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Fatigue performance of a cold-curing structural epoxy adhesive subjected to moist environments

Maria Savvilotidou, Thomas Keller, Anastasios P. Vassilopoulos *

Composite Construction Laboratory (CCLab), Ecole Polytechnique Fédérale de Lausanne (EPFL), Station 16, Bâtiment BP, CH-1015 Lausanne, Switzerland

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ABSTRACT

This paper presents the results of an experimental program designed to study the effects of aging and a wet environment on the fatigue behavior of epoxy resins used in bridge applications. Specimens were manufactured, cured, and treated, before the experiments, under a variety of conditions, in room and in water environments, in order to simulate the aging of adhesives in bridges for a period of up to 100 years. Experimental results indicated that a typical power law S-N equation could describe the fatigue stress vs. life behavior of the examined material under different gravimetric conditions. The slope of the curve was found to be in the range of that of other polymers and polymeric composite materials. The cyclic strain behavior and hysteresis loops were obtained under different gravimetric conditions and at different stress levels and the effects of both parameters on the viscoelastic behavior of the material have been thoroughly discussed. The fatigue failure surfaces were also recorded using a digital handheld microscope to reveal the damage mechanisms. The results of this work showed that the examined epoxy resin could sufficiently sustain fatigue loads with maximum cyclic stress levels of more than 25% of their quasi-static strength for more than 2 million cycles.

1. Introduction

Structural adhesives have been used in bridge construction since the 1960s [1,2], but mainly for strengthening or upgrading purposes [3,4]. Only lately have such materials been used for bonding critical structural components in bridge applications, such as the bonding of a glass fiber-reinforced polymer (GFRP) bridge deck onto steel girders presented in [5]. In contrast to mechanical connections, adhesives used in these applications allow the easy and rapid joining of different materials, without disturbing their integrity by drilling holes for bolted connections for example.

Due to the often large bonding surfaces and in most cases outdoor applications, cold-curing adhesives are employed for such joints. Bond lines of this type have to sustain millions of fatigue cycles [6], which however are of low cyclic stress level [7,8], during their operational lifetime. Normally, such joints in bridge construction are sealed to prevent exposure to humidity and UV radiation. Nevertheless, in view of the long service life of bridges, up to 100 years, damage to the sealing cannot be excluded, and the adhesive may thus be exposed to moisture or even stagnant water during several decades [9].

The fatigue performance (with or without the presence of environmental effects) of adhesives has usually been examined through the investigation of the performance of adhesively-bonded joints, probably due to the fact that in aerospace applications thin adhesive layers are used and their behavior is always investigated in relation to the adjacent adherends. Experiments on joints provide more consistent results than those on bulk adhesives. Thus, adhesively bonded joints are usually preferred, even when the adhesive behavior is of interest. However, the analysis of the results in terms of fatigue resistance may be complicated by the complex stress state developed in joint testing configurations [10].

When moisture is present, adhesive behavior is decisive for overall joint performance, since adhesives are the joint components more susceptible to water effects. Aggressive environments can adversely influence the bond strength; water molecules diffuse into adhesively-bonded joints and degrade both the interface and the adhesive itself [11–12], thus influencing the structural performance of the joints, especially when exposed to water environments over long periods [13–15]. Water effects on the adhesive's mechanical properties are diffusion-dependent and can be correlated to the extent of plasticization, as discussed in [9,13,16–18].

In civil engineering, as well as in wind energy, cold-curing adhesives are often used and thicker adhesive layers than those used in

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* Corresponding author.
E-mail address: anastasios.vasilopoulos@epfl.ch (A.P. Vassilopoulos).

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the aerospace industry are employed. Important characteristics of the bond lines found in these applications are large adhesive thicknesses and volumes, and multiaxial (fatigue) loading conditions. For example, the thickness of the bond lines in 70-m wind turbine blades can attain 30 mm [19], while that of the bond line between the sandwich bridge deck and the steel girders of a 12-m-long vehicular bridge can reach 8 mm [5]. In such cases, the fatigue behavior of the bulk adhesives, excluding the effects of any adherends, needs to be investigated in order to obtain valuable date for an appropriate structural design.

There is a scarcity of experimental data concerning the fatigue performance of bulk structural adhesives related to any engineering domain. As mentioned above, numerous studies exist on the fatigue behavior of adhesively-bonded joints with or without temperature and water effects. Less information exists regarding the investigation of the fatigue behavior of bulk adhesives [20–24], while very little exists on the fatigue behavior of bulk structural adhesives subjected to wet conditions [25].

Previous results show that the fatigue behavior of bulk adhesive specimens and neat resin specimens can be modeled by typical S–N equations, such as the exponential Basquin relationship [24]. S–N curves of neat epoxy specimens exhibit similar slopes to those of S–N curves of unidirectional glass fiber epoxy composite laminates, indicating that the fatigue behavior of the matrix is decisive for the behavior of unidirectional composite laminates.

Stress strain loops can be monitored during fatigue life to provide information about material stiffness fluctuations (usually derived from the slope of the loops), as well as the hysteretic fatigue behavior of the examined material, linked to structural changes, the development of cyclic creep, and self-heating (hysteretic heating) during fatigue loading [20,22,26–28].

During fatigue cycles, the material temperature increases due to self-heating. When the frequency is sufficiently low, and other conditions are fulfilled, e.g., thin specimens, cooled testing space, low damage accumulation, etc., the energy resulting from self-heating is usually released to the environment, causing no damage to the material's structural integrity. However, under certain conditions, self-heating can have detrimental effects on the material's lifetime [25]. Epoxy resins are sensitive to hysteretic heating effects as from the early life, while polyester seems more resistant to such effects up until the late stages, close to failure [25].

Cyclic creep strain is progressively accumulated under stress-controlled cyclic loading of polymer composites [26,29–30], as well as of epoxy polymers [22,27]. This strain is of a viscoelastic nature, as can be confirmed by its complete recovery after load removal [27]. The amount of the accumulated strain depends on the applied cyclic mean stress [22]; the former is diminished under inversed fatigue loading, $R = \sigma_{\min}/\sigma_{\max} = -1$, while it becomes significant for tension-tension $(0 < R < 1)$ and compression-compression $(1 < R < \infty)$ fatigue loadings [29].

An estimation of the material fatigue stiffness can be obtained by fitting the cyclic stress-strain data over each cycle [26]. This process allows the estimation of the secant stiffness variations during fatigue life. The fluctuations of this property over the material's lifetime have been intensively investigated for composite laminates, and adhesively-bonded joints, as a function of different parameters, such as the laminate stacking sequence, loading patterns, as well as a function of temperature and humidity. Nevertheless, there is no information on the fatigue stiffness variations, self-induced temperature and damage area development during the fatigue life of bulk epoxy adhesives for bridge applications, neither wet, nor subjected to water environments.

The above comprehensive literature review shows that, although there is a limited number of reports on the durability of resins and adhesives, there is no study investigating the fatigue performance of cold-curing structural adhesives for civil engineering applications, especially when they are subjected to wet environmental conditions.

This work investigates the fatigue behavior of a cold-curing epoxy structural adhesive, subjected to aggressive environments encountered in typical bridge applications. A wide range of cyclic loads is applied to the specimens in order to cover all regions of fatigue life, from the low-cycle fatigue to the operational lifetime of up to 10^7 cycles, under the serviceability load ranges of a bridge. Dry, saturated wet, and dried specimens are examined to investigate the water effects on their long-term behavior. The effects of water content on fatigue life, the fatigue damage accumulation, and the fatigue stiffness are described and thoroughly discussed. Specimens' failure surfaces are presented in order to relate observed failure mechanisms to water and fatigue loading effects.

2. Experimental procedures

2.1. Material and specimen preparation

A commercial cold-curing epoxy adhesive (Sikadur-330, supplied by SIKA Schweiz AG), commonly used in structural civil engineering applications, was selected for this study. Typical applications of this adhesive include the bonding of carbon fiber-reinforced polymer plates or the impregnation of fabrics to strengthen existing structures.

The adhesive was produced under laboratory conditions ($T = 21 \pm 3$ °C and $RH = 40 \pm 10\%$) with 4:1 resin to hardener mixing ratio, and then was poured into aluminum molds to produce specimens with the dimensions shown in Fig. 1, according to ASTM D638-14 [31]. According to the supplier data sheet, the viscosity of the product is approximately 6000 mPa s at 23 °C. To represent adhesive applications under on-site bridge conditions, no vacuum was applied to remove entrapped air.

The molds were left under laboratory conditions for seven days to allow the specimens to cure. All specimens were then post-cured at 60 °C for three days in a climatic chamber with a precision of 0.5 °C. After this post-curing process, the T_g, defined at the peak of the loss modulus vs temperature curve obtained by DMA, was estimated at approximately 75 °C [9]. The mechanical properties of these specimens correspond to properties obtained for the same adhesive if naturally aged for nine months at ambient temperature on a bridge as shown in [32].

2.2. Preconditioning

A first set of specimens (DRY) was used for the derivation of the reference quasi-static and fatigue data. Fatigue data for this set were collected after the aforementioned post-curing treatment over a period of nine months after the post curing. During this time, the specimens were stored under laboratory conditions, and as previously proved [32] their quasi-static properties were not significantly affected by such time periods.

The second set (WET) comprises the wet specimens, those that, immediately after post curing, were placed in a bath of demineralized water at 50 °C. The temperature of the bath was 50 °C to

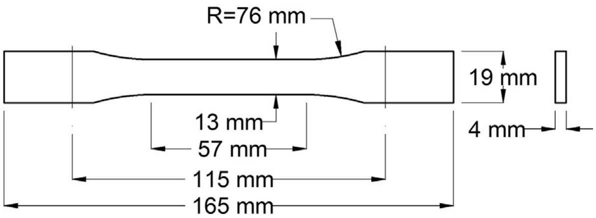

Fig. 1. Bulk specimen geometry.