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# 4 EFFECT OF ELEVATED TEMPERATURES ON THE MECHANICAL 5 PERFORMANCE OF PULTRUDED FRP JOINTS WITH A SINGLE ORDINARY 6 OR BLIND BOLT

Chao Wu<sup>1</sup>, Yu Bai<sup>2</sup> and J. Toby Mottram<sup>3</sup>

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# 9 ABSTRACT

10 Presented in this paper is a combined experimental and analytical modelling study of the strength of pultruded FRP single bolted double-lap joints subjected to tensile loading and 11 12 elevated temperatures. Dynamic mechanical analysis (DMA) and thermogravimetric analysis 13 (TGA) are conducted on the polymeric composite material to determine the glass transition 14 temperature and decomposition temperature, respectively. Based on the DMA and TGA 15 results, and to cover glass transition without any material decomposition, the six temperatures selected for the test program are +23 °C, +60 °C, +100 °C, +140 °C, +180 °C and +220 °C. 16 17 Three nominally identical joints are tensioned to failure at each temperature. A total of 36 18 double-lap joints are tested, comprising 18 joints fabricated with ordinary steel bolting and 19 the other 18 with novel blind bolting. A comparison is made based on load-displacement 20 curves, failure modes and maximum (ultimate) loads. It is found that both methods of 21 mechanical fastening experience a reduction of 85% in maximum load as the test temperature 22 increases from +23 °C to +220 °C. Three proposed empirical or mechanism-based models for 23 characterising strength under elevated temperatures are shown to provide good predictions 24 for the maximum loads obtained in the test program.

<sup>2</sup> Senior lecturer, corresponding author, Department of Civil Engineering, Monash University, Clayton, VIC 3800, Australia. Tel: +61 3 9905 4987; Email: yu.bai@monash.edu; Fax: +61 3 9905 4944.

<sup>&</sup>lt;sup>1</sup> Research Fellow, Department of Civil Engineering, Monash University, Clayton, VIC 3800, Australia. Email: chao.wu@monash.edu.

<sup>&</sup>lt;sup>3</sup> Professor, Civil Research Group, School of Engineering, University of Warwick, Coventry CV4 7AL, UK. Email: Toby.Mottram@warwick.ac.uk

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# 27 INTRODUCTION

Pultruded fibre reinforced polymer (PFRP) composites are thin-walled shapes that have constant cross-section along their straight length. Over the last 20 years they have witnessed increasing R&D (Mottram, 2015), and have been adopted in new all-FRP constructions (Bank, 2006; Pendhari *et al.*, 2008). Their attraction in civil engineering is mainly due to their advantages in reduced manufacturing cost, light weight, ease of installation, and low maintenance cost because of their resistance to harsh environmental conditions (Hollaway, 1993; Bakis *et al.* 2002).

35 Connecting PFRP shapes in structural systems is the key to providing structures that 36 are reliable and possess structural integrity (Bank, 2006; Mottram and Turvey, 2003). For 37 connecting elements, steel bolting is a preferred connection method because of its low cost, 38 ease of installation/disassembly and straightforward inspection procedures with manageable 39 quality control (Turvey, 2000; Mottram and Turvey 2003). In physical situations where 40 access for tightening the bolting from both sides is restricted (such as when connecting 41 tubular hollow shapes (Wu et al., 2014)), blind bolts, requiring access from one side only 42 offer a convenient engineering solution (Evernden and Mottram, 2006).

It is well known (Wong and Wang, 2007; Wang *et al.*, 2011; Bai *et al.*, 2008; Correia *et al.* 2013) that the mechanical properties of PFRP materials degrade when the temperature reaches and exceeds the glass transition temperature ( $T_g$ ). What is not well understood is the effect of elevated temperature on the mechanical response of bolted joints loaded to ultimate failure.

48 Although bolted joints in PFRP structures are subject to complex stress states 49 (Turvey 2000; Bank 2006), it has been shown in Girão Coelho and Mottram (2015) that to

50 understand bolted joint response and failure we first are to characterize PFRP joints under a 51 single stress condition. As an example of this strategy, Kim and Whitney (1976) investigated 52 the pin-bearing strength of laminated composites under hot-wet conditions. Three 53 graphite/epoxy laminates were kept in a humidity chamber at a relative humidity of 98% until 54 the specimens showed a weight increase of 1.5%. Then the specimens for a single steel bolted 55 joint were tested at a moderate temperature of 126.7 °C (260 °F). The results showed a maximum strength reduction of 40% after the aging conditioning. In a study by Scarponi et al. 56 57 (1997), single steel bolt joints of T300/934 carbon fibre/epoxy laminate were tested under 58 combined changes in temperature and lateral tightening torque. The test matrix included five 59 temperatures in the range -150 °C to +80 °C, with four tightening torques to the steel bolting 60 of 0 Nm, 5 Nm, 30 Nm and 50 Nm. It was found that the bearing strength reduced from 356 61 MPa to 313 MPa when the temperature increased from room temperature to +80 °C without a 62 bolt tightening torque. Tightening the single 9.4 mm diameter bolt to 50 Nm significantly 63 increased the bearing strength by over four times from 313 MPa to 1371 MPa, even when the 64 temperature was +80 °C. Hirano et al. (2007) studied the effects of temperature on the pinbearing strength of two carbon FRPs. In their test matrix the three temperatures adopted were 65 -100 °C, +25 °C and +150 °C. The change in failure mode was recorded over the temperature 66 67 range and strength decreased by a maximum of 41%. Although these previous studies have 68 contributed knowledge to the understanding of the thermal-mechanical response of FRP 69 bolted joints, they used aerospace carbon FRPs that respond to bolt bearing load differently to 70 PFRPs.

Turvey and Wang (2001, 2007a, b, 2009a, b), and Zafari and Mottram (2012) performed series of tests with bolted connections that showed that there were strength reductions when the material was PFRP. Single bolted joints (10 mm diameter hole and 9.8 mm diameter bolt (*D*)) were tested by Turvey and Wang (2007b) in batches of three under 75 tension load at room temperature, +60 °C and +80 °C. Bolt tightening was to the 'finger tight' 76 condition (defined as the tightness attained by the resistance to bolt tightening using human 77 fingers only; it will provide through-thickness lateral restraint once the bolted joint is loaded). 78 Two geometrical configurations for the double-lap joints were arbitrarily found to achieve 79 bearing failure or net tension failure at room temperature, respectively. All bearing-designed 80 joints showed the same consistent bearing failure mode at the elevated temperatures. A 81 reduction of 39% in bearing strength (there was lateral restraint with the steel bolting) was found at +60 °C and a higher decrease of 51% was found at +80 °C. More significant 82 83 reductions were determined with the net tension designed joints, with 49% reduction at +60 84 °C and 56% reduction at the highest temperature of +80 °C. This higher reduction for net 85 tension was associated with a change of the failure mode from tension (at room temperature) 86 to bearing (at  $+60 \degree C$  and  $+80 \degree C$ ).

87 Turvey and Wang (2009a) tested PFRP joints having two bolts in a single column at room temperature and +60 °C. The geometric configurations studied included three end (edge) 88 89 distance-to-bolt diameter ratios (E/D), two pitch distance-to-bolt diameter ratios (P/D) and 90 two side distance-to-bolt diameter ratios (S/D). A joint's ultimate load was defined as the 91 maximum load that it resisted, whereas the damage load was when there was first evidence of 92 a reduction for a change in joint stiffness in the (linear) load-displacement response. It was 93 found that changing the three geometric ratios had an effect on the thermal-mechanical 94 properties (damage load and ultimate load). Test results showed that when the temperature was increased to +60 °C, the average reduction in ultimate load was 17%, and for damage 95 96 initiation it was higher at 42%, regardless of the geometric configuration. It is interesting to 97 note that, when E/D was 4, P/D was 2 and S/D was 4, the maximum strength reductions were 98 recorded for ultimate load at 36% and for damage load at 59%. Zafari and Mottram (2012) reported a study for the pin-bearing strength of an PFRP material for the web of a wide flange 99

100 shape. Specimens were soaked in water for 3000 hours at +40°C before pin-bearing loading 101 at room temperature. The test matrix involved the presence of a clearance hole and four steel 102 pins (plain bolt shafts) for diameters of 10 to 25 mm. It was found that when the bearing load 103 was in the pultrusion direction, the average strength reduction was 30% for increasing shaft 104 diameter.

105 Previous studies with PFRP materials do provide some insight for understanding temperature effects on the mechanical behaviour of bolted joints. One limitation in their 106 107 scope of application is that the temperature has ranged up to +80 °C, and this only covers the 108 initial stage of the glass transition process. Work is required to understand mechanical 109 performance when elevated temperatures encompass the full range of glass transition and 110 toward the decomposition temperature. FRP structures may experience temperatures higher 111 than +80 °C in extreme events such as localized heating from a fire. To have the data to 112 design for safety, characterisation of the mechanical response of PFRP joints is essential over 113 a higher temperature range, including  $T_{g}$ .

114 Because blind bolts are convenient when access for ordinary bolting is poor, a novel 115 type of blind bolt has been included in the test program. Wu et al. (2014) reported on both 116 static and fatigue results for PFRP double-lap joints with this blind bolting, but not for 117 temperatures higher than room temperature. This paper presents new test results for joints with a single bolt to an elevated temperature of +220 °C. To establish the temperatures in the 118 119 test program, dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA) 120 tests were conducted with the PFRP material to determine  $T_{g}$  and decomposition temperature 121  $(T_{\rm d})$ . On the basis of these measurements, the experimental temperatures were selected to be +23 °C, +60 °C, +100 °C, +140 °C, +180 °C and +220 °C, which exceeded  $T_g$  without the 122 123 occurrence of material decomposition. A total of 36 PFRP joints were failed and their load-124 displacement curves were constructed. Damage and ultimate loads from the 18 ordinary and 125 18 blind bolted joints were compared. Three existing models for strength change with 126 temperature were assessed and compared in terms of their reliability and relevance to predict 127 the maximum loads for a single bolted joint at elevated temperatures.

# 128 EXPERIMENTAL PROGRAM

## 129 Materials

130 The PFRP plate with a thickness of 5.5 mm was supplied by Nanjing Xingya FRP Co. Ltd. The same polymeric composite material was used in the bolted joint study by Wu et al. 131 132 (2014). It consists of E-glass fibre reinforcement embedded in a polyester resin matrix. Fibre 133 volume fraction and fibre architecture were characterised according to ASTM D-3171, and 134 full details are reported in Wu et al. (2014). The overall fibre volume fraction is 48%. The 135 plate lay-up has a symmetric and balanced reinforcement scheme, with rovings in the core, 136 sandwiched between two layers of a continuous strand mat (CSM). Measured tensile 137 properties in directions longitudinal and transverse to the pultrusion direction are reported by 138 Wu et al. (2014), following tensile coupon tests in accordance with ASTM D 3039. The 139 longitudinal tensile modulus and strength are 32 GPa and 393 MPa, respectively, and in the 140 transverse direction these properties are lower at 5 GPa and 22 MPa, respectively. Using the 141  $10^{\circ}$  off-axis tensile test method, detailed by Chamis and Sinclair (1976), the mean in-plane 142 shear strength is 25.4 MPa from testing a batch of ten coupons.

The ordinary bolts, 45 mm in length, are made of M10 zinc-plated steel and supplied by Exafast. The steel grade is 4.6, with a nominal tensile strength of 400 MPa, which is equivalent to a Grade A bolt as specified in ASTM A307. The ordinary bolt has a tensile capacity of 13.9 kN and a single shear capacity of 9.3 kN, according to BS 5950. The measured diameter of the shank (smooth part) is 9.8 mm. The blind bolts, 60 mm in length, are M10 high tensile yellow-zinc plated, and were supplied by Blind Bolt Australia. The tensile capacity of the blind bolt is 12.9 kN and the single shear capacity over the thread is 23.2 kN. The measured shank diameter is 9.93 mm. The washers for both bolt types are zincplated fenders with inner diameter 10 mm, outer diameter 25.4 mm and thickness 1.75 mm.
For ordinary bolted joints, two washers were placed beneath bolt head and nut. For blind
bolting a single washer was used on the accessible side of the bolt. For the installation
process of a blind bolt the reader is invited to consult the detailed description given by Wu *et al.* (2014).

#### 156 **DMA Testing**

157 DMA was performed with the PFRP plate material to obtain the temperature-dependent 158 mechanical properties of storage and loss moduli. These test results enabled determination of 159 the glass transition temperature  $(T_g)$  required to know the elevated temperatures for the test 160 program. A Q800 dynamic mechanical analyser from TA Instruments was used in accordance 161 with ASTM D5023-07. Rectangular specimens of PFRP were cut with dimensions 60 mm by 162 12 mm, with the longer sides parallel to the direction of pultrusion. A specimen was tested in 163 a three-point bending set-up at a dynamic oscillation frequency of 1 HZ. Scanning was carried out over a temperature range from -40 °C to +300 °C, at four different heating rates of 164 3 °C/min, 5 °C/min, 7.5 °C/min and 10 °C/min. 165

166 As typical examples of DMA results, the three curves plotted in Fig. 1 are for the 167 storage modulus, E' (solid line), loss modulus, E'' (dashed line), and damping factor, given 168 as tan  $\delta$  (long-short dashed line), at a heating rate of 10 °C/min. E' represents the elastic 169 modulus of the  $60 \times 12 \times 5.4$  mm specimen in flexure. T<sub>g</sub> in this paper is defined as the 170 temperature at which the peak of the E'' is reached. In the second row of columns (2) to (5) in Table 1 are reported the  $T_{gs}$  for the four heating rates. It is seen from the DMA 171 characterization that  $T_g$  increased by 10 °C from 143 °C to 153 °C as the applied heating rate 172 increased from 3 °C/min to 10 °C/min. 173

174 TGA Testing

TGA was performed in order to determine the decomposition temperature,  $T_{d}$ . Testing was 175 176 carried out using an STA 409 PC/PG simultaneous thermogravimetry and differential 177 scanning calorimetry analyser from NETZSCH. Specimens were created by grinding the 178 PFRP material into a powder using a rasp. Samples were taken throughout the plate's 5.5 mm thickness to ensure that the fibre and resin content in the powder samples was representative. 179 The analyser took scans from room temperature up to 800 °C with the sample in a nitrogen 180 atmosphere, having a flow rate of 10 ml/min. As with the DMA testing, the four heating rates 181 182 were 3 °C/min, 5 °C/min, 7.5 °C/min and 10 °C/min.

183 Plotted as a solid curve in Fig. 2 is the remaining mass of the sample versus the 184 increase in temperature at the heating rate of 10 °C/min. The mass reduction rate curve, 185 shown as a dashed curve in the figure, was constructed from the derivation of the remaining 186 mass curve. According to Kale et al. (2006),  $T_d$  is determined when the maximum mass 187 reduction rate is achieved. For the four heating rates, the third and fourth rows in Table 1 report  $T_d$  and the corresponding remaining mass as a percentage. The PFRP plate is found to 188 decompose at a temperature in excess of 365 °C, and at 800 °C the remaining mass is 77.4% 189 190 (mainly the fibres and matrix additives). In the resin burn-off test procedure by Ye et al. 191 (1995) which is used to establish volume fractions of the constituents the required constant 192 furnace temperature (for 2 hours) is under 600 °C.

# 193 **PFRP Bolted Joints for Tensile Testing under Elevated Temperatures**

Fig. 3 presents the details and dimensions of the double-lap single bolted joints. Fig. 3a shows side and plan engineering drawings; photographs for the same views are given in Fig. 3b. All joints had the same total length of 306 mm and width of 80 mm. Dimensions chosen for the PFRP joint detailing (whiter plates illustrated in Fig. 3) were specified using the EUROCOMP Design Code (Clarke, 1996), and were found also to satisfy the Italian guidance given in CNR-DT 205/2007 (Anonymous, 2008) for the design of PFRP elements. 200 For the single bolted joint, D is 9.8 mm and the geometrical ratios are E/D of 4.0 and S/D of 201 4.0, for the width of 8D. The centrally placed hole has diameter 10.5 mm for a clearance hole 202 of about 0.7 mm. As encountered previously by Bai and Keller (2009), premature failure of 203 the outer CSM layers occurred when the PFRP plate was directly clamped by the testing 204 machine grips, especially when the polymeric composite was subjected to tensile loading 205 under elevated temperatures. To avoid this undesirable failure mode, a steel gripping fixture was added at both ends of the PFRP joint, as can be seen by the darker components in Fig. 3. 206 207 The whiter region of the specimen in Fig. 3 is the bolted joint assembled from three 5.5 mm 208 thick PFRP plates. With the steel fixtures in place the tensile loading could be reliably 209 transferred into the PFRP bolted joint. The steel plates of the same thickness were connected 210 to the PFRP joint by two 8 mm diameter bolts of steel (M8) grade 4.6 in a single row. The 211 measured diameter of the M8 bolting was 7.9 mm. The capacity of the connection between 212 steel and PFRP was designed to be stronger than that of a single 10 mm PFRP bolted 213 connection. It should be noted that, for all specimens, only the plain (smooth) shank of the 214 M10 bolt was in contact with the hole of the inner PFRP plate of the joint.

A clamping force of 3 Nm was applied to the single M10 bolt using a calibrated torque wrench. This relatively low tightening torque was chosen to negate the complication of a significant long-term reduction in clamping tension from material creep and viscoelastic relaxation (Cooper and Turvey, 1995; Mottram, 2005). To facilitate a fair comparison, the 18 joints with the blind bolting method of connection had the same dimensions and bolt torque as did the 18 ordinary bolted joints.

# 221 Experimental Set-up and Test Method

The experimental set-up is shown in Fig. 4. All tensile tests were carried out using an Instron 5982 Dual Column Testing System machine with a load capacity of 100 kN. For the temperature control at elevated temperatures the testing machine has an Instron 3119-408 225 Environmental Chamber with a maximum working temperature of +600 °C.

226 To monitor temperature, a K-type thermal couple with the sensitivity of  $\pm 1$  °C was 227 inserted between the outer and inner PFRP plates of the dummy specimen seen in Fig. 4. The 228 tip of the thermal couple was located close to the bolt region under investigation. The dummy specimen had identical detailing, and its constant temperature was considered to be the same 229 230 as that of the tested joint specimen. Fig. 4 shows reflective sticker markers attached to the 231 outer and inner PFRP plates. Their vertical separation of 70 mm set an initial gauge length for 232 measurement of the joint's displacement (extension) using an MTS LX 500 non-contact Laser Extensometer with a strain resolution of 1 µm; the scan rate was 100 scans/s. The 233 234 gauge length was the same in all 36 joints. The extension measured was for the relative 235 displacement of the outer and inner PFRP plates within the joint region; vertical deformation 236 in other regions was excluded.

237 After installation of the unloaded specimen, the door of the environmental chamber 238 was locked and the temperature inside was increased to the target temperature at a rate of 5 239 <sup>o</sup>C/min. The temperatures of the chamber and the specimen were continuously monitored. 240 When the target temperature was reached, it was kept constant for 30 minutes to ensure that 241 the temperature within the whole specimen was uniform and stable. The soaking time 242 selected was 10 minutes above the minimum recommendation of 20 minutes by Turvey and 243 Wang (2007a). The joint specimen was next loaded to ultimate failure under stroke control at 244 a constant rate of 0.5 mm/min. Load was applied in the pultrusion direction because this is 245 the main load carrying direction in PFRP structures (Bank 2006). Because the joint ultimate 246 failure occurred in a relatively short time (5 minutes to maximum tension) from the onset of 247 stroke controlled loading, the current study did not monitor any creep response.

From the DMA and TGA test results in Table 1,  $T_g$  ranged from 149 °C to 153 °C and  $T_d$  from 368 °C to 399 °C. Based on these findings the six test temperatures were +23 °C,

 $+60 \,^{\circ}\text{C}$ ,  $+100 \,^{\circ}\text{C}$ ,  $+140 \,^{\circ}\text{C}$ ,  $+180 \,^{\circ}\text{C}$  and  $+220 \,^{\circ}\text{C}$ , to cover  $T_{g}$ , and to ensure that no PFRP decomposition occurred. For each temperature, three identical joints, for each of the two bolting methods, were tested to obtain a measure of batch variability. The test program thus comprised 18 joints with ordinary bolting and 18 with blind bolting.

The aim of this paper is to obtain the thermal-mechanical degradation of a PFRP bolted joint against elevated temperatures. The influence of loading once the joint was at a target temperature is expected to be higher than if the test method followed a thermal-loading procedure, such as for the fire curve in ASTM E119.

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# 259 EXPERIMENTAL RESULTS

Table 2 reports the test results for the 36 specimens. In column (1) a label for the specimen is given. In the labelling scheme 'O' is for ordinary bolting and 'B' is for blind bolting. The number following the bolt type represents the target test temperature ( $+60 \,^{\circ}C$ ,  $+100 \,^{\circ}C$ ,  $+140 \,^{\circ}C$ ,  $+180 \,^{\circ}C$  or  $+220 \,^{\circ}C$ ); 'R' in the specimen label stands for room temperature, that is,  $+23 \,^{\circ}C$ . The number 1, 2 or 3 after the hyphen in the label is for the order of specimen testing in the batch of three.

266 The typical failure modes observed under elevated temperatures are presented in 267 Figs. 5a to 5f for ordinary bolting and in Figs. 6a to 6f for blind bolting. Because all joints failed within the inner PFRP (refer to Fig. 3), an outer PFRP plate on one side had to be 268 269 removed to expose the failure pattern for the photograph. It can be seen in the 12 photographs 270 that shear-out was the final failure mode, regardless of the value of elevated temperature and 271 the type of bolt. Inspection of the images shows that the surface colour of the PFRP material 272 gradually changed from 'white' to 'brown' with the increase of temperature from RT to +220 273 °C. A similar colour change was observed for PFRP reinforcing bars under extreme 274 temperatures by Robert and Benmokrane (2010). The work of Asmussen (1983), Burton (1993), Peutzfeldt and Asmussen (1990) and Tsotsis (1995) indicates that this colour change
is likely due to oxidation of the polymer matrix in an air atmosphere.

277 Plotted in Figs. 7 and 8 are typical load-displacement curves for O and B joints, 278 respectively. The displacement is the separation of the vertical markers as measured by the non-contact laser extensometer. The six temperatures are each represented by a single 279 specimen, with the curves coloured as follows: black for +23 C; red for +60 °C; green for 280 +100 °C; blue for +140 °C; pink for +180 °C; purple for +220 °C. Inspection of their 281 282 characteristics shows an obvious enormous reduction in joint stiffness and maximum load as temperature increased. For joints with ordinary bolting at +23 °C and +60 °C the tensile load 283 284 increased linearly to maximum load for ultimate failure. The load then fell away rapidly as 285 the shear-out mode allowed damage to progress and the axial displacement to reach 20 mm. 286 The lower load level of 2 to 3 kN that was maintained at displacements > 4 mm is mainly attributed to a resistance from the frictional forces created by the lateral restraint of the 3 Nm 287 bolt torque (and difference in thermal expansion). A second contribution (Abd-El-Naby and 288 289 Hollaway, 1993) to this residual load can be an interlocking mechanism from the shear-out 290 failure having through-thickness deformations. When the temperature exceeded +60 °C the 291 load-displacement curve is seen to be non-linear prior to the maximum load. In addition, the 292 load reduction after the peak was less rapid when the temperature exceeded +180 °C. As an 293 example, the purple load-displacement curve in Fig. 7 for +220 °C temperature is seen to 294 become almost horizontal after the maximum tension.

Fig. 8 indicates that the equivalent load-displacement characteristics of the blind bolted joints are generally similar to those determined with the ordinary bolted joints. At +23  $^{\circ}$ C and +60  $^{\circ}$ C, the load dropped suddenly after an initial linear increase to the maximum load. Then a residual load, at a similar level to that in the O joints, was obtained once the axial displacement reached 16 mm. A non-linear response before the maximum load is evident 300 when the test temperature is +100 °C. When the temperature increased above +140 °C there 301 was no sudden loss in stiffness, and once the maximum load was attained the load-302 displacement curve continued virtually horizontally at this residual load level.

303 In Table 2, columns (2) and (3) list the target test temperature  $(T_{target})$  and the 304 measured temperature ( $T_{\text{measured}}$ ) when the specimen was loaded (at 0.5 mm/min) to failure. 305 The maximum (peak) load  $(P_{max})$  recorded is reported in column (4). These maximum or 306 ultimate loads were extracted from the corresponding load-displacement curves. Columns (5) and (7) in Table 2 show the average measured temperature ( $T_{avg.measured}$ ), average maximum 307 load  $(P_{avg,max})$  from a batch of three specimens (e.g. OR-1 to OR-3), and the percentage 308 309 reduction in maximum load based on the average (column (6)) at RT. It can be seen from 310 either the  $P_{\text{max}}$  or  $P_{\text{avg.max}}$  results in Table 2 that both O and B joints experienced an obvious 311 degradation with the increase of temperature. For the ordinary bolted joints, it is seen that  $P_{\text{avg.max}}$  changes from 15.5 kN at room temperature to 13.3 kN at +60 °C, giving a 14% 312 313 reduction. At +100 °C the resistance is reduced by 38%, with  $P_{\text{avg.max}} = 9.65$  kN. The next 40 314 <sup>o</sup>C increase has a significant effect of lowering strength, as  $P_{\text{avg,max}}$  is 5.60 kN for a 74% 315 reduction. Further reductions in  $P_{avg,max}$  at +180 °C to 78% and at +220 °C to 85% have been 316 obtained with the O joints.

A similar reduction trend in  $P_{avg.max}$  is observed for B joints at the six test temperatures.  $P_{avg.max}$  at +60 °C is 11.9 kN, which is found to be only 3% below the RT average of 12.3 kN. A significant reduction of 37% occurs when the temperature increases to +100 °C. At +140 °C  $P_{avg.max}$  is 3.2 kN, only a quarter of its RT value. Further reductions at +180 °C to 79% and at +220 °C to 85% occur with the B joints, and it is noteworthy that these are precisely the same percentages as achieved with the ordinary joints. This finding indicates that joint strength at the highest temperature is independent of bolt type.

#### 325 DISCUSSION OF EXPERIMENTAL RESULTS

326 Although both O and B joints showed the same shear-out failure mode in Fig. 5 and Fig. 6, 327 respectively, they have different maximum loads. It is evident from the results in Table 2 that  $P_{\text{avg.max}}$  for the six B batches are lower, on average, by 23% than their six equivalent O 328 batches. From the determination of the change in maximum load using  $((P_{avg,max,O} -$ 329  $P_{\text{avg.max.B}}$  )/ $P_{\text{avg.max.O}}$  ×100% it can be seen that the relative difference is independent of 330 temperature. For example, at +23 °C,  $P_{avg.max.B}$  is 21% lower than  $P_{avg.max.O}$ . At the three 331 elevated temperatures of +100 °C, +180 °C and +220 °C, the relative differences are found to 332 333 be 20%, 24% and 19%, respectively. An overall relationship cannot be established because at +60 °C the magnitude of  $P_{avg,max,B}$  is 11% lower and, with a difference of 42%, there is a 334 335 second outlier at +140 °C.

336 To explain the strength differences between O and B joints, all 36 specimens were 337 disassembled to investigate the detailed interaction between the single blind or single 338 ordinary bolt with its bolt hole. It was found that the contact area between the blind bolt shaft 339 and PFRP plate was reduced due to the slot in the bolt's shaft. This reduction in the contact area is illustrated in Fig. 9 (after testing at +220 °C). The B bolt is found to have roving 340 341 reinforcement from the inner PFRP plate packed into the slot opening in the plain length of 342 the steel shaft. The reduced contact area resulting from the detailing of the blind bolt causes a 343 stress concentration state at the bolt hole, leading to failure at a lower strength of the B joint. 344 Similar observations and comments on PFRP joints with blind bolts have been reported by Wu et al. (2014). 345

According to the experimental results in Table 2 both O and B joints experienced a considerable reduction in strength of up to 85% when the temperature reached +220 °C. This reduction is associated with the matrix dominant failure mode of shear-out. For fire engineering this reduction could be relevant for ultimate limit state design. Note that with the 350 pultrusion composite process it could be impractical to avoid having a matrix dominant 351 failure in bolted joints because of the mechanical properties obtained from having the 352 standard fibre architecture and reinforcement types.

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# 354 MODELLING OF ULTIMATE LOADS UNDER ELEVATED TEMPERATURES

As seen from the 12 photographs in Figs. 5 and 6 the single bolted joints ultimately failed by the shear-out mode in the inner plate. The strength for this mode of failure can be predicted from using formula (Bank, 2006):

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$$P_{\rm sh} = 2 \times t \times E \times \tau_{\rm LT,T}.$$
 (1)

In Eq. (1), t is 5.5 mm for the thickness of the PFRP plate; E is 40 mm for the distance from 359 360 bolt centre to the free end of joint, as shown in Fig. 3;  $\tau_{LT,T}$  is the in-plane shear strength of 361 the PFRP plate material as a function of temperature T. Because two sheared surfaces are created for the failure mode,  $P_{\rm sh}$  is taken to be the shear force resistance of one surface 362 363  $(t \times E \times \tau_{LT,T})$  multiplied by 2. It should be noted that Eq. (1) is based on a number of assumptions, one of which is that forces generated by bolt clamping and by interaction 364 365 between inner and outer plates can be neglected. A second assumption is that the in-plane 366 shear strength is constant along the shear failure surfaces. Thirdly, it is assumed that the 367 temperature increase in the steel bolt does not have an effect on joint strength. To support this 368 assumption we observe that no yielding or damage in the steel bolting was observed. At 220 369 <sup>o</sup>C the modulus of elasticity of the steel will not have reduced by 10%, and so compared to 370 the PFRP's reduction the bolting appears rigid.

The unknown parameter in the strength equation is  $\tau_{LT,T}$  of the PFRP material at elevated temperatures. Several analytical models for closed-form expressions are proposed in the literature for strength characterisation of FRPs under elevated temperatures. They are either empirical equations based on curve fitting to experimental test results (Mahieux *et al.*  375 2001; Gibson et al. 2006; Feih et al. 2007; Correia et al. 2013) or based on kinetic description 376 of glass transition (Bai and Keller, 2009). Generally, it is found that the empirical models 377 yield close agreement with experimental results, probably implicit in applying the curve 378 fitting approach. Their weakness is that they lack a physical background, and must rely on the 379 availability of experimental data that is known to be relevant and reliable. On the other hand, 380 any mechanism-based model will require additional data from the physical description of the 381 glass transition stage (as the material state changes from glassy state to leathery state with the 382 breakdown of secondary bonds), and it can be challenging to precisely characterise the 383 required modelling parameters.

In this paper, two empirical models and one mechanism-based model are selected and compared for characterisation of the temperature-dependent in-plane shear strength in Eq. (1).

387 The empirical model of Feih *et al.* (2007) expresses strength as a function of 388 temperature by:

389 
$$\tau_{\text{LT,T}} = \left[\frac{\tau_{\text{LT,G}} + \tau_{\text{LT,L}}}{2} - \frac{\tau_{\text{LT,G}} - \tau_{\text{LT,L}}}{2} \tanh(\varphi(T - T_k))\right] R_{\text{rc}}(T)^n$$
(2)

390 where  $\tau_{LT,G}$  and  $\tau_{LT,L}$  are the in-plane shear strengths in a glassy state (a state at room 391 temperature) and in a leathery state (a state after glass transition and before decomposition), 392 respectively;  $\varphi$  and  $T_k$  are parameters obtained by curve fitting of experimental data;  $R_{rc}(T)^n$ 393 is a scaling function considering the mass loss during the decomposition process. Because 394 there is no FRP decomposition in the current bolted joint study this parameter is set to1.0.

For the second empirical model, a descriptive model proposed by Correia *et al.*(2013) is based on Gompertz's distribution. It has the expression:

397 
$$\tau_{\mathrm{LT,T}} = \left(1 - e^{Be^{CT}}\right) \times \left(\tau_{\mathrm{LT,G}} - \tau_{\mathrm{LT,L}}\right) + \tau_{\mathrm{LT,L}}$$
(3)

398 where  $\tau_{LT,G}$  and  $\tau_{LT,L}$  are as in Eq. (2); coefficients *B* and *C* are shape and scale parameters 399 determined from fitting the expression to experimental data. Correia *et al.* (2013) showed that 400 the model described by Eq. (3) gave a close prediction for the in-plane shear strength 401 (measured using the  $10^{\circ}$  off-axis test method) of a PFRP material.

402 Bai and Keller (2008) proposed a model based on the well-known rule of mixtures 403 as:

404 
$$\tau_{\text{LT},\text{T}} = \tau_{\text{LT},\text{G}}(1 - \alpha_{\text{G},\text{T}}) + \tau_{\text{LT},\text{L}}\alpha_{\text{G},\text{T}}(1 - \alpha_{\text{D},\text{T}}) + \tau_{\text{LT},\text{D}}\alpha_{\text{G},\text{T}}\alpha_{D,T} .$$
(4)

405 where  $\tau_{LT,G}$  and  $\tau_{LT,L}$  are the same shear strengths as in Eqs. (2) and (3).  $\tau_{LT,D}$  is the shear 406 strength at the decomposition state and  $\alpha_{G,T}$  and  $\alpha_{D,T}$  are the conversion degrees for the glass 407 transition and decomposition at temperature *T*, respectively. Since there was no PFRP 408 decomposition at +220 °C it is appropriate to let  $\alpha_{D,T} = 0$ . Parameter  $\alpha_{G,T}$  can be characterised 409 based on the kinetic theory via the Arrhenius equation, which is based on Maxwell– 410 Boltzmann distribution:

411 
$$\frac{d\alpha_{G,T}}{dT} = \frac{A_G}{\beta} \exp\left(\frac{-E_{A,G}}{RT}\right) (1 - \alpha_{G,T})^{n_G} .$$
 (5)

412 where  $A_G$  is the pre-exponential factor,  $E_{A,d}$  is the activation energy and  $n_G$  is the reaction 413 order. *R* is 8.314 J/mol.K for the universal gas constant and  $\beta$  is the constant heating rate at 3 414 °C/min. Bai and Keller (2008) explain in detailed how to establish values for the model's 415 parameters of  $A_G$ ,  $E_{A,d}$  and  $n_G$ .

416 In addition to the rule of mixtures model giving  $\tau_{LT,T}$  in Eq. (4), Bai and Keller 417 (2008) proposed the alternative of using an inverse rule of mixtures approach, which gives 418 the lower bound estimation for  $\tau_{LT,T}$ , via:

419 
$$\frac{1}{\tau_{\text{LT,T}}} = \frac{1 - \alpha_{\text{G,T}}}{\tau_{\text{LT,G}}} + \frac{\alpha_{\text{G,T}}}{\tau_{\text{LT,L}}}$$
(6)

To apply the three models given by Eqs. (2) to (6), the in-plane shear strengths at glassy ( $\tau_{LT,G}$ ) and leathery ( $\tau_{LT,L}$ ) states are required. According to the test results in Table 2 the average maximum load ( $P_{avg.max}$ ) at +220°C was a mere 15% of the RT value. Similarly, as the plot in Fig. 1 shows, *E*', from the DMA testing, gave the same reduction rate over the 424 same temperature range. Because the shear-out failure is a matrix dominant mode it may be 425 assumed that  $\tau_{LT,L} = 0.15 \tau_{LT,G}$ .

The required parameters for the three models are presented in Table 3. Those for the 426 427 two models by Feih et al. (2007) and Correia et al. (2013) are different for the O and B bolt types, and were obtained by curve fitting of the experimental results reported in Table 2. The 428 429 kinetic parameters for the third mechanism-based model from Bai and Keller (2008) are 430 independent of bolt type and using the DMA test results were calibrated through the modified 431 Coats-Redfern method (Coats and Redfern, 1964, 1965). Because the six batches with blind 432 bolting gave  $P_{\text{avg.max}}$ , on average, 23% lower than that of the six batches with ordinary bolted 433 joints, it was necessary to factor  $\tau_{LT,G}$  by 0.77 when predicting  $P_{sh}$  for blind bolting using the 434 upper and lower bounds, i.e. Eqs. (4) and (6) from Bai and Keller (2008).

435 Figs. 10a and 10b are constructed to compare the analytical predictions from the 436 three models with  $P_{\text{avg.max}}$ s taken from Table 2. Fig. 10a is for the ordinary bolting with a 437 batch's Pavg.max located at the centre of the solid black circle symbols. Fig. 10b is the 438 equivalent figure for the blind bolted joints with open circle symbols for the  $P_{avg,max}$ s. The 439 error bars in the figures represent one standard deviation (SD) in  $P_{\text{avg.max}}$ , with the SD 440 calculated for the Gaussian statistical distribution from the three  $P_{\text{max}}$  results in a batch. 441 Predictions for  $P_{\rm sh}$ , using the four Eqs. (2) to (6) for  $\tau_{\rm LT,T}$  in Eq. (1), are plotted as continuous curves over the temperature range from 0 °C to +250 °C. The Feih et al. (2007) model results 442 443 are represented by the red curve and the Correia et al. (2013) model by the green curve. The 444 blue dashed curves are for upper (Eq. 4) and lower (Eq. 6) bound predictions using the model 445 of Bai and Keller (2008).

It can be seen in Fig. 10 that the three models yield satisfactory predictions in relation to the experimental results. The superior predictions by the two models by Feih *et al.* (2007) and Correia *et al.* (2013) benefit from their modelling ability being formed from 449 calibration of parameters by curve fitting to the same joint strength data plotted in the figures.

450 In order to compare the reliability of the predictions by the three models, the 451 following statistical process was followed (taking O joints as the example). First, at a specific 452 temperature, the ultimate joint load was predicted based on each model through Eqs. (2) to (6). Then the ratio of the prediction divided by the experimental result was calculated. Since 453 454 there are 18 experimental results for ordinary bolted joints, each of the three models produced 455 18 ratios. Finally, using the Gaussian statistical distribution the SD and coefficient of 456 variation (CoV) for the 18 ratios were calculated as parameters that quantified the predictive 457 reliability of each model. The same process was followed using the results with the B joints. 458 The statistical analysis was performed for the rule of mixtures in the Bai and Keller (2008) 459 modelling approach, but not for the inverse rules of mixtures approximation.

For O joints the Feih *et al.* model gives a SD of 0.06 kN and CoV of 0.06, the Correia *et al.* model a SD of 0.01 kN and a CoV of 0.11. The Bai and Keller model marginally overestimates the  $P_{\text{max}}$ s for the O joints by 14%, giving a SD of 0.14 kN and a CoV of 0.13. Bai and Keller (2009) have previously reported a similar overestimation when using their upper bound approximation.

For B joints the SD and CoV increase to 0.17 kN and 0.16 for the Feih *et al.* model. The same trend is found with the Correia *et al.* model, with a SD of 0.25 kN and CoV of 0.22. The upper bound solution by the Bai and Keller model yields a relatively higher SD of 0.34 kN with a CoV of 0.30. However, the empirical models require different sets of parameters (see Table 4) calibrated by curve fitting from the corresponding experimental data for O joints or B joints. Accordingly, the outcomes of these two approaches would be highly dependent on the availability and reliability of experimental data.

The parameters required in the Bai and Keller model, using either the rule of mixtures or the inverse rule of mixtures bound approximation, can be conveniently

474 determined from a relatively small number of DMA data points by applying the modified 475 Coats-Redfern method (Coats and Redfern, 1964, 1965). Furthermore, only one set of 476 parameters (namely  $E_{A,d}$ ,  $A_G$ , and  $n_G$ ) needs to be calibrated, without the need for a curve 477 fitting procedure to experimental results. In addition, the upper and lower bound curves can give the strength range that should cover the experimental strength range. Because 478 479 experimental data is not always going to be available that corresponds to joint detailing to be designed in PFRP structures (Turvey, 2000; Bank, 2006), the mechanism-based model 480 481 provides a rational procedure for strength prediction in PFRP structures when subjected to 482 elevated temperatures.

483 It should be noted that these observations are made based on the shear-out mode, 484 which is commonly found with single bolted joints made with relatively highly orthotropic 485 PFRP material (Cooper and Turvey 1995; Turvey and Wang 2007b). The experimental 486 observations and the kinetic modelling methodology presented herein can provide the basis 487 for us to understand how temperature affects other modes and joints subjected to different 488 loading conditions. The justification for this extension of our work is that Bai and Keller 489 (2009) showed that the mechanical degradation of a polymeric composite laminate is 490 fundamentally associated with a glass transition process.

491

## 492 CONCLUDING REMARKS

For the first time, tensile testing for strength variation was conducted on PFRP double-lap single bolted joints subjected to elevated temperatures from room temperature to +220 °C. Both ordinary and blind steel bolts were used to assemble 18 joints of each bolt type in six batches to cover characterisation over the temperature range. The thermal-mechanical responses of the 36 joints were studied by way of load-displacement curves, mode of failure

and maximum (ultimate) loads. The experimental maximum loads were compared withpredictions by applying three analytical models and satisfactory agreement was obtained.

500

Based on the current study the following conclusions can be drawn:

- 501 1) DMA and TGA test results showed that the glass transition temperature of the 5.5 502 mm thick PFRP plate material was in the range +143 °C to +153 °C, and that the decomposition temperature ranged from +368 °C to +399 °C; the variation was 503 504 seen to be dependent on the heating rate. On the basis of the DMA and TGA data the six selected test temperatures chosen were  $+23 \,^{\circ}C$ ,  $+60 \,^{\circ}C$ ,  $+100 \,^{\circ}C$ ,  $+140 \,^{\circ}C$ 505 +180 °C and +220 °C, to cover glass transition and to ensure that no PFRP 506 507 decomposition occurred. Within this temperature range, all ordinary and blind 508 bolted joints failed with the shear-out mode in the inner PFRP plate. For 509 temperatures > 100 °C it was observed that the surface colour of PFRP changed 510 from a 'white' to a 'darker brown'; the degree of colour change increased with 511 temperature. It is believed that surface polymer matrix oxidation in the air 512 atmosphere was the cause of the distinct colour change.
- 513 2) Elevated temperatures were found to modify the characteristics of the loaddisplacement curves for both ordinary and blind bolted joints. At room 514 515 temperature (+23 °C), load increased linearly with joint displacement up to the 516 maximum load, followed by a sudden load drop to a lower level that was constant 517 to an axial displacement of 20 mm. As the temperature was increased the load-518 displacement curve became increasingly non-linear before the maximum load was reached. When the temperature was higher than +100 °C it was found that the 519 520 joint's load decreased gradually after maximum load as the axial displacement 521 grew to 20 mm.

522 The average maximum load of joints (batches of three nominally identical 3) 523 specimens) with ordinary bolts was 15.5 kN at +23 °C. It dropped by 14%, 38% and 64% at temperatures of +60 °C, +100 °C and +140 °C. A significant reduction 524 to 78% (3.39 kN) was obtained at +180 °C, and at the maximum constant 525 temperature of +220 °C the average maximum load was 2.4 kN or 15% of that at 526 room temperature. For the blind bolt the average maximum joint load at +23 °C 527 was lower at 12.3 kN than for the ordinary bolt. For whatever reason, the strength 528 reduction was only 3.1% at +60 °C; significantly less than with the ordinary 529 bolting. Above +60 °C, reductions were 37%, 74% and 79% at temperatures of 530 +100 °C, +140 °C and +180 °C, respectively. Finally, at +220 °C, the average 531 532 maximum load was a mere 1.84 kN for a reduction of 85%; the same maximum 533 reduction as obtained with ordinary bolting. It was found that the average maximum loads in batches of blind bolted joints were, on average, lower by 23%; 534 535 the lower strength was caused by the slot (for blind fixing) in the steel shaft 536 introducing a damaging stress concentration state into the PFRP plate.

Models leading to closed-formed equations by Feih et al. (2007), Correia et al. 537 4) (2013) and Bai and Keller (2008) were studied to predict the maximum (ultimate) 538 539 loads of 36 failed joints. It was shown that predictions by the three models over the full temperature range agreed well with the experimental strength results. 540 541 Using the rule of mixtures and the inverse rule of mixtures approximations, the 542 modelling by Bai and Keller (2008) gave predictions for upper and lower bounds 543 to the joint ultimate load. The key advantage of the Bai and Keller model is that it 544 is not semi-empirical and so calibration of parameters does not rely on curve 545 fitting to available experimental test results. The authors therefore recommend its application when undertaking initial design calculations for the safe design of 546

547 PFRP bolted joints that are to be subjected to elevated temperatures up to the 548 decomposition temperature.

549

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#### 677 LIST OF CAPTIONS

## 678 **Table Captions:**

- Table 1. Experimental results of DMA and TGA tests
- 680 Table 2. Bolted joint specimen labels and experimental results from tensile tests
- Table 3. Calibrated parameters for Eqs. (2) to (5).

682 **Figure Captions:** 

- 683 Fig. 1. DMA test results at a heating rate of 10 °C/min.
- Fig. 2. TGA test results at heating rate of 10 °C/min
- Fig. 3. Double-lap joints for tensile testing under elevated temperatures: (a) joint dimensions
- of unit in mm, and (b) assembled double-lap joint with single blind bolt
- Fig. 4. Experimental set-up for tensile testing of PFRP bolted joints under elevatedtemperatures
- 689 Fig. 5. Failure modes of PFRP joints with single ordinary bolt at elevated temperatures of: (a)

690  $+23 \text{ }^{\circ}\text{C}$ ; (b)  $+60 \text{ }^{\circ}\text{C}$ ; (c)  $+100 \text{ }^{\circ}\text{C}$ ; (d)  $+140 \text{ }^{\circ}\text{C}$ ; (e)  $+180 \text{ }^{\circ}\text{C}$ ; (f)  $+220 \text{ }^{\circ}\text{C}$ .

691 Fig. 6. Failure modes of PFRP joints with single blind bolt at elevated temperatures of: (a)

692  $+23 \degree C$ ; (b)  $+60 \degree C$ ; (c)  $+100 \degree C$ ; (d)  $+140 \degree C$ ; (e)  $+180 \degree C$ ; (f)  $+220 \degree C$ .

- Fig. 7. Load-displacement curves of PFRP joints with ordinary bolts under elevatedtemperatures
- Fig. 8. Load-displacement curves of PFRP joints with blind bolts under elevated temperatures Fig. 9. Reduced contact area for the blind bolt: (a) failed PFRP blind bolted joint with one side outer PFRP plate removed, showing the context of the joint for investigation in (b) when the inner PFRP plate was removed, it showed rovings of inner PFRP plate filling the slot of blind bolt and (c) the roving filling in the slot of blind bolt was more obvious after the blind bolt was totally removed from the joint. Both (b) and (c) indicate the contact of the slot of

- 501 blind bolt with the hole of inner PFRP plate, resulting in a reduced contact area of the inner
- 702 PFRP plate.
- 703 Fig. 10. Comparisons between model predictions and experimental results for PFRP bolted
- 704 joints with a single: (a) ordinary bolt; (b) blind bolt.
- 705

# **Tables:**

# 708 Table 1. Experimental results of DMA and TGA tests

	(1)		Heating rate (°C/min)			
			5.0	7.5	10.0	
		(2)	(3)	(4)	(5)	
$T_{\rm g}$ (°	°C)	142.9	145.0	149.8	153.3	
$T_{\rm d}$ (°	°C)	367.7	389.5	394.4	399.4	
Rem	naining mass (%)	77.4	81.9	85.8	89.0	
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	Target	Measured	Max. load	Average measured	Average Max. load	Max. load
Specimen	temperature	Temperature	$P_{\rm max}$ (kN)	temperature	$P_{\rm avg.max}$	reduction
label	$T_{\text{target}}(^{\circ}\text{C})$	$T_{\text{measured}}$ (°C)		$T_{\text{avg.measured}}$ (°C)	(kN)	(%)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
OR-1	23	24.2	15.01			
OR-2	23	24.3	16.69	24.3	15.5	0
OR-3	23	24.5	14.74			
O60-1	60	62.3	13.27			
O60-2	60	62.5	13.27	62	13.3	14
O60-3	60	61.7	13.36			
O100-1	100	103	8.66		9.65	38
O100-2	100	102	10.38	102		
O100-3	100	102	9.91			
O140-1	140	143	5.87			
O140-2	140	143	5.47	142	5.60	64
O140-3	140	142	5.45			
O180-1	180	180	3.67			
O180-2	180	184	3.57	181	3.39	78
O180-3	180	179	2.93			
O220-1	220	219	2.37			
O220-2	220	213	2.08	216	2.29	85
O220-3	220	217	2.41			
BR-1	23	24.3	12.78			
BR-2	23	24.5	11.47	24.5	12.3	0
BR-3	23	24.6	12.49			
B60-1	60	61.3	12.02			
B60-2	60	61.2	11.98	61	11.9	3.1
B60-3	60	61.5	11.63			
B100-1	100	102	8.48			
B100-2	100	101	6.74	102	7.77	37
B100-3	100	103	8.08			
B140-1	140	139	3.24			
B140-2	140	141	3.30	139	3.24	74
B140-3	140	139	3.18			
B180-1	180	176	2.65			
B180-2	180	180	2.41	177	2.56	79
B180-3	180	177	2.63			
B220-1	220	213	1.95			
B220-2	220	212	1.72	213	1.84	85
B220-3	220	215	1.87			

Table 2. Bolted joint specimen labels and experimental results from tensile tests

# Table 3. Calibrated parameters for Eqs. (2) to (5).

Model	Parameters			
Model	Ordinary bolted joints	Blind bolted joints		
Feih et al. (2007) - Eq. (2)	$\varphi = 0.0179 \ ^{\circ}\text{C}^{-1}, T_{\text{k}} = 110 \ ^{\circ}\text{C}$	$\varphi = 0.0159 {}^{\mathrm{o}}\mathrm{C}^{-1},  T_{\mathrm{k}} = 84.1 {}^{\mathrm{o}}\mathrm{C}$		
Correia <i>et al</i> . (2013) – Eq. (3)	$B = -9.24, C = -0.0245 ^{\circ}\text{C}^{-1}$	$B = -3.79, C = -0.0216 ^{\circ}\mathrm{C}^{-1}$		
Bai & Keller (2008) – Eq. (5)	$E_{\rm A,d} = 16500 \text{ kJ.mol}, A_{\rm G} = 4.56, n_{\rm G} = 0.61$			