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A MICROMECHANICAL BASED APPROACH FOR DYNAMICAL PROPERTIES EVALUATION IN CASE OF POLYMERIC COMPOSITE MATERIALS

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Abstract: *The paper aims to present a theoretical vs. experimental approach of the dynamic elastic moduli for multiphase polymeric based composite materials. The theoretical approach will be developed around several theoretical models from literature using a homogenization concept and the experimental results will be retrieved from DMA measurements on Al_2O_3 particles embedded along with random E-glass long fibres into a polymeric matrix, in different volume fraction. A sensitivity analysis was carried to accompany the investigations in order to provide quantitative measures to the influencing factors on the elastic property.*
Key words: micromechanics, dynamic, properties, composites, polymeric.

1. INTRODUCTION

Engineered materials emerged as a natural consequence in this explosion of new materials world, followed by the new or revised theoretical models in order to predict and tailor their material properties. The results of these steps have to be crosschecked by values retrieved using experimental techniques for each property.
Understanding polymeric composites behavior under a dynamic regime is not an easy task to accomplish or predict. Technical literature lack of intensive studies on this area mainly due to the high acquisition cost of the research equipment or restrictive accessibility to a data base on polymers properties. Nevertheless, there are several authors that were approaching this type of experimental investigation on the class of composite materials, in their classic formulae – one constitutive embedded into a matrix, in pursue of retrieving the dynamic elastic modulus, the most comprehensive material property [1-4]. The herein paper attempts to approach a new class of composite materials, namely multiphase polymeric based composites, from a theoretical perspective and DMA based experimental measurements in order to complete the material data base containing their mechanical and thermal properties [5-6].

2. THEORETICAL MODELS

A two step homogenization process is being employed in order to retrieve the dynamic elastic moduli of the multiphase polymeric based composite materials under discussion. According to the authors' studies and experience the elastic moduli in case of multiphase materials, no matter their static or dynamic values or reinforcement type (e.g. particle/fiber), provide different values if the approaching methodology employed differs, such is the multi-step homogenization or the overall approach [7-8].

2.1 1st homogenization step

The first homogenization is being applied to the particles and polymeric matrix, leading to the so called *equivalent matrix*, based on small volume fractions in which was embedded this complementary phase. Due to the dilute nature of the spherical particles, having an isotropic and non-dissipative behavior, the most suitable theoretical model to predict the elastic moduli is the Mori-Tanaka one:

$$\frac{K^*_{me}}{K^*_m} = 1 + \frac{V_p\left(\frac{K^*_p}{K^*_m} - 1\right)}{V_p + \xi(1 - V_p)} \quad (1)$$

$$\frac{G^*_{me}}{G^*_m} = 1 + \frac{V_p\left(\frac{G^*_p}{G^*_m} - 1\right)}{V_p + \varsigma(1 - V_p)} \quad (2)$$

where:

$$\xi = 1 + \frac{3(K^*_p - K^*_m)}{3K^*_m + 4G^*_m} \quad (3)$$

$$\varsigma = 1 + \frac{6(G^*_p - G^*_m)(K^*_m + 2G^*_m)}{3K^*_m + 4G^*_m} \quad (4)$$

are individual phases material based parameters. In the previous expressions K^*_{me} and G^*_{me} are the dynamic equivalent matrix bulk modulus and shear modulus, respectively, K^*_m and G^*_m are the dynamic bulk modulus and shear modulus of the matrix material, whereas K^*_p and G^*_p are the dynamic bulk modulus and shear modulus of the particles, all expressed in GPa, V_p accounts for the particle volume fraction.

The dynamic Young modulus of the equivalent matrix can be predicted using the well known relationship:

$$E^*_{me} = \frac{9K^*_{me}G^*_{me}}{G^*_{me} + 3K^*_{me}} \quad (5)$$

2.2 2nd homogenization step

The second homogenization was applied to the random fibers reinforced equivalent matrix. Two different micromechanical theoretical models, developed by Paul and Ishay-Cohen, were used to predict the dynamic Young elastic modulus of the overall composite material.

According to the Paul's model the expression can be written as:

$$E^*_c = E^*_{me}\left[\frac{1 + \left(\frac{E^*_f}{E^*_{me}} - 1\right)\sqrt[3]{V_f^2}}{1 + \left(\frac{E^*_f}{E^*_{me}} - 1\right)\left(\sqrt[3]{V_f^2} - V_f\right)}\right] \quad (5)$$

where the real and imaginary components can identified by doing some mathematical computation.

The second theoretical model used in the approach was the Ishay-Cohen, in the form:

$$E^*_c = E^*_{me}\left[1 + \frac{V_f}{\frac{E^*_f}{E^*_f - E^*_{me}} - \sqrt[3]{V_f}}\right] \quad (6)$$

the real and imaginary components resulting after several mathematical manipulation.

In the previous expressions E^*_c stands for the dynamic effective elastic modulus of the composite material, E^*_{me} is being the dynamic Young modulus of the equivalent matrix and E^*_f of the fibers, all expressed in GPa, whereas V_f accounts for the fibers volume fraction.

Generally, in all previous expressions:

$$P^* = P' + iP'' \quad (7)$$

stands for any material property (Young modulus - E, shear modulus - G or bulk modulus - K), being the complex form containing a real part, P', known as the storage component and an imaginary one, P'', known as the dissipative component, respectively.

The E-glass fibers embedded into the equivalent matrix, as well as the particles, were considered as having a non-dissipative behavior, being the main reason in neglecting the imaginary component. The polymer matrix is the only composite phase having a dissipative behavior.

3. EXPERIMENTAL RESEARCH

3.1 Materials

Samples were manufactured to form a multiphase structure made up from two different constitutive – fibers and particles - embedded in different volume fraction into a polymeric material. The composite matrix was chosen as being a polyester resin, Synolite 8388 P2, made by DSM Composite Resins (Switzerland), due to its good interfacing properties and the particles were Al_2O_3. The particles were embedded into the polyester resin different volume fraction (5% and 10%).

The 3rd phase chosen were E-glass type random fibers, Mat ES 33-0-25, made by Johns Manville, SUA, being characterized as having a 65% volume fraction. The additives used were chosen as being chemical compounds showing compatibility with the other phases and allowing polymerization process initiation and development.

A reference sample made up only from fibers, with the same volume fraction as the other ones, was manufactured to aid the experimental data comparison and the tailoring process with respect to the effective elastic properties of structures.

3.2 Experimental investigation

Samples of reference and multiphase composite materials (dimensions: 50x10x5 mm) were subjected to 3 point bending under a temperature imposed program, in air, using a DMA 242 C device, from NETZSCH, Germany.

The effective dynamic moduli of each class of composite materials and their temperature dependence were provided directly by the testing machine software called Proteus. The experimental investigations were done using the following settings: temperature program - 30^0 C to $160^0/200^0$ C, heating rate 2 K/min, frequency 1 Hz, and maximum dynamic force of 6.1 N. Oxidation processes are scarcely due to environment control inside the furnace.

3.3 Sensitivity analysis

The sensitivity analysis was carried out using the @RISK 5.5 from Palisade, U.S.A. As input parameters were chosen the volume fraction and the elastic properties of each constitutive, the latter considered as having each a normal distribution, the output being in this case the effective dynamic elastic moduli of the multiphase polymeric composite, in its modulus value. Using only 5 simulations and 5000 iterations will be enough to underline the major influencing constitutive elastic properties on the overall dynamic modulus of the multiphase composite as it can be seen from the resulting Tornado graphs.

4. RESULTS AND DISCUSSIONS

Figure 1 shows a digital microscope picture (magnification x500) of one of the multiphase composite samples containing 5% Al2O3 particles and 65% E-glass random fibers embedded in the polymeric resin. As it can be seen due to the dilute limits of the particles these are highly dispersed into the overall composite mass.

In Fig. 2 is being shown the experimental curves retrieved by imposing 2 successive heating cycles, the variations being associated to the sample elastic modulus behavior with temperature.

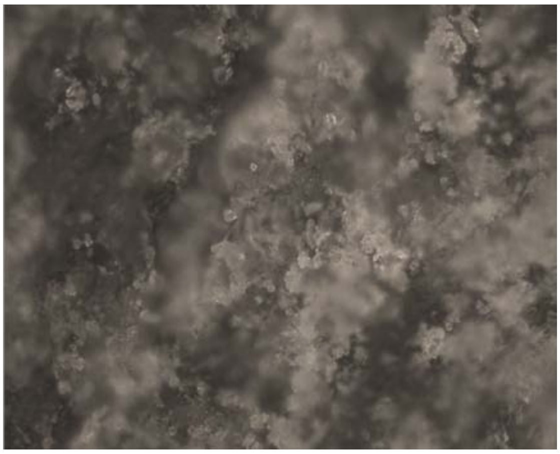

Fig. 1. Microscopical view of a multiphase composite sample (x500) – 5% particles, 65% fibres and polymeric matrix

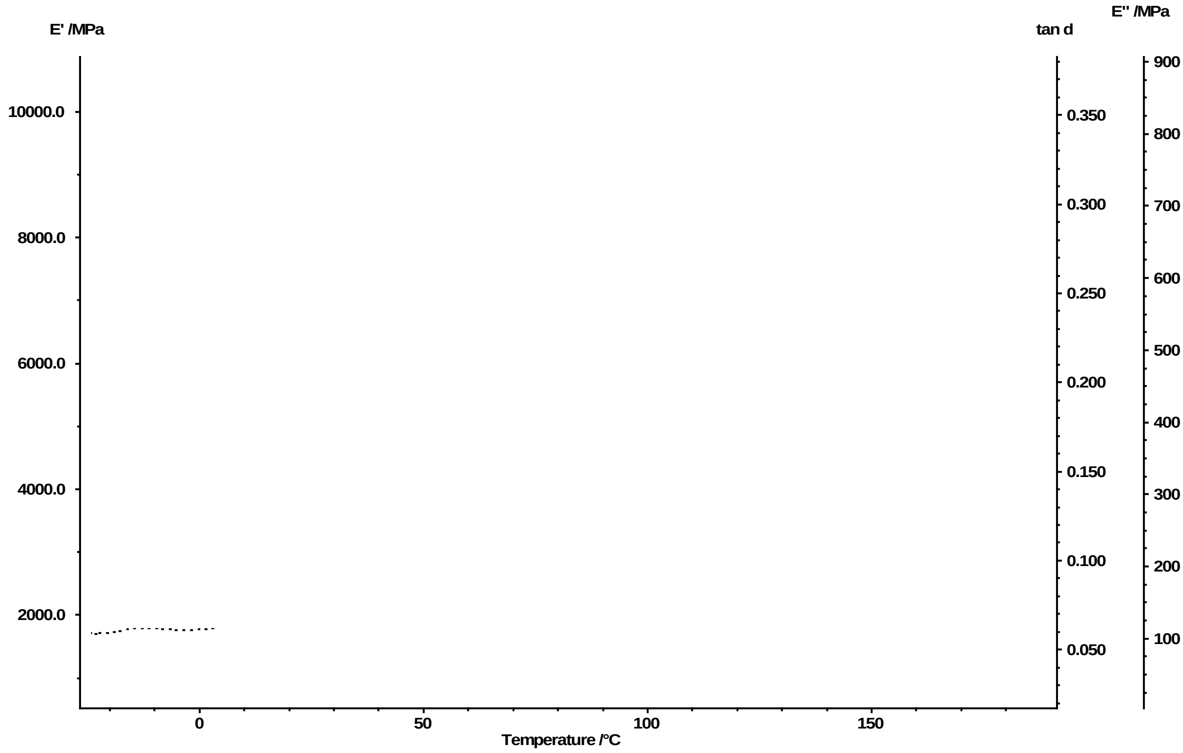

Fig. 2. Storage and loss components temperature variation as well as the loss factor for the 5% particles and 65% fibers multiphase composite sample

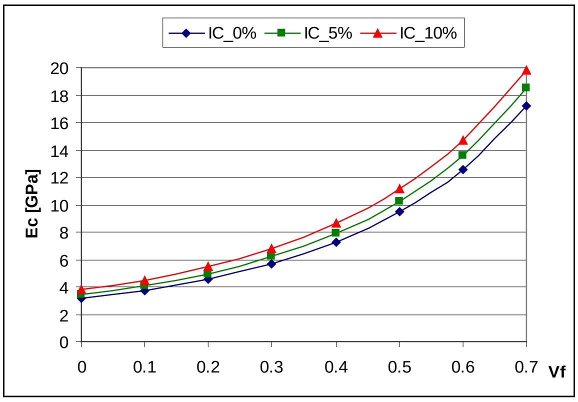

Fig. 3 Composite elastic modulus variation according to the Mori-Tanaka and Ishay-Cohen theoretical models

As it can be seen in the second heating step the composition is becoming more stable, the matrix material stiffer, the glass fibers are dominating the process and the retrieved dynamic modulus of the sample.

The material has at -20°C a storage modulus of 10.6 GPa. During the first heating, two transitions can be measured; an extrapolated onset at 58°C in the storage modulus curve. Corresponding, a peak in the loss modulus curve at 73°C and an onset at 72°C in the loss factor curve occurs. The second transition can be measured at 86°C (onset) in the E'-curve (real component), at 87°C (peak) in the E''-curve (imaginary component) and at 100°C in the tanδ-curve (loss factor). During the second heating the first transition disappeared.

In Fig. 3 were represented the theoretical values corresponding to the fibres volume fraction dependence of the elastic modulus in case of composite samples reinforced with 0%, 5% and 10% Al_2O_3 particle volume fractions along with the 65% E-glass random fibres predicted by using the 2 step homogenization concept based on Mori-Tanaka and Ishay-Cohen theoretical models.

According to the Mori-Tanaka and Paul theoretical models used as the second combination in the homogenization approach the variations resemble the ones plotted in the figure 3, having the same asymptotic behaviour but the values predicted for the effective elastic moduli of the composites samples being higher.

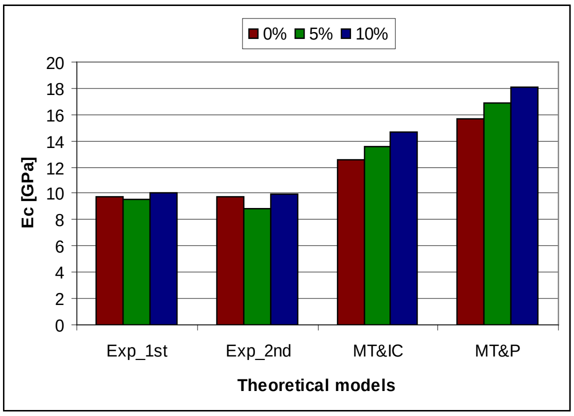

Fig. 4 Experimental – theoretical values for the multiphase composite samples

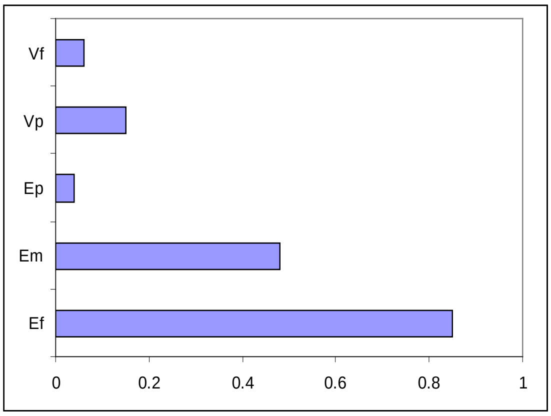

Fig. 5 Tornado graphs underlying the major influencing factors

Figure 4 corresponds to the comparative theoretical-experimental retrieved values by using the theoretical models from the previous chapter as well as the experimental values identified for each heating process.
As it can be seen the experimental values are less than the predicted ones and closely to the ones given by the Mori-Tanaka and Ishay-Cohen homogenization process.
In Fig. 5 is being plotted the values corresponding to a Tornado graphs after running a sensitivity analysis by using the @RISK 5.5 software from Palisade Co. As it can be seen the major influencing factor on the effective elastic composite sample considered (the same holds for all composite samples under study) are the fibers' elastic modulus followed by the matrix' elastic modulus. These founding are consistent with the theoretical predicted values and experimental retrieved ones.

5. CONCLUSIONS

Despite of time involved and energy consumed in the process of material characterization the results obtained offers a lot of professional satisfaction. The class of multiphase polymer based composites is increasingly developing due to the application potential in different fields, such as liquid tanks, electro-magnetically shielding panels, aggressive environmental standing structures or variable temperature standing components.
As was mentioned in the paper the dynamic property is not an easy task to accomplish and represent the most comprehensive material property. It covers information related to the: *molecular structure* – constitutive phases, relaxation mechanisms, free volume, etc.; *structure properties* – adhesion, temperature performance, long term behaviour, dimensional stability, etc. and *manufacturing conditions* – temperature, stress, strain, heat history, etc. All of these are influencing in some extent the dynamic mechanical property retrieved.

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