

CHARACTERIZING THE FIRE PERFORMANCE OF ADHESIVES USED IN GLUED-IN RODS CONNECTIONS

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ABSTRACT: Glued-in rods are an aesthetically and performant type of connection that has seen its usage increase in the last few years. However, there is still a lack of data concerning the fire performance of glued-in rods, limiting its integration in standards. Recent studies suggest that the adhesive is critical to the fire performance of glued-in rods, since its capacity is greatly reduced when the temperature exceeds its glass transition temperature (T_g), ranging between 45-65 °C for most structural adhesives. To validate these findings, dynamic mechanical analysis (DMA) tests were performed to better assess the T_g and other thermomechanical properties of the adhesives used in this research. Axial tension tests at stabilized temperature were performed on 67 glued-in rods specimens using five different adhesives (three epoxies and two polyurethanes) and various sets of temperatures at the glue line interface. Most of these specimens have shown a ductile failure mode for temperatures below the T_g of the adhesive and a brittle failure mode for temperatures above the T_g . This research helps determine guidelines for the fire design of glued-in rods and related testing, and ultimately leading to a design method for providing fire resistance to connections made of glued-in rods.

KEYWORDS: Girods, Fire behaviour, Adhesive, Glass transition temperature, Thermal resistance

1 INTRODUCTION

Glued-in rods, a connection system in which a metal rod is glued inside a wood element by means of a structural adhesive, are gaining in popularity in the construction industry due to their high bearing capacity, good slip stiffness, and great aesthetic. Since the metal rod is concealed inside the wood element, the connection is naturally protected in a fire situation. Wood has a predictable behavior under high temperatures, with a char depth given by Equation 1 [1] where $x_{c,n}$ is the notional char depth (mm), β_n is the notional char rate (0.70 mm/min for glulam) and t is the time (min). Wood also has a high thermal gradient that can be determined through Equation 2 [2], where T is the temperature (°C), T_0 is the initial temperature (typically taken as 20°C), T_p is the temperature at the base of the char layer (300°C), x is the position within the thermal penetration depth (mm) and α is the thermal penetration depth (35 mm).

$$x_{c,n} = \beta_n \times t \quad (1)$$

$$T = T_0 + (T_p - T_0) \left(1 - \frac{x}{\alpha}\right)^2 \quad (2)$$

However, in North America there is no consensus between experts on the design of glued-in rods, and existing standards does not cover all adhesive types, limiting its use in the construction industry [3]. While glued-in rod connections have been well characterized at ambient temperature, their thermomechanical behavior at

elevated temperatures using different adhesives has been understudied.

Previous research suggests that the adhesive is the critical element in glued-in rod connections when exposed to elevated temperatures since its capacity decreases drastically when the glass transition temperature (T_g) is exceeded [4-7]. The latter is defined as the temperature where permanent modifications occur in the molecular structure of the polymer, leading to a transition of a solid to rubberlike state of the adhesive [8-11]. At temperatures above the T_g , this results in a considerable loss of adhesion and cohesion in the adhesive layer, translating to a poor performance of the connection with an undesirable brittle failure mode [7, 12-15].

The T_g of an adhesive strongly depends on its type, formulation, curing time, curing temperatures and thermal history [16, 17]. The latter refers to the effect of post-cure, which can increase the T_g by creating additional crosslinks in the adhesive. Furthermore, the T_g is not a fixed temperature, but is rather a range of temperatures that can vary between different test methods, and even within the same test method [16]. For this reason, it is a difficult task to accurately assess the temperature at which the thermomechanical performance of glued-in rods starts to deteriorate. The Dynamic Mechanical Analysis (DMA) is a sensitive method commonly used to determine the range of the T_g for all adhesives tested according to three curves: 1) storage modulus, 2) loss modulus and 3) $\tan \delta$. The storage modulus, which is a quantification of the

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adhesive's stored energy [18], is an indication that the mechanical properties of the adhesive start decreasing at this temperature [19]. The thermomechanical behavior of glued-in rods can be determined through axial tension tests at stabilized temperatures [20], which informs the researcher on the stiffness, capacity, and failure modes of the connections at specific temperatures.

This research aims to characterize the performance of adhesives used in glued-in rods connections at elevated temperature. To do so, the effect of temperatures below the storage modulus, above the storage modulus and above the loss modulus on the resistance, stiffness and failure mode of the connection have been evaluated. The only variables in this research were the nature of the adhesive and the temperature of exposure. The findings will help determine guidelines for the fire design of glued-in rods and related testing.

2 EXPERIMENTAL

2.1 MATERIALS AND DIMENSIONS

Glued-laminated timber (glulam) made from Spruce-Pine of grade 12c-E per CSA O122 [21] provided by Art Massif were used in the fabrication of all glued-in rod specimens. The timber was conditioned at a temperature of 20°C and 65% relative humidity (RH) until their masses were at a constant value, meaning an equilibrium moisture content of $\pm 12\%$. Each glulam specimen had dimensions of 80 x 104 x 686 mm (length of 1372 mm when fully assembled), with holes of 19 mm in diameter and 402 mm in length drilled in the middle lamella by a computer numerical control (CNC). Threaded steel rods of 16 mm nominal diameter and 800 mm length of grade ASTM A307 [22] with minimal strength of 414 MPa were used. An example of a glued-in rod specimen is shown in Figure 1.

A total of five structural adhesives were used in the fabrication of glued-in rods connections: three epoxies (EPX1, EPX2 and EPX3) and two polyurethanes (PUR1 and PUR2) which were deemed, per the adhesive suppliers, suitable for glued-in rods application. The identity of each adhesive is not provided due to non-disclosure agreements. Once injected into the connection, the adhesive had a 1.5 mm thickness around the steel rod.

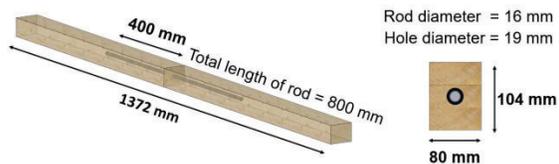


Figure 1: Glued-in rod specimen

2.2 FABRICATION OF GLUED-IN RODS

Two 9.5 mm diameter injection/ejection holes were drilled perpendicular to grain at 380 mm from the middle of the glued-in rod specimen. A threaded steel rod was inserted inside the 19 mm drilled hole of a glulam

specimen. Prior to concealing the connection with a second glulam, butyl tape was added between both timber elements to limit the spread of the adhesive at this butt joint and have a preliminary bonding. A tight contact between the connection was ensured by two clamps. The adhesive was then continuously inserted through the injection hole until it ejected through the ejection hole. Both holes were closed by 9.5 mm diameter timber dowels to seal the assembly and limit heat flow during the heating of the specimens. The fabrication process is shown in Figure 2. Once fabricated, the glued-in rod specimens were untouched for 48h to allow the cure of adhesive at room temperature. The latter were then transferred to a conditioning room at 20°C and 65% RH for a minimum of 10 days, ensuring the same conditions for all specimens. A total of 67 glued-in rod specimens, consisting of 13 specimens per adhesive plus two additional samples for the EPX3 adhesive, with final dimensions of 80 x 104 x 1372 mm was fabricated.

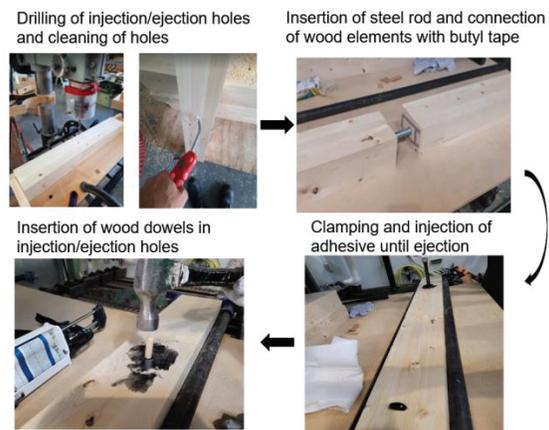


Figure 2: Fabrication process of glued-in rods

2.3 CHARACTERIZING ADHESIVES

To better assess the T_g and other thermomechanical properties of the adhesives used in this experimentation, DMA tests were performed. A DMA Q800 machine from TA Instruments was used, with a heating rate of 3°C/min until 150°C and a strain of 0.1%. The T_g obtained from these tests were used to determine the various sets of target temperature at which the glued-in rods were exposed for the mechanical testing. A total of 30 DMA tests were performed, meaning each adhesive was tested six times.

A second DMA run was performed on all adhesive samples previously tested, therefore fully crosslinked, to evaluate the effect of post-cure on the T_g from the storage modulus and loss modulus.

2.4 EXPERIMENTAL SET-UP AND INSTRUMENTATION

Four steel plates were fastened to the glued-in rod specimens using 16 self-tapping screws of 120 mm in length fixed at a 45° angle of the longitudinal direction at

each extremity. The number, length and angle of screws were determined to offer a greater resistance than the steel rod at ambient conditions. They have been calculated according to the withdrawal resistance equation from the Canadian Construction Materials Centre (CCMC) Evaluation report 13677-R [23]. To limit the heat transfer at both ends from the high thermal conductivity of the steel plates, the latter were wrapped into ceramic fiber blankets, as shown in Figure 3a.

A total of 8 type K thermocouples (TC) were positioned inside and outside the specimen to monitor the temperature in real time: two TC at the glue line interface (1 and 4), four TC at 15 mm around the glue line interface (2, 3, 5 and 6), one TC on the surface (7) and one TC in the oven (8), as shown in Figure 3b and 3c. A firestop caulking was used to seal the holes drilled for the insertion of each TC.

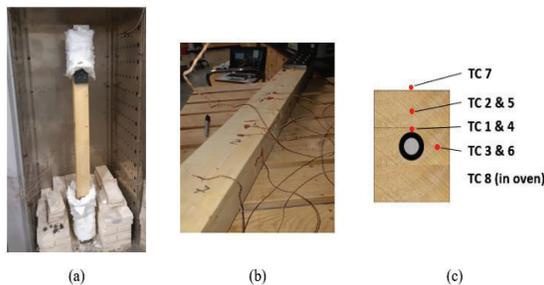


Figure 3: Heating experimental set-up

2.4 AXIAL TENSION TEST AT STABILIZED TEMPERATURE

The glued-in rod specimen was inserted inside an oven preheated at 200°C, regardless of the target temperature, to expose each specimen to the same heating conditions. The oven temperature was set to be below the auto-ignition temperature of the glulam, while being sufficiently high for an adequate heat transfer. The target temperatures used in this research were: 1) 21°C (ambient), 2) just below the T_g found through the storage modulus, 3) between the storage and loss modulus, and 4) above the loss modulus specific to each adhesive. Once the average of both TC at the glue line interface (1 and 4) reached the predetermined target temperatures, the specimen was transferred and fixed to the axial tension test bench within 10 minutes.

Four 25 mm Linear Variable Differential Transformer (LVDT) lasers were installed (two on each side of the large face) at 12.5 mm from the center of the specimen, as shown in Figure 4a. The LVDT lasers allowed to adequately measure the slip of the connection from all sides when axially loaded. Each specimen was subjected to an axial displacement rate of 0.5 mm/min until failure of the connection. Pin-pin end conditions were used to connect the specimen to minimize additional bending moment applied on the glued-in rod (Figure 4b). The temperature within the specimens was constantly monitored throughout the entire duration of the axial

tension test to ensure that the glue line's temperature remained near the target.

Upon the failure of the specimen during the axial tension test, the failure mode was determined, and load-slip curves were generated, giving information on the strength of the connection (F_{max}), i.e. the peak of the load slip curves, and the stiffness of the connection (k_i). The latter was calculated following Equation (3) provided by EN 26891 [24] standard, where v_i is the slip at $0.4F_{max}$.

$$k_i = \frac{0.4F_{max}}{v_i} \quad (3)$$

Two additional tests were performed on the EPX3 specimen (EPX3-14 and EPX3-15) to determine the effect, if any, of post-cure on the thermomechanical behavior of glued-in rods. The latter were previously heated at the T_g of the fully crosslink adhesive (65°C) for three hours. These specimens rested for 12 hours at ambient conditions before heating in the oven for a second time and performing the axial tension test.

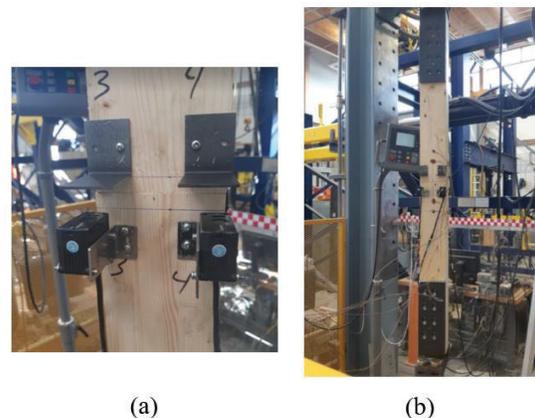


Figure 4: Axial tension test set-up

3 RESULTS

3.1 DMA TESTS

A total of six DMA tests were performed for each adhesive to determine the T_g through the storage modulus, loss modulus and $\tan \delta$. The former was identified by the intersecting the tangent at the top of the curve and at its inflection point, while the loss modulus and $\tan \delta$ were valued at the peak of their respective curves, as show in Figure 5.

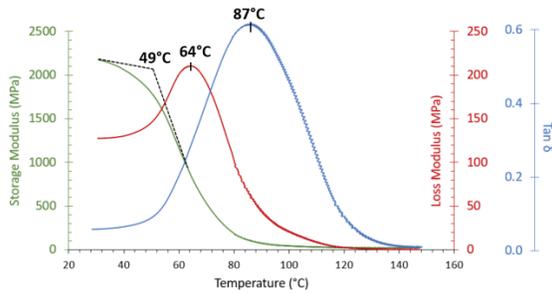


Figure 5: DMA test of EPX1-3 specimen

Table 1 displays the average T_g values of the storage modulus and loss modulus of each adhesive tested. Since the mechanical and physical properties of adhesives are already significantly affected at temperatures above the loss modulus, results for the T_g found through the $\tan \delta$ are not displayed and will not be considered hereafter.

Table 1: T_g values based on storage and loss modulus

Specimen	Storage modulus (°C)	Loss modulus (°C)
EPX1	52	67
EPX2	48	59
EPX3	51	60
PUR1	56	71
PUR2	24	34

Table 1 shows that the storage modulus of all epoxies (EPX) samples was similar, with a difference of less than 4°C between each other. A greater dissimilarity was found for the loss modulus of the latter, with an 8°C difference at most. The polyurethane (PUR) specimen 1 had the highest T_g found through the storage modulus and loss modulus, while the PUR2 specimen demonstrated the poorest values of all adhesives. In addition to the reference specimen tested at ambient conditions, these results were used to determine the target temperatures each glued-in rod specimen was exposed during the axial tension test at stabilized temperature.

The post-cure effect did not have the same influence on the T_g of all adhesives. The EPX1, PUR1 and PUR2 adhesive specimens showed a decrease in the average T_g found through the storage modulus compared to the first heating cycle. The EPX2 and EPX3 adhesives demonstrated a considerable positive impact in their storage modulus, with an increase of +7°C and +15°C, respectively. As for the T_g found through the loss modulus, the EPX1 specimen showed a reduction while the EPX2, EPX3, PUR1 and PUR2 specimen had an increase ranging from +5°C to +19°C. Figure 6 displays the difference in the T_g between the first and second heating cycle for all adhesives. Since the EPX3 was the most positively affected by the post-cure, it was chosen to

determine the effect on the thermomechanical properties of previously heated glued-in rod connections.

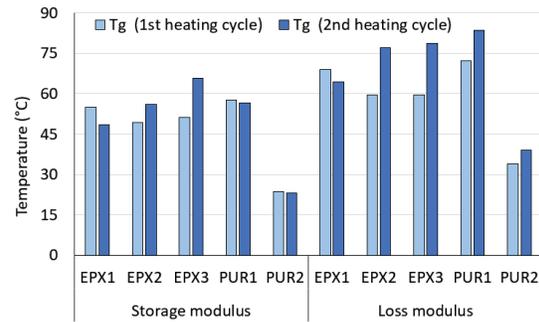


Figure 6: Influence of post-cure on T_g

3.2 MECHANICAL BEHAVIOR OF GLUED-IN RODS AT DIFFERENT TEMPERATURES

The reference values and failure mode for each glued-in rod specimens fabricated with a different adhesive were determined from an average of three specimens per adhesive tested at ambient temperatures. The average resistance and stiffness at 21°C for the EPX1, EPX2 and EPX3 specimen were respectively 75 kN and 177 kN/mm, 72 kN and 161 kN/mm, as well as 74 kN and 180 kN/mm. While the PUR1 specimen had a similar average resistance and stiffness than the EPX specimens, i.e. 74 kN and 178 kN/mm respectively, the PUR2 specimen had average values of 73 kN and 62 kN/mm. Most specimens had a ductile failure mode in the steel rod (Figure 7a), except for the PUR2 specimens which all failed in a brittle manner in the adhesive layer (Figure 7b).

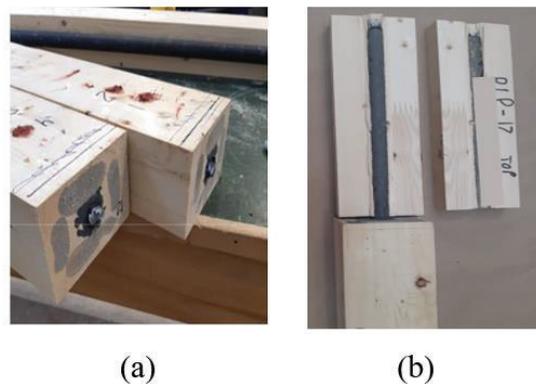


Figure 7: (a) Ductile failure and (b) brittle failure mode

When tested at temperatures just below the storage modulus specific to each adhesive, the resistance of almost all specimens dropped by less than 4%, regardless of the adhesive. However, the specimens tested closest to the storage modulus showed a decrease of between 23% and 29% for the EPX1, EPX3 and PUR1 adhesive, while the EPX2 adhesive demonstrated a reduction of 17%. At this temperature the failure mode remained ductile in most cases, while a brittle failure mode in the wood component

occurred for in the EPX2 specimen. Given that the T_g found through the storage modulus of the PUR2 was relatively low, no tests were performed at this temperature for the latter.

When tested at temperatures just above the storage modulus, the capacity of the glued-in rod specimens remained similar than the reference values, with a reduction of less than 5%. As for the stiffness, a reduction of between 59% and 70% was observed in the EPX1, EPX2, EPX3 and PUR1 specimen when compared to their respective reference value. The PUR2 specimens showed a stiffness reduction of 23%. A ductile failure mode occurred for all EPX1 and EPX3 specimen, while the adhesive failed in all EPX2, PUR1 and PUR2 specimens.

As the temperature increased closer to the T_g based on the loss modulus, the capacity of the EPX1, EPX2 and EPX3 decreased by 28%, 22% and 4%, respectively when compared to the reference values. The PUR1 specimen showed the largest decrease with 41%. The stiffness of those specimens decreased by between 66% and 77%. The PUR2 specimen had a reduction in its capacity and stiffness by 14% and 36%, respectively. All specimens demonstrated a brittle failure mode in the adhesive layer.

The mechanical properties of all specimens further decreased when tested at temperatures above the loss modulus, which was around 79°C. Almost all specimens had lost more than 50% of their initial strength, and more than 74% of their initial stiffness. In the case of the PUR2 specimens, it lost 65% of its initial strength and 57% of its initial stiffness. A brittle failure in the adhesive layer occurred in all specimens. The temperature (T) in °C, capacity (cap) in kN and stiffness (stiff) in kN/mm are displayed in Table 2 and 3 for the EPX and PUR, respectively. Figures 8 to 12 shows the evolution of the capacity, stiffness and failure modes of all specimens as the temperature increases.

Table 2: Test temperature, capacity and stiffness of EPX specimens

#	EPX1			EPX2			EPX3		
	T	cap	stiff	T	cap	stiff	T	cap	stiff
1	21	74	192	21	72	157	21	75	160
2	21	75	156	21	73	163	21	75	194
3	21	75	184	21	72	162	21	73	187
10	47	71	126	42	71	144	43	74	162
11	45	72	150	43	63	133	45	72	133
4	55	72	68	51	71	144	54	70	76
5	55	73	60	57	64	40	52	73	82
6	53	72	63	54	68	49	54	72	74
12	62	66	41	59	61	55	60	71	61
13	68	54	29	60	56	44	65	63	38
7	73	42	34	86	32	38	78	41	42
8	77	48	31	73	33	38	77	38	44
9	79	36	33	79	37	42	77	44	37

Table 3: Test temperature, capacity and stiffness of PUR specimens

#	PUR1			PUR2		
	T	cap	stiff	T	cap	stiff
1	21	75	174	21	73	63
2	21	75	203	21	74	58
3	21	73	157	21	74	65
10	47	73	134	28	72	48
11	58	71	64	29	72	56
4	56	73	76	54	36	27
5	54	73	88	53	41	28
6	56	73	64	50	41	27
12	65	64	45	36	63	40
13	68	44	23	40	51	39
7	80	34	27	77	25	27
8	80	34	28	76	28	26
9	77	32	29	77	26	24

When evaluating the effect of post-cure, i.e. the thermomechanical behavior of a glued-in rod specimen with a fully crosslinked adhesive, the EPX3-14 specimen had an increase of 14% in the resistance and an increase of 99% in the stiffness compared to the EPX3-13 specimen which did not go through two heating cycles. Both specimens were tested with a glue-line temperature of 65°C. The EPX3-15 specimen also demonstrated an increase in the capacity and stiffness even when compared to specimens tested with a lower glue-line temperature, as shown in Figure 13.

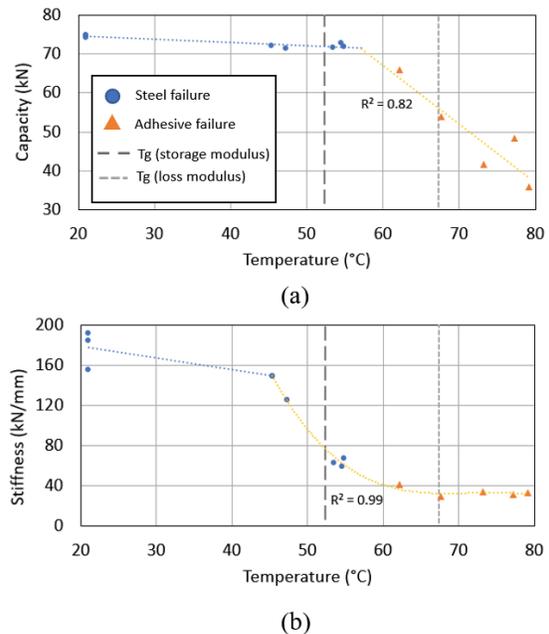
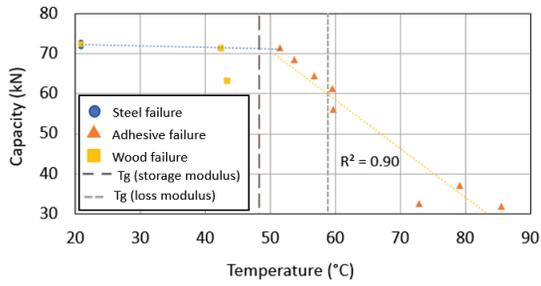
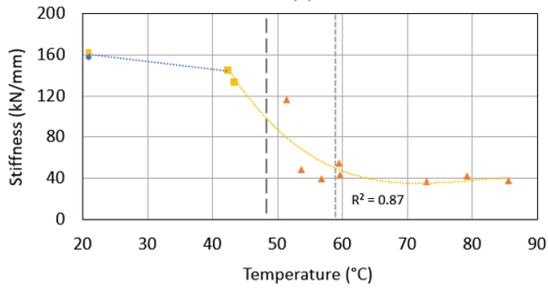


Figure 8: (a) Capacity and (b) stiffness evolution of EPX1

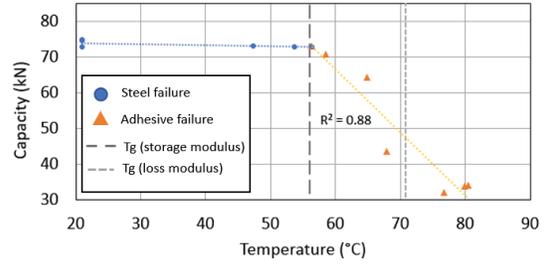


(a)

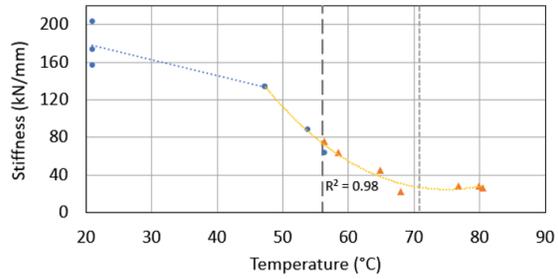


(b)

Figure 9: (a) Capacity and (b) stiffness evolution of EPX2

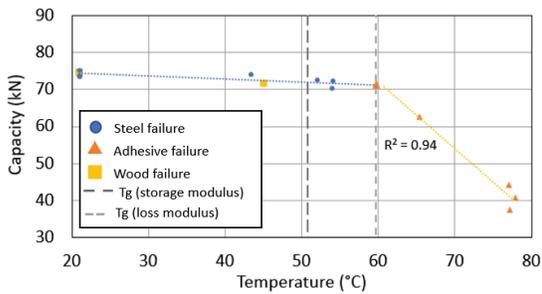


(a)

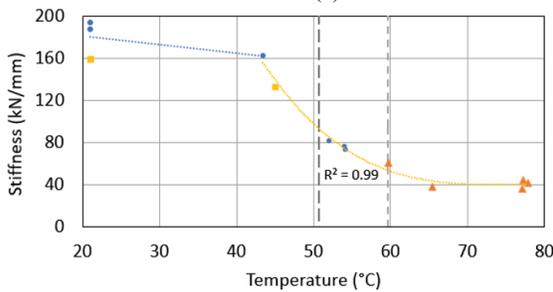


(b)

Figure 11: (a) Capacity and (b) stiffness evolution of PUR1

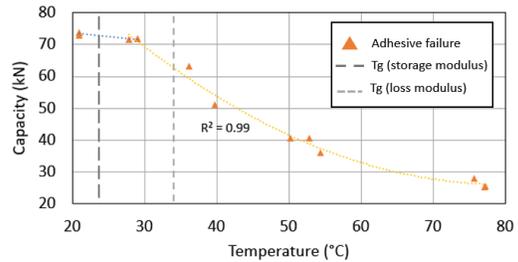


(a)

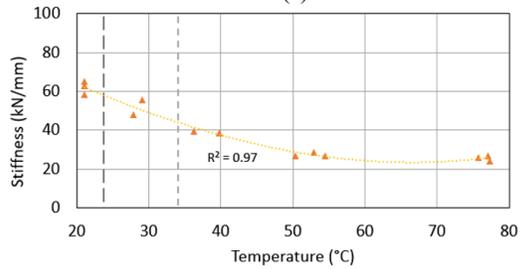


(b)

Figure 10: (a) Capacity and (b) stiffness evolution of EPX3



(a)



(b)

Figure 12: (a) Capacity and (b) stiffness evolution of PUR2

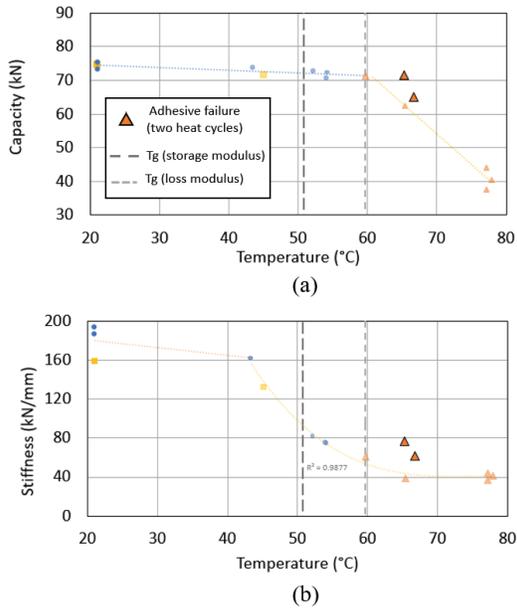


Figure 13: Effect of post-cure on the (a) capacity and (b) stiffness of glued-in rods with EPX3

4 DISCUSSION

The DMA tests revealed that the T_g of all adhesives, except that of the PUR2, was similar. Therefore, the thermomechanical behavior of each glued-in rod specimens could be easily compared, and correlations can be drawn. A significant increase in the capacity and stiffness occurred in both specimens that were exposed to two heating cycles, i.e. where a post-cure effect was intentionally evaluated. The latter suggest that the method chosen to evaluate the thermomechanical properties of the glued-in rods did not enable the adhesive to fully crosslink, leading to representative results.

The results of the axial tension test suggest that the mechanical properties of the adhesive, thus the glued-in rod connections, are considerably affected when the glueline interface is exposed to elevated temperatures. As it can be observed in this research, the T_g found through the storage modulus of a DMA test is a good indication that the connection will not have the same mechanical behavior, regardless of the adhesive. In fact, all specimens have shown that when exposed to temperatures below the storage modulus, only a small decrease of the capacity occurred ($\leq 4\%$) while a more noticeable decrease in the stiffness was observed ($\leq 29\%$). However, the failure mode remained ductile in most specimens.

When the glueline temperature exceeded its T_g found through the storage modulus, the stiffness of all adhesives dropped by up to 70% compared to their initial stiffness. The failure mode occurred in the adhesive layer rather than the steel rod in three adhesive specimens, while it remained ductile in the other two adhesives tested. As the temperature at the glueline interface increased closer to

the T_g of the loss modulus specific to each adhesive, all specimens had a brittle failure mode in the adhesive layer. This suggests that the thermomechanical behavior glued-in rods at this temperature is dependent on the nature of the adhesive.

The loss of capacity and stiffness of glued-in rods at elevated temperatures can be attributed to the increased mobility of molecules in the internal structure of the adhesive, leading to a rubbery state of the latter. This also translated into an undesired failure in the adhesive layer.

These observations align with the results of Verdet *et al.* [7], Lahouar *et al.* [8], Lartigau *et al.* [9], Di Maria *et al.* [12] and Luo *et al.* [15]. However, the findings of this research have determined that the T_g found through the storage modulus of a DMA test would be a reliable limit to impose for the temperature at the adhesive interface. These observations could pave the way for provisions and guidelines for the fire design of glued-in rods.

For example, to design a glued-in rod with the EPX1 adhesive (i.e. with a T_g from storage modulus of 52°C) required to provide a fire resistance rating of 1h without using protective materials, the designer would:

1. Determine the depth of the char layer from Eq.1:

$$x_{c,n} = 0.7 \times 60 = 42 \text{ mm}$$

2. Determine the position at which the temperature would reach the T_g from the storage modulus after 1h of fire from an adaptation of Eq.2:

$$x = \frac{35}{\sqrt{\frac{52 - 20}{300 - 20}}} = 103.5 \text{ mm}$$

3. Add both values:

$$\text{Min. dimension} = 42 + 103.5 = 145.5 \text{ mm}$$

Therefore, a glued-in rod fabricated with the EPX1 and a cross-sectional dimension greater than 146 x 146 mm would be adequate to resist a 1h fire scenario. This would assume that the butt-joint between the glulam elements would be properly fire-stopped to prevent fire penetrating and directly affect the steel rod.

5 CONCLUSION

Even though glued-in rods are a type of connection that is gaining in popularity, their thermomechanical behavior has been understudied, thus limiting its inclusion in standards around the world. Many researchers have suggested that the T_g of the adhesive used was the critical temperature at which the mechanical properties of the glued-in rods will deteriorate. Yet, the T_g is a range of temperature that can vary according to the method of determination chosen or the parameters used within a method. The results suggest that a Dynamic Mechanical

Analysis is an accurate method to inform on the properties of adhesives at different temperatures by determining the T_g through the storage modulus, loss modulus and tan δ. The objective of this research was to characterize the performance of adhesives used in glued-in rod connections at elevated temperatures. The following key observations have been found throughout this research for different temperatures at the connection's glue line interface:

1. All glued-in rod specimens had over 95% of their initial load-carrying capacity when the temperature at the bondline was lower than their respective T_g found through the storage modulus of a DMA test;
2. Except for the PUR2 specimen, no failure occurred in the adhesive layer below their respective storage modulus, while 3/5 adhesive specimens failed at temperatures just above the storage modulus;
3. All glued-in rod specimens had a brittle failure in the adhesive layer when the latter reached temperatures close to their respective loss modulus;
4. The stiffness of glued-in rods is most affected by elevated temperatures, with a decrease of up to 70% at temperatures just above the storage modulus;
5. The axial tension test at stabilized temperature did not enable a considerable post-cure effect, thus providing representative results on the thermomechanical properties of glued-in rods.

Therefore, the T_g determined through the storage modulus of a DMA test would be a reliable limit to impose for the temperature at the adhesive layer. Above the latter, the thermomechanical behavior of the connection is dependent on the nature of the adhesive, which could lead to unsafe connections. It is recommended to take precautions, i.e. protect with gypsum board, fire protective coatings or increase the section dimension, to limit a temperature rise above the T_g found through the storage modulus of a DMA test at the bondline.

Future research on the fire performance of glued-in rods should include an exposure to a standard fire curve, more repeatability at temperatures between the storage modulus and loss modulus, and/or evaluate the fire performance of glued-in rods subjected to a bending moment.

Some limits of this research include the grade of the ASTM A307 rod, since only a minimal tensile strength of 414 MPa is required by the standard. Furthermore, the charring at the butt joint of the connection, which could expose the steel rod to high temperature and increase the heat conductivity along the rod was not considered.

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