STABILITY OF ROOM TEMPERATURE CURE EPOXY ADHESIVES FOR TIMBER STRUCTURES UNDER CREEP LOADING IN TENSION AND SHEAR

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ABSTRACT: Ambient temperature cure adhesives are commonly used for the repair and reinforcement of timber in buildings and may also be used where bonded-in steel or composite pultruded rods and plates are employed to make connections in timber structures. The thixotropic behaviour of such adhesives is an essential factor when overhead repairs or reinforcements are being made in the field. Shear thinning characteristics are an important feature of such adhesives but glass transition temperatures (Tg) tend to be low and generally not much greater than room temperature. This paper addresses the issue of why connections bonded-in with thixotropic epoxy adhesives do not fail under constant creep loads in the field despite temperatures exceeding the material’s Tg. A series of creep tests have been performed on adhesive samples under constant creep load in a dynamic mechanical thermal analyser (DMTA) whereby the temperature is increased in steps up to 80°C. The adhesive behaves as an elastic solid below Tg, as a viscoelastic solid just above Tg and as a low modulus, partially cross-linked, rubbery polymer at higher temperatures. Creep resistance is therefore a function of the cross-linked molecular structure of these adhesives. The assumption that the adhesives will creep uncontrollably above Tg is unfounded. The results of testing a miniature wood/adhesive/wood sandwich in shear in the DMTA are also reported, confirming the rubbery behaviour of the adhesive above Tg.

KEYWORDS: Ambient cure epoxy adhesives, thixotropy, glass transition temperature, creep, DMTA, timber.

1 INTRODUCTION

The application of adhesives and sealants in construction is widespread and thermosetting and thermoplastic polymers are key constituents of wood panel products, glue-laminated timber and double-glazed timber windows. Adhesives are increasingly being used to bond together primary elements of timber structures including bridges and buildings where threaded steel or pultruded composite rods are bonded into timber sections to transfer load. Bonded-in rods may be retrofitted in the reinforcement of timber bridges and used for the construction of new timber bridge decks or used to bond new sections onto the end of decayed beams (Figure 1) for the repair of historic buildings [1,2]. A precise quantity of adhesive is injected into oversized drilled holes or routed slots in the timber and the rods or pultrusions are inserted into the holes or pressed into the slots.

Unlike aerospace structures, which are mostly based on high temperature cure epoxy matrix composites cured in autoclaves at about 180°C, timber structures are bonded together with ambient temperature cure adhesives, which are also mostly based on epoxy adhesives. In the case of aerospace epoxies a highly cross-linked structure is developed in a matter of a few hours and the adhesives behave elastically and rigidly over their working temperature range. Ambient temperature cure, low exotherm epoxies for bonding timber are left to cure for days rather than hours to ensure that adequate cross-links have formed within the adhesive and that the adhesive is well bonded to the timber and the rod. However, a heavily cross-linked structure is unlikely so these adhesives may be characterised by a glass transition temperature, Tg, at the transition between elastic and viscoelastic behaviour.

Figure 1: CFRP plates bonded into timber for beam end repair [1]. ©Rotafix Ltd.
Some ambient temperature cure epoxy timber adhesives are heavily filled with ceramic micro-particles such as mica, bentonite and silica [3] which raise the Young’s modulus and the glass transition temperatures to above 50°C. Other adhesives are designed to be thixotropic (shear thinning) in their uncured state, especially where overhead injection of adhesive is necessary. Once cured the Tg of shear thinning adhesives is often of the order of 30-40°C although post curing will increase Tg to a modest extent [4]. Specifiers of adhesives may be unwilling to select an adhesive for bonding in rods with a low Tg on the basis that creep under constant load is likely to occur at elevated temperatures and under high stresses [5]. However Richter and Steiger [6] report no significant loss in strength and only minor creep deformation in epoxy adhesives used to bond CFRP to wood up to 50°C. Experience, over many years, of using epoxy adhesives to bond in rods in construction applications demonstrates that no failures have occurred in creep. The paper reports on the mechanical response of a low Tg, thixotropic epoxy adhesive subjected to multi-stage creep loads in a dynamic mechanic thermal analyser (DMTA). Properties are evaluated in tension and also in shear using a DMTA specimen with a unique micro-shear geometry.

2 EXPERIMENTAL METHODS

2.1 MATERIALS

The adhesive system under investigation is marketed as Rotafix Structural Adhesive (RSA) and is manufactured by Rotafix (Northern) Ltd of Abercraf, South Wales, UK. RSA is a thixotropic, non-Newtonian two-part, epoxy gap-filling adhesive system that is generally used in the thickness range 2-12 mm. It is formulated to form a strong bond between timber and GFRP or CFRP pultruded rods. The base adhesive is a mixture of diglycidylether of bisphenol-A + (DGEBA/F) and mono- and di-functional reactive epoxy diluents together with treated silica fume nanoparticles which control thixotropy. The curing agent contains polyetheramines combined with a rheology modifier. The nanostructure of RSA is imaged in Figure 2 using transmission electron microscopy.

The dispersion of nanosilica particles is quite even (Figure 2a) because they are coated with a hydrophobic silane to prevent gross clustering. Particle sizes are ~ 20nm in diameter (Figure 2b).

2.2 DYNAMIC MECHANICAL THERMAL ANALYSIS (DMTA)

The assessment of the dynamic mechanical properties of the RSA adhesive and deformation under creep load was performed with a Tritec 2000 DMTA manufactured by Triton Technology of Keyworth, Nottinghamshire, UK. The clamping arrangement for adhesive and laminated specimens is shown in Figures 3a and 3b respectively. All measurements of creep properties were made in tensile mode as shown. The DMTA was used in dynamic mode to perform thermal scans from -60°C (below the glass transition temperature) to 100°C in order to measure the storage modulus (E’), loss modulus (E") and the loss tangent (tan δ) as a function of temperature. Specimens were 3mm thick by 5mm wide with a gauge length between the clamps of 15mm. A frequency of 1Hz was employed with a heating rate of 2°C/min and liquid nitrogen was used as the cryogenic coolant. The glass transition temperature was deduced by drawing tangents to the elastic and viscoelastic portions of the storage modulus characteristic and recording the temperature at their point of intersection.

Creep tests on RSA specimens were performed in static mode using software that allowed a constant force to be applied to the specimen in tension. The temperature was held at 20°C for periods of 30 or 120 minutes and then ramped up in steps of 5°C with the dwell periods repeated until 80°C was reached. The output from the tests was deformation as a function of temperature and time and the deformation was converted into tensile creep strain based on the original gauge length. Creep was also performed in shear using the specimen geometry described in Section 2.3.

Figure 2: Nanostructure of RSA adhesive (a) scale bar = 2µm (b) scale bar = 0.1µm.

Figure 3: Tritec 2000 DMTA showing (a) RSA sample clamped in tensile mode and (b) veneer/RSA/veneer sandwich clamped in tensile mode to perform shear tests.
2.3 SPECIMEN GEOMETRY FOR CREEP IN SHEAR

The shear modulus of materials is well known to be less than the tensile modulus and in the case of bonded-in rods the interaction between the rod and its adhesive envelope is essentially in shear. Hence a miniature shear specimen was designed to simulate the bonded interface (Figure 4) by laminating an RSA adhesive layer (2mm) between two beech wood veneers (each 0.5mm thick).

Figure 4: Miniature laminated veneer/adhesive/veneer specimen with offset cuts for creep in shear.

The beech wood veneers were attached to glass plates with double-sided adhesive tape and the RSA adhesive was mixed and pressed between the glass plates. Following cure of the adhesive the specimens were cut from the laminate using a fretsaw. Offset cuts were then made through one layer of veneer and through the adhesive thickness on opposite sides of the specimen. Hence an isolated zone of adhesive, 6mm in length (Figure 5), could be subjected to static shear in the DMTA through the application of a tensile force to the ends of the specimen.

Figure 5: Dimensions of the miniature laminated veneer/adhesive/veneer specimen.

Shear strain was estimated by dividing the specimen elongation by the thickness of the adhesive layer.

3 EXPERIMENTAL RESULTS

3.1 THERMOMECHANICAL PROPERTIES OF RSA IN TENSION

The thermomechanical properties of RSA, measured in the DMTA, are presented in Figure 6 where the storage modulus and loss tangent are plotted as a function of temperature.

Figure 6: DMTA thermal scan for RSA in tension.

RSA behaves as a classic viscoelastic material with a storage modulus of 1.3GPa in the elastic zone at -60°C falling, via the viscoelastic transition, to ~0.02GPa in the rubbery zone above a temperature of approximately 55°C. The glass transition temperature is calculated to be 34.5°C based on the intersecting tangent construction discussed in Section 2.2. The loss tangent characteristic peaks at 50.5°C with a peak tan δ value of 0.96 which reduces to a value slightly greater than zero at 100°C. Hence at first sight the exposure of RSA to temperatures greater than 35°C might be assumed to result in significant creep as a result of the substantial fall in the dynamic storage modulus. However the application of static load in a multi-stage creep experiment demonstrates that this is not the case.

3.2 MULTI-STAGE CREEP RESULTS IN TENSION

RSA specimens were loaded with a static force of 1.75 N (tensile stress = 0.12 MPa) and the temperature was raised in 5°C steps from 25°C up to 80°C with a dwell time of 30 minutes at each temperature step (Figure 7). At 25 and 30°C the strain experienced in the sample is very small but at 35°C there is a clear indication of time-dependent creep with acceleration in creep rate at 40°C.

Figure 7: DMTA thermal scan for RSA in tension with 30min dwell times.

The shape of the creep increments is not classically exponential but nevertheless the creep rate diminishes
with time at the end of each creep increment (Figure 8) suggesting that an equilibrium strain will be reached.

**Figure 8:** DMTA thermal scan for RSA in tension with 30min dwell times showing creep steps between 30 and 50°C.

At 45°C, following a small increase in strain in response to the increase in temperature of 5°C the strain settles down to a constant strain value. Thereafter each step of increase in temperature results in a small increase in strain which settles down to constant value (Figure 9).

**Figure 9:** DMTA thermal scan for RSA in tension with 30min dwell times showing creep steps between 60 and 75°C.

In summary the RSA adhesive behaves as an elastic solid below Tg and as a viscoelastic material, creeping to a limit, between Tg and (Tg + 15°C). Above (Tg + 15°C) the strain in RSA increases by approximately 0.2% for each 5°C increase in temperature which is equivalent to a thermal expansion coefficient $\alpha$ approximately equal to $4.10^{-4}$°C$^{-1}$.

**Figure 10:** Strain plotted versus temperature for RSA unloaded and clamped in tension mode.

In order to understand this expansion effect two experiments were performed in the DMTA to measure the thermal expansion coefficient of RSA unloaded. In one experiment the temperature was ramped up with no load applied to the sample which was clamped in tension mode and the strain measured (Figure 10). In the second case a tension sample was exposed to temperature steps of 1°C ramped up in increments at one minute intervals with a very small constant load (0.002N) applied to maintain control of the experiment in creep mode. The creep programme in the DMTA allows a maximum of 40 creep steps so the data generated is more limited. Strain is plotted versus temperature in Figure 11.

**Figure 11:** Strain plotted versus temperature for RSA clamped in tension mode using the creep programme.

In Figures 10 and 11 there is a change in slope at approximately 45 to 50°C corresponding to the end of the viscoelastic zone at ~(Tg + 15°C). The data in Figure 11 is converted into a plot of thermal expansion coefficient versus temperature and a peak value of $\alpha$ occurs at ~42°C.

**Figure 12:** Thermal expansion coefficient plotted versus temperature for RSA based on data from Figure 10.

There is a good correlation between the $\alpha$ values predicted from Figure 9 and those plotted in Figure 12. Hence once viscoelastic deformation has occurred from ~45°C onwards, most of the extension is due to thermal expansion. To place this behaviour in context, heavily cross-linked epoxies have thermal expansion coefficients of 0.4 to 0.6.10$^{-4}$°C$^{-1}$ and natural rubber ~2.2.10$^{-4}$°C$^{-1}$.

It is worth considering whether RSA behaves as an elastic material (very unlikely), a rubber, a partially cross-linked rubber or a viscous polymer above Tg. A fully cross-linked rubber (e.g. polyisoprene) is expected to display entropy-driven behaviour above Tg such that the rubber will contract under constant creep load as the temperature is increased whilst the rubber will expand when heated below Tg. Evidence from Figures 7 to 9 suggests that above (Tg + 15°C) RSA expands in increments as the temperature increases in 5°C steps.
rather than contracting so it certainly does not behave like a fully cross-linked natural rubber. Evidence is therefore sought of semi-rubbery behaviour with partial cross-linking.

In Figure 13 the previous creep experiment was repeated with the same 5°C temperature steps but with longer dwell times of 120 minutes. As before little deformation occurs until the 35°C step but at the end of the 45°C step and at the end of each step thereafter the creep strain settles down to an almost constant value.

As the creep steps are increased from 50°C up to 80°C an interesting trend develops. The initial increases in strain which might be expected to result from thermal expansion are followed by a linear decline in strain at each constant temperature step. This trend, shown in an the expanded plot (Figure 14) for the steps between 60 and 75°C, demonstrates that there is strain recovery under constant creep stress which is likely to be associated with a partially cross-linked structure.

This entropy-driven contraction could be retarded by other features of the molecular structure that behave viscoelastically. Silica fume is used to control the thixotropy of the uncured adhesive in conjunction with mono-functional and di-functional reactive epoxy diluents. Hence a loosely cross-linked rubbery structure is feasible and rubber-like behaviour is manifested with time. The effect is also more obvious for the higher temperature steps as one might expect.

Inspection of Figures 7 and 13 for the 30min and 120min dwell periods respectively indicates that the final creep strain at the end of the 80°C step is very similar with a value of just over 3.5%. Hence the final equilibrium strain at 80°C is independent of the thermal history, again indicating partial cross-linking and some rubbery character to the deformation.

### 3.3 MULTI-STAGE CREEP RESULTS IN SHEAR

Novel miniature shear specimens (Figures 4 and 5) were subjected to multi-stage creep in the DMTA and the clamped specimen is imaged in Figure 2b. The specimens were loaded in tensile mode but the offset cuts caused shear strains to develop across the central adhesive zone which had a thickness of ~2mm, length of ~6mm and width of ~5mm. These quantities were measured accurately for each specimen.

The same temperature versus time history was imposed on the specimen as for the tensile sample in Figure 6, with an applied constant static force of 1.75N (shear stress = 0.06MPa). Shear strain and temperature versus time are plotted in Figure 15 from 20 to 80°C.

At the end of the creep history the strain reaches a value of 7% which is not unexpected as the shear modulus of
RSA is lower than the tensile modulus. In Figure 15 a slight reduction in shear creep strain is evident in the last 200 minutes of the experiment. The plot is expanded in Figure 16 for the dwell periods between 60 and 75°C. There is evidence of creep recovery within each loading step as there was for tensile creep. The magnitude of the strain steps at high temperatures is much less than it was for the tensile creep experiments (Figures 7 and 9) because the shear sample will be little affected by thermal expansion as the temperature increases in steps. The shear creep experiments were repeated with 120 minute dwell periods and the results are plotted in Figure 17.

The overall trends are similar to those of Figure 15 for the 30 minute dwell periods but the initial stages of shear creep are condensed by the longer timescale. From 60°C onwards there is a slight rising trend in the creep strain which is amplified in Figure 18. It should be appreciated that the strain scale covers a very small range of values. An initial increase in strain at the onset of each increase in temperature is followed by a further increase in shear creep strain for the 75 and 80°C steps. This behaviour is a function of the geometry of the shear test.

\[ \varepsilon(t) = \varepsilon_0 \exp\left(-\frac{t}{\tau}\right), \]

where \( \varepsilon_0 \) is the equilibrium strain and \( \tau \) is the characteristic retardation time which is the ratio of the dashpot viscosity to the spring constant. Evidence from Figures 6 and 11 suggests that this model is inadequate for the description of creep in RSA in the temperature range \( T_g \) to \( (T_g + 15°C) \) where the material appears to behave as a partially cross-linked, semi-viscoelastic material. An equation with two additive exponential creep terms representing two simultaneous deformation mechanisms is likely to be more appropriate for modelling the behaviour of RSA in creep.

4 DISCUSSION

This paper has been concerned with the time- and temperature-dependent properties of a thixotropic, ambient cure epoxy adhesive used for bonding rods into timber structures. Pull out of rods bonded into wood cubes with RSA [4] requires a shear stress of \(~6\text{MPa}\) to failure so the shear stress applied to the DMTA samples is only 1% of this value. Working shear stresses in the adhesive layer surrounding bonded-in rods will be much less than 6MPa especially where the bonded-in lengths are extensive (up to 3m for glulam beam repairs). Although the DMTA can apply a creep force of up to 10N it is still necessary to run laboratory scale tests on bonded interfaces at design stresses. In current work the thick adherend shear test (TAST) [7] is used to assess timber to timber bonded interfaces in creep under constant load as a function of temperature and relative humidity in an environmental chamber. The same creep conditions will be imposed on pultruded and threaded steel rods bonded into timber substrates. Despite the small stresses applied to tensile and shear creep tests in the DMTA, the results have demonstrated that the RSA adhesive behaves as a low modulus partially cross-linked rubber above \( T_g \) and that creep occurs to a limit. The low modulus is an advantage in a construction context where the temperature in roof spaces may rise to 50°C or more. The inherent energy absorbing capability of the adhesive layer is an advantage under conditions of shock load and in earthquake zones. Conventional viscoelastic polymers are modelled with combinations of springs and dashpots. The simple Kelvin-Voigt model for creep comprises a spring and dashpot in parallel. Creep strain as a function of time \( t \),

5 CONCLUSIONS

- The creep properties of Rotafix Structural Adhesive (RSA) have been evaluated up to a temperature of 80°C. RSA is a thixotropic, solvent-free, ambient cure epoxy adhesive which is employed for bonding rods and plates into timber structures.
- A Dynamic Mechanical Thermal Analyser (DMTA) has been used to apply a constant...
creep load to RSA in tension and the temperature was increased in 5°C steps up to 80°C. Dwell times of 30 and 120 minutes were employed.

- The same temperature history was applied to a unique miniature laminated creep shear specimen comprising an adhesive layer sandwiched between two beech veneers.
- In the tensile creep experiments viscoelastic creep occurred up to a limit but beyond (Tg + 15°C) further strain appeared to be related to the thermal expansion of the specimen confirmed by dilatometric measurements made in the DMTA.
- In the shear creep experiment the creep strains were higher as a result of the lower modulus of the adhesive in shear. However as for the tension tests creep occurred up to a limit.
- It can be concluded that under small applied tensile and shear loads the thixotropic RSA adhesive, with a Tg of ~35°C, is capable of withstanding creep loads at well above Tg without extensive viscous deformation. The RSA is thought to behave as a partially cross-linked rubber above Tg.

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