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Thermal expansion of epoxy and polyester polymer mortars—plain mortars and fibre-reinforced mortars

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Abstract

The study was conducted in order to determine the coefficient of thermal expansion of two specific binder formulations of epoxy and unsaturated polyester polymer mortars. The variation of this parameter with temperature was also analysed. Polymer concrete and mortars have been observed to have lower coefficients of thermal expansion at lower temperatures than at higher temperatures. Plots of strains vs. temperature are often bilinear, indicating a sharp change in the coefficient of thermal expansion (International Congress on Polymers in Concrete, July 1995). To determine how this discontinuity varies for these two materials, specimens of both formulations were tested for several temperature ranges between −20 and 60 °C. In addition, to determine the influence of fibre reinforcements on thermal expansion of polymer mortars, epoxy polymer mortars reinforced with both carbon and glass chopped fibres were also tested for thermal expansion.

It was concluded that, for both formulations, the variation of thermal expansion with temperature follows a parabolic law rather than a bilinear law. The reinforcement of chopped glass fibres (1%) has no significant effect on thermal expansion of epoxy polymer mortar, while the inclusion of carbon fibres (2%) on the same mortar formulation has a reducing effect on thermal expansion of this composite material for temperatures above room temperature.

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1. Introduction

Coefficients of thermal expansion are widely used for design purposes and to determine if failure by thermal stresses may occur when a solid body, composed of two different materials, is subjected to temperature variations.

Polymer concretes and mortars are often used to repair Portland cement concrete structures. It is well known that polymer concrete generally has a higher coefficient of thermal expansion than Portland cement concrete. In addition, the variation of thermal expansion of polymeric materials within the common temperature ranges is higher than that which occurs for hydraulic concrete. Therefore, thermal behaviour analysis of these repair materials, in terms of thermal expansion, is of crucial importance.

1.1. Usual test methods for coefficient of thermal expansion of polymer concrete and mortars

Different methods, based on different standards [2–4], are being used to determine the linear coefficients of thermal expansion of polymer concrete and mortars, and it seems there is no consensus between them. Length change measurement method, specimen geometry and the temperature range analysed are the main discussion subjects.

The test method described in ASTM standard E228-85 [2], covers the determination of linear thermal expansion of solid materials over the temperature range of

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−180–900 °C, using vitreous silica dilatometers. The method can be applied to mortars and composites, among other solid materials. A transducer (dial gauge or differential transducer) is commonly used for measuring the difference in length and a calliper is used to measure the reference length of the specimen. Length of specimen is dependent on the transducer accuracy, and the tube and rod type of dilatometer apparatus determines the dimensions of the specimen cross-section. The accuracy of this method is strongly dependent on the precision of length and temperature measurements, as well as the calibration of the dilatometer. In addition, the method requires a special device, not commonly available in research laboratories, and the specified specimen geometry is not easily obtained by a casting process.

Recently, the ASTM Standard test method for linear shrinkage and coefficient of thermal expansion of chemical-resistant mortars, grouts and monholitic surfaces, C531, was amended in order to extend its application to polymer concretes [3]. According to this method, thermal expansion of the material is evaluated through the mean coefficient of thermal expansion between room temperature and 100 °C. Length change is measured, through a micrometer, between special metal studs embedded in each end of the specimens (bars of square cross-section 25 × 25 mm and 250 mm length). The measurements are performed after the conditioning of the specimens, for at least 16 h, at each temperature level. The precision of this method has not yet been established. However, it is obvious that it will depend on user skill. The measurements at the highest temperature (100 °C) must be done very quickly, and the specimens must be removed, one at a time, at a rate that does not allow the temperature of the oven to drop below the established temperature. This method presents yet another drawback, which is the need to use special moulds that enable the moulding of bars with the metal studs embedded at each end. On the other hand, the mean coefficient between 23 and 100 °C, determined by this method, gives only a rough idea of thermal expansion behaviour of resin concretes and mortars.

RILEM PC-13 standard, presented by Technical Committee 113: Test-Methods for Concrete–Polymer Composites [4], also specifies a test method for coefficient of thermal expansion of polymer concrete and mortar. In this method, lengths are measured by means of a length comparator, which must be capable of pressing on gauge studs embedded on the sides of the specimens, at a gauge length of 250 mm. Prismatic specimens, with square cross-section of 100 and 400 mm length, are recommended.

Thermal expansion is analysed over the temperature range of 20–80 °C, with measurements stepwise at 20 °C intervals. A thermostatic water bath, with sufficient capacity for the immersion of the specimens, is used to control the temperature. Water temperature is raised at a rate of 20 °C/h and held constant at each step for 2 h. The mean coefficient of thermal expansion is calculated as the slope of the straight line that best expresses the relationship between thermal expansion and water temperature. This standard is not very clear about the way measurements should be done: with the length comparator also immersed in the thermostatic bath, removing the specimens to make the measurements, or providing studs with length enough to allow the placement of the length comparator outside the bath. Regardless of this last remark, and considering all possible interpretations, this test procedure does not seem very practical.

Some researchers, based on existing standards applicable to ceramic tiles and cement pastes, developed their own methods for determination of the coefficient of thermal expansion of polymer concrete and mortars [5,6]. However, these methods also present the inconveniences resulting from the mechanical process used to measure length changes of the specimens. Thermal strain of polymer concretes and mortars is normally not a linear function of temperature. Methods of measuring the expansion coefficient by averaging the thermal strain over a large temperature range of many degrees may result in failure to determine the variation in the coefficient of thermal expansion of these materials.

1.2. Alternative test method for coefficient of thermal expansion of polymer concrete and mortars

Previous studies, conducted by Wheat and co-workers [1], showed that length change measurements, needed to determine the coefficients of thermal expansion of polymer concretes, could be made by electrical strain gauges. This measuring process allows for continuously monitoring thermal strain as a function of temperature. Therefore, information on how coefficient of thermal expansion changes with temperature is obtained.

Ideally, a strain gauge bonded to a test part would respond only to the applied strain in the part, and be unaffected by other surrounding variables. Unfortunately, the resistance strain gauge, as all sensors in general, is somewhat less than perfect. The electrical resistance of the strain gauge varies not only with strain, but with temperature as well. This temperature-induced resistance change is independent of, and unrelated to, the strain induced by mechanical and/or thermal stresses in the test object to which the strain gauge is bonded. It is purely due to temperature change, and is thus called the thermal output of the gauge.

Two concurrent and algebraically additive effects cause this phenomenon, sometimes referred to as ‘temperature-induced apparent strain’. First, the electrical resistivity of the grid conductor is temperature dependent; and, as a result, the gauge resistance varies with temperature. The second contribution to thermal output is due to the differential thermal expansion between the grid conductor and the test part or substrate material to which the gauge is bonded. Because the grid is, by design, strain sensitive, the gauge exhibits a resistance change proportional to the differential expansion. The errors due to thermal output can become extremely large as temperatures deviate from the arbitrary reference temperature (ordinarily, room temperature). In addition to thermal output errors, the relation between strain and resistance change, the 'gauge factor', varies with temperature, introducing more measuring errors.

However, using self-temperature-compensated strain gauges, which employ specially processed alloys, it is possible to minimize the thermal output over a wide temperature range, as long as they are applied to test materials with thermal expansion coefficients for which they are intended. Depending upon the test temperature, higher accuracy can be obtained making additional corrections for the thermal output and gauge factor variation in the strain measurement. Engineering data sheets accompanying each package, series and lot of self-temperature-compensated strain gauges supplies polynomial equations for the corresponding thermal output curve and gauge factor variation with temperature [7]. It should be noted that the thermal output data are specifically applicable to the strain gauges of the designated lot number, applied to test material with the same coefficient of thermal expansion as the type of material used in determining the supplied thermal output curves.

To measure strains induced only by thermal effects, in a material not mechanically strained, self-temperature-compensated strain gauges indicated for use with test materials without thermal expansion can be applied. This way, the effect of resistance change with temperature is compensated; the thermal expansion of the grid conductor is also taken into account, but not the thermal expansion of the test part. Therefore, strain data obtained from tests are only due to the thermal expansion of test material.

Ordinary strain gauges, not self-temperature-compensated, can however be applied to measure strains induced by thermal effects, as done by Wheat et al. [1], and also recommended in ASTM D5335 [8]. However, this practice requires the use of a compensating (dummy) gauge bonded to a test material that has a known reproducible thermal expansion. On the other hand, this procedure yields less accuracy, as the thermal output error, due to the differential thermal expansion between the grid conductor and the test part, still remains.

In this research work, to measure the strains induced by temperature changes on polymer mortar specimens, strain gauges with 'constant alloy in self-temperature compensated form for structural materials with coefficient equal to zero', were used. This process of length change measurement provides more consistent and reliable results. Thermal expansion of polymer mortars was analysed over a temperature range of −15–60°C, with readings every 12 °C/13 °C steps. Prismatic specimens, with square cross-section of 40 and 100 mm length were used, but other geometries are possible (as long as the specimens have minimum dimensions at least twice the length of the strain gauges).

The method used in this study to analyse thermal expansion of polymer mortars is very simple to apply, provides good accuracy and allows a higher freedom in the choice of specimen geometry, presenting itself as a good alternative to conventional methods.

2. Experimental procedure

2.1. Binder formulations and test specimens

Plain mortar formulations were prepared by mixing foundry sand with an unsaturated polyester and an epoxy resin. Resin content was 20% by mass and no filler was added in either formulations.

The polyester resin used in this investigation was S226E (NESTE®), an unsaturated orthophtalic polyester diluted in 44% styrene. The resin system is pre-accelerated by the manufacturer and the initiator used was methyl ethyl ketone peroxide (2 phr).

The epoxy resin system was eposil 551 (SILICEM®), based on a diglycidyl ether of bisphenol A and an aliphatic amine hardener. This system has low viscosity, and is processed with a maximum mix to hardener ratio of 2:1.

Thermal and mechanical properties of both resins are presented in Table 1.

Foundry sand used in this study was a siliceous one, with very uniform grains and a mean diameter, d_{50}, of 342 microns. The sand was dried before adding to polymeric resins in an automatic mixer.

Fibre-reinforced epoxy polymer mortars were prepared in the same way as plain epoxy polymer mortars, with the incorporation of 1 and 2% by weight of chopped glass and carbon fibres, respectively.

The chopped carbon fibre used was HTA 5131 (TENAX®), with an epoxy size of 1.3%, and the chopped glass fibres were provided by PPG®, with no sizing and soaked in a 2% silane A174 aqueous solution. All chopped fibres considered were 6 mm in length. The mean coefficients of thermal expansion of these reinforcements, between 25 and 300 °C, are respectively, for glass and carbon fibres, 5.5 µm/m °C and −0.1 µm/m °C (values given by the supplier).

Previous studies by the authors [9–12], with an extensive experimental program, allowed an optimisation of the material formulation, as well as a complete mechanical characterization of binder formulations that are now being used in the present work.

Both reinforced and plain polymer mortars, with these binder formulations and mix proportions, were mixed

Table 1
Thermal and mechanical properties of polyester and epoxy resins

Resin properties (after one week at 25 °C)	Polyester resin (NESTE-S226E)	Epoxy resin (EPOSIL-551)
Glass transition temperature: DMA iso 6721-5	87 °C	45 °C
Heat distortion temperature: HDT iso 75	50 °C	34 °C
Tear strength[a] ISO 527	58 MPa	40 MPa
Flexural strength[a] ISO 178	119 MPa	70 MPa

[a] Mechanical properties given by the supplier.

and moulded to prismatic specimens 40 × 40 × 160 mm, according to RILEM standard TC113/PC2 [13]. All specimens were allowed to cure for 7 days at room temperature and then post-cured at 80 °C for 3 h, before being tested for thermal expansion.

2.2. Length change measurement

Length change measurements were made by strain gauges with constantan alloy in self-temperature compensated form for structural materials without thermal expansion. Flexible gauges with a cast polyamide backing, completely encapsulated, with 6.35 mm grid length were used. Three specimens of each mortar type were instrumented with one strain gauge placed longitudinally on one of their lateral faces.

In order to test the accuracy of the method for posterior validation, an aluminium alloy reference specimen (2017 4.0Cu–0.6Mg–0.7Mg–0.5Si), with a known coefficient of thermal expansion (mean coefficient of thermal expansion, between 20–100 °C: 23.6 μm/m °C), was also instrumented and tested [14].

Electrical strain gauge data were taken using two data loggers with a continuous acquisition system (spider-8) in eight channels. Strain gage data were registered with the gage factor setting of the strain indicator at 2.00. Corrections for the gage factor variation and for the thermal output were performed after data acquisition.

2.3. Specimens conditioning procedure

Three instrumented specimens of each type of plain and reinforced polymer mortar, as well as the aluminium alloy specimen, were placed in a climatic chamber at room temperature for 8 h. Strain indication on the data logger was then balanced for zero strain on all channels. Afterwards, the temperature was lowered by steps of 12.5 to −15 °C, then it was increased in six increments until +60 °C, and finally it was decreased by three steps to room temperature. The heating/cooling rate between temperature levels was approximately 0.28 °C/min, and in each step, temperature was held constant for 7.75 h, in order to allow the specimens to reach thermal equilibrium. All specimens were subjected to a total of three of these cycles.

2.4. Data treatment

Strain data used for determination of the mean coefficients of thermal expansion, for each temperature range, were taken from the last strain measurements at each temperature level or step. At those points, thermal equilibrium was already reached and the strain gage readings were constant. The obtained strain values were then corrected for the thermal output and gage factor variation according to the following equations:

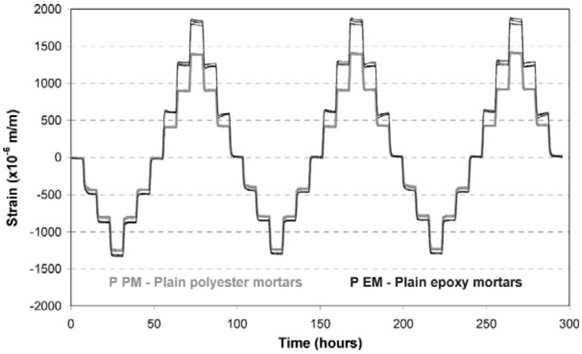

Fig. 1. Strain data of plain polymer mortars acquired by data logger throughout the three temperature cycles.

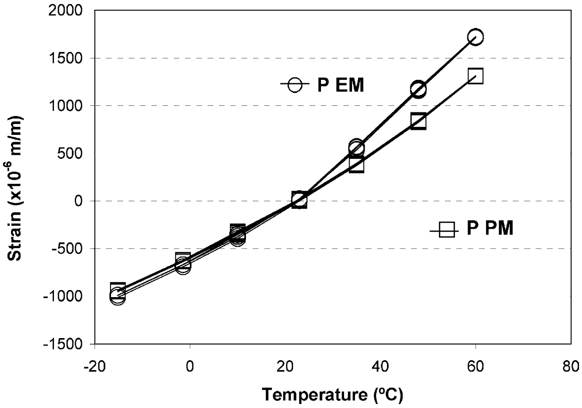

Fig. 2. Evolution of strains of plain epoxy (PEM) and polyester polymer mortar (PPM) specimens with temperature, during the three cycles.

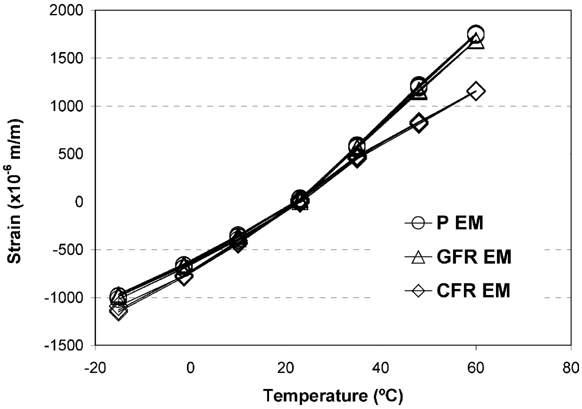

Fig. 3. Evolution of strains with temperature of plain (PEM), glass fibre-reinforced (GFREM) and carbon fibre-reinforced epoxy polymer mortars (CFREM), during the three cycles.

Table 2
Mean coefficients of thermal expansion of polymer mortars and aluminium alloy reference specimen

Temperature ranges [T_i, T_{i+1}]	Mean coefficient of thermal expansion α±S.D. (μm/m °C)				
	AL Alloy2017	PPM	PEM	GFREM	CFREM
[−15 °C, −2 °C]	22.36 ± 0.21	22.97 ± 0.06	23.45 ± 0.03	22.79 ± 0.08	26.46 ± 0.02
[−2 °C, +10 °C]	22.73 ± 0.26	25.39 ± 0.64	26.39 ± 0.76	25.88 ± 0.73	30.42 ± 0.72
[+10 °C, +23 °C]	22.36 ± 0.13	26.68 ± 0.54	29.94 ± 0.75	29.22 ± 0.61	33.97 ± 1.09
[+23 °C, +35 °C]	23.12 ± 0.09	30.86 ± 0.09	44.45 ± 1.50	45.51 ± 1.21	37.75 ± 0.44
[+35 °C, +48 °C]	23.10 ± 0.11	34.94 ± 0.20	47.33 ± 0.63	45.65 ± 0.26	27.88 ± 0.42
[+48 °C, +60 °C]	23.46 ± 0.10	39.08 ± 0.38	45.89 ± 0.62	43.97 ± 0.54	27.27 ± 0.88

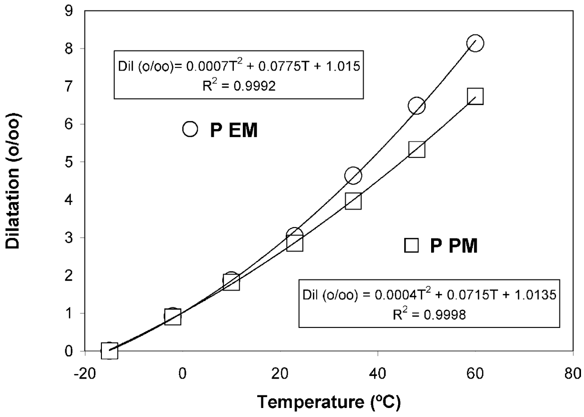

Fig. 4. Dilatation–temperature curves for both formulations of plain polymer mortars.

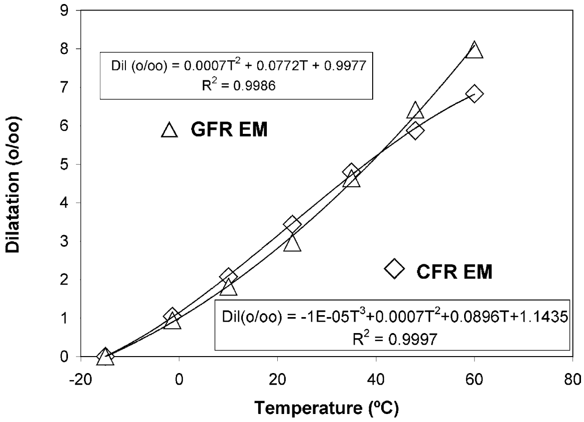

Fig. 5. Dilatation–temperature curves of fibre-reinforced epoxy polymer mortars.

$$\varepsilon = [\varepsilon_A - \varepsilon_{T/O}(T_i)].F^*/F(T_i) \qquad (1)$$

$$\varepsilon_{T/O}(T_i) = A_0 + A_1.T_i + A_2.T_i^2 + A_3.T_i^3 + A_4.T_i^4 \qquad (2)$$

$$F(T_i) = F_0.[1 + V_{\%}(T_i)/100] \qquad (3)$$

where ϵ_A, indicated strain, uncorrected for either thermal output or gage factor variation with temperature; ϵ, strain magnitude corrected for both thermal output and gage factor variation with temperature; $\epsilon_{T/O}(T_i)$, thermal output at temperature T_i; A_0, A_1, A_3, A_4, polynomial coefficients of thermal output curve of gage strain; $F*$, instrument gage factor set; $F(T_i)$, strain gage factor at temperature T_i; F_0, room temperature gage factor; $V_{\%}(T_i)$, variation of gage factor for temperature $T_{i.}$

The coefficients of thermal expansion, for each temperature range, were calculated by first fitting straight lines between adjacent points of consecutive measurements of corrected strain values and corresponding temperatures (Ei,Ti), and then determining the respective slopes, as described in Eq. (4).

$$\alpha_{(Ti,Ti+1)} = [\varepsilon(T_{i+1}) - \varepsilon(T_i)]/[T_{i+1} - T_i] \qquad (4)$$

where $\alpha_{(Ti,Ti+1)}$, mean coefficient of thermal expansion for temperature range between T_i and T_{i+1}; $\epsilon(T_i)$, $\epsilon(T_{i+1})$, correct strain magnitude at temperature T_i and T_{i+1}, respectively.

For each polymer mortar formulation and for each temperature range, the mean coefficient of thermal expansion considered corresponds to the average of eighteen values of $\alpha_{(Ti+1,Ti)}$ obtained as mentioned above ($2\alpha_{(Ti+1,Ti)}$ per cycle × 3 cycles × 3 specimens).

Dilatation–temperature curves were obtained considering, for each temperature range, the expansion of an elementary volume of test material when temperature is raised from –15 to 60 °C. For each temperature range, dilatation was calculated considering the correspondent coefficient of thermal expansion, as described in Eq. (5):

$$\Delta V_{(Ti,Ti+1)} = 3[\Delta T_{(Ti,Ti+1)}.\alpha_{(Ti,Ti+1)}] + 3[\Delta T_{(Ti,Ti+1)}.\alpha_{(Ti,Ti+1)}]^2 + [\Delta T_{(Ti,Ti+1)}.\alpha_{(Ti,Ti+1)}]^3 \qquad (5)$$

where $\Delta V_{(Ti,Ti+1)}$, expansion of an elementary volume of test material when temperature is increased from T_i until T_{i+1}. $\Delta T_{(Ti,Ti+1)}$, temperature variation between T_i and T_{i+1}.

3. Discussion of results

3.1. Mean coefficients of thermal expansion

Strain data acquired by data logger throughout the three cycles are plotted in Fig. 1, showing the different behaviour of polyester and epoxy plain polymer mortars when subject to temperatures changes. These data are not yet treated. In general, polyester mortars recover to equilibrium quite fast after temperature changes, while epoxy mortars require more time to stabilize and strain measurements present slightly more scatter.

For each type of polymer mortar, the evolution of strains and temperature during the three cycles are shown in Figs 2 and 3. Strains vs. temperature data for specimens of the same formulation are very similar, and when plotted on the same graph become almost coincident. In order to make the obtained results more clear, only the strain–temperature curve of one specimen of each type is presented.

Table 2 presents the mean coefficients of thermal expansion obtained for each temperature range, according to Eq. (4), with corresponding standard deviations.

As expected, both formulations of plain polymer mortar present higher coefficients of thermal expansion at higher temperatures. For temperatures between –15 and 10 °C, thermal expansion of plain polymer mortars are very similar, but above 10 until 60 °C, the increase rate of coefficient of thermal expansion of epoxy mortars is higher than that for polyester mortars.

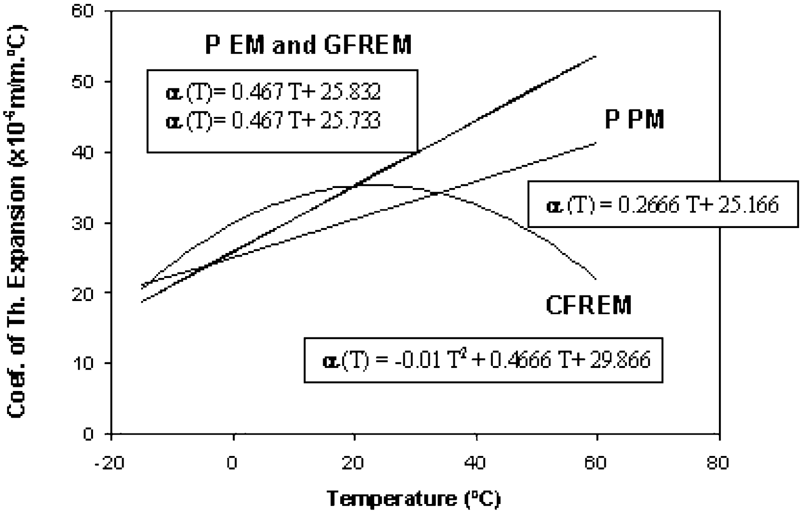

Fig. 6. Variation laws of instantaneous linear coefficient of thermal expansion with temperature of plain and fibre-reinforced polymer mortars.

The reinforcement of chopped glass fibres (1%) has no significant effect on thermal expansion of epoxy polymer mortars. On the other hand, for temperatures above room temperature, the incorporation of chopped carbon fibres (2%) has a strong reducing effect on the coefficient of thermal expansion of this composite material. The negative coefficient of carbon fibres is, certainly, the responsible factor for the partial hindering of positive expansion of epoxy mortars. For reasons we cannot explain, for temperatures below room temperature, chopped carbon fibres reinforcement has a slight raising effect on thermal expansion of epoxy mortars.

3.2. Dilatation–temperature curves

Dilatation–temperature curves are plotted in Figs 4 and 5. These curves were determined considering the expansion of an elementary volume of test material when temperature is raised from –15 to 60 °C. Isotropic behaviour for the test material was assumed. For each temperature range, dilatation was calculated considering the correspondent coefficient of thermal expansion, as already described in Section 2.4.

Dilatation–temperature curves, between –15 and 60 °C, of both formulations of plain polymer mortars and of glass fibre-reinforced epoxy mortars are more accurately predicted through an approximation to a parabolic law than a bilinear law. A third order polynomial law is, however, a better approximation for dilatation temperature curves of carbon fibre-reinforced epoxy mortars.

Considering the corresponding approximations, the variation of instantaneous coefficients of thermal expansion with temperature follow a linear law for plain polymer mortars and glass fibre reinforced epoxy mortars, and a parabolic law for carbon fibre-reinforced epoxy mortars, as illustrated in Fig. 6.

For practical use, dilatation–temperature curves can be approximated by bilinear laws. In this case, for plain polymer mortars and glass fibre-reinforced epoxy mortars, discontinuity points are located at 23 °C, while for carbon fibre reinforced epoxy mortars, this discontinuity is located approximately at 35 °C. Corresponding mean coefficients of thermal expansion, for temperatures below and above discontinuity points, are presented in Table 3.

3.3. Validation of the method

Evolution of strains as a function of temperature for the reference specimen used in this study, an aluminium alloy (1207), is plotted in Fig. 7. The known literature value of the coefficient of thermal expansion of this material is the mean coefficient between 20 and 100 °C: $\alpha_{(20\,°C,\,100\,°C)} = 23.6\ \mu\text{m/m}\ °C$.

Using the measured values of strain and temperature (T_i, ϵ_i), to calculate by interpolation and extrapolation the expected values of $\epsilon_{20\,°C}$ and $\epsilon_{100\,°C}$, and determining the corresponding slope of the straight line fitted between the two points, (20 °C, $\epsilon_{20\,°C}$) and (100 °C, $\epsilon_{100\,°C}$), the expected value for the mean coefficient of thermal expansion of the aluminium alloy, between 20 and 100 °C, obtained by this method, is 23.48 µm/m °C.

This calculation allowed the approximated assessment of the accuracy of the method. A small difference of 0.5% was found between the literature value and the expected value obtained by this method.

Table 3
Mean coefficients of thermal expansion of polymer mortars considering a bilinear law for dilatation–temperature curves

Polymer mortars	Mean coeff. of thermal expansion (μm/m °C)	
	−15 °C < T<23 °C	23 °C < T<60 °C
P PM	25	34
P EM	27	46
GFREM	26	45
	−15 °C < T<35 °C	35 °C < T<60 °C
CFREM	32	28

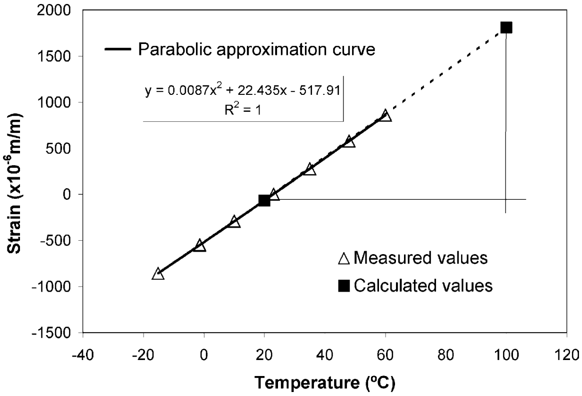

Fig. 7. Evolution of strains with temperature of aluminium alloy reference specimen. Calculation of expected value of its mean coefficient of thermal expansion between 20 and 100 °C.

4. Conclusions

The coefficients of thermal expansion of two specific binder formulations of polyester and epoxy polymer mortars were determined. The effect of chopped glass and carbon fibres reinforcement on the thermal expansion of epoxy polymer mortars was also analysed. Electrical self-temperature-compensated strain gages were used to measure length changes. The mean coefficients of thermal expansion were calculated and the variations of this parameter with the various temperature ranges considered were analysed.

Based on test results, the following conclusions can be drawn:

- Both formulations of plain polymer mortars present higher coefficients of thermal expansion at higher temperatures. For temperatures ranges between –15 and 10 °C, thermal expansion of both binder formulations are very similar, but above 10 °C, the increase rate of coefficient of thermal expansion of epoxy mortars is higher than that found for polyester mortars.
- The reinforcement of chopped glass fibres (1%) has no significant effect on thermal expansion of epoxy polymer mortars, while the incorporation of chopped carbon fibres (2%), for temperatures above room temperature, has a strong reducing effect on the coefficient of thermal expansion of this composite material.
- For both formulations of plain and reinforced polymer mortars, the variation of thermal expansion with temperature follows a polynomial law rather than a bilinear law. Therefore, it can be concluded that coefficients of thermal expansion of these materials vary continuously within the temperature range between −15 °C and +60 °C.
- However, for practical use, when a higher precision

is not required, the following values for the mean coefficients of thermal expansion of epoxy and polyester mortar formulations may be considered: for temperatures ranges below room temperature, the coefficient values of 25 and 27 μm/m °C for polyester and epoxy mortars, respectively; and for temperatures ranges above this point, the correspondent values of 34 and 46 μm/m °C.

The determination of the coefficient of thermal expansion of polymer mortars by this method is quite precise, with excellent accuracy for practical purposes. The method does not require any specific devices or special equipment and it is very simple to implement, which makes it a good alternative to the conventional methods.

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