

# DEVELOPING AND CHARACTERIZING NEW MULTIPHASE COMPOSITE MATERIALS FOR AUTOMOTIVE BRAKE ROTORS

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**ABSTRACT** – The paper focuses on development and material properties measurements of new structural combinations of particle-particle multiphase polymeric composite materials. The composites were manufactured using a self-developed technology, embedding in different volume fraction iron oxide and black carbon into a polymeric matrix with the purpose of obtaining a new material with improved mechanical and thermal properties to be used for automotive brake rotors. The paper focuses on one of the most important thermal property, the linear coefficient of thermal expansion (CTE), which represents the structures' stability related indicator. This thermal property will be measured using a differential dilatometer DIL 402 PC from Netzsch (Germany). Based on the experimental data retrieved, several manufacturing and structural composition related influencing factors will be identified with the purpose of tailoring further material combinations for automotive brake rotors.

## 1. INTRODUCTION

Understanding the behaviour of composite materials relative to the properties of their constitutive, filler and matrix, is desirable not only for the practical purpose of predicting the material properties but also for the fundamental knowledge required in developing new materials or tailoring the existing one for an imposed condition.

Coefficient of thermal expansion (CTE) is the material property of primary influence on dimensional stability as it represents the materials response to changes in temperature. Consequently, materials with low CTE are highly desirable for precision structures under different environmental conditions where thermal gradients and temperature fluctuations could cause substantial change in dimensions.

The problem of relating effective thermal properties of particle reinforced composite materials to constituent properties has received considerable attention. Theoretical models, at macro and micro scale, were developed to aid the overall thermal properties prediction (e.g. coefficient of thermal expansion, thermal conductivity etc.) accompanied by numerical predictions such as the finite element method, in the form of the bounds or model trends, for isotropic or anisotropic phases [1], [8-10]. The mathematical complexity of the analysis associated to the developed theoretical models ranges from the simple law of mixture to the methods using thermo-elastic energy principles.

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. 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 [3-4], [7]. Consequently, a micromechanical based theoretical approach accompanied with experimental research to aid the characterization and tailoring process of a structure that may prove a multifunctional character is the main purpose of the herein paper.

The samples were manufactured using a self-developed technology and were made up as a particle reinforced or 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

A variety of micromechanical based theoretical models have been developed to explain the effect of fillers on the thermal expansion behaviour of the composite materials. There has been progressive development of more advanced models, comparatively with the basic rule of mixture, that use the fundamental mechanical properties of each phase to predict the expansion behaviour of the overall composite based on fundamental energy principle. Among these the Benveniste, Kerner or Turner emerged as new theoretical models developed especially for particle reinforced composite materials.

In Table 1 are being provided the expressions of few micromechanical based theoretical models to be used in overall composite materials CTE prediction, theoretical models that proved to provide values that are close related to the experimental retrieved data.

**Table 1** Micromechanical based theoretical models developed for CTE prediction

Theoretical model	Associated formulae
Benveniste	$\alpha_c = V_p \alpha_p + (1 - V_p) \alpha_m + \frac{V_p (1 - V_p) (\alpha_p - \alpha_m) (K_p - K_m)}{\frac{3K_p K_m}{4G_m} + V_p K_p + (1 - V_p) K_m}$
Kerner	$\alpha_c = \alpha_p V_p + \alpha_m V_m + V_p V_m (\alpha_m - \alpha_p) \frac{\frac{1}{K_m} - \frac{1}{K_p}}{\frac{V_p}{K_p} + \frac{V_m}{K_m} + \frac{3}{4G_m}}$
Levin	$\alpha_c = (1 - V_p) \alpha_m + V_p \alpha_p + \frac{\alpha_p - \alpha_m}{\frac{1}{K_p} - \frac{1}{K_m}} \left[ \frac{1}{K_c} - \left( \frac{1 - V_p}{K_m} + \frac{V_p}{K_p} \right) \right]$

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**Rosen-Hashin**

$$\alpha_c = \alpha_p + (\alpha_m - \alpha_p) \left[ \frac{\frac{1}{K_c} - \frac{1}{K_p}}{\frac{1}{K_m} - \frac{1}{K_p}} \right]$$

**Turner**

$$\alpha_c = \frac{V_m K_m \alpha_m + V_p K_p \alpha_p}{V_m K_m + V_p K_p}$$


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In all previous expressions,  $\alpha_c$  stands for the effective CTE of the particle reinforced composite material, expressed in  $K^{-1}$ ,  $\alpha_p$  and  $\alpha_m$  for the individual phases (particles and matrix) CTE's, expressed in  $K^{-1}$ , whereas  $V_p$  and  $V_m$  are the phases' volume fraction. The mechanical properties of each constitutive are being present in terms of the bulk moduli,  $K_p$  or  $K_m$ , expressed in GPa, or as Young longitudinal moduli,  $E_p$  or  $E_m$ , expressed in GPa. Supplementary,  $\nu_p$  and  $\nu_m$  stands for the Poisson ratio of each constitutive, particle and matrix material.

The previous expressions were used herein by applying a two step procedure, namely by using it two times for predicting the CTE for each class of particle-particle multiphase polymeric composite materials. A simple computer algorithm was developed to aid this process of material property prediction.

### 3. EXPERIMENTAL RESEARCH

#### 3.1. MATERIALS

The composite samples were manufactured using a self-developed manufacturing technology, by mixing together two different types of particles (e.g. iron oxide and black carbon) in different volume fraction, into a polymeric material namely polyvinyl acetate. The inclusions were characterized as having a medium size of 80  $\mu m$  in case of the iron oxide particles and 10  $\mu m$  in case of the carbon particles and were mixed to form a two or multi-phase structure. The samples were formed as having a cylindrical shape (diameter – 20 mm, height – 5 mm), 5 pieces for each class of composite materials as a minimum requirement in statistical data analysis. The composite classes are as follows:

- 0% C, 80 % Fe, 20% polymer resin;
- 10% C, 70% Fe, 20% polymer resin;
- 15% C, 65% Fe, 20% polymer resin;
- 20% C, 60% Fe, 20% polymer resin.

In the previous, the polymeric resin is considered to include the chemical compounds contained into the additives added to help the polymerization process. The carbon particles were impossible to be mixed solely into the polymeric resin to form a sample by using the manufacturing technology developed and used herein.

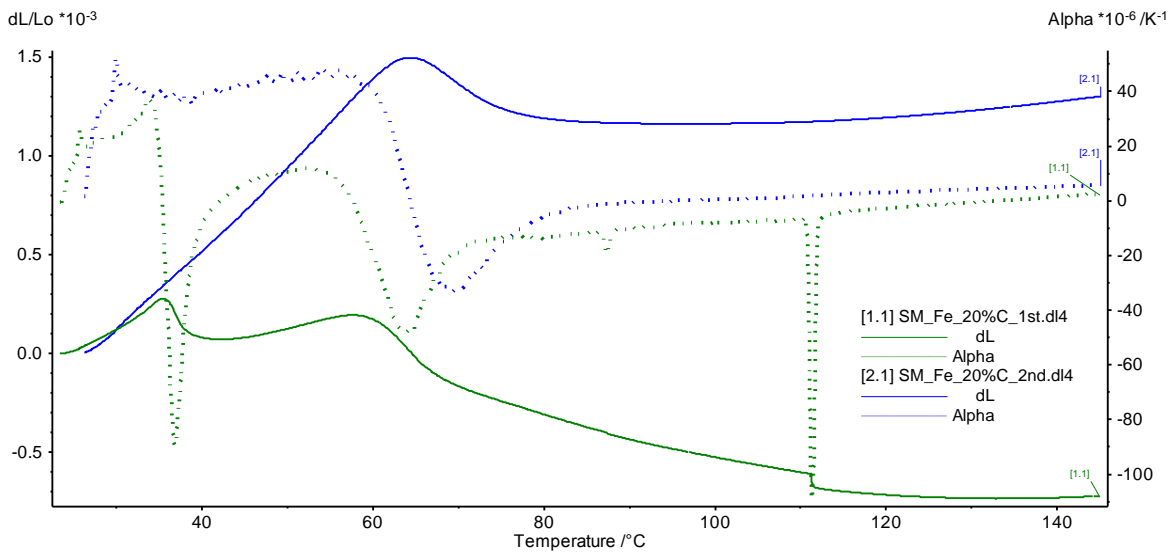
#### 3.2. TESTING PROCEDURE AND DEVICES

The CTE measurements were performed using a DIL 420 PC differential dilatometer from NETZSCH GmbH (Germany). The particle-particle combinations of multiphase composites were shaped as cylinders of 10x25  $mm^2$ . 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.

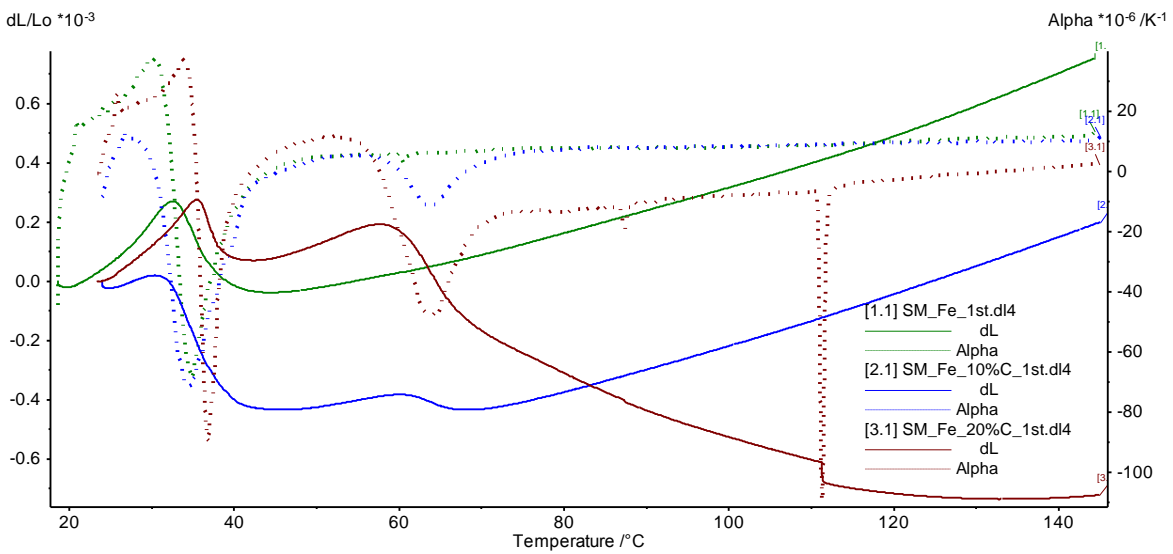
The measured experimental data were sent to a PC via an USB cable, the acquisition software – Proteus Analysis (from the same manufacturer) - displaying information regarding the thermal strain variation with the imposed thermal range. Further experimental data manipulation allows linear or technical 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<sub>2</sub> specimen under identical conditions. The thermal regimes imposed were as follows: successive thermal heating (2 thermal cycles); thermal regime variation – monotonically heating up to 150<sup>0</sup>C.

**4. RESULTS AND DISCUSION**



**Fig. 1** Instantaneous CTE and thermal strain vs. temperature variation retrieved from two successive thermal cycles applied on the 20% C & 60% FeO<sub>2</sub> multiphase composite sample



**Fig. 2** Thermal strain and instantaneous coefficient of thermal expansion for the reference sample, 10% and 20% C reinforced multiphase composites – 1st thermal cycle

As it can be seen from Fig. 1 and Fig. 2, the thermal strain field vary from sample to sample and from thermal cycle to the other thermal cycle applied upon one type of multiphase polymeric composite material individually. This behaviour is normal and relay on the internal chains established within the molecules, either of the constitutive or polymer matrix, or mixed. Other influencing factors could be associated with the internal voids or imperfect interfaces as a natural consequence of the manufacturing process. This can be sized in the instantaneous coefficient of thermal expansion as suddenly decreasing of its variation with temperature, as it can be seen around 35<sup>0</sup>C and 65<sup>0</sup>C.

In the same figures it can be seen that around 110<sup>0</sup> C the composite samples are experiencing a suddenly decrease in the variation of their instantaneous coefficient of thermal expansion field. This behaviour can be regarded with the polymeric matrix, the polymer chains going thorough a process of broken and rearranging its connections, the temperature being close related with the glass temperature transition of the polymeric material.

In Table 2 are being given the experimental retrieved data for three types of particle-particle polymeric composite samples under the investigation. The experimental data correspond to two successive heating cycles applied upon the composite samples with the purpose of characterize their behaviour under temperature change regimes.

**Table 2** Instantaneous CTE values for particle-particle multiphase polymeric composite samples subjected to successive heating thermal cycles

Temperature [ <sup>0</sup> C]	Reference sample [x10 <sup>-6</sup> / <sup>0</sup> C]		10% C particles multiphase composite [x10 <sup>-6</sup> / <sup>0</sup> C]		20% C particles multiphase composite [x10 <sup>-6</sup> / <sup>0</sup> C]	
	1 <sup>st</sup> cycle	2 <sup>nd</sup> cycle	1 <sup>st</sup> cycle	2 <sup>nd</sup> cycle	1 <sup>st</sup> cycle	2 <sup>nd</sup> cycle
20 (heating)	14.087	10.996	10.996	13.459	10.599	13.861
50 (heating)	4.7076	4.2966	4.2966	10.834	2.6179	4.3809
100 (heating)	8 .2254	8.3001	8.3001	10.928	8.0652	13.025
150 (max.)	9.5383	4.7654	8.8357	10.902	8.7654	5.5987

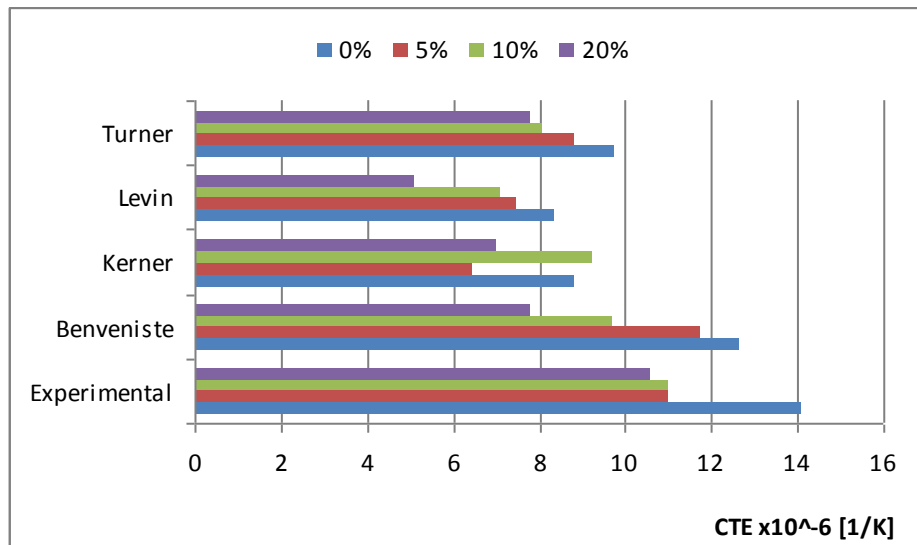
As it can be seen, the CTE values are different from thermal cycle to the next thermal cycle, and from sample to sample. The differences can be regarded to the fact that the more are the composite samples subjected to thermal cycles the more stable became their internal structure, the previous actually acting as an external thermal treatment were applied upon the samples.

The higher become the volume fraction of the second particle type constitutive (in our case carbon particles), the lower the overall CTE of the multiphase polymeric structure. As the temperature changes the instantaneous coefficient of thermal expansion experience different values that are not heading a linear variation as it is natural.

One can split the temperature interval in three main regions: *region I* - up to 50<sup>0</sup>, *region II* – between 50<sup>0</sup>C and 100<sup>0</sup>C, and *region III* – up to 150<sup>0</sup>C. In the *region I* the composite sample are following a natural behaviour and their instantaneous CTE variation with temperature is conducted by the internal behaviour of the structure as it came from the manufacturing process, with ups and downs at some temperature values.

With respect to the *region II*, the composite structure becomes more stable, the only influencing factor on the CTE variation being the one that corresponds to intrinsic property of the polymeric materials – their glass transition. For the *region III* – the thermal field and instantaneous CTE are experiencing a smooth variation, the internal structure and the intrinsic material properties of the constitutive having no evident influence.

In Fig. 3 are being represented the predicted values obtained for each class of multiphase composite material under the study using the micromechanical based approaches tackled herein along with the experimental retrieved data.



**Fig. 3** Instantaneous linear coefficient of thermal expansion – experimental vs. theoretical comparison (for all the particle-particle polymeric composite samples, function of the black carbon volume fraction)

As is can be seen there are differences among the values that can be regarded with the fact that the theoretical models do not encompass the presence of the void or the imperfect interfaces among the particles, nor describe the possible thermal chains within the structures. These theoretical models can be used during the new structures design process due to the fact that are pretty good in predicting the overall material property behaviour of the composite samples.

## 5. CONCLUSIONS

Thermal properties, such are the thermal conductivity or coefficient of thermal expansion, play an important role in the design, manufacturing and practical applications of multiphase composite materials, as the latter emerged as new materials with outstanding properties and resistance to different environmental regimes.

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 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, 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.

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