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Long-Term Shear Creep Behavior of Polymer-Based Injection Mortar Systems

Patrick R. Bradler[1], Joerg Fischer[1], Reinhold W. Lang[1], Roman Wan-Wendner[2]
[1]Institute of Polymeric Materials and Testing, Johannes Kepler University
Altenberger Straße 69, 4040 Linz, Austria
patrick.bradler@jku.at
[2]Institute of Structural Engineering, University of Natural Resources and Life Sciences
Gregor-Mendel-Straße 33, 1180 Vienna, Austria

Abstract – To assess the lifetime of resin-based injection mortar systems, a fundamental knowledge of their long-term behavior is necessary. In this research, the long-term shear creep modulus of two commercially available polymer-based injection mortar systems was investigated. Besides an epoxy resin based injection mortar system (EP), a vinyl ester resin based injection mortar system (VE) was utilized. Therefore, an accelerated characterization method providing shear creep modulus data considering various material states was implemented. Due to the sensitivity of the injection mortar systems to curing state and moisture content, the shear creep master curve was generated by testing under stable material conditions. Hence, upper bound material conditions with a defined curing state (cured) and moisture content (dry) were introduced. For this reference state I, shear creep curves were determined at different temperatures. Based on the time-temperature shift methodology, for both injection mortar systems shear creep master curves were generated for up to 50 years. To allow for an evaluation of the influence of curing state and moisture content, reference state II and reference state III were defined as lower bound material conditions. Due to the long-term instability of these reference states, short-term tests were utilized to evaluate reduction factors enabling a shift of the shear creep master curve of reference state I. Together, these results provide an evident effect of curing state and moisture content. For both injection mortar systems, the effect of curing state was rather small and the influence of moisture content was more pronounced.

Keywords**:** Shear Mode, Creep Master Curve, Injection Mortar, Vinyl Ester Resin, Epoxy Resin, Polymer, Anchoring.

1. Introduction

Polymer based injection mortar systems are frequently used in the building industry [1]. These materials exhibit several advantages compared to conventional mortar systems (e.g., short curing times) [2]. As the use of injection mortar is mostly in long-term applications, a good understanding of the materials is required. While most injection mortars are loaded in mixed mode, for most applications the shear loading is predominant. Therefore, this loading mode is of utmost interest for certification, simulation and lifetime prediction.

While in previous research, the tensile creep behavior of polymer based injection mortar systems was investigated [3, 4], the present research focuses on the development of shear creep master curves. Since the properties of the injection mortar systems are strongly dependent on curing state and environmental conditions, it was crucial to include the effect of curing state and moisture.

2. Background

Thermosets are frequently used for long-term applications in the building and construction sector (e.g., resin based anchoring systems) [5]. For product lifetime prediction, a good knowledge of the long-term behavior of the applied materials is required. Curing state [6, 7] and moisture uptake [8] are strongly affecting the long-term properties of thermosets and thermoset based compounds.

To gain creep data for a duration of up to 50 years, the thermo-mechanical behavior of polymeric materials allows the use of a time-temperature shift procedure [9, 10]. Based on the measurements of creep modulus curves at various temperatures in appropriate testing times a creep master curve can be generated by time-temperature shifting. Due to a change in temperature, but consistency of all other measurement parameters, creep curves are measured in an experimental window with low measurement times and can be shifted horizontally to higher times (see Fig. 1). A reference temperature is required, which is not shifted (e.g., 23°C in Fig. 1) and the creep curve measured at a higher temperature is horizontally shifted until having a congruent range with the preceding creep curve. This empirical shift allows for the establishment of a creep master curve for a longer period [11, 12].

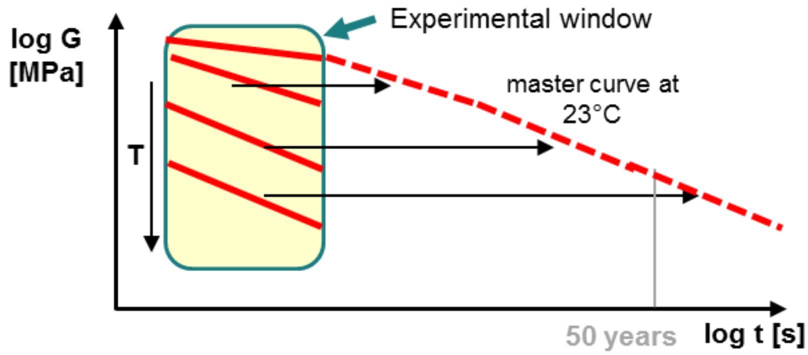

Fig. 1: Schematic illustration of the time-temperature shift concept for generating a creep master curve [9, 13].

3. Experimental

One epoxy resin based injection mortar system (EP) and one vinyl ester resin based injection mortar system (VE) was investigated. Both injection mortar systems (IMS) are commercially available. As the properties of the polymer-based IMS are strongly dependent on curing state and environmental conditions, three different reference states were defined (see Table 1). The upper bound creep curve is represented by reference state I, achieved by preconditioning at 110°C for 24 h for EP. Conversely, VE has to be preconditioned at 150°C for 96 h. Reference state I, designated as “cured & dry”, is hardly reached during regular processing and service conditions. However, it is required to conduct the creep shear tests with cured and dry specimens to ensure stable test conditions without effects of post curing and moisture change during the creep test period. Reference state II, representing the first lower bound creep curve and a material state of incomplete curing, is similar for both materials with a testing after a curing time of 24 h at room temperature. Reference state III, representing the second lower bound and a material state of high moisture content, is achieved by using materials from reference state I, which were additionally immersed in water at 80°C. To realize an adequate moisture content, the specimens were pre-conditioned for 3 h for EP and for 30 h for VE. The resulting moisture content represents a water uptake of 27 d at room temperature. Due to the instable material states of both lower bound reference states II and III, long-term creep shear tests at various temperatures are not possible. Hence, for these two reference states only short-term shear tests were performed to create reduction factors for shifting the shear master creep curve for reference state I.

Table 1: Overview of the reference states.

Reference state	Tempering	Moisture	Designation
ref. state I-EP	110°C, 24 h	dry	cured & dry
ref. state I-VE	150°C, 96 h	dry	cured & dry
ref. state II	-	as received	standard-cured
ref. state III-EP	110°C, 24h	wet	cured & wet
ref. state III-VE	150°C, 96h	wet	cured & wet

As alluded to above, the shear creep curves were measured for reference state I at different temperatures. Out of these results and by using empirical time-temperature shifts, the shear creep master curve for reference state I was generated and depicted in a double logarithmic plot of modulus against time (see Fig. 2). Since it is impossible to determine shear creep curves for the reference states II and III, the creep master curves for these reference states were created by applying reduction factors and semi-empirical modulus shifts. Therefore, short-term shear tests were conducted with specimens from all reference states. Based on a comparison between the values obtained for the lower bound and upper bound conditions, the reduction factors for the two lower bound reference states were evaluated. By simply multiplying the two specific reduction factors with the shear creep modulus data for reference state I, the shear creep master curves for the reference states II and III were realized.

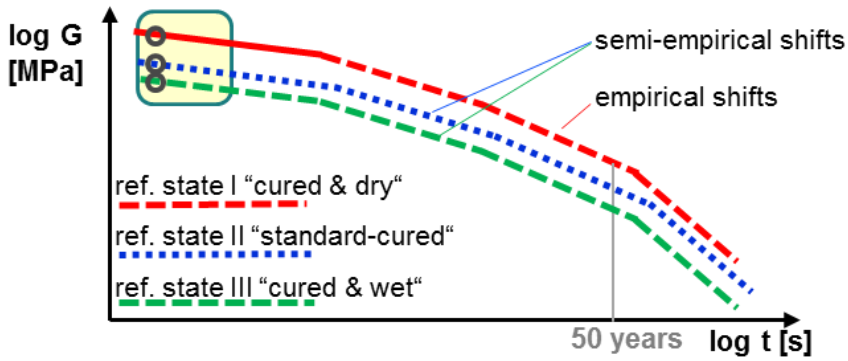

Fig. 2: Test concept with the different reference states.

The long-term shear creep tests and the short-term shear tests were performed on a universal testing machine of the type Z20 (Zwick Roell, Germany) equipped with a self-developed shear equipment (see Fig. 3) inspired by ASTM D5379 [14]. The specimens used for all tests were double V-notched Charpy test specimen according to ISO 179-1 [15]. The creep experiments were performed at different temperatures between room temperature and 60°C. The creep stress was set to 7 MPa for EP and to 3.5 MPa for VE. The strain was measured using a camera system and the digital image correlation software Aramis (Gom, Germany). The testing speed for the short-term monotonic tests was 1 mm/min. All tests were conducted at a constant temperature of 23°C. Using the monotonic test data, the secant moduli values were calculated between 0.05 MPa and the stresses chosen for the creep tests (i.e., 7 MPa for EP and 3.5 MPa for VE). The reduction factors for reference state II and III are the ratio of the secant modulus from the short-term tests of reference state II and reference state III referred to the secant modulus of reference state I (upper bound). In general, the long-term shear creep tests were repeated two times, while for short-term shear tests five repetitions were carried out. The deviations of long-term and short-term tests was consistent below 7 %.

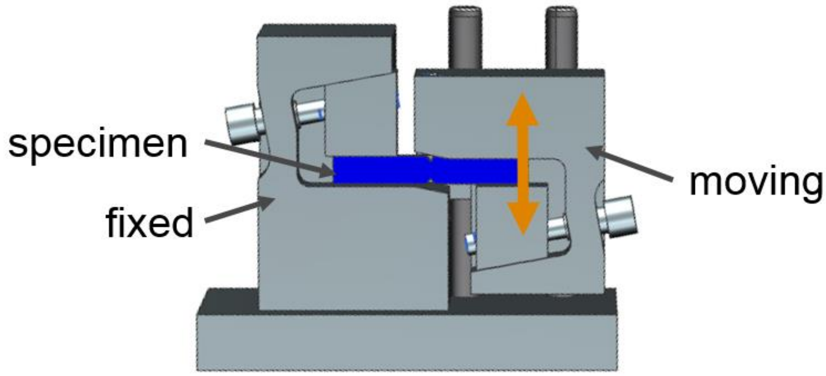

Fig. 3: Shear test equipment with specimen and an indication of the possible moving direction.

4. Results and Discussion

The shear creep curves for reference state I of EP and VE for different temperatures and the generated shear creep master curves for the temperature 23°C are depicted in Fig. 4. With increasing temperatures, a shift to lower modulus values was obtained. For EP, a change in the slope was detected between 35°C and 40°C. Starting at 40°C, higher slopes were received. Conversely, for VE, within the applied temperature range the slopes of all determined creep curves were similar. While for EP the horizontally time-temperature shifting of the creep curves resulted in a shear modulus master creep curve for longer than 50 years, for VE solely a shifting for up to 1 year was possible. Latter limitation was due to a shrinkage of the VE specimens, which was probably caused by a combination of post-cure and/or moisture reduction. However, a linear extrapolation was performed to predict the shear moduli for up to 50 years. The shear creep modulus master curves for EP and VE provide after 50 years shear creep modulus reductions of 88% and 52%, respectively.

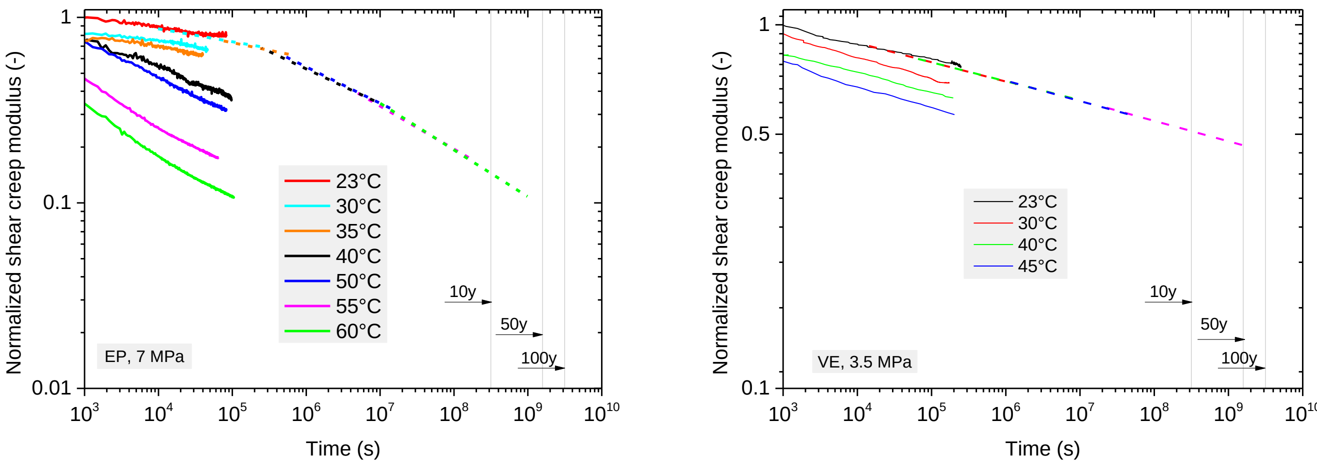

Fig. 4: Normalized shear creep curves for EP (left) and VE (right) at different temperatures and the generated master curve for reference state I.

Although, reference state I is hardly reached during regular processing and service conditions, the generation of the shear creep master curve was only possible for this material state. In order to cover the application relevant material behavior, worst-case material states such as incomplete curing and moisture uptake were considered in this research. For these reference states II and III, constant material conditions can only be ensured in short-term tests. The results for the monotonic shear tests are illustrated for both grades in Fig. 5. Out of these curves, the secant moduli were calculated for the evaluation of the reduction factors. For both resins, the highest shear strength was determined in reference state I. The strain-at-break values for the reference state II were much smaller compared to the other two reference states. This behavior may be attributed to a diminished bonding between the insufficient cured resin matrix and the filler particles. Comparing the two different grades, EP exhibited much higher strain-at-break values.

Table 2 depicts the reduction factors calculated from the secant moduli for both materials and all reference states. EP reveals a decrease of 7% for the “standard-cured” reference state II and a resulting reduction factor of 93%. The reduction factor of the “cured & wet” reference state III is 86%. Thus, a high moisture uptake is strongly affecting the material performance of EP. VE shows the same ranking with a decrease of 4% for reference state II and a deterioration of 7% for reference state III. Compared to the reduction factors of EP, the influence of the different reference states is less pronounced for VE. This finding is contrary to previous studies in tensile mode, which have indicated that the influence of the reference states is more pronounced for VE [4].

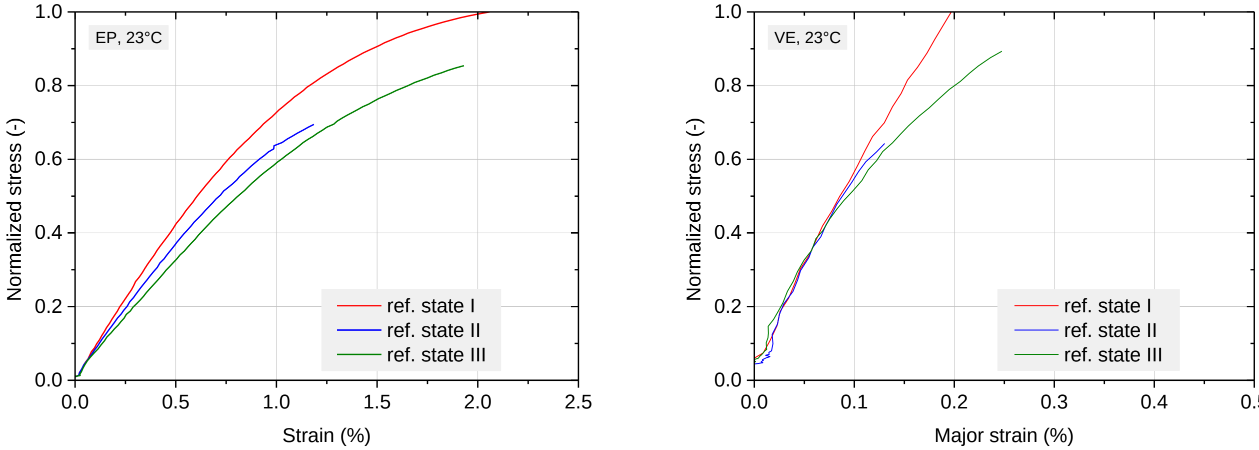

Fig. 5: Normalized stress-strain curves for the different reference states for EP (left) and VE (right).

Table 2: Overview of the reduction factors for both materials.

Reference state	EP	VE
ref. state I	100%	100%
ref. state II	93%	96%
ref. state III	86%	93%

Fig. 6 depicts the shear creep master curves for EP and VE including the bandwidth of the material performance, which was derived by a shift of the master curves to lower modulus values. The shift is dependent on the reduction factors that are illustrated as grey lines at short times representing the secant moduli of the three reference states. In lifetime simulations, the creep modulus decrease corresponding to the worst-case scenario should be emphasized. For both resins, reference state III represents the worst material performance. Compared to the initial creep modulus values determined in reference state I for EP and VE, the application of the reduction factor for reference state III resulted in shear creep modulus reductions of 91% and 56% after 50 years.

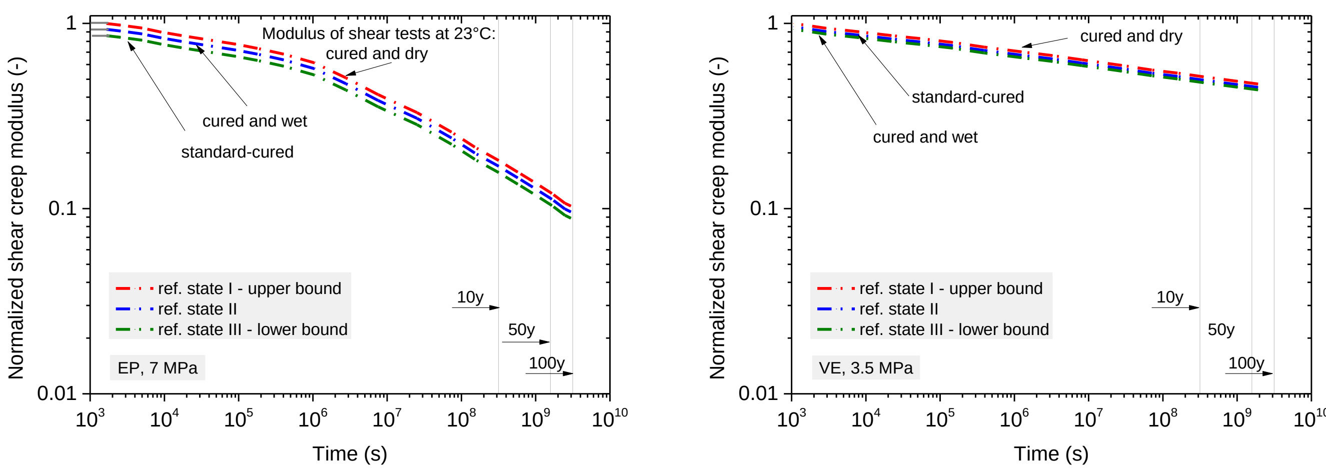

Fig. 6: Bandwidth of normalized shear creep modulus master curves for EP (left) and VE (right) accounting for the creep behavior for all three defined reference states.

5. Summary

In the present paper, the long-term shear creep behavior of two commercially available resin based (epoxy (EP) and vinyl ester (VE)) injection mortars systems (IMS) is described. Focus was given on the development of a shear creep modulus master curve. Moreover, the influence of incomplete curing and moisture on the long-term material performance was investigated. Therefore, three different reference/material states were defined. While the long-term shear tests were conducted with "cured and dry" specimens (reference state I), short-term shear tests were performed for the incomplete cured ("standard-cured") specimens (reference state II) and for the "cured and wet" specimens (reference state III).

Shear creep modulus master curves were generated for reference state I. The EP resin based IMS showed a more pronounced decrease of the modulus with an increasing slope in the master curve at higher times. On the contrary, the VE resin based IMS revealed a nearly linear decrease of the modulus over time. The short-term shear tests, capturing the remaining strength for the lower bound reference states II and III exhibited reduction factors of 93% for reference state II (incomplete curing) and 86% for reference state III (moisture) for the EP resin based IMS. Tests with the VE based IMS showed a reduction factor of 96% for reference state II and 93% for reference state III. Finally, based on the reduction factors the shear creep master curves for both materials were shifted to lower moduli, accounting for the bandwidth of the deterioration of material long-term performance caused by incomplete curing and moisture.

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