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Predicting, measuring and tailoring thermal properties of morphological and structural modified polymeric composite materials

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1. Introduction

The development of heterogeneous engineered materials, such as the multiphase polymeric composites, requires combined knowledge and skills, both theoretically and experimentally, in order to provide a material combination with desired thermo-mechanical and physical properties for structural components from a given application. Nonetheless, these new emerging materials have to possess improved mechanical, thermal or electrical properties comparatively with their classical counterparts to prove their consistency, development and manufacturing (Curtu & Motoc Luca, 2009; Motoc Luca et al., 2010).

Multiphase polymeric composites have found a niche into engineering applications as materials for structural components in aero-space (e.g. rocket nozzles, fuel tanks,), civil engineering (e.g. liquid tanks), mechatronics (e.g. electronic packaging, sensors/actuators), electrical (e.g. electrical contacts, electrical shields), automotive (e.g. drive shafts, cylinders, brake rotors) or manufacturing (e.g. bearings, pistons) industries.

All the engineering applications of these multiphase polymeric composite materials require controlled thermal expansion characteristics in order to match those of other components and low values of the property to attain a good dimensional stability. With respect to the stability issue, this can be viewed from two perspectives: a change in the geometrical form – materials' CTE (i.e. CTE - coefficient of thermal expansion) is playing a key role and a change in mechanical properties – a mismatch between the constitutive have a dominant effect (Tan & et al., 1999; Dimitrienko, 1997, a & b).

Technical literature provides numerous experimentally related papers on the effective CTE for composite materials, manufactured as metal or polymeric based matrices. Most of these papers are approaching the 2-phase combination, either with fibres or particle reinforced as inclusions of the composite structure leaving an unexplored field with respect to the multiphase composite structures (Dey & Tripathi, 2008; Tognana & et. al., 2009). The previous holds for the theoretical based micromechanical approaches as well, the papers focusing on different methods developed to predict the effective properties of the composite structures, at the representative volume element or representative unit cell levels, based on double or multi-inclusion homogenization schemes.

These approaches are aiming the general objective of predicting the interaction between the microstructure and the effective (overall) properties (Nemat-Nasser & Hori, 1999).

In order to design a proper multiphase composite material for the previous mentioned engineering applications, to withstand different environmental condition, a lower CTE is desired. Following this objective, an attempt has been made to study and present the CTE behaviour for self-made multiphase polymeric composite materials combinations, to identify, to size and to tailor the major influencing factors in order to address thermal management issues.

The paper aims to present a comparison approach, from theoretical and experimental perspectives, of the CTE and the influencing factors on the thermal behaviour (e.g. manufacturing conditions, material morphology, material structure, environmental conditioning, etc.) for classes of two and multiphase polymeric composites. The samples were manufactured using self-developed technologies and were made up as a particle reinforced, fibre reinforced or particle-fibres and particle-particle combinations reinforced polymeric composites. The fillers used were different materials (metallic, ceramics) with different particle sizes and were embedded in different volume fraction, solely or in combinations into polymeric matrices.

2. Theoretical approaches

The fundamental knowledge on the CTE material properties define and use 3 different types of coefficients of thermal expansion: linear, superficial and volumetric. These CTE coefficients are constant only over specific temperature intervals and are defined function of this temperature range. With respect to the composite materials there are difficulties in predicting, through modelling, the CTE property due to the constitutive – either fibres or particles – embedded into another type of material that has its own behaviour subjected to the same environmental conditions.

The micromechanical approach methodology was developed as a useful tool for predicting the material properties – mechanical, thermal or electrical – of composite materials, based on the volume/mass ratio of the phases, geometrical parameters (e.g. size, length and shape), distribution, type of phases, etc., in the form of:

$$\text{effective material property} = f(\text{structural parameter})$$

Technical literature provides several theoretical models that were used for predicting the effective CTE property of composite materials function of its individual phases with/without of interaction among them. The mean homogenization schemes were implemented already in specialized modelling software. Among these methods are the well known Mori-Tanaka and double inclusion micromechanical approaches (Pindera & et al. 2009). The Mori-Tanaka method was developed in 1973, based on the approximate solution provided by the Eshelby, each inclusion from the RVE behaving as if it were isolated into the real matrix. The assumptions were made considering the body infinite and subjected to the average matrix strains in the RVE as the remote strain (see Fig. 1). The Mori-Tanaka theoretical model was used successfully for predicting the effective properties of composite materials having their inclusion phase less than 25%, even was proved that it works very well for higher values.

The double inclusion method was proposed by Nemat-Nasser and Hori and relays on the following principle: each inclusions (I) having the stiffness C_1 is surrounded with the real matrix of stiffness C_0 , the outside area being fulfilled by a reference medium with C_r stiffness. In other words, the composite is being replaced with a fictitious reference matrix containing embedding the inclusions and the real matrix of the composite structure as was depicted in Fig. 2. For linear elastic two-phase composites the double inclusion model gives good prediction of the effective material properties no matter the size, volume fraction and individual constitutive material properties of inclusion phase. These two theoretical micromechanical based models will be used herein in order to predict the mechanical and thermal effective material properties of the multiphase composite polymeric composite materials under investigation.

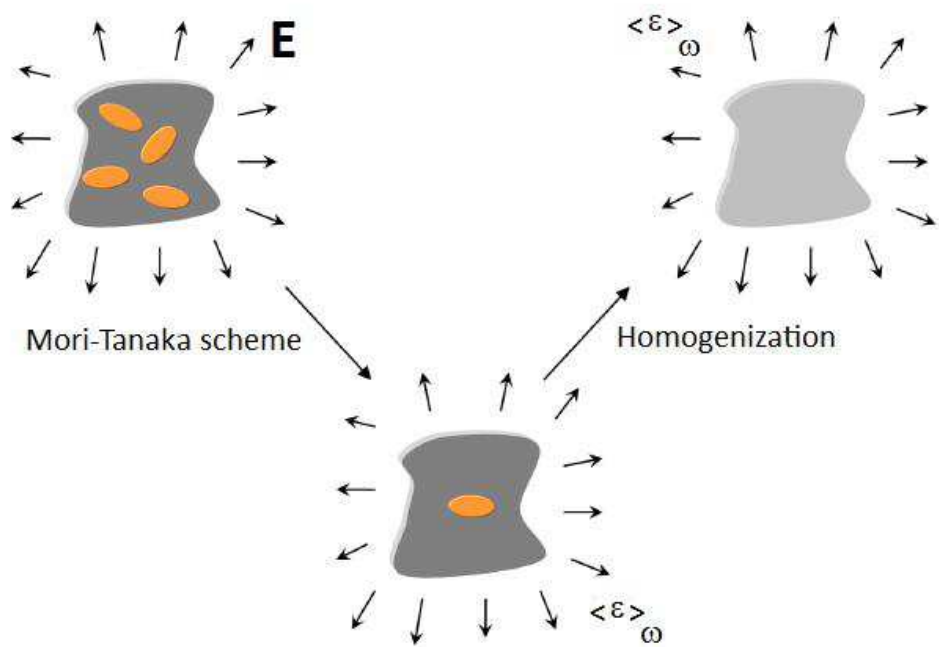


Fig. 1. Schematic illustration of the Mori-Tanaka model (from Nemat-Nasser & Hori, 1999)

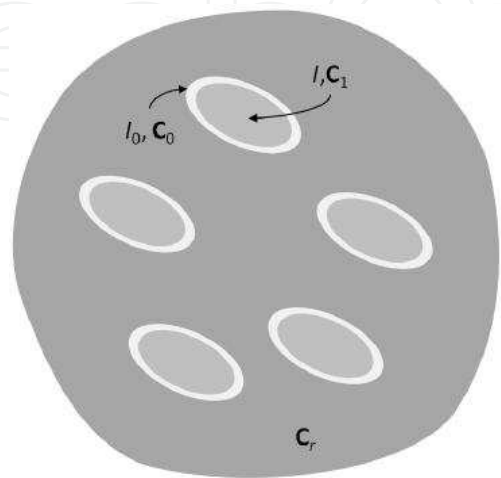


Fig. 2. Schematic illustration of the double inclusion method

3. Experimental research

3.1 Materials. Manufacturing issues

The particle-fibre multiphase composite samples were manufactured as having random fibres and particles embedded in different volume fraction into a polymeric matrix. The matrix material is commercially known as SYNOLITE 8388 P2 from DSM Composite Resins (Switzerland), a polyester resin type. The particle inclusions considered were 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 or technical pure iron particles, mixed within the polyester resin mass in 5% and 10% volume fraction, respectively. The 3rd phase chosen were E-glass type random fibres, commercially available under MultiStrat™ Mat ES 33-0-25 trade name, from Johns Manville, SUA, mixed as having a 65% volume fraction in the overall composite volume. The reference sample was made without any particle content and used for comparison purpose.

With respect to the second combination chosen, particle-particle type multiphase composite materials, the particles were technical pure iron having 50 μm as a mean value of the particle size in combination with black carbon particles of 1 μm in diameter, embedded into different volume fraction (e.g. black carbon – 0%, 5%, 10%) into a PVAc (e.g. polyvinyl acetate) polymeric matrix material having a 30% content in the mixture, from DSM Composite Resins (Switzerland). As in the previous case, a reference sample was manufactured, containing no carbon particles. A self-conceived manufacturing configuration was used for the particle-particle multiphase composites samples to be able to use a controlled temperature environment to aid the polymerization process as well as a compression head to aid the sample compaction.

3.2 Testing procedure and devices

The CTE measurements were performed using a DIL 420 PC differential dilatometer from NETZSCH GmbH (Germany). The particle-fibre reinforced multiphase composite samples were shaped into rectangular bars of about 5x5x25 mm³ whereas the particle-particle combinations of multiphase composites were shaped as cylinders of 10x25 mm² (dxh). For all samples the transversal external surfaces were polished to guarantee plan-parallel surfaces for precise positioning within the measuring head.

The samples are positioned horizontally on two quartz beds. The measured experimental data were sent to a PC via an USB cable, the acquisition software – Proteus Analysis (from the same manufacturer) - displaying information regarding the thermal strain variation with the imposed thermal range. Further experimental data manipulation allows linear or technical CTE retrieval vs. temperature range or time.

The temperature variation was set up having different trends in time, with a heating rate of 1 K/min, into a static air atmosphere. To eliminate the systems errors, the dilatometer was calibrated by measuring a standard SiO_2 specimen under identical conditions.

The thermal regimes imposed were as follows, successive heating (2 thermal cycles) being imposed for all the multiphase polymeric composite samples:

- linear – monotonically rise from 20°C up to 250°C for the particle-fibres multiphase combination;
- triangular – monotonically heating and cooling up/down to/from 150°C for the particle-particle multiphase combination;

4. Results and discussion

Figure 3 shows the measuring head containing the thermocouple positioned in the sample neighbourhood along with the quartz beds and pushrod used to hold the composite samples. In Fig. 4 and Fig. 5 are being shown the microscopic views (x500 magnification) taken with a digital microscope for the 10% Fe & 65% E-glass fibres multiphase polymeric composite sample and for the 10% C & 60% Fe particles multiphase combination, respectively.

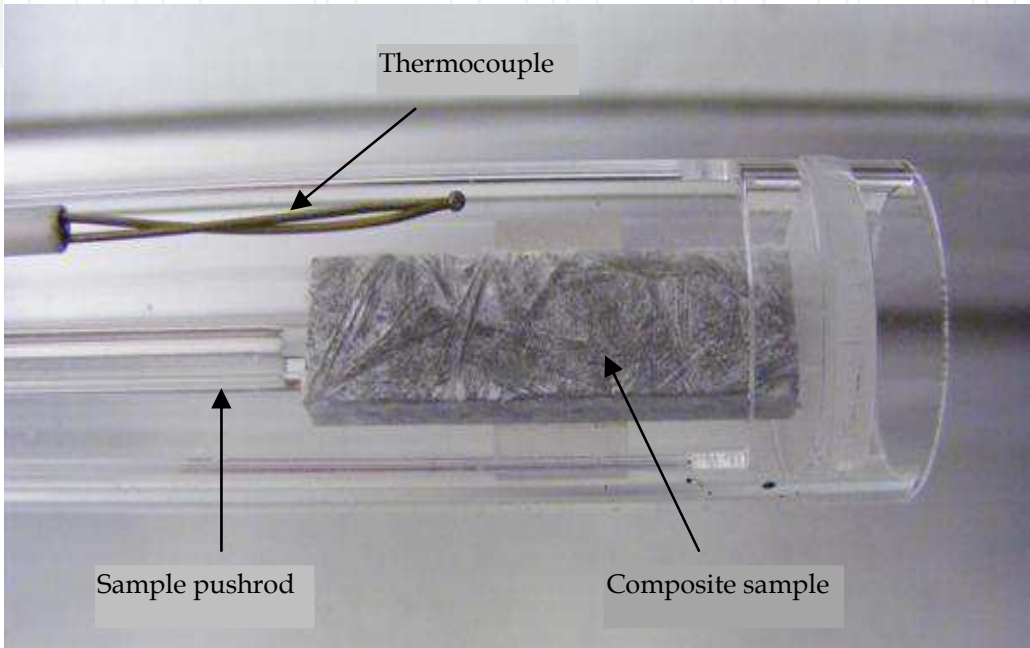


Fig. 3. The measuring head – thermocouple and composite sample positions

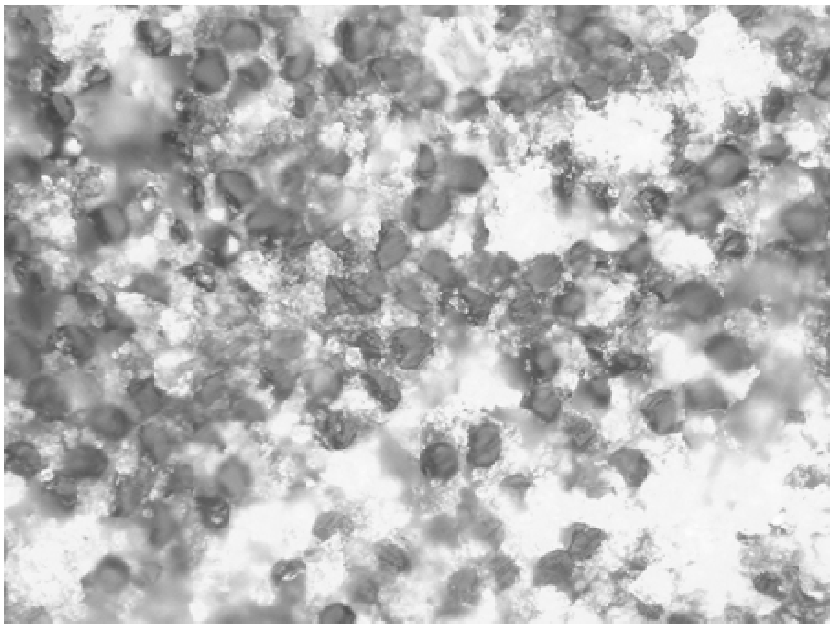


Fig. 4. Microscopic view for the 10% Fe & 65% E-glass fibres multiphase composite sample

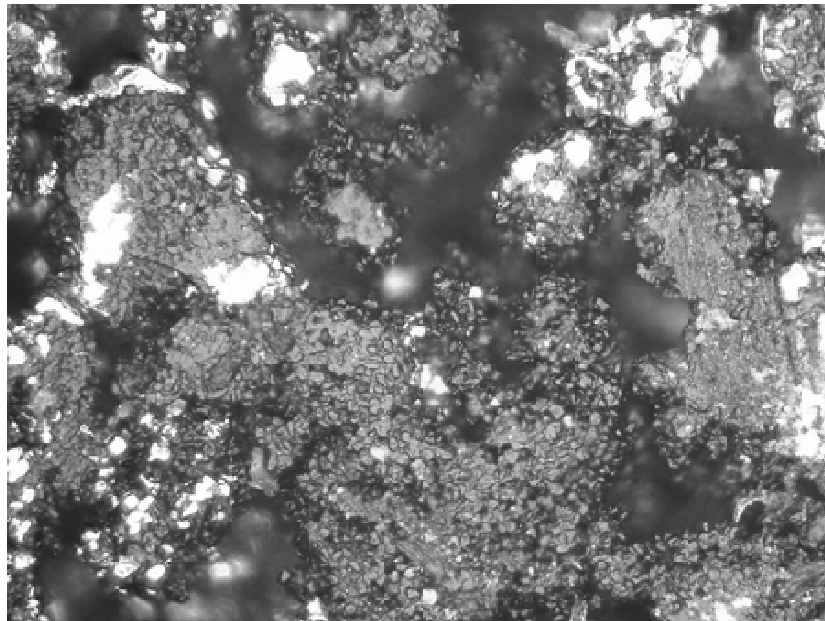


Fig. 5. Microscopic view for the 10% C & 60% Fe particles multiphase composite sample

As it can be seen, in case of the particle-fibres multiphase structure the particles were embedded within the overall composite structure as having a dilute nature, being widespread into the mixture, whereas in case of the particle-particle multiphase combination, due to the manufacturing process, an agglomeration of the iron particles can be seen, leading to a clustering effect within the overall compound. The latter was responsible for the conductive paths during the electrical measurements done to fully characterize the multiphase composite samples, the subject being approached by the authors elsewhere.

Fibre-particle multiphase composite materials

In Table 1 are provided the values of the instantaneous CTE recorded experimentally, whereas in Table 2 are given the values of the temperatures at which were recorded the maximum values of the CTE for all the particle-fibres multiphase polymeric composite samples under experimental investigations experiencing a single linear heating cycle. These temperature values can be viewed as an overall behaviour of the polymeric multiphase composite samples as experiencing a linear increase of a temperature in their surrounding environment and not necessarily as a compulsory measure for the composites' performance. These differences can be also sized on the experimental curves corresponding to the instantaneous coefficient of linear expansion, from Fig. 6 to Fig. 8. Three different regions can be identified for all the composite samples, according to the curve shapes - *region I* up to 50°C, *region II* - from 50°C to 100°C, and *region III* - from 100°C to 250°C.

Also, as it can be seen, the higher the particle content the different get the thermal strain fields developed within the composite structure. For all the composite samples an abrupt decrease in the instantaneous CTE variation it can be seen around the 55°C, changes that can be regarded to the polymeric matrix material whose chains it must be broken and reshaped differently.

Usually, within the literature are reported the CTE values for a specific temperature. The idea of reporting a mean value over a temperature range lied on the fundamental principles

of thermal dilatations – measuring coefficients of linear expansions has to provide a linear variation of the property and any variations from this has to be regarded to the internal structure of the material under study or other influencing factors.

Figure 6 is showing both thermal strain and instantaneous coefficient of thermal expansion for the reference sample containing only the 5 layers of E-glass random fibres subjected to two heating thermal cycles. Within the *region I* the CTE values are almost linearly, in both cases, differences between the curves being relatively higher and revealing the environmental conditioning influence, whereas *region II* is revealing an abrupt decrease in the instantaneous CTE values based on the polymer chains reordering and chemical modifications as a part of an unfinished cure process. *Region III* is revealing a smother linearly variation on both experimental curves, differences among the values being not as higher as in the first region. This variation can be regarded less to the polymer behaviour at temperature higher than its glass temperature but to the fibres reinforcements thermal behaviour.

The *successive heating cycles* do have an influence on both thermal strains and instantaneous coefficient of thermal expansion of the composite materials, as it can be seen from the same previous figure. The experimental data retrieved for the reference sample reveal the polymer behaviour under temperature variations, the more cycling the most stable the overall composite, the resin polymerization reaching its final stage. A maximum value of the linear CTE was recorded at 22.1°C with an onset at 45.4°C for the first thermal cycling, whereas in case of the second cycling the peak was reached at 55.9°C.

The *particle types*, either ceramic (e.g. Al_2O_3) or metallic (e.g. Fe) and *particle volume fraction* do not have a major influence on the overall linear CTE of the multiphase composite material mainly due to the reduce volume fraction in which were embedded. This behaviour can be sized in the Fig. 7 and Fig. 8.

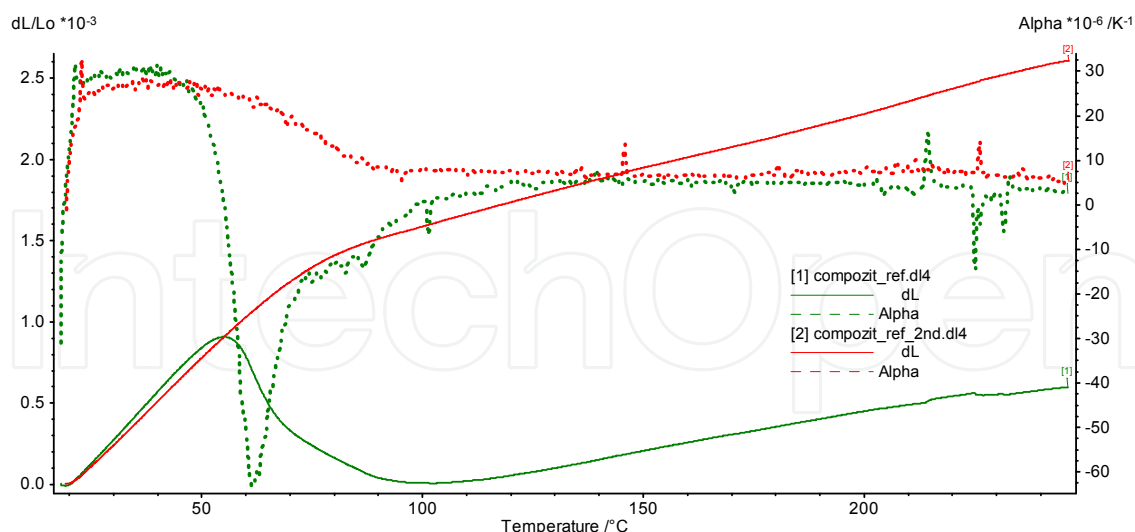


Fig. 6. Thermal strain and instantaneous coefficient of thermal expansion for the reference sample subjected to successive heating under a linear increase of temperature

It is acknowledged from literature that increasing the particle volume fraction the overall linear CTE experience a slightly decrease. This behaviour is being used to address stability

issues. Unfortunately, as it can be seen from the values provided in Table 2, the instantaneous linear CTE slightly rise with the particle volume fraction content for the same temperature range. From authors’ point of view this behaviour does not necessarily has to be regarded as a drawback but *manufacturing* related influencing factors has to be identified, such as non-uniform dispersion of the particles over the entire volume of the polymeric composite.

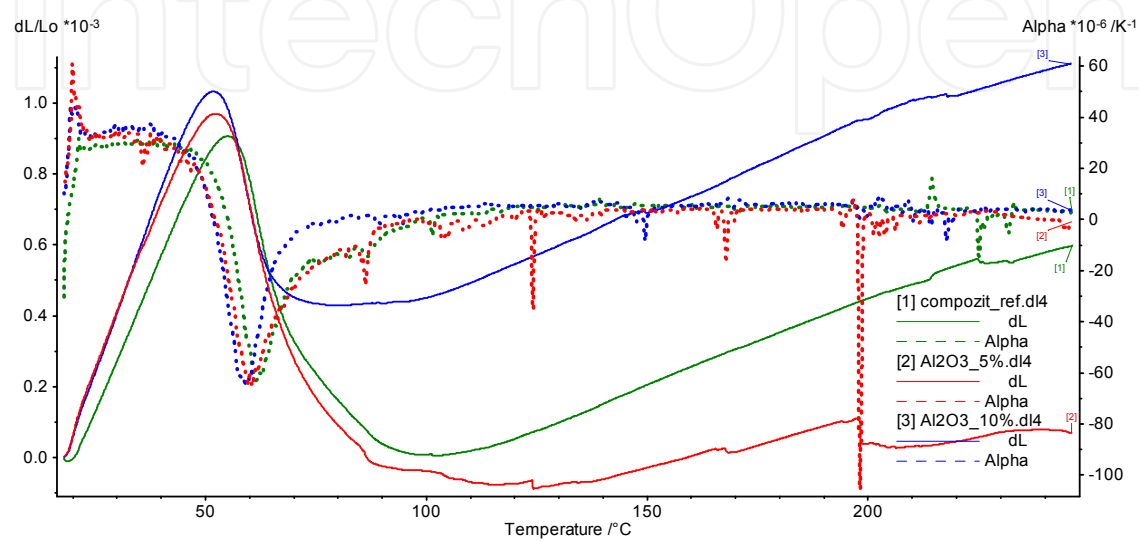


Fig. 7. Thermal strain and instantaneous coefficient of thermal expansion for the reference sample, 5% and 10% Al₂O₃ reinforced multiphase composites – linear thermal regime

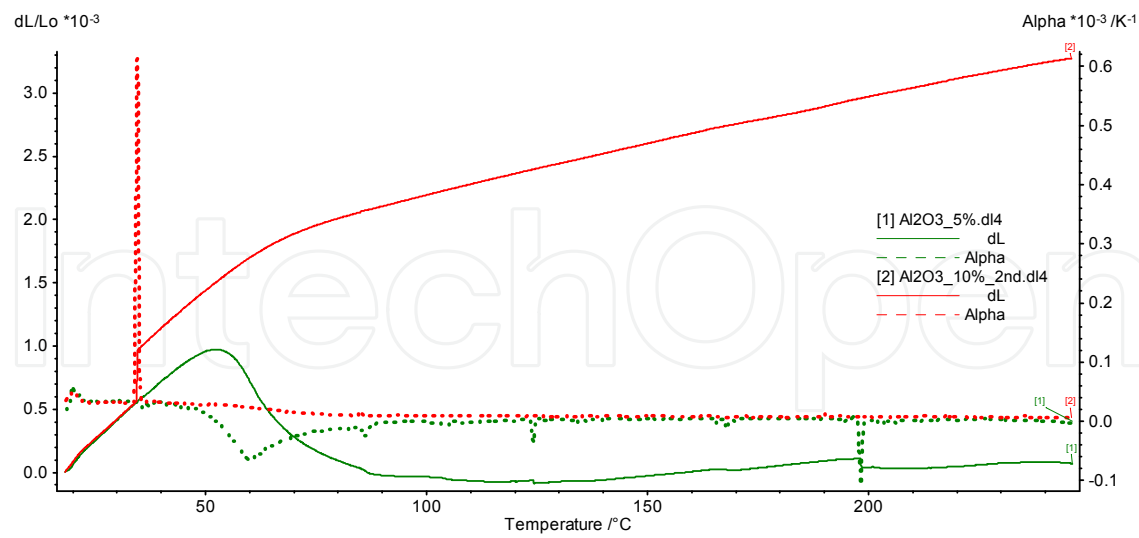


Fig. 8. Thermal strain and instantaneous coefficient of thermal expansion for the 5% and 10% Al₂O₃ reinforced multiphase composites subjected to heating/cooling linearly

In the same table, over the entire temperature range the overall CTE for each composite sample, individually, is experiencing a decrease.

Temperature [°C]	Reference sample [x10 ⁻⁶ /°C]	5% particles multiphase composite [x10 ⁻⁶ /°C]		10% particles multiphase composite [x10 ⁻⁶ /°C]	
		Al ₂ O ₃	Fe	Al ₂ O ₃	Fe
20	31.434	36.789	31.903	41.237	29.624
50	21.809	13.326	25.045	12.579	24.826
100	1.2586	2.8613	4.4431	3.8404	4.6403
150	5.2118	3.4754	4.8478	4.4835	4.7307
200	4.2617	2.1328	4.9412	1.3774	4.6980
250	2.6055	1.5667	2.4167	3.4394	2.5823

Table 1. Instantaneous CTE values for all the particle-fibres multiphase polymeric composite samples subjected a single heating thermal cycle

	Reference sample	5% particles multiphase composite		10% particles multiphase composite	
		Al ₂ O ₃	Fe	Al ₂ O ₃	Fe
Temperature [°C]	55.3	52.4	123.4	52.0	118.8
CTEmax[x10 ⁻³ /°C]	0.9050	0.9679	0.0967	1.030	0.1634

Table 2. Peak values of the CTE and the temperatures values associated as were recorded experimentally

The predicted values based on the micromechanical approach were subjected to comparison with the experimental retrieved data. In Fig. 9 is being shown two RVEs corresponding to the reference composite sample (only E-glass fibres) and to a 5% particle reinforced multiphase polymeric composite sample. The RVEs were generated using a commercial software called DIGIMAT and the predicted CTE values based on the double inclusion and Mori-Tanaka homogenization concepts. In Fig. 10 were plotted the predicted values vs. the experimental retrieved values for iron particles reinforced multiphase composite materials at the reference temperature of 20°C. As it can be seen the experimental values closer approaches the predicted values based on the Mori-Tanaka homogenization scheme, even the double inclusion predicted values are more precisely.

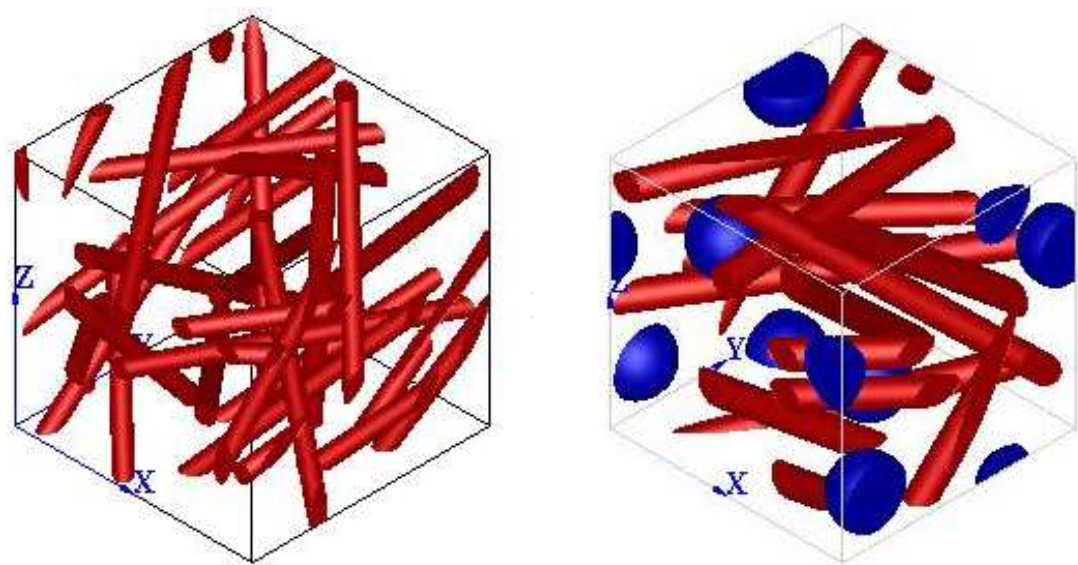


Fig. 9. Representative volume elements for random fibres reinforced polymeric composite (left) and multiphase fibre-particle reinforced polymeric composites (right)

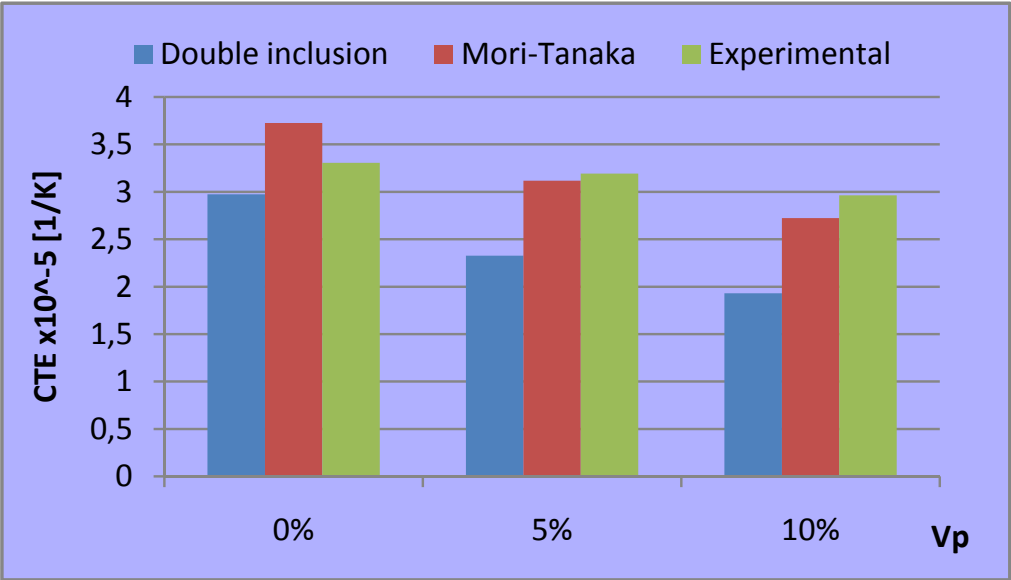


Fig. 10. Theoretical vs. experimental CTE values for the multiphase particle-fibre type polymeric composite materials (65% E-glass fibres, 0% to 10% Fe particles)

Particle-particle multiphase composite materials

In Table 3 are provided the values of the instantaneous CTE recorded experimentally, whereas in Table 4 are given the values of the temperatures at which were recorded the maximum values of the CTE for all the particle-fibres multiphase polymeric composite samples under experimental investigations experiencing a single linear heating cycle. These differences can be also sized on the experimental curves corresponding to the instantaneous coefficient of linear expansion, from Fig.11 to Fig. 13. For these cases the analysis will be carried out on the *heating* and on the *cooling* thermal trends -up to 150°C, down from 150°C, respectively.

Also, as it can be seen, the higher the particle content the different get the thermal strain fields developed within the composite structure. For all the composite samples an abrupt decrease in the instantaneous CTE variation it can be seen around the 35°C in the heating step, changes that can be regarded to the polymeric matrix material whose chains it must be broken and reshaped differently.

For all the particle-particle multiphase composite polymeric samples the second thermal cycles lead to a smoothly variation in the instantaneous CTE over the temperature range. From manufacturing point of view, the first heating cycle can be viewed as a thermal treatment applied upon the composite samples, as a supplementary aid to the polymerization process.

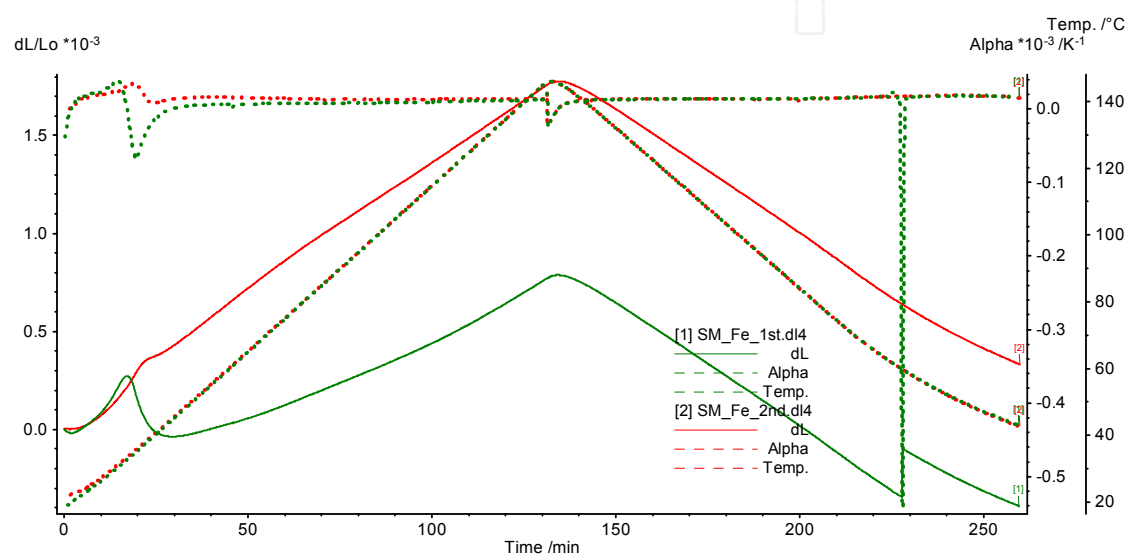


Fig. 11. Thermal strain and instantaneous coefficient of thermal expansion for the reference sample subjected to successive heating under a linear increase/decrease of temperature

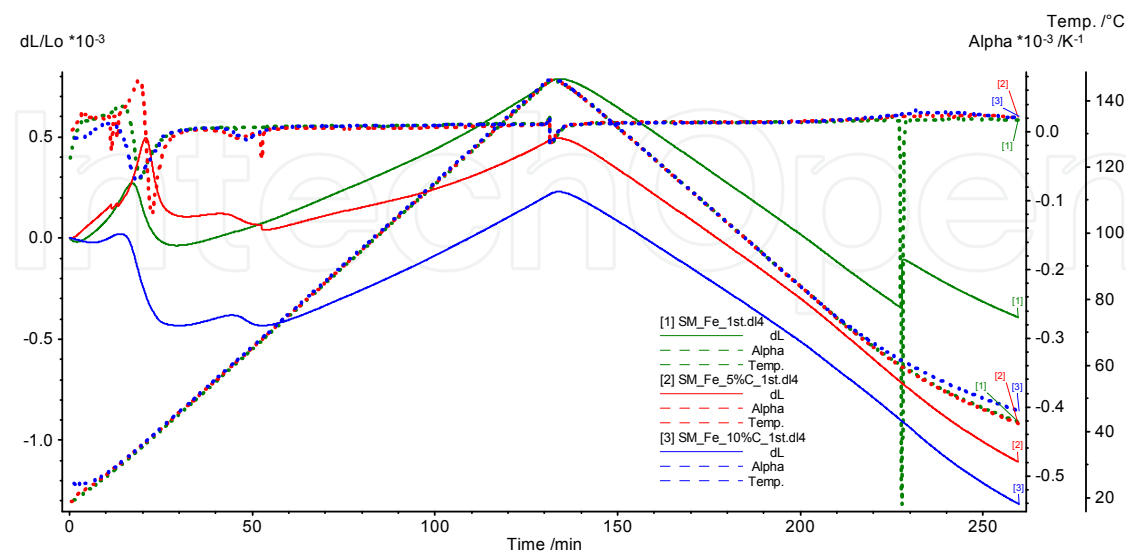


Fig. 12. Thermal strain and instantaneous coefficient of thermal expansion for the reference sample, 5% and 10% C reinforced multiphase composites - 1st thermal cycle

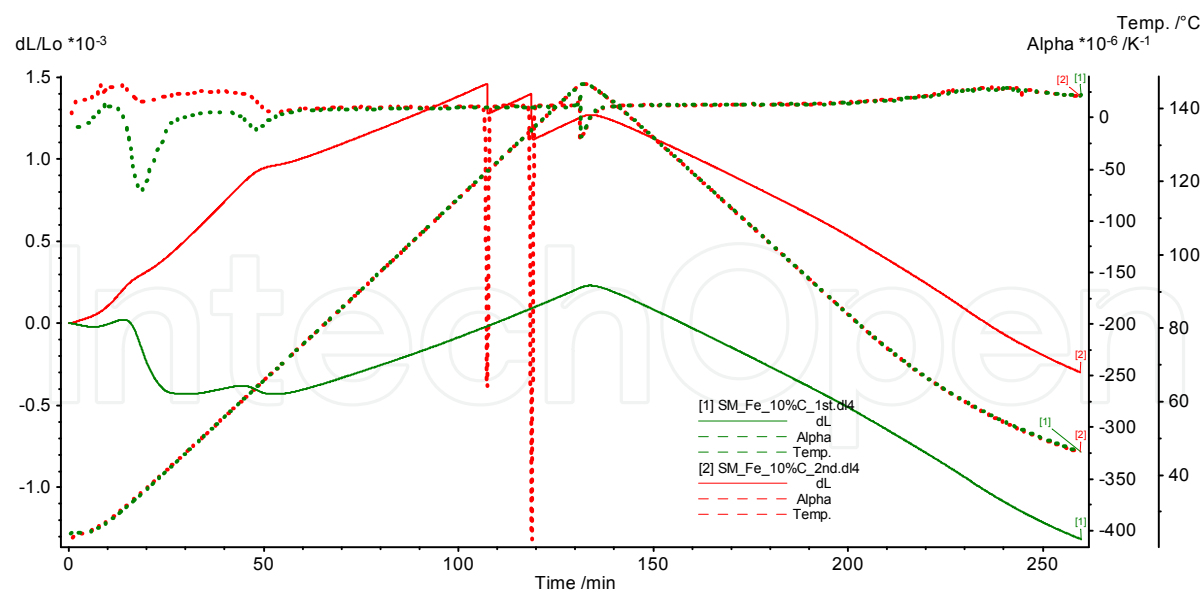


Fig. 13. Instantaneous coefficient of thermal expansion and thermal strain variation over the time for two successive thermal cycles applied to the 10% C & 60% Fe multiphase composite








Temperature [°C]	Reference sample [x10 ⁻⁶ /°C]		5% C particles multiphase composite [x10 ⁻⁶ /°C]		10% C particles multiphase composite [x10 ⁻⁶ /°C]	
	1 st cycle	2 nd cycle	1 st cycle	2 nd cycle	1 st cycle	2 nd cycle
20 	14.087	12.899	11.742	13.459	10.996	15.548
50 	4.7076	14.637	2.0925	10.834	4.2966	2.4086
100 	8 .2254	12.088	4.1026	10.928	8.3001	9.6639
150 	9.5383	10.833	4.7260	10.902	4.7654	10.915
100 	13.091	12.962	13.236	14.269	12.359	12.465
50 	17.538	17.946	23.458	23.759	24.122	24.859
20 	15.880	16.010	20.810	20.946	20.097	20.479

Table 3. Instantaneous CTE values for all the particle-particle multiphase polymeric composite samples subjected a single heating thermal cycle

The increase of the carbon black volume fraction within the multiphase structure has an influence on the overall instantaneous CTE of the composite, actually leading to a decrease of the experimentally recorded values, as it can be seen in Table 3.

Composite samples	Heating		Cooling	
	T [°C]	CTEmax [x10 ⁻³ /°C]	T [°C]	CTEmax [x10 ⁻³ /°C]
Reference composite 70% Fe	32.6	0.2695	42.7	-0.3942
	145.5	0.7860	-	-
Multiphase composite 5% C, 65% Fe	36.7	0.4935	42.3	-1.1101
	145.8	0.4919	-	-
Multiphase composite 10% C, 60% Fe	146	0.2271	46.2	-1.3163

Table 4. Peak values of the CTE and the temperatures values associated as were recorded experimentally – first heating thermal regime

In Fig. 14 is being shown two RVEs corresponding to the reference composite sample (only Fe particles) and for a 5% C & 65% Fe particles reinforced multiphase polymeric composite sample. The RVEs were generated using the same software as previously and the predicted CTE values based on the double inclusion and Mori-Tanaka homogenization concepts. In Fig. 15 were plotted the predicted values vs. the experimental retrieved values for the multiphase particle-particle reinforced composite materials at the reference temperature of 20°C.

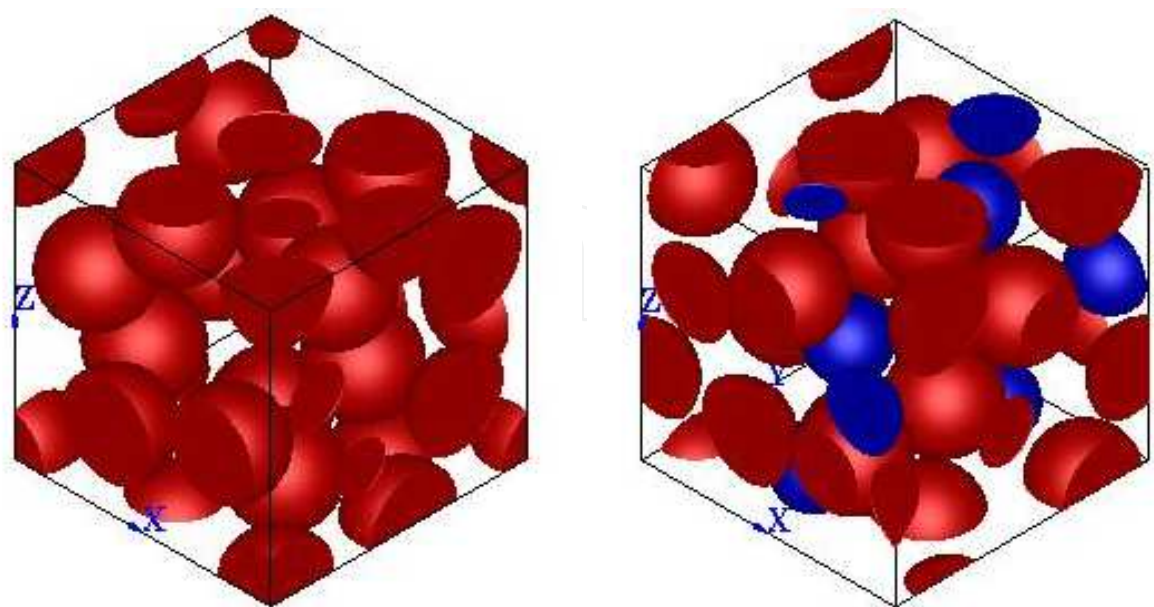


Fig. 14. Representative volume elements for random particle reinforced polymeric composite (left) and multiphase particle-particle reinforced polymeric composites (right)

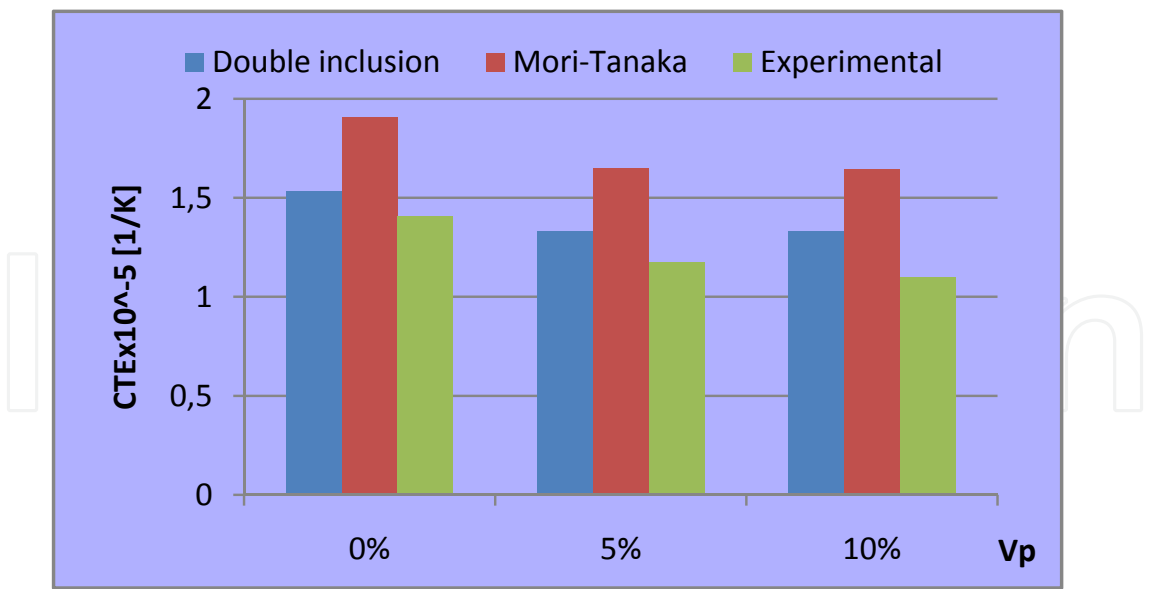


Fig. 15. Theoretical vs. experimental CTE values for the multiphase particle-particle type polymeric composite materials (70% to 60% Fe particles, 0% to 10% C particles)

For all the particle-particle multiphase composite samples in the cooling down step of the thermal regime imposed, the retrieved CTE values are higher than their counterparts from the heating step. This behaviour is somehow natural due to the fact that the temperature within the oven do not experience a suddenly temperature decrease. Also, it can be seen the fact that in the cooling step the instantaneous CTE values are almost the same over a large temperature range, denoting stability in the composites' behaviour.

This also can be sized on the peak values retrieved for all the particle-particle polymeric composite samples. In the heating step of the first thermal cycle, from 20°C up to 150°C, two different peaks at different temperature values are being registered whereas in the cooling down step, from 150°C to 20°C only one peak is being revealed. With respect to the second thermal regime applied a single peak is being registered for all the samples, in the cooling down step. Interesting is the fact that the temperatures values are almost the same for all the multiphase composite samples as it can be seen from the values provided in Table 4.

As it can be seen from Fig. 15 the predicted values based on the double inclusion method closely approaches the experimentally recorded values, not as in the case of the particle-fibre reinforced polymeric composites. This observation it cannot be generalized to hold for these multiphase combinations due to the lack of the experimentally values and composite structures subjected to study.

5. Conclusion

The instantaneous coefficient of linear thermal expansion in case of a multiphase polymeric composite material is not an invariant value being very sensitive to the heterogeneities and thermal regimes at which is subjected the measured sample. Its temperature dependence reflects phase changing, degree of polymerization, differences in internal structure, external environmental conditioning and the evolution of the internal thermal strain. Inclusion type, volume fraction, size and distribution within the overall composite structure have their influences on the predicted and experimentally measured CTE values. Adding inclusions

such as particles into a 2 phase composite either fibre or particle reinforced lower the overall effective CTE property and provide stability to the multiphase composite structure.

The herein analysis did not take into account the presence of voids or other micro-defects nor of the inclusions interaction within the multiphase composite structure even their presence or influence it can be sized in the experimentally retrieved values of the effective CTE material property.

Further studies are under development on multiphase composites made of different combinations, arrangements, particle sizes or fibre length, with or without being subjected to extreme environmental conditions. Different thermal regimes will be also considered as viable alternatives for developing a protocol for thermal residual stresses recovery. Supplementary, the presence of porosities, inclusions or interaction among the constitutive will be included into the modelling models.

Thermal management issues can be addressed after thermal properties measurements on these multiphase composite structures based on a tailoring process for different combinations among the phases, theoretical predictions and an optimization approach.

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