

16 **1. INTRODUCTION**

17 The Polyisocyanurate (PIR) foam polymer differs from the traditional Polyurethane (PUR) foam in the
18 stronger molecular structure that is achieved in its manufacturing procedure, consisting on highly cross-linked
19 isocyanurate rings [1,2]. These enhancements are traduced in the thermal stability, flame retardance and
20 mechanical behaviour of PIR [3,4], turning it more suitable than PUR to be used as a foam material in situations
21 in which these properties play a relevant role [5,6].

22 Polymeric foams are commonly used as core materials of composite sandwich panels in civil and military
23 structural applications [7,8]. In the former applications, which are the scope of the present research, the foam
24 material should combine stiffness with low thermal conductivity, fire resistance performance and lightness [9,10].
25 Having these properties, the foam maintains the integrity during its service life, acts as a thermal insulation material
26 and fire retardant, and has minor impact on the total weight of the structure [11,12]. These requirements explain
27 the use of rigid closed-cell polymeric foams (as PUR and PIR) in the sandwich panels, which are stiffer and more
28 heat flow resistant when compared to flexible open-cell foams [13].

29 When the sandwich panels are used as structural elements in a floor or a wall, the foam is mainly subjected
30 to shear stresses, whose influence in its integrity should be considered when the panels are being designed [14]. In
31 this context, the foam density usually depends on the configuration of the panel and the number of ribs providing
32 shear strength. In the panel structural design, the adoption of with ribs and low-density foams is often preferred,
33 as opposed to the solution where only high-density foams are adopted and ribs are absent. This is the case in the
34 work reported in [7], where a sandwich panel consisting of thin, interconnecting, fibre reinforced polymer (FRP)
35 facing sheets that form a bidirectional gridwork infilled with a polymeric foam, is described as the optimal solution.
36 Moreover, due to significant permanent loads on the panels, the shear creep behaviour of the foam and its
37 deformations should be accounted for and checked as well [15]. In this matter, the influence of temperature in the
38 shear creep effect can be significant [16].

39 Typically, the main function expected for PIR foams is thermal insulation. Therefore, publications
40 concerning their thermal response and fire resistance are abundant in literature [17–24]. However, other properties
41 of PIR still need to be assessed, particularly regarding its structural integrity and mechanical behaviour as a foam
42 material in composite structural sandwich panels. This paper intends to provide new knowledge within this scope.
43 At first, a literature review is made in order to provide the background on the relevant treated subjects. Then, the
44 experimental program developed is detailed, and its results are presented and discussed. The results obtained are

45 later used to calibrate modelling parameters of an analytical approach for predicting shear creep deformation of
46 PIR when subjected to expected stress levels and temperatures. Finally, the main conclusions of the work are
47 summarized.

48

49 **2. LITERATURE REVIEW**

50 Several studies were carried out to assess the compressive (e.g. [25,26]), tensile (e.g. [27]) and shear
51 strengths (e.g. [28]) of PUR foam. Creep behaviour of PUR is highlighted in the work of Huang and Gibson [29],
52 which considered the empirical Findley's power law [30,31] for the modelling of the PUR creep behaviour. More
53 recently, El Ghezal *et al.* [32] modelled PUR creep through a micromechanical approach. The temperature effect
54 on the mechanical properties of PUR is already well described in the works of Crawford *et al.* [33] and Garrido *et*
55 *al.* [34]. The influence of temperature on the creep behaviour of PUR subjected to compression and shear was
56 investigated by Briody *et al.* [35] and Garrido *et al.* [16], respectively. A significant influence was observed in
57 both studies.

58 Some research works were designed to assess the mechanical properties of PIR for compression and tensile
59 behaviour. Javni *et al.* [36] assessed the thermal response of PIR along with its compressive strength. Later, Stirna
60 *et al.* [37] developed an experimental campaign to observe PIR behaviour subjected to compressive and tensile
61 stresses, with deformations measured in the directions parallel and perpendicular to the applied load. In the last
62 years, some further developments were carried out, particularly the study of Andersons *et al.* [38] in which PIR
63 stiffness and strength were analysed considering their anisotropic performance. Continuing their research, the same
64 authors [39] evaluated the fracture toughness through experimental tensile tests, and the obtained results were
65 considered in a micromechanical approach that accounted for the microscopic properties of the PIR foam.

66 In terms of PIR shear behaviour and shear creep behaviour, information available in literature is absent.
67 This paper presents an experimental campaign aiming to obtain results on PIR shear strength, shear creep
68 behaviour and the influence of temperature in its creep response.

69

70 **3. EXPERIMENTAL PROGRAM**

71 The experimental program carried out in this study is encompassed in the research project "Easyfloor–
72 Development of composite sandwich panels for building floor rehabilitation". The sandwich panels developed in

73 the project contain several improvements over the most common solutions used nowadays [40]. In this context,
74 PIR was included in the sandwich panels as the foam core material due to its enhanced insulation properties.

75 One of the main tasks in this research project is the individual mechanical characterization of the materials
76 forming the sandwich panel. Thus, the present experimental program on the PIR foam comprised two main types
77 of tests: i) failure tests to assess the behaviour of PIR under pure quasi-static monotonic-instantaneous shear
78 loading; and, ii) creep tests, where PIR is subjected to a pure shear constant load over time. The load applied in
79 the creep tests is a percentage of the average ultimate load obtained in instantaneous failure tests. The objective of
80 the creep tests is to determine PIR creep response for different load amplitudes and service temperatures.

81 Prior to the shear tests on the PIR foam (failure and creep), which were designed to fulfil the principal
82 objectives of this study, some other tests were carried out to characterize the PIR material. All the PIR specimens
83 tested correspond to a rigid low-density closed-cell foam. The foam manufacturing was implemented through the
84 mechanical mixing (at 3000 rpm and 20 °C) of isocyanate, polyol, surfactant, catalysts, water and the blowing
85 agent (CO₂). The isocyanate index is 160. According to the technical datasheet of the product, the typical
86 properties of this PIR foam are: density of 40 - 50 kg/m³ (EN 1602); thermal conductivity <0.024 W·K-1/m
87 (EN 12667); closed-cell content >90% (ISO 4590); compressive strength >270 kPa (EN 826); dimensional
88 stability (70 °C, 90% RH / -20 °C) ≤0.5% / ≤1% (EN 1604); fire behaviour E(Euroclass, EN13501-1), B2
89 (Building Material, DIN 4102-1); water absorption ≤0.5 kg/m² (EN 1609).

90

91 **3.1 Material characterization**

92 For the material characterization of the PIR foam, compression and direct tensile tests were carried out
93 following the applicable normative rules. For the compression tests, the specifications of standard
94 ASTM C365 [41] were adopted, resulting in an average compressive strength of 0.22 MPa (CoV = 10.67 %) and
95 elastic modulus of 5.81 MPa (CoV = 11.53 %) for the 5 specimens tested (see **Figure 1(a)**). The direct tensile tests
96 followed the guidelines on the standard ASTM C297 [42], and an average direct tensile strength of 0.22 MPa (CoV
97 = 15.52 %) and elastic modulus of 4.79 MPa (CoV = 10.03 %) were obtained for the 5 specimens tested (**Figure**
98 **1(b)**). The uniaxial compression and tensile tests were performed parallel to the foam rise direction.

99 The viscoelastic thermal response of the material was measured through a classic Dynamic Mechanical
100 Analysis (DMA) test [34], in order to evaluate the effect of temperature on PIR creep. In this test, prismatic
101 specimens with 10 mm width, 4 mm thickness and 60 mm length were used. The dynamic load was applied in a

102 dual cantilever setup at a frequency of 1 Hz, and the samples were heated at a rate of 1 °C/min, under an inert
103 atmosphere (N₂), from -50 °C to 200 °C.

104 **Figure 2** shows the typical results obtained in the DMA tests performed on PIR foam specimens, in terms
105 of the evolution of the storage modulus E' , the loss modulus E'' and the loss factor $\tan(\delta)$, where $\delta =$
106 $\arctan(E''/E')$. With the temperature increasing between -50 °C and 200 °C, a couple aspects can be identified
107 that sustain the PIR foam as a polymer with an increased thermal stability, as follows:

- 108 • Firstly, a sudden drop in the storage modulus is not seen;
- 109 • Secondly, a maximum peak value is not observed for the loss modulus, which seems to decrease
110 continuously with temperature. Furthermore, that continuous decrease with temperature is more
111 pronounced for temperatures approximately between 0 °C and 40 °C, matching a wide range of in-
112 service temperature values that are expected in a sandwich panel;
- 113 • Thirdly, regarding the loss factor, it shows a slight continuous and approximately linear decrease for
114 temperatures varying between 0 °C and 40 °C, reflecting the behaviour observed for E' and E'' .

115 These statements match the observations of Javni *et al.* [36] concerning DMA tests carried out in rigid
116 foams with a high isocyanate index. A high level of isocyanate is a characteristic of the PIR foams, and it is
117 responsible for their strong molecular structure consisting on highly cross-linked isocyanurate rings. The results
118 of **Figure 2** also resemble those achieved in DMA tests on thermoset polymers with crystalline structure [43], in
119 which the molecules are arranged in an almost perfectly regular and periodic form.

120 Differences can be noticed when comparing the results of the present DMA tests (in PIR specimens) with
121 PUR results available in literature [34], for the same temperature range (-50 °C to 200 °C): i) the rigid PUR foam
122 has a typical sudden decrease in E' ; and ii) E'' continuously increases in the range of common in-service
123 temperatures until a maximum peak is reached. A significant continuous reduction trend in storage modulus from
124 the beginning to the end of the temperature range tested is observed, which means that PIR becomes more
125 deformable with increasing temperature, in terms of elastic response. However, the loss modulus shows a
126 decreasing trend with increasing temperature, indicating a reduction in viscoelasticity as temperature increases -
127 the loss factor values have varied positively for PUR (+60%) and negatively for PIR (-33%). This fact may indicate
128 an overall higher thermal stability of PIR within frequent in-service temperatures.

129

130 **3.2 Monotonic-instantaneous shear tests up to the failure**

131 The shear tests up to the failure on PIR followed the recommendations of ASTM C273 [44] and
132 ISO 1922 [45]. The test fixture developed and the test configuration are illustrated in **Figure 3(a)** and **Figure 3(b)**,
133 respectively. As can be seen, two of the PIR faces are bonded to “L” shape elements made of two UNP 120 steel
134 profiles. 3-dimensional bearing joints were used to avoid bending moments in the system. These joints ensure the
135 verticality of the load line of action and the absence of bending moment transmission at both supports. Then, with
136 the plates moving in opposite directions, away from each other, a transverse displacement (distortion) of the planes
137 parallel to the bonded faces of the PIR foam is imposed (see **Figure 3(b)**).

138 The PIR foam was bonded to the UNP120 steel profiles with “Sika 32 EF”, which is a two-component
139 polyurethane adhesive. Initially, the steel profiles were cleaned with acetone. Then, the two components of the
140 adhesive were mixed and applied to both the steel profiles and the PIR specimens faces. At first, while on steel
141 profile was resting horizontally, one face of PIR was glued to it. Soon after, the other steel profile was placed on
142 the free top face of PIR. Consequently, a slight compression was applied on the foam due to the steel profile self-
143 weight. Simultaneously, the adhesive in excess emanating from the bonding was removed. Finally, the specimens
144 were stored into a climatic chamber, with controlled temperature (20 °C) and relative humidity (55%). Although
145 only 4 hours were specified as enough curing period by the adhesive’s manufacturer technical data, the specimens
146 rested in the climatic chamber for 1 to 7 days until they were tested.

147 Prismatic specimens with 360 mm length, 120 mm width and 30 mm thickness were used. In total, 6
148 specimens (F1 to F6) were tested in quasi-static monotonic-instantaneous loading up to failure, under displacement
149 control at a rate of 0.50 mm/min, in a universal testing machine. One LVDT was used to record the shear
150 displacement δ developed between the plates, as shown in **Figure 3(b)**. The LVDT has a measuring range of
151 ± 25 mm and a linearity error of ± 0.12 mm. The applied force was measured using a load cell of 200 kN (± 0.12 kN).

152 The geometry and dimensions defined for the specimens meet the specifications of the standards
153 ASTM C273 [44] and ISO 1922 [45]. The standards prescribe that the width b should be higher than 50 mm, so
154 that a good adhesion of the PIR to the plates can take place. For the specimen length L , the condition $L \geq 12 \times t$ is
155 met, where t is the specimen thickness. This latter condition minimizes the effect of the secondary (tensile)
156 stresses at the specimen ends. Consequently, the applied transverse displacement can inflict pure shear along most
157 of the foam length and shear failure is most likely to occur, represented by a diagonal crack crossing the specimen’s

158 core. In this context, the standards state that tests which portray a cohesive failure of the adhesive, or of the PIR
159 foam in the contact area with the adhesive, should be rejected.

160 The appropriateness of the setup of **Figure 3**, along with the foam specimen dimensions specified by the
161 standards, was tested by the authors in a previous work [46], where shear failure occurred in 4 of the total 6
162 specimens assessed. In that experimental campaign, the principal strain patterns were measured through Digital
163 Image Correlation (DIC), and pure shear could be depicted at most of the specimen's length. At the end, these tests
164 allow the determination of the PIR foam mechanical properties, according to Equations (1) to (3),

$$\tau_u = \frac{F_u}{L \times b} \quad (1)$$

165

$$\gamma = \frac{\delta_u}{t} \quad (2)$$

166

$$G = \frac{\Delta\tau}{\Delta\gamma} \quad (3)$$

167 where τ_u is the ultimate shear strength, γ_u is the ultimate shear strain, G is the shear modulus, F_u is the maximum
168 force value measured and δ_u its corresponding shear displacement. According to ASTM C273 [44], the shear
169 modulus G should be calculated as the "slope of the initial portion" of the shear stress-strain curve. In the present
170 study, G is calculated in the interval between $\gamma = 0.01$ and $\gamma = 0.025$.

171

172 3.3 Shear creep tests

173 For the shear creep tests, a new test setup was specifically developed in the present study, since a constant
174 shear stress over time is to be applied on the PIR foam. **Figure 4** depicts the outlook of the test setup and its main
175 features:

- 176 • Additionally to the LVDT, shear displacement was also measured through a dial gauge attached to the
177 steel UNP120 profiles supporting the PIR foams (see **Figure 4(a)**). The LVDT is placed in one face of
178 the test fixture and the dial gauge on the opposite face. The dial gauge was added, as a redundant
179 measuring system, because the long duration of the creep tests increased the probability of a failure
180 occurring in the data acquisition system associated with the LVDTs;

- 181 • The top (superior) bearing joint of the test fixture is connected to a UNP200 steel beam in equilibrium
182 (see **Figure 4(b)**). The beam is linked (through steel chains) to the bearing joint on one end and at the
183 other end is loaded with rock blocks (sustained load). In turn, the rock blocks were piled in order to
184 reach the intended creep load on the PIR foam specimens. Therefore, weight of all the components of
185 the testing system causing load was considered in the estimation of the creep load to be applied, namely
186 the steel beam and steel plates;
- 187 • Room temperature and relative humidity RH were maintained approximately constant throughout the
188 tests, by executing the tests into a climatic chamber containing a heating/refrigeration system.

189 A total of 12 specimens were tested: i) 6 at a room temperature equal to 20 ± 1.5 °C (with RH = $55 \pm 6\%$)
190 and subjected to 3 load amplitudes (20%, 40% and 60% of the average shear strength measured in the shear tests
191 up to the failure); ii) the other 6 specimens were tested at 30 ± 1.5 °C (RH = $55 \pm 6\%$) and subjected to 3 load
192 amplitudes as well (20%, 30% and 40% of the average shear strength). Thus, 2 specimens were considered for
193 each test series, so that variability could be taken into account. Temperature ranging between 20 °C and 30 °C is
194 very common in temperature-controlled environments, like residential and office buildings. The load levels match
195 the ones considered in research works [16,29] available in literature that assessed the shear creep behaviour of
196 PUR (10% to 40% of the average shear strength). The 60% level was added in order to observe if creep failure
197 could occur.

198

199 **3.4 Post-shear creep tests**

200 Six months after the conclusion of shear creep tests at 30 °C, the 6 specimens were subjected to a monotonic-
201 instantaneous shear load up to failure. The setup and procedure implemented in these tests are like the tests
202 described in Section 3.2. This option aimed at assessing post-shear creep behaviour of these specimens and
203 compare with the ones that did not suffer creep effects.

204 Finally, it should be mentioned that all the tests, namely, monotonic-instantaneous shear tests, shear creep
205 tests shear and post-shear creep tests were performed with the foam rise direction perpendicular to the steel plates.

206

207 4. RESULTS

208 The results obtained in the monotonic-instantaneous shear tests up to failure and the shear creep tests are
209 presented and analysed. Since the displacements measured in the LVDTs and in the dial gauges were very similar,
210 only the first ones were used for the evaluation of the shear strains.

211

212 4.1 Monotonic-instantaneous shear tests up to failure

213 In the shear tests up to failure performed on the 6 PIR foam specimens, two failure modes were identified
214 (**Figure 5**):

- 215 • The first failure mode – FM1 – (**Figure 5(a)**) is characterized by a shear crack parallel to the steel
216 plates (UNP profiles – see **Figure 3**) at the interface between PIR foam and steel. In all the tests that
217 portrayed this failure mode, the crack arose in the vicinity of the contact area of the PIR foam with the
218 adhesive, while the adhesive itself remained intact. In general, at the bonded zone the adhesive filled
219 the open cells and voids of the PIR and, consequently, the specimen strength is positively affected
220 around the contact area with the adhesive. This explains the existence of a thin layer of PIR foam
221 attached to the UNP profiles. The crack always started at the extremities, since the boundary conditions
222 caused tensile stress concentrations near the steel plates, [46]. Then, the crack evolved along the
223 interface PIR/steel plates;
- 224 • The second failure mode – FM2 – (**Figure 5(b)**) is another type of cohesive shear failure that PIR has
225 experienced, characterized by the formation of a shear crack, transverse to the steel plates, at
226 approximately specimen's half-length. The crack is diagonal ($\approx 45^\circ$ to the load line of action) due to the
227 pure shear stress state generated by the test configuration, which inflict an inclined principal tensile
228 stress in the foam (see in [46]).

229 **Figure 6** shows the shear stress *versus* strain ($\tau - \gamma$) curves obtained in the 6 shear tests performed. Half of
230 the specimens experienced failure mode FM1 and at remaining ones FM2:

- 231 • In the case of FM2, the typical mechanical response [28,34] of a polymeric foam subjected to shear
232 stress state can be recognized: i) an initial linear elastic branch, ii) a non-linear phase, and iii) a
233 hardening elasto-plastic linear branch. The transverse shear failure takes place when the maximum
234 shear strain of the foam is reached. This failure mode is brittle and occurred suddenly;

235 • Some differences can be perceived in the $\tau - \gamma$ curve of the specimens that depicted FM1. In these
236 tests, the non-linear phase ii) does not stabilize into the hardening elasto-plastic phase iii), denoting
237 that the shear crack parallel to the steel plates continues to grow until the specimen collapses. Here,
238 stiffness degradation caused by cracking is governed by the shear crack parallel to the steel plates in
239 the vicinity of the contact area between the PIR foam and the adhesive.

240 **Table 1** presents the main results acquired in the tests. Considering only the specimens that displayed the
241 failure mode FM2 (transverse shear crack), the average values were: i) shear strength $\tau_u = 0.19$ MPa; ii) shear
242 modulus $G = 2.77$ MPa; and iii) ultimate shear strain $\gamma_u = 0.193$. Moreover, a small variability can be verified in
243 failure mode FM2 test results. In contrast, the tests that showed failure mode FM1 only have a small variability in
244 the G modulus, since cracking has not yet appeared (elastic phase). In those tests, the average values of τ_u and γ_u
245 were significantly lower.

246 As was explained in Section 2, no data could be found so far in literature, regarding shear strength of a PIR
247 foam, to be compared with the results of **Table 1**. Shear tests were performed on PUR foam having a higher
248 density: 87.4 kg/m^3 in tests performed by Garrido *et al.* [16] and 62 kg/m^3 in Witkiewicz and Zielinski [28] tests,
249 in which the obtained shear strength average values were 0.32 MPa and 0.35 MPa, respectively.

250

251 4.2 Shear creep tests

252 The reference shear strength τ_u value of **Table 1** used for the shear creep tests was 0.19 MPa, the average
253 value obtained in the tests which experienced cohesive transverse shear failure (FM2). Tests where a crack parallel
254 to the steel plates (FM1) was formed were not considered, since this type of failure mode is not governed by the
255 shear strength, as expected in a pure shear test.

256 The evolution of the PIR foam shear strain (γ) over time is illustrated in **Figure 7** for the different load
257 amplitudes applied. Creep compliance (J), strain per unit of stress ($J = \gamma / \tau$), evolution is depicted in **Figure 8**. For
258 a better understanding, **Table 2** (for the specimens at 20 °C) and **Table 3** (for the specimens at 30 °C) include the
259 values acquired at the loading instant γ_e , just before unloading γ_c , after unloading γ_{un} and at the end of the test γ_{end} .
260 Moreover, **Table 2** and **Table 3** also include the expected elastic shear strain, $\gamma_{e,expected}$, calculated considering the
261 average value of $G = 2.77$ MPa from **Table 1**. Specimens at 20 °C were unloaded at 2184 hours and the
262 corresponding tests were completed at 3168 hours, while the ones settled at 30 °C were unloaded at 3000 hours

263 and tests were concluded at 4080 hours. This difference between the tests at 20 °C and at 30 °C was implemented
264 so that unloading at different ages (2184 hours and 3000 hours) could be tested.

265 During some periods, technical problems occurred and for that reason no displacement values on the
266 specimens were measured. Those gaps can be seen in the graphs of **Figure 7** and **Figure 8** and correspond in the
267 20 °C tests to the intervals: 1005 – 1497 and 2682 – 2861 hours. In the 30 °C tests the gaps can be found in the
268 following intervals: 786 – 818, 1479 – 1853 and 3328 – 3840 hours.

269 All the tests revealed the first 2 stages of the typical creep material behaviour [47]:

- 270 • Until approximately $t = 200$ hours, primary creep, in which the strain rate is high due to the deformation
271 caused by the internal rearrangement of the material microstructure. This rearrangement is also the
272 reason why the strain rate decreased with time;
- 273 • Then, the strain rate diminished to a minimum and became approximately constant as the secondary
274 stage of creep began. The internal material microstructure does not change significantly at this stage.

275 In the third stage of creep, characterized by an exponential growth of deformation, the applied load inflicts
276 damage in the material and failure is expected to occur. This third stage was not observed in the tests carried out
277 and none of the specimens failed due to creep. This evidence reinforces the idea that PIR is an enhanced foam
278 material, since failure was detected in the recent work of Garrido *et al.* [16] on a rigid high-density ($\rho = 87.4 \text{ kg/m}^3$)
279 closed-cell PUR foam. The PUR specimen that failed in that study was subjected to a creep load corresponding to
280 44% of the shear strength and at 28 °C. That specimen attained creep shear strains of $\gamma = 0.0543$ (between
281 measurements taken after 402 and 477 hours of creep), which exceeded the average failure strain reported for that
282 foam in the static failure tests ($\gamma = 0.043$). Those load *versus* temperature conditions are not as severe as the ones
283 implemented in the present experimental campaign, where a maximum creep shear strain of $\gamma = 0.134$ was attained
284 at 2184 hours, less than the ultimate shear strain measured in the static tests ($\gamma = 0.193$).

285 The shear strain values and the strain rate always increased with the load amplitude applied. Furthermore,
286 the results obtained were very similar for tests with the same load amplitude and room temperature, except for the
287 tests on specimens subjected to 60% of the shear strength. After unloading, the influence of load amplitude was
288 also relevant, as the absolute value of the strain rate was higher on the most loaded specimens, when the
289 comparison is established between tests performed at the same room temperature. The specimens at 30 °C had a
290 higher strain rate absolute value after unloading mainly because the load was removed later (3000 hours) than the
291 specimens at 20 °C (2184 hours). In this matter, creep behaviour after unloading followed an inverse path compared

292 to loading, similar to a primary creep stage: the strain rate was high and decreased with time but had negative
293 values. Then, the tests were ended (stopped) when the beginning of the secondary stage took place.

294 Some conclusions about the viscoelastic linearity of the PIR foam can be derived from **Figure 8**, where the
295 creep compliance values calculated for the tests are displayed. As can be seen, creep compliance values follow the
296 same path of the shear strain values (**Figure 7**), with the exception of one of the specimens subjected to 60% of
297 the shear strength (at 20 °C). Specimen C-L60-T20-2 showed creep compliance values close to the specimens
298 subjected to 40% of the shear strength, a fact that is most likely attributed to the high variability occurring when
299 load amplitudes are high, such is the case.

300 **Figure 8** also reveals that creep compliance values increased with the shear stress applied, indicating
301 nonlinear creep. In this context, a load amplitude corresponding to 30% of PIR shear strength was enough to induce
302 a nonlinear creep response, a lower threshold value compared to the values obtained by Huang and Gibson [29]
303 and Garrido *et al.* [16] in a high-density PUR foam. Huang and Gibson [29] concluded that the PUR foam tested
304 had a linear viscoelastic behaviour below half the shear strength, while Garrido *et al.* [16] stated that a shear stress
305 of 44% of the shear strength was high enough to cause nonlinear creep.

306 Finally, analysing the influence of temperature in the results, the values of the elastic shear strain γ_e were
307 not significantly different between the tests at 20 °C and at 30 °C. Then, after loading, it can be noticed that the
308 specimens tested at 20 °C had a lower strain rate. However, that primary creep stage lasted longer, and with time
309 the shear strain became slightly higher than the shear strain measured in specimens tested at 30 °C. In the tests of
310 Garrido *et al.* [16] on a PUR foam, the observed behaviour was contrary, with higher creep deformations achieved
311 by the specimens when the temperature increased. However, as was mentioned before, PIR is an enhanced material
312 in terms of thermal stability. The DMA test performed in the present work showed (see **Figure 2**) that the loss
313 factor slightly decreases when the temperature increases, in the range of the common foam service temperatures.
314 This fact is apparently the most plausible explanation for the better creep response detected in the specimens tested
315 at 30 °C.

316

317 **4.3 Post-shear creep tests**

318 **Table 4** presents the results obtained in the 6 post-shear creep tests. The average values of G , τ_u and γ_u
319 obtained in the monotonic-instantaneous shear tests (without creep effects) are taken as reference values, and
320 therefore (see **Table 1**): $G_{ref} = 2.77$ MPa; $\tau_{u,ref} = 0.19$ MPa; and $\gamma_{u,ref} = 0.193$. Then, comparing the post-creep results

321 with the reference values, the applied creep load did not cause damage on the shear strength and stiffness of the
322 PIR specimens. Material hardening can be detected in the post-creep mechanical properties, with higher values
323 achieved for the shear modulus and ultimate shear strength. Material hardening also manifests itself in the less
324 ductile and more brittle post-creep failure of the specimens, with lower values achieved for the ultimate shear
325 strain.

326

327 5. ANALYTICAL MODELLING OF CREEP BEHAVIOUR

328 Accurate simulations of PUR subjected to shear creep loading by using the Findley's power law [30,31]
329 was already demonstrated in literature [16,29], and therefore it is also considered in the present work for the
330 analytical modelling of the PIR foam shear creep behaviour. In this section, the Findley's power law formulation
331 is presented. Then, the parameters of the power law are determined based on the results obtained in the
332 experimental campaign. The calibration of the parameters follows two approaches: i) shear strain of the PIR foam
333 due to the creep effect is assumed to be independent of temperature; and ii) temperature influence is considered
334 on the creep response.

335

336 5.1 Findley's power law

337 The Findley's power law is an empirical equation in which the shear strain due to the creep effect, γ_{cr} , varies
338 as a power of the time (in hours) after loading is applied, t :

$$\gamma_{cr} = m \times \left(\frac{t}{t_0} \right)^n \quad (4)$$

339 In this formulation, the total shear strain is calculated from the following equation:

$$\gamma = \gamma_e + m \times \left(\frac{t}{t_0} \right)^n \quad (5)$$

340 where m is a creep amplitude coefficient depending on the applied stress and n is a time exponent coefficient,
341 independent of stress and foam density. The coefficient n is assumed to be a material dependent for a given
342 hygrothermal condition [29,48]. The values of both coefficients are calibrated from the experimental results
343 obtained. t_0 is the time unit considered, so the time parameter can be normalized and the dimension consistency of
344 Equation (5) assured. $t_0 = 24$ hours (1 day) is considered in the present work.

345 Calibration of the Findley's power law parameters from the experimental results was performed using the
 346 Nonlinear Generalized Reduced Gradient optimization algorithm of Lasdon *et al.* [49]. The automatic
 347 implementation of this algorithm is included in the Microsoft Excel Solver tool and its background theory can be
 348 consulted in detail in Reference [50]. The coefficients m and n are calculated aiming to minimize the sum s :

$$s = \sum_{i=1}^t |\gamma_{i,\text{exp}} - \gamma_{i,\text{num}}| \quad (6)$$

349 where γ_{exp} is the shear strain measured in the experimental tests, and γ_{num} is the shear strain given by the Findley's
 350 power law. The sum has the time step of 1 day.

351 For the unloading phase, the adopted analytical modelling of the foam relaxation can be useful for
 352 predicting, at a certain time, the residual deformation after the application of a creep load. To achieve that purpose,
 353 a primary analysis revealed that a satisfactory approximation between experimental and modelling values could
 354 be obtained considering the Findley's power law as well. The implemented analysis demonstrated that:

- 355 • After the unloading instant, the calculation of the creep deformation recovery from the Findley's power
 356 law should contemplate time after unloading, i.e. t is replaced by $t - t_{\text{un}}$, where t_{un} (in days) is the age
 357 at which unloading is done;
- 358 • The creep amplitude coefficient at the unloading phase, hereinafter designated as m_{un} , must assume a
 359 negative value, and a smaller absolute value compared to the loading phase;
- 360 • A modification in the time exponent coefficient n for the unloading phase is not necessary, and the
 361 value considered in the loading phase can be maintained. This fact sustains the notion, already
 362 mentioned, that n is a material constant for a given hygrothermal condition.

363 Thus, at the unloading stage, the calculation of the creep deformation recovery $\gamma_{\text{cr,un}}$ from the Findley's
 364 power law can be given by the following equation:

$$\gamma_{\text{cr,un}} = m_{\text{un}} \times \left(\frac{t - t_{\text{un}}}{t_0} \right)^n \quad (7)$$

365 Since the elastic deformation γ_e is completely recovered after unloading, Equation (5) is reduced to:

$$\gamma = \gamma_e + m \times \left(\frac{t_{\text{un}}}{t_0} \right)^n - \gamma_e + m_{\text{un}} \times \left(\frac{t - t_{\text{un}}}{t_0} \right)^n \quad (8)$$

366 or

$$\gamma = m \times \left(\frac{t_{un}}{t_0} \right)^n + m_{un} \times \left(\frac{t - t_{un}}{t_0} \right)^n \quad (9)$$

367

368 5.2 Calibration assuming creep behaviour independent of temperature

369 As could be observed in the shear strain results of **Figure 7** and in the DMA test of **Figure 2**, creep
370 deformations in PIR foam are slightly higher at 20 °C compared to 30 °C. The difference is not very significant,
371 and seems plausible to assume in modelling, at first hand, a simpler approach in which temperature does not
372 influence creep behaviour in the range of 20 °C to 30 °C.

373 **Figure 9** reveals the results achieved with this procedure, and a very satisfactory fitting can be identified
374 between experimental and modelling values. Especially for the tests at 30 °C, which present less variability
375 compared to the ones at 20 °C. **Table 5** shows the values calibrated for the coefficients m and n . The calibration
376 error is calculated through the following expression:

$$\text{error} = \frac{\sum_{i=1}^t |\gamma_{i,\text{exp}} - \gamma_{i,\text{num}}|}{\sum_{i=1}^t \gamma_{i,\text{exp}}} \quad (10)$$

377 **Figure 10** depicts the values calibrated for the coefficient m in function of the applied shear stress (τ / τ_{max}).
378 The trend curve that provided the best fit, among others that were tested, was the following empirical power law
379 with a regression factor $R^2 = 0.998$:

$$m = a \times \left(\frac{\tau}{\tau_{\text{max}}} \right)^b \quad (11)$$

380 with $a = 0.0452$ and $b = 1.684$.

381 As was proved in the tests of Huang and Gibson [29], the coefficient m is directly proportional to the applied
382 stress when creep is linear. However, linear creep could not be detected in the experimental campaign carried out
383 in the present work, and this fact seems to explain the power law variation of m in **Figure 10** with the applied
384 stress.

385 Then, it was later concluded in the calibration procedure of m_{un} that: i) m_{un} is directly proportional to m ;
386 and ii) determination of m_{un} must include the age t_{un} (in days) at which unloading is done. At the end, the following
387 empirical expression was derived for m_{un} :

$$m_{un} = -0.004 \times m \times \left(\frac{t_{un}}{t_0} \right) \quad (12)$$

388 or

$$m_{un} = -1.808 \times 10^{-4} \times \left(\frac{\tau}{\tau_{max}} \right)^{1.684} \times \left(\frac{t_{un}}{t_0} \right) \quad (13)$$

389 when Equation (11) is incorporated.

390 The results of this modelling approach for the unloading stage are depicted on **Figure 9**. As can be seen,
391 the power law curves reflect the strain rate tendency for all the stress levels and for both tests at 20 °C and 30 °C.
392 For the elastic strain γ_e , it was considered $\gamma_e = \tau / G$, with $G = 2.77$ MPa. The value adopted for the shear modulus
393 G is the average value obtained in the shear failure experimental tests which depicted a transverse shear failure
394 mode (see **Table 1**).

395 **5.3 Calibration assuming creep behaviour dependent on temperature**

396 An analytical modelling approach is presented in this section in which the calculation of creep shear strain
397 from the Findley's power law depends on temperature. For that purpose, and since the tests were developed at two
398 temperatures (20 °C and 30 °C), some parameters of the Findley's power law in this approach become now linearly
399 dependent on temperature. A linear dependency between temperature and the viscoelastic behaviour of the PIR
400 foam was depicted in the DMA test of **Figure 2**, for a temperature range between 0 °C and 40 °C.

401 In this analysis, it was assumed that creep behaviour is dependent on temperature, and therefore a
402 calibration procedure was implemented, as in Section 5.2, to obtain values for the coefficients m and n for each
403 test series (see **Table 6**). In the following, Equation (14) could be derived for coefficient n , depending on
404 temperature T (in Kelvin):

$$n = -0.001248 \times T + 0.6855 \quad (14)$$

405 The best fit for the coefficient m variation with the stress level is a power law similar to the one presented
406 in Equation (11), whose representation for the tests at 20 °C and at 30 °C are depicted in **Figure 11**. In that
407 procedure, it was noticed that coefficient b of the power law (see Equation (11)) did not vary significantly with
408 temperature, and good results could be achieved through the consideration of a constant value ($b = 1.6245$).

409 For coefficient a , a linear expression was adopted in function of temperature T (in Kelvin), according to
410 Equation (15):

$$a = -0.00025 \times T + 0.117 \quad (15)$$

411 Finally, **Figure 9** illustrates the new Findley's power law curves corresponding to this approach, where the
412 influence of temperature is considered in the creep shear strain analytical calculation. A slight improvement occurs,
413 with the model curves closer to the experimental values. This enhancement is more perceptible in the tests at 20
414 °C, particularly the ones carried out with $\tau / \tau_{\max} = 0.4$. For other temperature ranges, if they are within 0 °C to 40
415 °C, the advantages regarding the use of this temperature dependant approach are expected to be more evident.

416

417 **6. CONCLUSIONS**

418 An experimental campaign was developed to assess shear strength and the shear creep behaviour of a rigid
419 low-density closed-cell PIR foam, commonly used in the insulation of structural sandwich panels. Temperature
420 influence on the creep deformations was also evaluated through standardized pure shear tests performed at 20 °C
421 and at 30 °C, for different stress levels (20% to 60% of the average shear strength). Then, an analytical model
422 based on the Findley's power law was calibrated to the experimental results, for both loading and unloading stages.
423 In this context, two modelling approaches were considered: i) a temperature independent approach, suitable to use
424 in the 20 °C to 30 °C service temperature range; and ii) a temperature dependent approach, which is expected to
425 give more accurate results for wider service temperature ranges (within 0° C to 40 °C). The main conclusions that
426 can be derived from the work in this paper are the following:

- 427 • PIR is a thermosetting polymer with a very high thermal stability, particularly in the common service
428 temperature ranges. The DMA test carried out in the present study showed results similar to those
429 typically seen in thermosetting polymers with crystalline structure, known for their almost perfectly
430 regular and periodic molecular arrangement. The DMA revealed that from 0 °C to 40 °C the loss factor
431 of PIR decreases slightly with the temperature increase, indicating an improvement in the viscoelastic
432 behaviour of PIR in that temperature range;
- 433 • Even for creep loading corresponding to 60% of the average shear strength, no PIR foam specimen
434 failed during the creep tests, which were maintained until at least 90 days after loading was applied;
- 435 • A stress level corresponding to 30% of the PIR average shear strength was enough to induce a nonlinear
436 creep response;
- 437 • Creep deformations were slightly higher in the tests performed at 20 °C compared to the ones
438 performed at 30 °C, matching the viscoelastic observed in the DMA test of the PIR foam;

- 439
- Material hardening can be detected in the post-creep mechanical properties, with higher values
- 440 achieved for the shear modulus and ultimate shear strength, and lower values achieved for the ultimate
- 441 shear strain;
- Analytical modelling of the PIR foam creep behaviour through the Findley's power law provided good
- 442 results and a very satisfactory fitting to the experimental values was noticed;
- In the temperature range between 20 °C and 30 °C, Findley's power law parameter calculation could
- 443
- 444 be extended in order to account temperature's influence in the PIR foam creep deformations.
- 445
- 446

447 **ACKNOWLEDGEMENTS**

448 This work is part of the research project "EasyFloor – Development of composite sandwich panels for

449 rehabilitation of floor buildings", involving the company ALTO – Perfis Pultrudidos, Lda., CERis/Instituto

450 Superior Técnico and ISISE/University of Minho, supported by FEDER funds through the Operational Program

451 for Operational Program for Competitiveness and Internationalization (POCI) and the Portuguese National

452 Innovation Agency (ANI) - project no. 3480 (POCI-01-0247-FEDER-003480). The second author acknowledge

453 the grant SFRH/BSAB/150266/2019 provided by FCT, financed by European Social Fund and by national funds

454 through the FCT/MCTES. Acknowledgments are extended to LEST – Laboratório de Estruturas for their material

455 support. The contribution of the Department of Mechanical Engineering of the University of Minho is also

456 gratefully acknowledged in the person of Engineer Pedro Ribeiro. Lastly, the authors would like to thank SIKA

457 Company for supplying the adhesive.

458

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DOI: 10.1016/j.conbuildmat.2019.07.337

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581 temperature.

582

583

Table 1 - Results of the shear failure tests on PIR foam specimens.

Specimen	ρ [kg/m ³]	G [MPa]	τ_u [MPa]	γ_u [-]	Failure mode ⁽¹⁾
F1	38.81	2.68	0.13	0.075	FM1
F2	37.67	2.75	0.16	0.139	FM1
F3	38.43	2.77	0.19	0.190	FM2
F4	35.58	2.81	0.18	0.178	FM2
F5	38.19	2.64	0.19	0.173	FM1
F6	36.62	2.74	0.19	0.211	FM2
Average	37.55	2.73	0.17	0.161	All
CoV (%)	2.98	2.06	13.6	27.4	
Average	36.88	2.77	0.19	0.193	FM2
CoV (%)	3.19	1.03	3.1	7.1	
Average	38.22	2.69	0.16	0.129	FM1
CoV (%)	1.22	1.69	16.3	31.5	

⁽¹⁾ FM1 – Failure mode governed by a shear crack parallel to the steel plates; FM2 – Failure mode governed by a shear crack transverse to the steel plates.

584

585

586 **Table 2** - Values of shear strain γ over time t (in hours) in the shear creep tests on the PIR foam at 20 °C.

Specimen	Creep load (% of shear strength)	γ_e	$\gamma_{e,expected}$	γ_c	γ_{un}	γ_{end}
		($t = 0$)	($t = 0$)	($t = 2184$)	($t = 2184$)	($t = 3168$)
C-L20-T20-1	20	0.012	0.013	0.027	0.015	0.012
C-L20-T20-2		0.013		0.026	0.015	0.011
C-L40-T20-1	40	0.028	0.027	0.065	0.042	0.033
C-L40-T20-2		0.028		0.070	0.044	0.035
C-L60-T20-1	60	0.042	0.040	0.134	0.093	0.077
C-L60-T20-2		0.036		0.099	0.068	0.055

587

588

589 **Table 3** - Values of shear strain γ over time t (in hours) in the shear creep tests on the PIR foam at 30 °C.

Specimen	Creep load (% of shear strength)	γ_e ($t = 0$)	$\gamma_{e,expected}$ ($t = 0$)	γ ($t = 2184$)	γ_c ($t = 3000$)	γ_{un} ($t = 3000$)	γ_{end} ($t = 4080$)
C-L20-T30-1	20	0.012	0.013	0.025	0.026	0.013	0.008
C-L20-T30-2		0.012		0.026	0.028	0.015	0.011
C-L30-T30-1	30	0.021	0.020	0.042	0.044	0.025	0.015
C-L30-T30-2		0.021		0.044	0.047	0.026	0.016
C-L40-T30-1	40	0.029	0.027	0.061	0.065	0.034	0.020
C-L40-T30-2		0.026		0.065	0.070	0.041	0.029

590

591

592 **Table 4** - Comparison of results obtained in the post-shear creep tests with the (before creep) shear tests, up to
593 failure (reference values).

Specimen	G [MPa]	τ_u [MPa]	γ_u [-]	Failure mode ⁽¹⁾	G / G_{ref}	$\tau_u / \tau_{u,ref}$	$\gamma_u / \gamma_{u,ref}$
C-L20-T30-1	2.89	0.20	0.169	FM2	1.04	1.05	0.88
C-L20-T30-2	3.24	0.22	0.190	FM2	1.17	1.16	0.98
C-L30-T30-1	3.05	0.20	0.175	FM2	1.10	1.05	0.91
C-L30-T30-2	3.08	0.20	0.174	FM2	1.11	1.05	0.90
C-L40-T30-1	2.89	0.19	0.184	FM1	1.04	1.00	0.95
C-L40-T30-2	3.33	0.21	0.140	FM2	1.20	1.11	0.73
Average	3.08	0.20	0.172	all	1.11	1.07	0.89
CoV (%)	5.32	4.64	9.23		5.41	4.80	8.91
Average	3.12	0.21	0.170	FM2	1.12	1.08	0.88
CoV (%)	4.92	3.88	9.66		4.99	4.11	9.34

⁽¹⁾ FM1 – Failure mode governed by a shear crack parallel to the steel plates; FM2 – Failure mode governed by a shear crack transverse to the steel plates.

594

595

596 **Table 5** - Coefficients m and n calibrated from the experimental tests, assuming creep behaviour independent of
597 temperature.

m	n	Creep load (% of shear strength)	Temperature [°C]	Error [%]
0.0031		20		5.50
0.0094	0.3076	40	20	5.66
0.0199		60		4.21
0.0031		20		1.95
0.0058	0.3076	30	30	1.83
0.0094		40		2.26

598

599

600 **Table 6** - Coefficients m and n calibrated from the experimental tests, assuming creep behaviour dependent on
601 temperature.

m	n	Creep load (% of shear strength)	Temperature [°C]	Error [%]
0.0032		20		2.75
0.0099	0.3197	40	20	2.85
0.0191		60		3.37
0.0030		20		1.96
0.0058	0.3072	30	30	0.86
0.0093		40		0.88

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606 **Figure 2** - Typical results of the DMA test carried out in the PIR foam (storage modulus, loss modulus and loss
607 factor from -50 °C to 200 °C).

608 **Figure 3** - Test setup for the assessment of the PIR foam shear strength: (a) test fixture (dimensions in [mm]);
609 (b) test configuration with the LVDT.

610 **Figure 4** - Setup of the shear creep tests on the PIR foam: (a) front view; (b) schematic representation (dimensions
611 in [mm]).

612 **Figure 5** - Failure modes identified in the tests performed to assess shear strength of the PIR foam: (a) FM1 - shear
613 crack parallel to the steel plates; (b) FM2 - transverse shear crack.

614 **Figure 6** - Shear stress – strain curves acquired in the PIR foam failure tests.

615 **Figure 7** - Results of shear strain over time obtained in the shear creep tests on the PIR foam for different load
616 amplitudes: (a) at 20 °C; (b) at 30 °C.

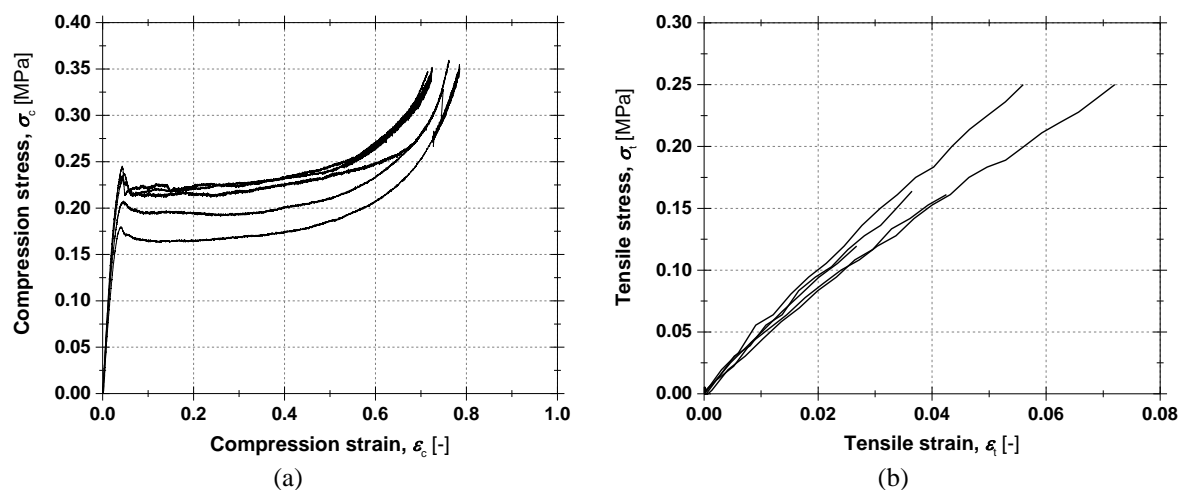
617 **Figure 8** - Results of creep compliance over time obtained in the shear creep tests on the PIR foam for different
618 load amplitudes: (a) at 20 °C; (b) at 30 °C.

619 **Figure 9** - Findley's power law numerical values adjusted to the experimental creep shear strain values: (a) at
620 20 °C; (b) at 30 °C.

621 **Figure 10** - Variation of the Findley's power law coefficient m as a function of the applied creep load (τ / τ_{max}),
622 assuming temperature does not affect creep behaviour.

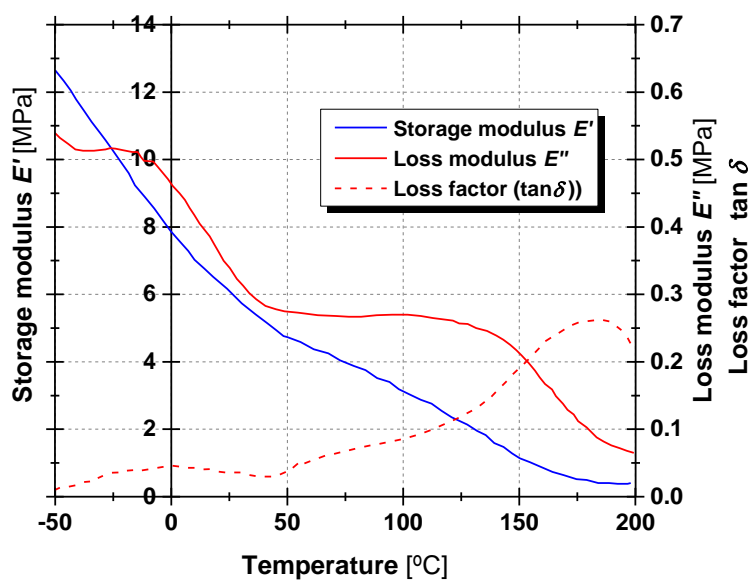
623 **Figure 11** - Variation of the Findley's power law coefficient m as a function of the applied creep load (τ / τ_{max}),
624 assuming temperature affects creep behaviour.

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626 **Figure 1** - Stress – strain curves acquired in the PIR foam failure tests: (a) compression; (b) tensile.

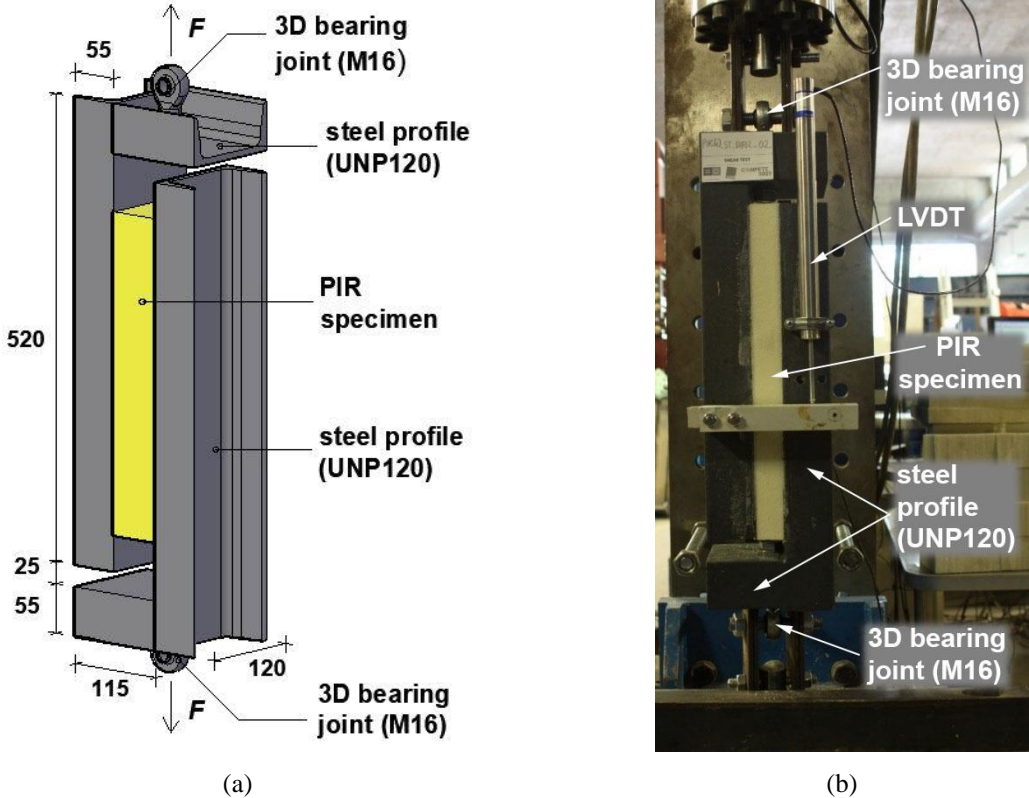
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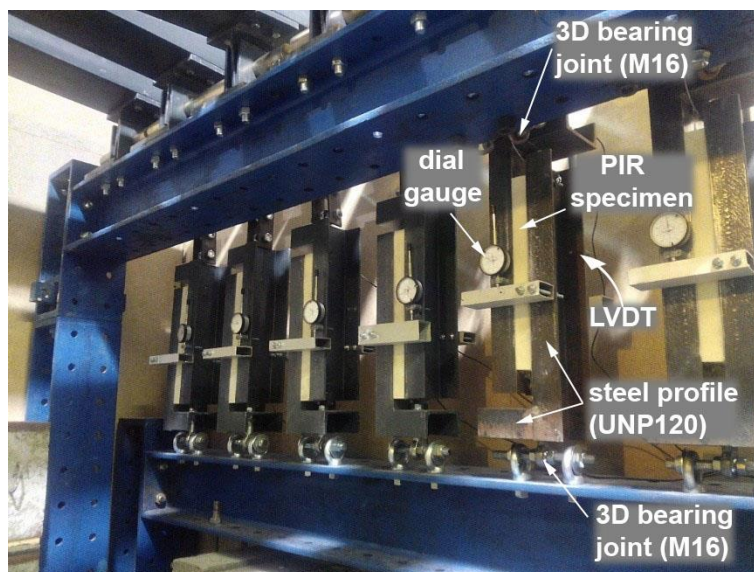
629 **Figure 2** - Typical results of the DMA test carried out in the PIR foam (storage modulus, loss modulus and loss
630 factor from -50 °C to 200 °C).

631



632 **Figure 3** - Test setup for the assessment of the PIR foam shear strength: (a) test fixture (dimensions in [mm]);
633 (b) test configuration with the LVDT.

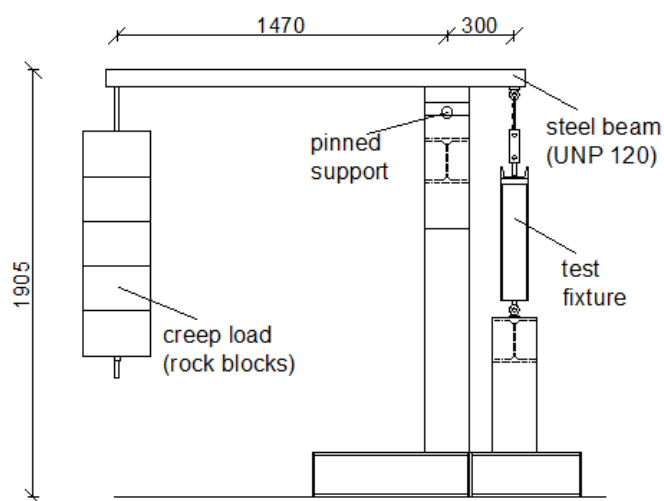
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(a)



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(b)

Figure 4 - Setup of the shear creep tests on the PIR foam: (a) front view; (b) schematic representation

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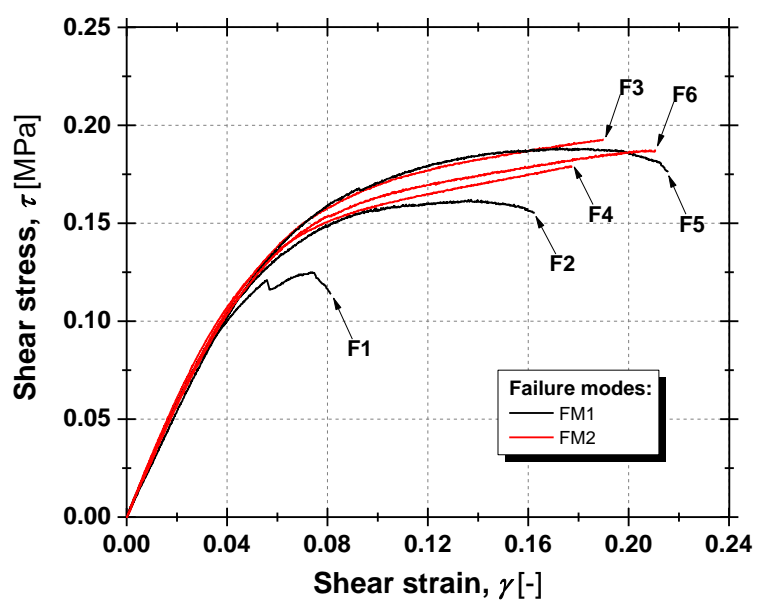
(dimensions in [mm]).

641



642 **Figure 5** - Failure modes identified in the tests performed to assess shear strength of the PIR foam: (a) FM1 -
643 shear crack parallel to the steel plates; (b) FM2 - transverse shear crack.

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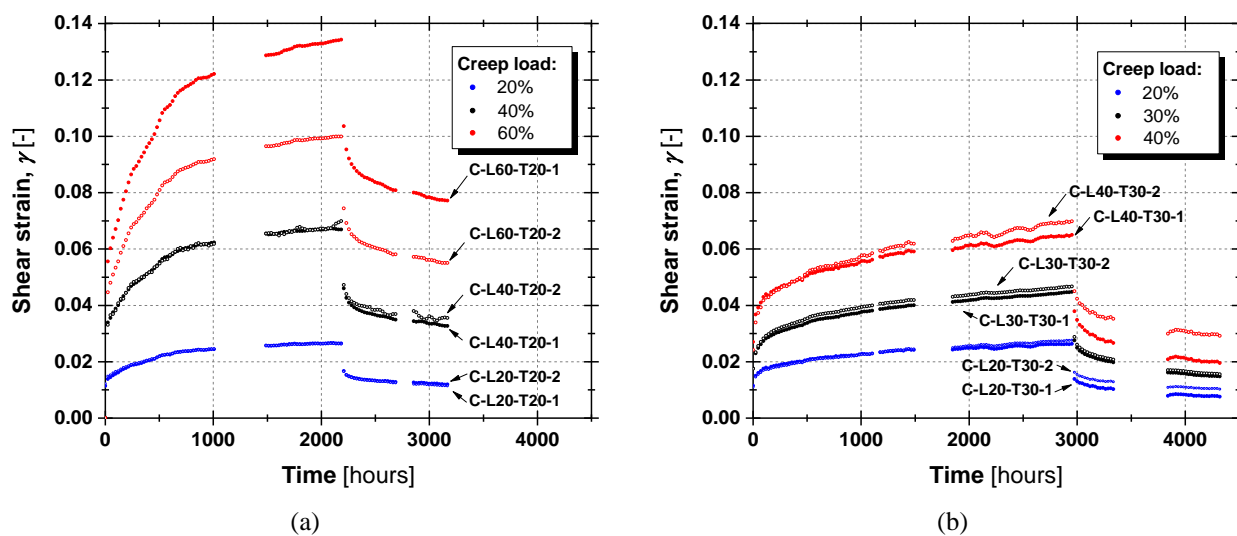


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Figure 6 - Shear stress – strain curves acquired in the PIR foam failure tests.

647



648 **Figure 7** - Results of shear strain over time obtained in the shear creep tests on the PIR foam for different load
649 amplitudes: (a) at 20 °C; (b) at 30 °C.

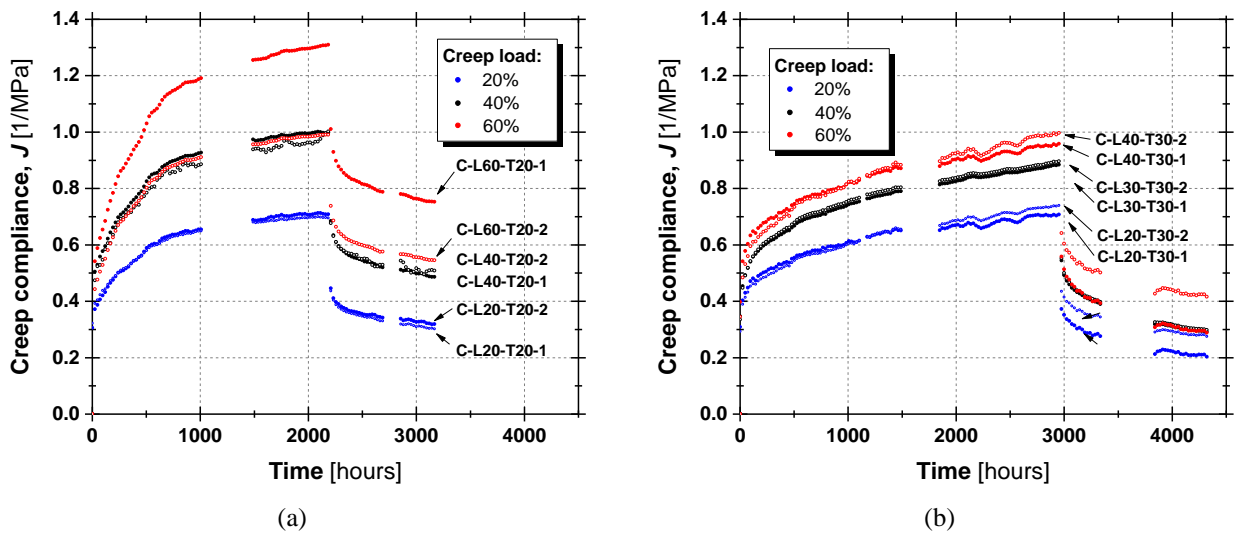
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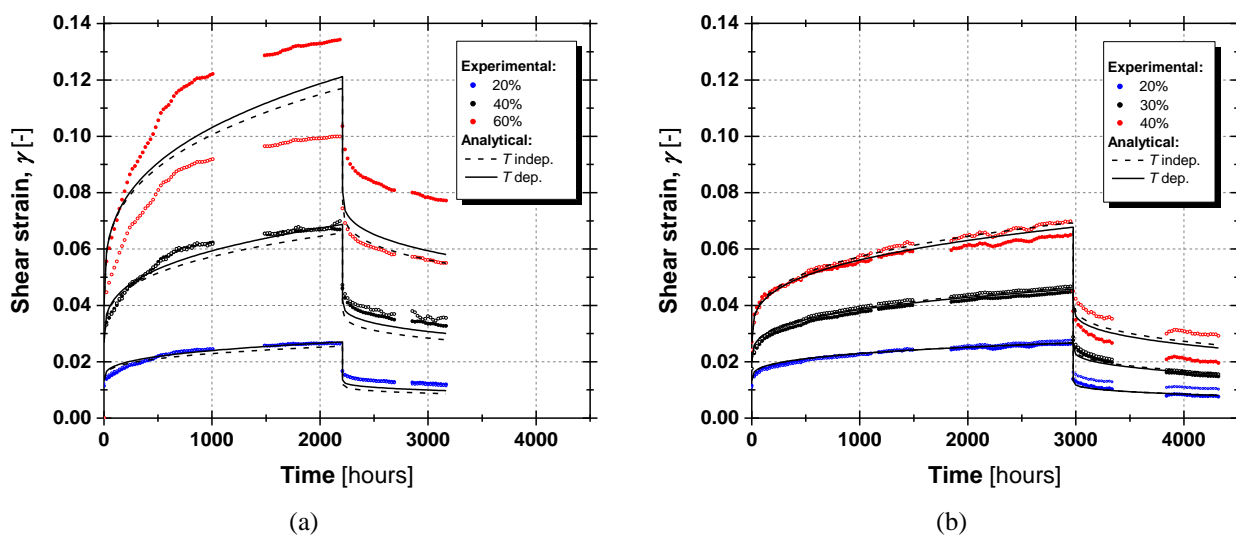
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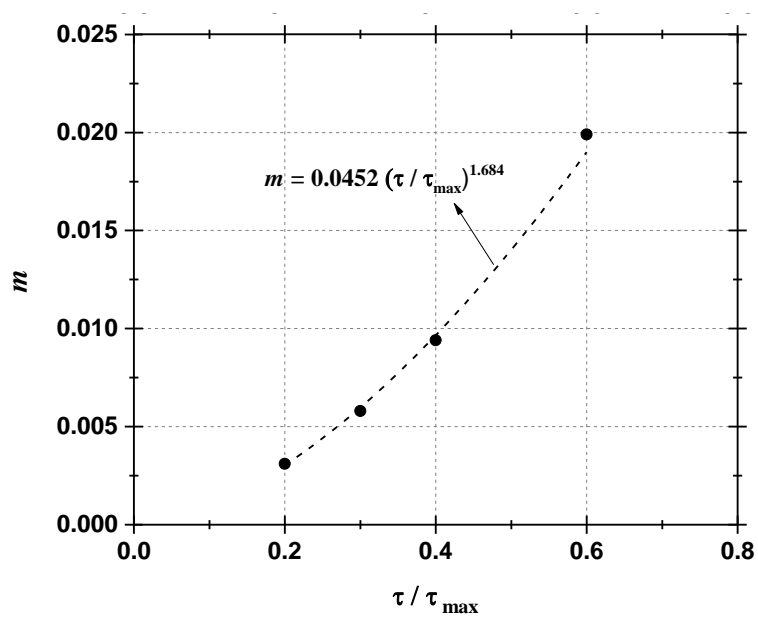
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657



658 **Figure 9** - Findley's power law numerical values adjusted to the experimental creep shear strain values: (a) at
659 20 °C; (b) at 30 °C.

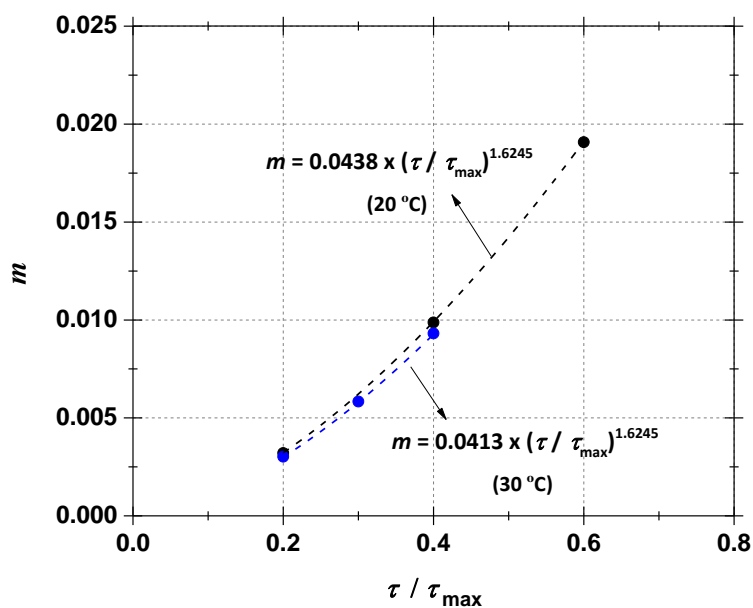
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661

662 **Figure 10** - Variation of the Findley's power law coefficient m as a function of the applied creep load (τ / τ_{\max}),
663 assuming temperature does not affect creep behaviour.

664



665

666 **Figure 11** - Variation of the Findley's power law coefficient m as a function of the applied creep load (τ / τ_{\max}),
667 assuming temperature affects creep behaviour.

668