Theoretical - Experimental Comparisons of Multi-phase Composite Materials Elastic Coefficients Retrieved from Tensile, Compressive and Bending Tests. Influencing Factors

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The paper presents a theoretical-experimental approach of a particular class of composite materials known as multiphase composites in order to retrieve its effective elastic coefficients and identify the major influencing factors on these variations. Moreover, in the paper is being presented a particular homogenization concept based on the well known theoretical predictions from the micromechanics of composite materials approaches, the Mori-Tanaka that applies to the particle reinforced composites and Halpin-Tsai that applies to the random fibre composites. The theoretical values were used against the experimental data, the later being retrieved during a simple tensile, compression and three point bending tests at room temperature on samples made using a self-developed manufacturing technology. The samples were ceramics and metal reinforced particles of various volume fraction embedded along with random E-glass fibres into a polyester matrix.

Keywords: composite, multi-phase, elastic coefficient, micromechanics, testing

Technical literature reveals the recent trends with respect to the development and material characterization approach of more complex structures as is the case of multi-phase composite materials. The latter represents a new generation of materials characterized as having different physical and mechanical properties, sometimes higher than those of solely particle or fibre reinforced composite ones [1, 2, 3, 8], Nonetheless, the research done in the area of multi-phase composites is far from satisfactory or fully accomplished especially with respect to the behaviour of these structures under different loading conditions or the understanding of the connections among their microscopic/macroscopic properties (e.g. volume fraction of inclusions, fillers dispersion within the matrix etc.).

With respect to the evaluation of the effective elastic coefficients of these multi-phase composite materials, technical literature does not reveal theoretical models, excepting the simplified ones that will be presented within this paper, to be used and to account the complexity of the combination – particles and fibres embedded into a matrix. Consequently, the mechanics of materials generalized model can be used in case of multi-phase composite materials for prediction of its effective elastic coefficients only in case of unidirectional fibres, case that implies some restrictions [4, 11].

The Halpin-Tsai theoretical model is well known as a model that provides good agreement with the experimental data in case of long, random fibres for fibre reinforced composite materials, but as is described do not lead to the best solution in case of multi-phase combination. It is natural that, in such circumstances, a new methodology has to emerge to allow the characterization of this class of materials, a new approach like the one developed by the authors of this paper. The idea is a simple one and involves a two step homogenization process based on two well known theoretical models such as: Mori-Tanaka theoretical model – a generalized self-consistent scheme for particle's reinforced composite materials effective elastic properties prediction, independently on the particles' size distribution and the Halpin-Tsai model, respectively.

Supplementary, the theoretical predictions were compared with some experimental data retrieved during the tensile, compressive and 3-points bending tests carried out on samples manufactured as being multi-phase composite structures. The samples were manufactured on a self-developed technology bases, the phases consisting of ceramics and metal particles embedded along with long, random E-glass fibres into a polymeric matrix. The data, either from theoretical prediction or experimentally retrieved were not compared with similar data from technical literature due to the lack of the latter and restricted access in case of availability.

Theoretical models

Next, will be presented the expressions of three theoretical models which will be used in the present study to predict the effective tensile Young's modulus of the approached multi-phase composite configurations: the mechanics of materials generalized model, the classical Halpin-Tsai theoretical model and a two step homogenization technique based on Mori-Tanaka and Halpin-Tsai models used to develop an expression of the effective elastic coefficient [6]. Before proceeding, there is a remark that states the fact that within the paper will be not presented the mathematics that usually accomplish the theory. Moreover, the indices used have the following meaning: f – fibres, p – particles, m – matrix, me –equivalent matrix, c – composite, V_f – fibres volume fraction, V_p – particle volume fraction, E – Young modulus, G – shear modulus, K – compressibility modulus, ν – Poisson ratio, etc. Supplementary, each phase is considered as being isotropic and void content is considered as being negligible.

The mechanics of materials generalized theoretical model

Applied to the composite materials having two different types of inclusions embedded into a matrix that has the

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role of connecting phase, usually with inclusions' elastic modulus higher in comparison with that of the matrix, this theoretical model represents the case of 3D model developed and presented in the literature for the particular case of unidirectional fibres reinforced composite materials [4, 9, 10]. The expression of the longitudinal effective elastic coefficient of a multi-phase composite material can be written as follows:

$$\frac{E_c}{E_m}=1-V_f\cdot\left(1-\frac{1}{1-\left(1-\frac{E_m}{E_f}\right)}\right)-\sqrt[3]{\frac{V_p^2}{2}}\cdot\left(1-\frac{1}{1-\sqrt[3]{2\cdot V_p^2}\cdot\left(1-\frac{E_m}{E_p}\right)}\right). \quad (1)$$

The Halpin-Tsai theoretical model

It is well know that the elastic modulus in case of random fibres reinforced composite materials can be predicted by the aid of relation:

$$E_c=\frac{3}{8}\cdot E_1+\frac{5}{8}\cdot E_2, \quad (2)$$

where E_1 and E_2 represents the longitudinal and transversal, respectively, elastic moduli for unidirectional, discontinuous fibre reinforced composite materials, moduli that can be written function of fibres' diameter and length by the aid of Halpin and Tsai expressions:

$$E_1=E_m\cdot\frac{1+2\cdot\eta_L\cdot V_f\cdot\frac{l_f}{d_f}}{1-\eta_L\cdot V_f}; \quad (3)$$

$$E_2=E_m\cdot\frac{1+2\cdot\eta_T\cdot V_f}{1-\eta_T\cdot V_f}; \quad (4)$$

where η_L and η_T can be written as:

$$\eta_L=\frac{\frac{E_f}{E_m}-1}{\frac{E_f}{E_m}+2\cdot\frac{l_f}{d_f}}; \quad (5)$$

$$\eta_T=\frac{\frac{E_f}{E_m}-1}{\frac{E_f}{E_m}+2}. \quad (6)$$

In the previous expressions the l_f and d_f are the length and diameter respectively, of the fibres, E_f being the Young's elastic modulus of the fibres depending on the fibre' diameter as well.

The Mori-Tanaka and Halpin-Tsai combined theoretical models

In order to be able to predict, closer to reality, the effective elastic modulus of the multi-phase composite materials such are those in this study, a two step homogenization procedure was initiated due to the different theoretical methods that are used and well known from the literature.

Consequently, in the first step were subjected to the homogenization process the particles, in their dilute concentration, and the matrix leading to the so called homogeneous equivalent matrix. The effective elastic properties of the combinations were predicted using the Mori-Tanaka theoretical model that applies to the small volume fraction of particles embedded into a matrix material. In this case the compressibility and shear elastic moduli can be predicted from the following expressions:

$$K_{me}=K_m+\frac{V_p\cdot K_m\cdot\left(K_p-K_m\right)}{K_m+\xi\cdot\left(1-V_p\right)\cdot\left(K_p-K_m\right)}; \quad (7)$$

$$G_{me}=G_m+\frac{V_p\cdot G_m\cdot\left(G_p-G_m\right)}{K_m+\varsigma\cdot\left(1-V_p\right)\cdot\left(G_p-G_m\right)}. \quad (8)$$

where ζ and ς are expressed as:

$$\xi=\frac{1+\nu_m}{3\cdot\left(1-\nu_m\right)}; \quad (9)$$

$$\varsigma=\frac{2\cdot\left(4-5\cdot\nu_m\right)}{15\cdot\left(1-\nu_m\right)}. \quad (10)$$

In the 2-nd step of the homogenization process the phases were considered to be the discontinuous fibres, randomly distributed and the equivalent matrix from previous step, leading to a homogeneous composite medium for which its effective elastic properties have been retrieved applying the Halpin-Tsai theoretical model.

In this case, the expressions used are similar to those of (2) – (6) excepting of the values that has to be used for the matrix, meaning the fact that one has to consider the values of the equivalent matrix predicted by the aid of (7) – (10) expressions.

Experimental part

Materials

In table 1 are listed the material characteristics for each individual phase of the multi-phase composite structures considered within the present study. Consequently, the composite matrix was chosen as being a polyester resin, Synolite 8388 P2, made by DSM Composite Resins (Switzerland), chosen due to its good interfacing properties. The particle inclusions were considered: ceramic materials (with a high content of Al_2O_3), made from a natural stone, characterized as having a relatively high purity and provided by Alpha Calcite, Germany under the Alfrimal registered trade-mark and technical pure iron, respectively. Both particle types were mixed within the polyester resin mass in 5% and 10% respectively, volume fraction. The 3rd phase was E-glass type random fibres, known as MultiStratTM Mat ES 33-0-25, made by Johns Manville, USA, 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. During the experimental tests of the multi-phase composite samples, the individual materials comprising the phases were not evaluated/characterized due to the fact that the acquisition process of raw materials was accompanied by the technical data files containing their chemico-physical characteristics.

With respect to the samples manufacturing, it can be underlined the fact that was used a classical technique, namely the manual forming, in which 5 layers of fibre mat were used along with the particles, matrix and additives mix.

Table 1
MATERIAL CHARACTERISTICS

Materials/Characteristics	Matrix	E-glass mat	Particles	
			Fe	Al_2O_3
Transversal elastic modulus [GPa]	3.7	72.4	196	385
Poisson ratio	0.39	0.2	0.33	0.21
Length [mm]	-	50	-	-
Diameter [μm]	-	12.5	100	24

Samples' testing

Samples of bi- and multi-phase composite materials, of 250 . 25 . 5 mm dimensions particular to the 2nd type samples according to the SR EN ISO 527-4 standard, were subjected to tensile and compression loadings, at room temperature, using a Lloyd LS 100 Plus device, at 1 mm/s speed. Another 8 samples of each composite class were subjected to a 3-point bending tests. For the latter, the samples were having 150 . 10 . 5 mm according to the SR EN ISO 178 and tested using a Lloyd LR 5K device. The elastic coefficients of each class of composite materials, with and without particles, were provided directly by the testing machine software – called Nexygen. The values of the elastic coefficients provided herein were considered the mean values from the experimental data.

Results and discussion

In figure 1 are being outlined the shape and the manner in which the ceramic particles (5% volume fraction) and fibre reinforced composite samples reached their failure states after the tensile tests. As can be seen, all the samples failed at 1/3 from their total length, in good agreement and according with the strength of materials principles. In figure 2 is being presented a closer view of one of the ceramic particles reinforced multi-phase composite material in order to underline the failure mechanism and the fact that the fibres do not break entirely in that area. The figure 3 corresponds to a closer view of a sample of 5 % volume fraction of iron particles composite type, subjected to a 3-point bending test, at room temperature, while in figure 4 is being plotted a process curve as was acquired during the test.

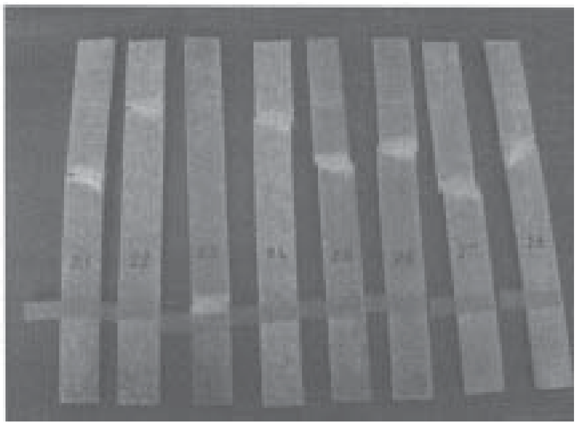

Fig. 1 Al_2O_3 (5% volume fraction) particle reinforced multi-phase composite samples after the tensile tests

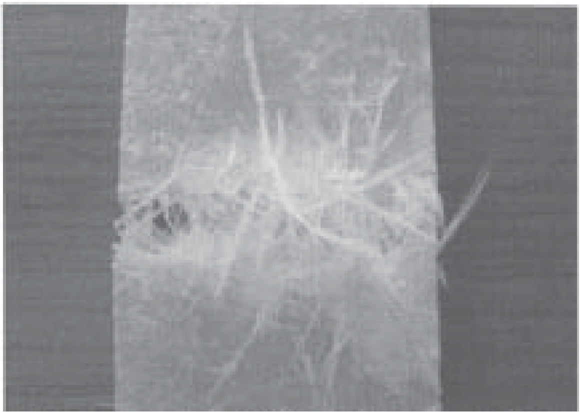

Fig.2. Al_2O_3 (5% volume fraction) particle reinforced multi-phase composite sample after a tensile test (closer view)

Fig.3. Al_2O_3 (5% volume fraction) particle reinforced multi phase composite sample after a 3-point bending test (closer view)

In figures 5 and 6 are being represented the Young's elastic coefficients for the samples of ceramic and iron particles respectively, as were predicted by the aid of the theoretical models presented at section 2 of the present study. From the plotted data it can be seen that for both classes of materials the experimental values go closer to the values corresponding to the mechanics of materials approach. The same finding can be sized in figure 7 as well, were the data correspond to the elastic coefficients relative variations of both types of particle inclusions considered in the tests, fact that it is somehow surprisingly due to the fact that the homogenization models proposed and based on the Mori-Tanaka & Halpin-Tsai theoretical models was developed to aid the characterization into a better way. In this context, it can be mentioned the fact that the literature provides some other theoretical approaches, namely some semi-empirical ones such as those known as Cox and Krenchel models, in which are being involved, apart from the already used characteristics, some correction coefficients depending on the physical properties of fibres and which are valid only for random and short dispersed ones [3, 5].

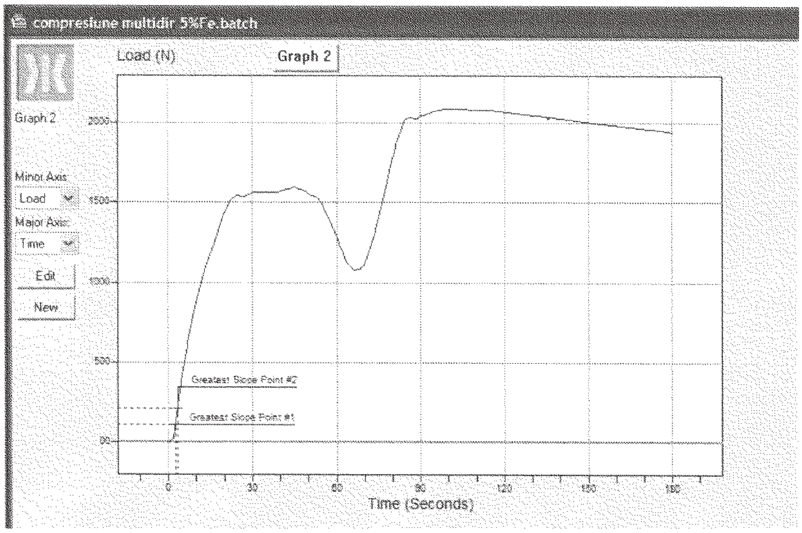

Fig. 4. Process curve acquired during a compression test on 5 % volume faction of iron particles of multi-phase composite sample

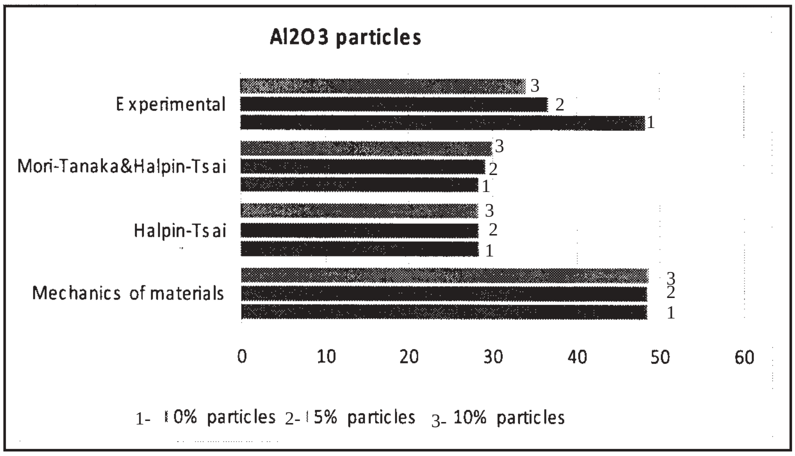

Fig. 5. Elastic coefficients variations for Al_2O_3 particles in case of multi-phase composite samples– theoretical vs. experimental comparison

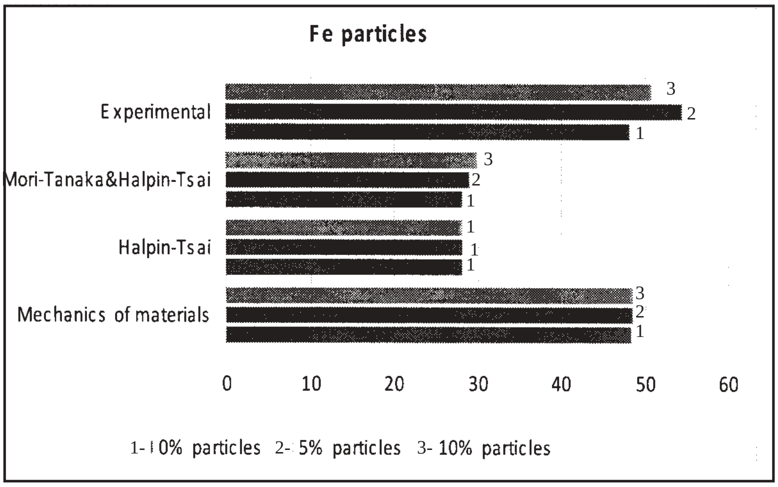

Fig. 6. Elastic coefficients variations for iron particles in case of multi-phase composite samples– theoretical vs. experimental comparison

The figures 8 and 9 correspond to the values predicted using the homogenization theoretical model proposed by the authors vs. the experimental retrieved data from the tensile and compressive respectively tests, at room temperature.

The discrepancies between the values lead to the necessity of identifying the sources of these errors, sources that were not restricted only to the manufacturing process, encompassing even the conditions in which the experimental trials were done. In such circumstances, it is necessary to recall the fact that the theoretical models do not take into account the voids influence, voids that certainly exist within the samples as a result of the manufacturing process. During the experimental research were not carried measurements in order to size the void content among the samples.

Supplementary, the mixing process of phases, even it was done by the aid of a professional mixer, it can be suspected of leading to particle agglomerates of which non-uniform distribution in the entire mass of composite material may lead to variations of the elastic coefficients. This fact can be sized analyzing the minimum and the maximum values of the elastic coefficients for each set of experimental retrieved data.

With respect to the way in which were provided the values of elastic coefficients by the testing machine software it can be mentioned the fact that it was used the slope option of stress-strain curve (the tangent elastic

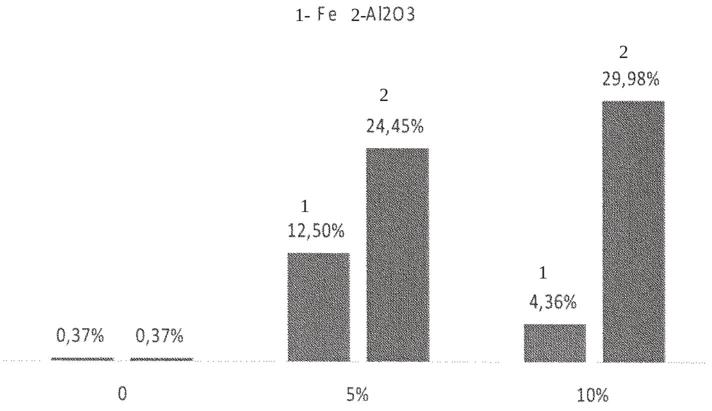

Fig. 7. Relative variations of the longitudinal elastic coefficients in case of Fe and Al_2O_3 particle reinforced composites compared with mechanics of materials theoretical model

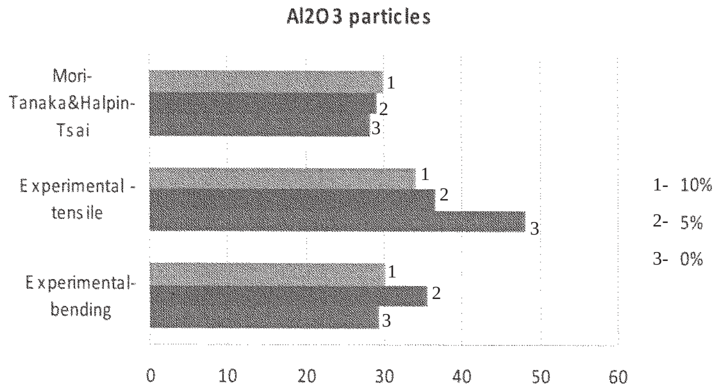

Fig. 8. Elastic coefficients variations for the Al_2O_3 particles reinforced multi-phase composites from tensile and compressive tests vs. theoretical values given by the Mori-Tanaka & Halpin-Tsai model

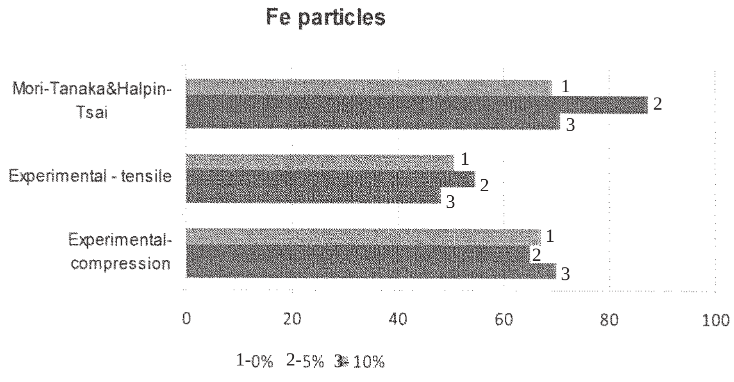

Fig. 9. Elastic coefficients variations for the iron particles reinforced multi phase composites from tensile and compressive tests vs. theoretical values given by the Mori-Tanaka & Halpin-Tsai model

modulus), even latter was identified the fact that a two-point option was a better choice for this class of materials with a complex structure. The speed at which were carried out the experiments does not lead to significant changes of the values retrieved even in case of choosing a higher value (e.g. 5 mm/s).

Conclusions

Theoretical vs. experimental data comparison within the frame of the present study described previously for this special class of composite materials, namely multi-phase ones, lead to the possibility of identifying some internal and external influencing factors allowing the behaviour characterizing of these materials on imposed loading conditions. With respect to the *internal influencing factors* it can be underlined the following aspects: the theoretical models do not take into account the voids and porosities influence in the prediction of the elastic coefficients even this is the case of their appearance inside the samples during the manufacturing process and inclusion dispersion behaviour within the matrix structure. Due to the fact that the present study does not encompass research data with respect to the void content it is impossible to predict the degree in which they are influencing the variation presented. Supplementary, the present study does not contain data on the way in which the particle-matrix-fibre interfaces irregularities are influencing the variation of the elastic coefficients even the technical literature provides some references with respect to the fact that this aspect lead to stress concentration in the contact area [2].

With respect to the *external influencing factors* the highest contribution on the elastic coefficients variations are due to the material, volume fraction, length and diameter of inclusions, either particles or fibres. During the experimental research carried out in this study was not underlined the way in which the fibres' length and diameter are influencing the elastic coefficients of the multi-phase structures due to the fact that authors had no access to other type of fibres (theoretical predictions were made for other fibre material types but inconclusive). A closer look at the theoretical and experimental values presented within this study for this special class of composite materials – multi-phase - underlines the fact that there are some discrepancies among the data. In such circumstances, it can be mentioned the fact that the theoretical models cannot considered as reference models leading to the best results – the statements from their beginning are simplified and with respect to the experimental data, the environment in which they were retrieved may not be the proper one for this class of composite materials.

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