

Experimental investigation on creep behaviour of an epoxy adhesive

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ABSTRACT: Epoxy adhesives have been extensively used in structural strengthening. This leads to a great concern in assessing their long-term performance since epoxy resins present viscoelastic behaviour. This work aims to better understand the long-term behaviour of a specific epoxy adhesive due to the creep effects. Therefore, an experimental program comprising tensile creep tests was carried out, divided in two series: (i) series S1 - epoxy specimens subjected to different stress levels for a predefined initial age; and (ii) series S2 - epoxy specimens subjected to equal stress levels but loaded at different ages. The paper presents the main results obtained so far, highlights relevant and corresponding conclusions.

1 INTRODUCTION

During the last three decades, several strengthening techniques with fibre reinforced polymers (FRP) have been proposed and used to improve the overall performance of existing structures and, namely in what concerns their load carrying capacity. In most of these techniques, such as externally bonded reinforcement (EBR) or near-surface mounted (NSM), the FRP materials are fixed to the structural member through epoxy adhesives. The adhesive is a load-bearing material that can transfer stresses without the loss of structural integrity. The main advantages of using structural adhesives are: (i) uniform stress distribution over a large area, minimizing the stress concentrations; and (ii) the connection of different materials, reducing the overall weight (Feng *et al.*, 2005). According to the literature, structural epoxy adhesives exhibit relevant creep deformations when subjected to sustained loads (Meyers *et al.*, 2008). This leads to a great concern in assessing their long-term load-bearing performance since epoxy resins present a viscoelastic nature, thus exhibiting a time-dependent behaviour (Feng *et al.*, 2005; Guedes, 2011). Moreover, the long-term behaviour of epoxy adhesives has been described as being strongly affected by the applied stress level and environmental exposure conditions, such as temperature and humidity (Costa *et al.*, 2012; Costa *et al.*, 2013).

Costa and Barros (2012) performed experimental tensile creep tests with an epoxy adhesive in which they observed considerable deformations in the tested samples due the creep effect during a period of 1000 hours of loading. The specimens were submitted to a constant stress state over time (stress level of 60%) under controlled environment (20°C and 60%RH). Meshgin *et al.* (2009) also carried out an experimental study in order to evaluate the response of epoxy at the concrete-FRP interface when subjected to sustained shear stress. The results showed that a significant creep deformation occurred in the epoxy within relatively short time period, as

compared to much lower delayed deformation observed in concrete. The magnitude of the applied shear stress and time-before-loading were significant factors in the shear creep behaviour of epoxy at the concrete-FRP interface.

The present work aimed to assess the long-term behaviour of a commercially available structural epoxy adhesive for application in the context of retrofitting existing concrete structures. Because of the high influence of this material in the overall structural response of strengthened structures along time, a specifically driven experimental programme comprising tensile creep tests, divided in two series, was prepared: (i) series S1, where the epoxy specimens were submitted to different stress levels at 7 days of age; and, (ii) series S2, in which the same level of stress was applied to distinct test specimens at distinct ages of loading. This paper describes the experimental program and presents the details of the main results obtained.

2 EXPERIMENTAL PROGRAM

2.1 *Material – epoxy adhesive*

The epoxy adhesive studied has the trademark “S&P Resin 220”, having been produced by S&P® Clever Reinforcement Company. This epoxy adhesive is a solvent free, thixotropic and grey two-component (Component A = resin, light grey colour and Component B = hardener, black colour). According to the manufacturer, after mixing the two components, the homogenized compound density is 1.70 to 1.80 g/cm³ and has the following mechanical properties: compressive strength >70 MPa; Young’s modulus >7.1 GPa; shear strength >26 MPa; adhesive tensile strength (at the end of 3 days at 20°C, when applied on concrete or on S&P laminate CFK) >3 MPa. The recommended application temperature is between +10°C and +35°C. Scanning electron microscope (SEM) with energy dispersive spectroscopy was used to determine the chemical composition of the constituents of the epoxy, which is described in detail elsewhere (Sena-Cruz *et al.*, 2013). From this analysis some mineral fillers, such as quartz, feldspar (SiAlK), barite (SBaO) and other silicates were observed.

2.2 *Experimental procedure*

In this experimental programme, 42 specimens of epoxy adhesive were prepared/tested. From this set of specimens, eight of them were subjected to sustained loads in order to study creep effects. The remaining ones were monotonically tested up to the failure in direct tensile. The tested specimens were manufactured according to ISO 527-2:2012. To assess the mechanical properties of the hardened adhesive, tensile tests were carried out according to ISO 527-1:2012. The geometry of the tested specimens is shown in Figure 1(a): the specimens are 4 mm thick, 10 mm wide in the region of interest, and have 115 mm of clear distance between grips. The specimens were cast into Teflon moulds, inside a climatic chamber at 20°C of temperature and 55% of relative humidity. All the three produced mixtures (one for series S1 and two for series S2) were made upon use of epoxy adhesive from the same container.

The experimental program was divided in two parts, namely series S1 and series S2. Each series includes four specimens submitted to a constant tensile stress in order to study the creep effect. Both creep tests (S1 and S2) were carried out in a climatic chamber, where the room temperature and relative humidity were 20°C and 55%, respectively. The main differences between series S1 and series S2 are the applied stress level and the ages of loading. All the specimens of series S1 were loaded at 7 days of age: two specimens were loaded at 20% of their maximum ultimate load and the remaining two specimens were loaded at 40% of the ultimate load. In the case of series

S2, all specimens were submitted to a constant stress level of 30% of the maximum ultimate load at different ages, namely, 1, 2, 3 and 7 days of age. As can be seen in Table 1, the specimens were labeled by S, C and D, i.e. S1_C20_D7 stands for series 1 (S1) subjected to a creep stress of 20% of the ultimate strength (C20) applied at 7th day (D7). A and B distinguish specimens in the same conditions. In monotonic tests, the labeling follows the same pattern.

The creep tests were performed using a mechanical system, where the specimens were subjected to constant stress level through application of gravity loads. The mechanical system was based on a lever structure (Meshgin *et al.*, 2009). In order to evaluate the dead weight for application during the creep tests, monotonic tensile tests up to the failure were performed with equal specimens at same age. The monotonic tests were carried out in a universal testing machine under displacement control, at a rate of 1 mm/min, according to EN ISO 527-1:2012. The applied load was assessed by a load cell with 50 kN of maximum admissible load (with a linearity less than 0.5%). The mid-height axial strain was measured with a TML strain-gauge (SG) type BFLA-5-3-3L (5 mm of measuring gauge length). Five samples were tested for each age.

After demoulding the specimens, their thickness and width were measured with a digital clipper (0.01 mm of precision) in the three distinct sections (A, B and C), identified in Figure 1a. Based on such measurements, the average cross-section area was calculated. Thereafter, the creep specimens were instrumented with two strain-gauges (type TLM BFLA-5-3-3L), glued at the middle height of the specimen, in opposite faces as shown in Figure 1b, connected to NI Compact DAQ, using NI 9235 strain-gauge module for data acquisition. After knowing the results for the ultimate strength, dead weights were carefully evaluated for each series, then the specimens were carefully loaded (see Figure 1c).

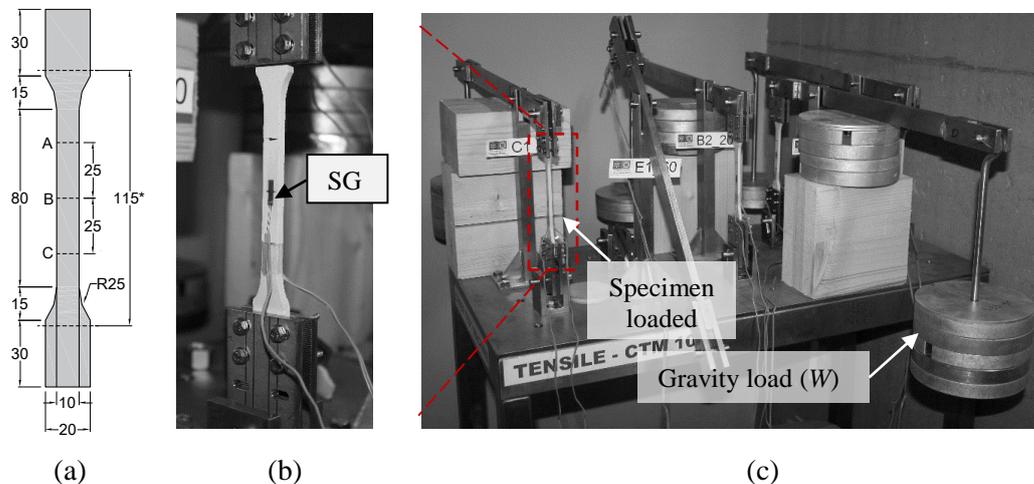


Figure 1 – Specimen and creep test setup: (a) Geometry of specimen according to ISO 527-2:2012 (*distance between grips); (b) detailed view of a specimen and grips; (c) overview of mechanical creep tests including the gravity load.

The main properties of creep tests are shown in Table 1. For each specimen tested, the measured cross-sectional area (A) was considered and the corresponding applied stress (σ) was defined, based on the results of monotonic tensile tests (section 3.1). The gravity load to be applied to each sample (W) was calculated based in Eq. 1.

$$\sigma = \frac{3 \cdot W \cdot g}{A} \quad (1)$$

However, it was not possible to apply exactly the predefined stress level with the available standard weights at the lab, then an approximation had to be used. In Table 1 one can find the percentage of the real creep stress applied ($\%f_{ult}$), based on dead weight effectively applied ($W_{applied}$). Likewise, the stress applied experimentally ($\sigma_{applied}$) was calculated. These values were very close to the percentages initially establish: 20% and 40% in the case of series S1 and 30% in the case of series S2.

The duration of creep tests of series S1 was about 85 days (2040 hours). Afterwards, the specimens were unloaded and the creep deformation recovery was monitored. The creep tests of series S2 are still ongoing at the date of submission of this paper (April 2015), with schedule to keep running for a total duration of at least 1000 hours. This period of time is recommended by ISO 899-1:2003 and ASTM D 2990-09 standards.

Table 1 – Properties of tensile creep tests of series S1 and S2.

Series	ID specimen	A	$W_{applied}$	$\sigma_{applied}$	$\%f_{ult}$
		[mm ²]	[kg]	[MPa]	[%]
S1	S1_C20_D7_A	46.77	7.30	4.59	19.9
	S1_C20_D7_B	44.73	7.29	4.79	20.8
	S1_C40_D7_A	45.98	14.38	9.19	39.9
	S1_C40_D7_B	45.57	14.28	9.22	40.0
S2	S2_C30_D1	44.60	8.39	5.54	29.7
	S2_C30_D2	42.77	10.51	7.23	29.9
	S2_C30_D3	44.43	10.18	6.74	29.9
	S2_C30_D7	44.76	11.21	7.37	29.9

3 RESULTS AND DISCUSSION

3.1 Tensile properties

Figure 2 represents the stress-strain curves obtained at various ages for both series. Table 2 includes the main properties obtained in the monotonic tensile tests, namely, ultimate tensile stress (f_{ult}), elastic modulus (E_{adh}) and ultimate strain (ϵ_{ult}). In series S1, a slight increase of the ultimate strength of epoxy adhesive can be observed between the test performed at the age of 7 days and 85 days (age of unloading of the creep tests of series S1), approximately 11%. However, the evolution of the elastic modulus was negligible (about 3%), as can be seen in Table 2. Young's modulus was calculated as the slope of the secant line between strain values of 0.05% and 0.25% on the stress-strain curve obtained in monotonic tensile tests, according to the recommendations of ISO 527-1:2012. Results of series S1, tested at the end of 7 days of curing, are in accordance with the ones found in the literature (Sena-Cruz *et al.*, 2013).

In Figure 2b it can be observed the curing process evolution of epoxy adhesive from the increase of stiffness at different ages by the slope difference of the curves. This difference was lower between 2 and 7 days of age rather than between 1 and 2 days. From this data is possible to verify that curing reactions happened until at least 3 days of age. As can be seen in Table 2, this effect

was shown in the values of Young's modulus of series S2. The Young's modulus at the age of 1 day was about 4 GPa and increased about 50% up to 3 days of age.

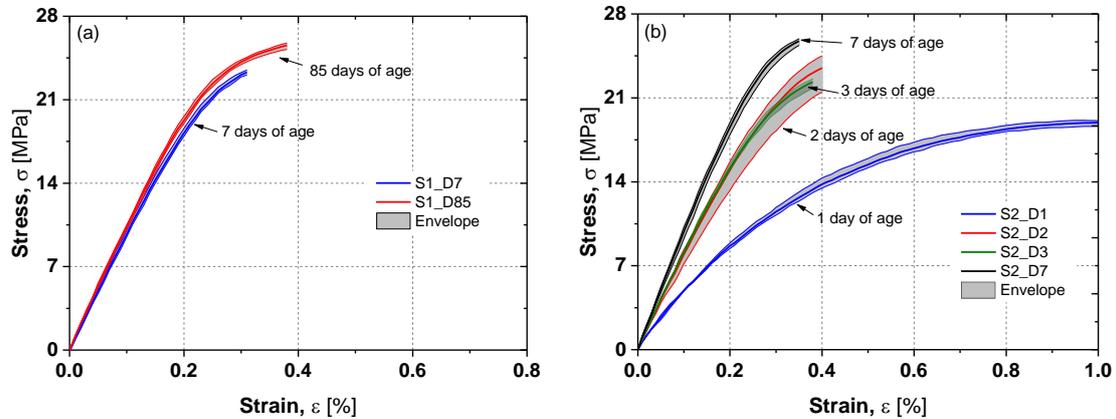


Figure 2 – Stress *versus* strain obtained from tensile tests at different ages: (a) series S1; and (b) series S2.

Table 2 – Results of the tensile tests of series S1 and S2 at different ages (average values).

Series	ID	A_{average} [mm ²]	f_{ult} [MPa]	E_{adh} [GPa]	ϵ_{ult} [%]
S1	S1_D7	46.06 (2.6%)	23.06 (4.4%)	8.27 (1.2%)	0.313 (12.8%)
	S1_D85	44.70 (3.7%)	25.61 (1.4%)	8.54 (1.0%)	0.442 (8.4%)
S2	S2_D1	45.10 (0.6%)	18.65 (3.2%)	3.73 (3.8%)	0.955 (15.6%)
	S2_D2	43.93 (1.5%)	24.17 (3.9%)	6.99 (6.0%)	0.496 (16.6%)
	S2_D3	44.90 (0.9%)	22.51 (3.2%)	6.93 (2.1%)	0.432 (9.6%)
	S2_D7	45.53 (2.6%)	24.64 (7.5%)	8.44 (2.2%)	0.342 (17.7%)

The maximum ultimate stresses of both series were very similar, with only the exception of specimen tested at the age of 1 day. On the other hand, just from at the age of 7 days similarity between Young's modulus of series S1 and S2 was observed. Then, one can observe that the evolution of maximum ultimate strength was attained more quickly than stiffness of epoxy adhesive. These results are in agreement with others research works (Fernandes *et al.*, 2015).

3.2 Creep behaviour

3.2.1 Series S1

Figure 3a shows the creep response of the specimens of series S1 submitted to a constant stress level and the recovery stage after unloading. The respective creep compliance-time curves are represented in Figure 3b. In Figure 3a, it is possible to observe good coherence between specimens A and B (same type of specimen at the same conditions). The values of elastic strains calculated based on E-modulus obtained in monotonic tensile tests were marked with points in the same figure (represented in Figure 3a). It can be observed the relation between the elastic strains calculated for the series S1_C40 are not exactly the double of series S1_C20. These variations can be related with a slight misalignment between lever and specimen in mechanical system itself,

minor variation in the cross-section geometry of the specimens and the intrinsic variations in the material properties.

During the experiment period under applied load, the evolution of creep strain was affected by temperature variation within the climatic chamber ($\pm 2^\circ\text{C}$) due to the high thermal expansion coefficient of epoxy adhesives ($\approx 41\text{E-}06/^\circ\text{C}$). Therefore, compensation of the measured strain was necessary to take into account the temperature effect. Creep coefficient was calculated based on the ratio $(\epsilon_{\text{max}} - \epsilon_{\text{elast}})/\epsilon_{\text{elast}}$. For the present experimental program, the calculated creep coefficients were 1.2 and 1.7 for specimens with 20% and 40% of stress level, respectively. These values and the difference between the creep compliances observed in Figure 3b would possibly point to a lack of homogeneousness of the viscoelastic behaviour of this epoxy adhesive. For a linear viscoelastic material, the creep compliance is independent of the stress levels which means that the compliance-time curves at different stress levels should coincide (Guedes, 2011).

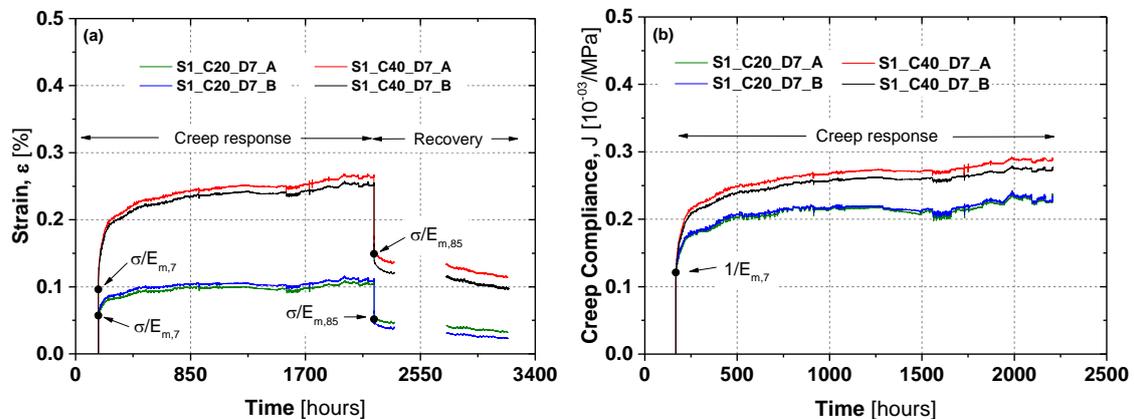


Figure 3 – Creep tests of series S1: (a) creep response and recovery of specimens; (b) creep compliances.

After unloading (at the age of 85 days), the elastic strain was practically recovered, with exception of the specimen S1_C20_D7_A, as shown in Figure 3a. According to the linearity of a viscoelastic material, this implies that the linear superposition principle was verified (Guedes, 2011). During the phase of recovery, the specimens presented also a kinetic evolution of deformation similar to the creep response upon loading. Considering the strain recovery up to the moment of writing this paper, the specimens of 20% and 40% of stress level recovered in average 58% and 37% of strain, respectively.

3.2.2 Series S2

Figure 4a shows the evolution of strains during the creep tests of the specimens loaded at different ages. The values of the elastic strains represented as σ/E were calculated based on the E-modulus obtained from monotonic tensile tests at different ages. These values were very close to the elastic strains measured in the creep experimental tests. Although the elastic strain of specimen loaded at 1 day of age seems very high, the magnification of the response at the first minutes show the opposite (see Figure 4c). The evolution of creep strain of this specimen was only considerable until up to 80 hours, approximately. In fact, two relevant phenomena occurred simultaneously from the instant of loading: curing reactions of material and creep effected. This behaviour can be also observed in the remaining specimens (with different ages), but less significant. The creep coefficient decrease at ages of loading more advanced (see Figure 4b), as it happens in others materials such as concrete.

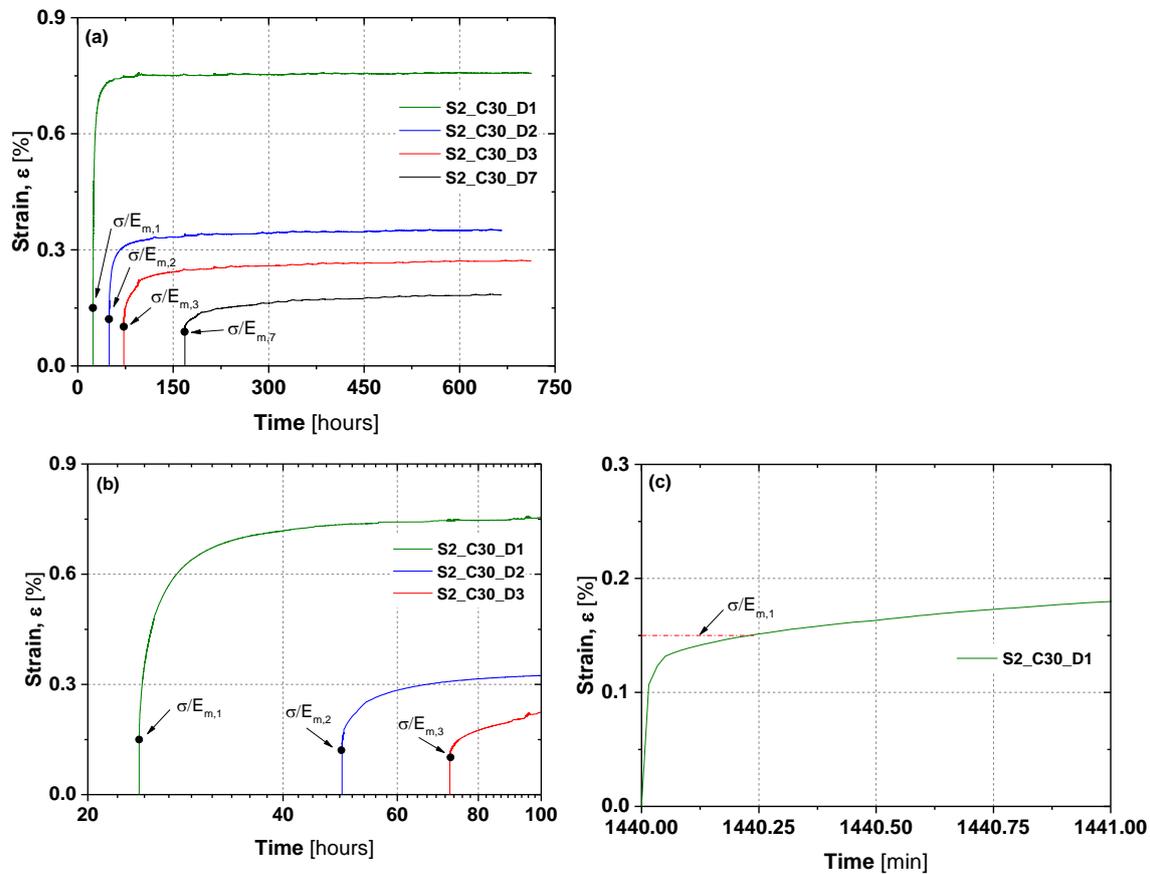


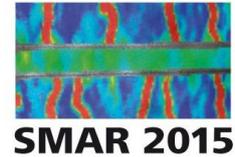
Figure 4 – Creep test at different ages: (a) specimens loaded at 1, 2, 3 and 7 days of age; (b) detail of creep response of 1, 2 and 3 days of age; (c) detail of elastic strain of the specimen loaded at 1 day of age.

4 CONCLUSIONS

The present paper gave contributions on the assessment of creep behaviour of a structural epoxy adhesive used in FRP strengthening applications. Experimental tests were performed in two series, in which the first different stress levels were assessed with a cured epoxy adhesive, and in the second different ages of loading were evaluated. The main conclusions were the following: (i) in both series the obtained elastic strains were coherent with the ones calculated by monotonic tensile tests; (ii) specimens loaded at the end at 7 days of age showed a behaviour that deviated from homogeneity; (iii) after the unloading of the specimens, the elastic strain were practically recovered, allowing to verify the linear superposition principle; (iv) the creep coefficient was decreased for ages of loading more advanced. Analytical simulations related with the experimental results here explained are ongoing. The simulations have been carried out with mechanical models that can describe the viscoelastic polymer behaviour, to better understand the physical phenomena of creep and the relationship between stress and strain for viscoelastic materials.

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