an amplitude displacement:
\[
X_0 = x, A_i = \frac{F_0}{k} A_i \quad (14)
\]
where \( \eta = \omega / p \), resulting the amplification factor \( A_1 \) and that the phase angle \( \theta \), such as:
\[
A_i = \frac{1}{\sqrt{(1-\eta^2)^2 + (2\zeta\eta)^2}} \quad (15)
\]
\[
\theta = \tan^{-1} \frac{2\zeta\eta}{1-\eta^2} \quad (16)
\]

In this case, the frequency response function \( H(i\omega) \), becomes:
\[
H(i\omega) = \frac{A_i}{p^2} \quad (17)
\]

Considering the structural attenuation system, the complex elasticity modulus is:
\[
E^* = E' + iE'', \quad G^* = G' + iG'' \quad (18)
\]
where: \( E' \) and \( G' \) are the longitudinal and transversal
dynamic elasticity modulus (or storage module), \( E' \) and \( E'' \) longitudinal and transversal loss elasticity modulus.[4],[5],[6].

In this case, the hysteresis attenuation factor \( g \) is:

\[
g = \tan \delta = \frac{E''}{E'} = \frac{G''}{G'}\quad (19)
\]

The values of \( G', G'' \) and \( \tan \delta \) dependent on the molecular structure, being strongly influenced by the deformation rate, the deformation, temperature and temperature changing speed.

Taking the vibrating probe sizes: \( S \) - thickness (0.25 ... 0.3 mm), \( L \) - length in contact with the resin 60 mm and \( l \) - width of 12 mm and area \( A = l \times L \), velocity \( \nu (\omega) \), or acceleration \( a (\omega) \) is measured along the probe. In this case, the output signals are the probe’s displacement amplitude, which vibrates in the composite material and the phase angle between the excitation force and the measured acceleration.

By definition, the system’s relaxing \( Q (s) \) is the ratio of voltage Laplace transform and distortion Laplace transform, namel:

\[
Q(\omega) = \frac{\sigma(\omega)}{\varepsilon(\omega)}\quad (20)
\]

**Fig. 3. Cross section area where the sensor is located: 1, 5-fiber reinforcement, 2, 4-layer of resin, 3 - sensor (vibrating probe).**

Considering that the probe is placed between two layers of resin (Fig. 3), the form factor \( b \) expresses the impedance based on relaxation with the equation:

\[
b = \frac{F}{\nu} = \frac{\tau}{\dot{\gamma}}\quad (21)
\]

where: \( F \) is the excitation force, applied to the vibrating probe, \( \nu \) – probe’s velocity, \( \tau \) - shear tension; \( \dot{\gamma} \) - shear strain rate.

Since:

\[
\frac{\tau}{\dot{\gamma}} = \frac{F/A}{(\nu/h_1 + \nu/h_2)} = \frac{F}{\nu} \left[ h_1 \cdot h_2 \right] \quad (22)
\]

then:

\[
b = \frac{\tau}{\dot{\gamma}} = \frac{F}{\nu} = \frac{\nu}{F} \left[ \frac{h_1 \cdot h_2}{A(h_1 + h_2)} \right] = \frac{h_1 h_2}{A(h_1 + h_2)} \quad (23)
\]

where: \( A \) is the probe’s surface area in contact with the material (resin), \( h_1 \) and \( h_2 \) the thickness of the resin layer between the probe and the reinforcement fiber. If \( h_1 = h_2 = h \), then:

\[
b = \frac{h}{2A}\quad (24)
\]

The relationship between relaxanťa and impedance is:

\[
Q(\omega) = i\omega \frac{h}{2A} Z(\omega)\quad (25)
\]

For the geometry in Figure 7.5., where the resin is sheared between the probe and the layers of reinforcement fiber, relaxing is the complex shear modulus \( G'(\omega) \), such as:

\[
G' = G' + iG''\quad (26)
\]

where \( G' \) is storage modulus; \( G'' \) - loss modulus.

Therefore, the studied system can be simplified to the diagram in Figure 4.

**Fig. 4. Simplified model of the studied system: C - resin damping coefficient; S - shear force, \( F_0 e^{j\omega t} \) exciter force.**

For this model, the motion equation (1) becomes:

\[
M \ddot{x} + C \dot{x} + Kx = F_0 e^{i\omega t}\quad (27)
\]

where: \( M \) is the system’s mass which includes the vibrating probe as well as it’s mounting system to the vibrator, including the mass of the vibrator (\( M = 16.8 \) kg), \( c \) - viscous damping coefficient of the resin, \( K \) - spring constant.

Noting: \( Kx = 2S \), and \( 2S = \frac{2AG}{h} \), \( x = G' x \quad (28)\)

Applying the Fourier transform to the equation (29), according to equation (17), the frequency response function \( H(\omega) \) is given by:

\[
H(\omega) = \frac{\tilde{X}(\omega)}{\tilde{F}(\omega)} = \frac{\omega^2}{\sqrt{(G' - M\omega^2)^2 + (C\omega)^2}}\quad (30)
\]

where: \( F(\omega) \) is the input, that is exciter force measured with a piezoelectric force transducer;
$\ddot{X}(\omega)$ - the excitation system response, that is the system acceleration measured with the piezoelectric accelerometer.

The resonant frequency of the system is:

$$G' - M \omega^2 = 0; \quad H(\omega) = \frac{\omega}{C} \quad (31)$$

Therefore, using equations (30) and (31) it can be determined, after the experiments, the complex shear modulus and viscous damping coefficient of the resin.

3. Experimental results

Figure 5 presents the measuring system used to monitor real-time processing of the studied composite material, the dynamic mechanical analysis and the thermal analysis.

The system performs the thermal analysis of the composite material in real time, providing information on the evolution of the composite temperature during processing and spectral analysis (frequency analysis of the studied composite system), which provides information on the composite processing stage and the evolution in time of its dynamic mechanical properties.

![Diagram of the experimental stand](image)

**Fig. 5.** Experimental stand used to monitor real-time processing of polymer matrix composites: 1 - force transducer, 2 - accelerometer, 3 - vibrating probe, 4 - mould; 5 - Composite material; 6 - Oven, 7 - temperature sensor LM 335Z, 8 - thermocouple type K

The matrix of the studied composite material is composed of an epoxy resin type DGEBA - Epilox T19-36 and the hardener in liquid stage, H 10-30. The reinforcement material used was an E fiberglass roving fabric with continuous filaments - EWR-W-300,[3],[4],[5],[7].

Experiments were carried out both on the resin-hardener system and samples of the composite material made from a total of 20 layers of fiberglass with [0/90]2o topology at various treatment temperatures: at room temperature and at 40°C. It was intended to monitor the developed temperature during the treatment of the composite at these temperatures, with the temperature sensor LM 335 Z. Therewith through the vibrating probe, which moving inside the composite under the action of electrodynamic excitation were highlighted the effects of treatment temperature over the phase transformations of the composite material, while changing the resonance frequency and viscous damping dynamical mechanical characteristics of the composite material.

For real-time monitoring of composite material processing a virtual instrument was created using the LabVIEW software that can achieve real-time dynamic mechanical analysis. The excitation signal as well as the sensor’s response signal acting on the studied composite material are filtered and processed by this LabVIEW instrument using a Hanning weighting function. Next the signal amplitudes and phase shift between them are measured. The frequency spectrum was recorded throughout the processing of the composite material at different temperatures using a data acquisition card AT-MIO-16E-2. All the experiments were carried out following the same procedure. The sensor was operated by a constant amplitude sinusoidal excitation signal of 5 V, generated by the virtual device. The gathered data was processed with MATLAB and Excel software, using relations (29) and (31).
An example of typical frequency response spectrum of the composite system treated at 40 °C is shown in Figure 6:

Fig. 6. The frequency spectrum of the composite system treated at 40 °C, acquired at T = 110 min.

It was noted that in the beginning of the experiment (t = 0) when the temperature inside the composite material was the same as the ambient temperature, the peak resonant frequency (transfer function $H(\omega)$), occurs at $f = 9.8$ Hz. In the treatment process of polymer matrix composites, the dynamic mechanical properties are controlled by two factors: temperature T and the degree of curing $\alpha$. As the treatment process progresses in time, the resin curing process also progresses, thereby the three-dimensional molecular links are realised leading to increased viscosity and hence the rigidity of composite material. Under these conditions the tip frequency response function $H(\omega)$ changes its position towards higher frequency values, $f = 14.8$ Hz, at the point $t = 110$ min and the composite temperature $T = 40.2$ °C.

Next there will be presented the graphs for the excitation frequency variation, the plane shear modulus G and viscous damping coefficient C, for the resin-hardener studied system T19-36 / H 10-30 and for the composite material.[4],[5],[6].

For the resin-hardener system, the frequency resonance variation graph $f$ expressed in Hz based on time, determined by the vibrating probe is shown in Figure 7. Over this graph was overlaid the temperature developed during the polymerization reaction system, to better illustrate the movement in time of the transfer function frequency peak $H(\omega)$ over the curing system.

Fig. 7. Variation of peak frequency and temperature of the resin-hardener system in time

Figures 8. and 9. present the graphs where the variation in time of the shear G and the viscous damping coefficient C are shown, calculated with the relations (29) and (31) and figure 10 has the variation in time of the damping coefficient of the composite material treated at ambient temperature.
Fig. 10. Changes over time for the damping coefficient of the composite material treated at room temperature.

Fig. 11. Changes in time for the parameters G and C of the composite treated at 40 °C.

Viscous damping coefficient C shows two peaks: first peak corresponds to the softening point of the resin-hardener system, when the exothermic maxim of the polymerization reaction occurs, and the second peak represents the thickeners point. This condition is accompanied by an increase in shear modulus until the system curing is complete.

Experiments on the composite material were carried out in the same manner as on the resin-hardener system. From figure 10 it is noted that the presence of reinforcing fibers can decrease the thickeners time, towards the thickeners time of the resin-hardener system,[2],[4],[5],[7].

The peak frequency resonance variation graph as well as the G and C composite parameters treated at 40 °C are shown in figure 11.

Analyzing the above graphs reveals that with the increaseof the treatment temperature of the composite material, the second peak of the damping coefficient moves towards the origin, meaning an acceleration in the thickening process of the composite material. You can also see that the treatment temperature does not affect in a very large manner the final value of the shear modulus, but the point when the maximum exothermic polymerization reaction is produced is highlighted by decreasing its value.

4. Conclusions

The experimental results show that this type of sensor (vibrating probe) based on mechanical impedance analysis may be used to detect viscoelastic transitions produced in the treatment of composite materials with thermosetting resin matrix, being able to detect the changes in apparent shear modulus and damping coefficient, accompanied by the gelation and vitrification of resin. It can be said that the mechanical impedance analysis method can be successfully applied to monitor the curing process of polymer matrix composites. The method supported by all devices on the basis of which has been developed can be used to characterize real-time status of any composite curing.

References: