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Influence of the engagement ratio on the shear strength of an epoxy adhesive by push-out tests on pin-and-collar joints: Part II: Campaign at different temperature levels

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ABSTRACT

Previous research led to the conclusion that the Engagement Ratio (i.e. the coupling length over the coupling diameter, *ER*) does not significantly affect the shear strength of an anaerobic adhesive (LOCTITE 648). Conversely, *ER* is effective on the response of an epoxy adhesive (LOCTITE 9466), with a beneficial effect for $ER > 1$. The aforementioned campaigns have been performed at room temperature, whereas, the effect of *ER* combined to that of temperature is still unexplored. The subject of this paper consists in the experimental investigation of the impact of *ER* on the strength of LOCTITE 9466 at higher temperatures. Decoupling tests have been performed, considering three levels of temperature (40 °C, 60 °C and 80 °C). Pin-and-Collar samples have been prepared, considering four levels of *ER*. A fixture device has been designed, to prevent misalignments and to reduce heat dissipation during the pushing-out phase.

The statistical processing of the data led to the conclusion that *ER* retains its effectiveness up to the temperature of 40 °C with strength enhancement for *ER* beyond 1. Conversely, at the highest levels of temperature, a strength drop to approximately 44% occurs, and the effect of *ER* is no longer significant to compensate this decrease. Moreover, a highly significant negative interaction was detected between *ER* and temperature.

1. Introduction

Recent technological achievements, easier manufacturing and processing, lightweight constructions, are mainly due to the development of adhesives [1]. Many applications are available in petroleum, aviation and aerospace industries [2–4]. Adhesives proved to be a valid alternative to conventional frictional joints, where the required interfacing pressure and friction are provided by suitable coupling tolerances. However, drawbacks often arise from the higher manufacturing and assembly costs and from the generation of a significant stress field, affecting the hub. It has been shown that anaerobic adhesives make it possible to significantly increase the active surface in a friction coupling (from approximately 20–30% to almost 100%), and therefore its overall resistance [5–8]. Previous studies [9] have been focused on the possible influence of the Engagement Ratio (*ER*), i.e, the ratio between the coupling length and the coupling diameter (L_c/D_c) on the joint strength. For this purpose, experimental campaigns have been conducted on press fitted and adhesively bonded (hybrid) joints with anaerobic adhesive and the tools of Design of Experiment (*DOE*) have been applied to tackle the problem. The result was that *ER* does not significantly affect strength at the 5% significance level. Epoxy adhesives have a wide application in the automotive industry, as a higher versatility can be granted in car design and manufacturing [10]. Regarding the effect of joint length or proportioning on the adhesive response, the effect of the joint length on the singular stress field near the interface end was studied in [11] with reference to lap joints. The impact of *ER* on the shear strength of an epoxy adhesive was experimentally studied in [12] with reference to differently proportioned pin-and-collar samples. The processing of the results, proved that *ER* significantly affects the strength of a LOCTITE 9466 adhesive at room temperature and that, unlike for an anaerobic bonding [9], the strength of an epoxy adhesive can be enhanced, when *ER* is increased to values higher than one.

One of the issues in the application of epoxy adhesives in the automotive industry and in many other fields stands in the operation temperature, being generally higher than the room temperature. Epoxies are often brittle and temperature sensitive, since the adhesive gets softened at incremented values of temperature. This question was tackled in some papers that highlighted a strength decrease, following a higher temperature with

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List of symbols

A	Coupling surface [mm^2]
C	Number of columns (levels) in the *ANOVA* [-]
D_C	Coupling diameter [mm]
$F_{Ad.}$	Decoupling force [N]
$F_{calc.}$	Fisher's ratio [-]
L_C	Coupling length [mm]
$\tau_{Ad.}$	Adhesive static shear strength [MPa]
R	Number of rows (replications) in the *ANOVA* [-]

List of acronyms

ANOVA	Analysis of Variance
ER	Engagement Ratio
LSD	Fisher's Least Significant Difference
MSQ	Mean Squares (general term)
MSBC	Mean Square Between Columns
MSW	Mean Square Within Columns
SSBC	Sum of Squares Between Columns
SSQ	Sum of Squares (general term)
SSW	Sum of Squares Within Columns
TSS	Total Sum of Squares
p-v.	p-value

respect to room conditions [1,10,13–17]. In Ref. [1] tensile tests were performed on standard specimens made of an epoxy adhesive. Tests were repeated at different temperature levels with five replications: one-factor *ANOVA*, were used to point out a significant impact on the strength and on the stiffness responses of the studied adhesive. This outcome is confirmed also in [10], while the great challenge in the development of adhesives that retain a good mechanical performance at high temperature from several points of view, including strength and thermal expansion, is well highlighted in [17].

Specifically regarding LOCTITE 9466, despite studies on the effects of surface treatments or on the dynamic response [18–21], specific campaigns on temperature effect are missing. Moreover, studies dealing with the combined effect of *ER* and temperature on epoxy adhesives, are not present in literature. Considering these two factors together is very important to investigate the possible interaction between them, and to derive useful suggestions for the designer. This is confirmed by several references [22–23] dealing with *DOE*, and has an important outcome in this specific case, arising from the need of determining if the effect of *ER* highlighted at room temperature is maintained in the whole temperature range.

Therefore, the subject of this paper is to determine the effect of *ER* in combination with that of temperature on the LOCTITE 9466 bi-component epoxy adhesive. This goal has been tackled experimentally, running campaigns at different temperature levels, with subsequent processing of the data by *DOE* techniques. The analysis has been refined by the application of multiple comparison tests and of orthogonality, following the conventional *ANOVA*. A two-way *ANOVA* has finally been applied to evaluate the importance and the significance of the interaction.

2. Materials and methods

According to [9,24], the main International Standards, dealing with pin-and-collar characterization, for anaerobic and epoxy adhesives are ISO 10123 and ASTM D4562–01 [25–26]. The suggested proportioning for the pin and the collar parts is substantially the same with a resulting *ER* very close to 0.9. With the aim of exploring a sufficiently large range for *ER*, it seemed to be reasonable to consider an interval from one half to the double of the reference value. This approach is also in agreement with that followed in [9] for anaerobic adhesives and in [12] for the same adhesive at room temperature, which makes it possible to compare the results. The drawings of the specimens, accounting for four levels of *ER* (0.4; 0.9; 1.3; 1.7) made of C40 UNI EN 10083-2 steel, are shown in [12]. For statistical evidence reasons, 10 Pin-And-Collar specimens of a total population of 40, were tested for each *ER* level.

The bi-component high strength epoxy adhesive LOCTITE 9466 (physical and mechanical proprieties in [27]) has been used to join the parts. The temperature application range, according to the manufacturer, may vary from 20 °C to 120 °C, even if the expected strength severely drops beyond 80 °C. This response of the adhesive is also related to its glass transition temperature, namely the temperature that separates a low-strain from a rubber-like behavior, which is in the order of 62 °C according to [27].

The tests have performed using the same self-aligning testing rig described in [12], designed to be efficient at tackling two issues: avoiding misalignment and reducing heat dissipation during decoupling. In particular, it was important to reduce the temperature drop due to conduction and irradiation, whose occurrence was expected during the pushing-out test. Its expected duration was between 40 and 70 s, depending on the collar height, and considering the specimen mounting procedure. According to several Refs., such as [28–32] PVC has a very low thermal conductivity coefficient, around 0.19 W/(m K), much lower than that of steel (C40), in the order of 50 W/(m K). For this reason, the fixture is equipped with a PVC insert, which is particularly efficient at retaining heat during the decoupling procedure. A drawing of the device is shown in [12].

The shear strength $\tau_{Ad.}$ has been computed as in Eq. (1), where, $F_{Ad.}$ is the peak decoupling force, A indicates the coupling area and D_c and L_c are the coupling diameter and length [12].

$$\tau_{Ad.} = \frac{F_{Ad.}}{A} = \frac{F_{Ad.}}{\pi \cdot D_c \cdot L_c} \quad (1)$$

3. Experimental procedure

As mentioned above, LOCTITE 9466, according to the datasheet by the manufacturer [27], can be used up to the temperature of 120 °C, however its mechanical properties experience a steep decrease and assume very low values between 80 °C and 120 °C. Therefore, it seemed to be reasonable to set the maximum investigated temperature level to 80 °C. Intermediate levels at 40 °C and 60 °C have been added, to cover the range between 20 °C (room temperature) and 80 °C with the uniform spacing of 20 °C. A further motivation to this choice was the opportunity to have one level (40 °C) below the glass transition temperature of the adhesive, another level above (80 °C), and a final one in the order of the same threshold (60 °C). The campaigns at 40 °C, 60 °C and 80 °C have involved samples with the aforementioned 4 different levels of *ER* with 10 replications.

The same specimens of the previous campaign at room temperature have been re-used for the here-described campaigns at increased temperature. This approach is justified by the outcomes of previous researches [9,12], provided that both pins and collars are accurately cleaned, before being involved in the subsequent coupling procedure. LOCTITE7063 multi-purpose cleaner has been used for the cleaning task. Afterwards, both pins and collars have been, carefully, measured by micrometers (for internal and external surfaces), thus determining the actual values of the coupling diameters. The samples have been arranged in pairs, in order to reduce the scattering of clearance, maintained within the recommended values in [25–26].

The pins and the collars have been coupled as described in [12], following the bi-component adhesive preparation and overall recommendations in [27]. All the specimens have been cured for 7 days at room temperature [27]. A photo of the specimens just after the coupling task is depicted in Fig. 1.

A lab oven has been used to increase the sample temperature up to the desired level. All the samples have been inserted, only after that a steady-state temperature was reached inside the oven and here maintained for 24 hours.

The pushing-out tests have been run in the same conditions as described in [12]. The environmental temperature has also been controlled and maintained around 20 °C. The samples have been withdrawn from the oven and mounted on the self-aligning heat-retaining device just before each test.

An important issue regarded the requirement that the results were not affected by the order of the trials, in order to ensure their consistency. Following Refs. [22–23], this question should have been tackled by a full randomization of all the tests. However, many difficulties would have arisen from this approach, in particular from the need for three different ovens, simultaneously set at the three different temperatures. Moreover, mixing the tests at different temperatures, the result of each test may have been affected by that of the previous one, due to the different heating (or cooling) of loading devices. Therefore, it seemed to be more reasonable to arrange the tests at the three different temperature levels in three distinct blocks to be performed separately. Within each block, the tests were completely randomized, alternating the use of the fixtures as in [12]. The tests at the temperature of 40 °C have been executed first, whereas the other campaigns followed in increasing order for temperature. Moreover, to avoid the tests at incremented temperatures may be affected by the deterioration of the previously used fixtures, the horizontal PVC surface and the punch, which were supposed to withstand the highest amount of load during decoupling, were respectively reworked and replaced. Following this approach, exactly the same conditions were warranted for each campaign at the three temperature levels. A picture of the specimens in the oven (placed in the randomized order) is shown in Fig. 2.

Fig. 1. An overview of the specimens, after pin–collar coupling, before the campaign at 40 °C.

Upon decoupling, temperature drop has been checked by a type-K contact thermocouple. In particular, both the temperature of the pin and that of the collar have been measured with the subsequent computation of an averaged value. A summary of the retrieved values, with reference to the test at 40 °C, is reported in Table 1. It must be pointed out that the oven temperature has been slightly increased beyond 40 °C (up to 44 °C), to compensate the expected abrupt temperature drop in the short time between specimen withdrawal from the oven and its insertion in the fixture. Some preliminary tests have been performed to estimate the minimum required increment. The lowest values in Table 1 are generally due to the longer duration of the test (specimens with higher height and *ER*) or to a delay before temperature measurement, when difficulties occurred upon specimen removal from the fixture. Regarding the other temperature levels (60 °C and 80 °C), the temperature in the oven was also incremented (by 5 °C) with respect to its nominal value, in order to prevent the aforementioned temperature drop effect upon sample withdrawal. The pin and collar temperatures after the test were generally lower, by

Table 1
The detected temperature drops in the campaign at 40 °C.

Sample number in the decoupling sequence	Actual value of *ER*	Average temperature after decoupling [°C]	Sample number in the decoupling sequence	Actual value of *ER*	Average temperature after decoupling [°C]
1	1.76	37.0	21	1.32	43.5
2	0.87	37.0	22	0.88	42.6
3	1.32	37.5	23	0.44	41.7
4	0.44	37.3	24	1.31	43.2
5	1.75	37.0	25	1.75	43.7
6	1.75	39.3	26	0.87	43.1
7	0.87	37.3	27	1.74	40.4
8	0.87	37.2	28	0.87	43.5
9	1.31	39.8	29	0.44	42.8
10	1.74	39.7	30	1.31	43.0
11	0.44	38.1	31	0.88	43.1
12	0.44	38.9	32	0.44	43.1
13	1.32	41.0	33	1.32	43.4
14	0.44	39.3	34	0.87	43.4
15	1.31	41.7	35	0.44	42.2
16	1.75	40.3	36	1.31	42.4
17	0.87	41.9	37	0.44	41.8
18	0.88	41.4	38	1.75	43.3
19	1.75	43.1	39	1.32	41.4
20	0.44	42.9	40	1.75	43.0

Fig. 2. Samples in the oven in the randomized order.

Table 2
Test sequences and results of the campaigns at 40 °C, 60 °C and 80 °C.

N. (40 °C)	Pin diameter [mm]	Collar diameter [mm]	Collar length [mm]	Clearance [mm]	*ER* [-]	*A* [mm^2]	$F_{Ad.}$ [kN]
1	12.64	12.67	22.25	0.03	1.76	884.6	23.1
2	12.66	12.72	11.09	0.06	0.87	442.1	11.8
3	12.65	12.69	16.70	0.04	1.32	664.7	17.8
4	12.65	12.70	5.63	0.05	0.44	224.2	5.4
5	12.64	12.67	22.23	0.03	1.75	883.8	25.0
6	12.64	12.68	22.24	0.04	1.75	884.5	21.5
7	12.66	12.74	11.13	0.08	0.87	444.1	11.3
8	12.66	12.72	11.08	0.06	0.87	441.7	11.9
9	12.65	12.69	16.67	0.04	1.31	663.5	18.6
10	12.66	12.74	22.21	0.08	1.74	886.1	22.8
11	12.65	12.72	5.65	0.07	0.44	225.2	5.4
12	12.65	12.70	5.59	0.05	0.44	222.6	4.3
13	12.65	12.69	16.70	0.04	1.32	664.7	21.2
14	12.65	12.72	5.63	0.07	0.44	224.4	5.2
15	12.65	12.70	16.66	0.05	1.31	663.4	20.6
16	12.66	12.73	22.22	0.07	1.75	886.2	22.3
17	12.66	12.72	11.10	0.06	0.87	442.5	10.0
18	12.66	12.72	11.16	0.06	0.88	444.9	10.6
19	12.64	12.68	22.24	0.04	1.75	884.5	25.5
20	12.66	12.73	5.62	0.07	0.44	224.1	4.9
21	12.64	12.67	16.70	0.03	1.32	663.9	18.1
22	12.66	12.72	11.14	0.06	0.88	444.1	11.0
23	12.66	12.72	5.58	0.06	0.44	222.5	4.6
24	12.65	12.70	16.67	0.05	1.31	663.8	19.6
25	12.64	12.68	22.25	0.04	1.75	884.9	24.2
26	12.66	12.73	11.10	0.07	0.87	442.7	10.6
27	12.68	12.75	22.24	0.07	1.74	888.4	22.8
28	12.66	12.72	11.07	0.06	0.87	441.3	11.4
29	12.66	12.73	5.57	0.07	0.44	222.1	5.2
30	12.69	12.77	16.68	0.08	1.31	667.1	15.5
31	12.66	12.73	11.15	0.07	0.88	444.7	11.3
32	12.66	12.72	5.65	0.06	0.44	225.2	5.1
33	12.64	12.68	16.69	0.04	1.32	663.8	18.8
34	12.66	12.72	11.11	0.06	0.87	442.9	7.0
35	12.65	12.72	5.59	0.07	0.44	222.8	5.2
36	12.66	12.73	16.71	0.07	1.31	666.4	17.1
37	12.66	12.73	5.60	0.07	0.44	223.3	5.7
38	12.65	12.68	22.25	0.03	1.75	885.3	25.0
39	12.63	12.67	16.69	0.04	1.32	663.3	19.1
40	12.64	12.68	22.21	0.04	1.75	883.3	24.7
N. (60 °C)	Pin diameter [mm]	Collar diameter [mm]	Collar length [mm]	Clearance [mm]	*ER* [-]	*A* [mm^2]	$F_{Ad.}$ [kN]
1	12.66	12.73	11.09	0.07	0.87	442.3	9.9
2	12.64	12.68	22.25	0.04	1.75	884.9	14.3
3	12.66	12.73	16.71	0.07	1.31	666.4	9.0
4	12.65	12.72	11.10	0.07	0.87	442.3	8.8
5	12.64	12.68	22.24	0.04	1.75	884.5	14.6
6	12.65	12.72	11.11	0.07	0.87	442.7	8.8
7	12.66	12.75	22.21	0.09	1.74	886.5	18.9
8	12.67	12.77	16.68	0.10	1.31	666.6	12.8
9	12.64	12.68	22.25	0.04	1.75	884.9	16.9
10	12.68	12.80	16.77	0.12	1.31	671.2	11.3
11	12.66	12.74	22.20	0.08	1.74	885.7	8.9
12	12.65	12.72	11.10	0.07	0.87	442.3	9.2
13	12.66	12.73	11.09	0.07	0.87	442.3	8.1
14	12.64	12.68	22.21	0.04	1.75	883.3	16.8
15	12.65	12.72	5.63	0.07	0.44	224.4	5.6
16	12.66	12.73	16.65	0.07	1.31	664.0	12.9
17	12.66	12.75	22.24	0.09	1.74	887.7	18.3
18	12.65	12.73	5.60	0.08	0.44	223.3	4.5
19	12.63	12.67	22.23	0.04	1.75	883.4	16.6
20	12.66	12.73	11.09	0.07	0.87	442.3	7.8
21	12.66	12.74	11.13	0.08	0.87	444.1	7.7
22	12.65	12.73	5.57	0.08	0.44	222.1	4.4
23	12.66	12.73	5.58	0.07	0.44	222.5	5.0
24	12.66	12.73	5.59	0.07	0.44	222.9	4.2
25	12.65	12.72	5.63	0.07	0.44	224.4	4.4
26	12.65	12.73	5.59	0.08	0.44	222.9	4.4
27	12.64	12.70	16.67	0.06	1.31	663.5	12.9
28	12.66	12.73	5.62	0.07	0.44	224.1	4.7
29	12.63	12.67	16.69	0.04	1.32	663.3	13.0
30	12.65	12.70	16.66	0.05	1.31	663.4	13.1
31	12.64	12.68	16.69	0.04	1.32	663.8	12.5

Table 2 (*continued*)

N. (60 °C)	Pin diameter [mm]	Collar diameter [mm]	Collar length [mm]	Clearance [mm]	*ER* [-]	*A* [mm²]	$F_{Ad.}$ [kN]
31	12.64	12.68	16.69	0.04	1.32	663.8	12.5
32	12.64	12.68	22.24	0.04	1.75	884.5	14.2
33	12.65	12.73	5.65	0.08	0.44	225.2	4.4
34	12.66	12.74	22.22	0.08	1.74	886.5	17.9
35	12.63	12.67	16.70	0.04	1.32	663.7	13.1
36	12.65	12.72	11.08	0.07	0.87	441.6	8.4
37	12.65	12.72	5.65	0.07	0.44	225.2	4.0
38	12.66	12.73	11.10	0.07	0.87	442.7	7.8
39	12.64	12.70	16.67	0.06	1.31	663.5	12.9
40	12.65	12.72	11.07	0.07	0.87	441.2	6.4
N. (80 °C)	**Pin diameter [mm]**	**Collar diameter [mm]**	**Collar length [mm]**	**Clearance [mm]**	***ER* [-]**	***A* [mm²]**	**$F_{Ad.}$ [kN]**
1	12.65	12.70	5.63	0.05	0.44	224.2	4.1
2	12.65	12.70	16.70	0.05	1.31	665.0	14.3
3	12.64	12.67	22.25	0.03	1.76	884.6	15.3
4	12.66	12.73	5.58	0.07	0.44	222.5	4.0
5	12.67	12.74	22.20	0.07	1.74	886.1	15.5
6	12.66	12.72	11.16	0.06	0.88	444.9	7.4
7	12.66	12.73	11.08	0.07	0.87	441.9	7.0
8	12.65	12.70	16.67	0.05	1.31	663.8	11.3
9	12.66	12.72	11.10	0.06	0.87	442.5	7.9
10	12.64	12.68	22.24	0.04	1.75	884.5	15.6
11	12.66	12.71	11.10	0.05	0.87	442.3	8.2
12	12.64	12.67	16.69	0.03	1.32	663.5	10.3
13	12.66	12.73	22.21	0.07	1.74	885.8	18.5
14	12.64	12.69	5.63	0.05	0.44	224.0	3.5
15	12.66	12.72	5.59	0.06	0.44	222.9	3.0
16	12.64	12.68	16.67	0.04	1.31	663.0	12.6
17	12.65	12.71	5.65	0.06	0.44	225.1	3.7
18	12.66	12.73	22.24	0.07	1.75	887.0	15.1
19	12.65	12.69	16.70	0.04	1.32	664.7	12.1
20	12.66	12.72	11.11	0.06	0.87	442.9	7.0
21	12.65	12.71	11.14	0.06	0.88	443.8	8.3
22	12.65	12.70	11.09	0.05	0.87	441.6	7.4
23	12.66	12.72	5.57	0.06	0.44	222.1	3.8
24	12.67	12.75	22.22	0.08	1.74	887.2	15.3
25	12.66	12.72	5.65	0.06	0.44	225.2	3.7
26	12.66	12.72	5.59	0.06	0.44	222.9	2.5
27	12.64	12.68	22.24	0.04	1.75	884.5	15.7
28	12.66	12.73	16.65	0.07	1.31	664.0	10.4
29	12.66	12.72	5.62	0.06	0.44	224.1	3.2
30	12.66	12.72	11.10	0.06	0.87	442.5	6.0
31	12.64	12.69	16.66	0.05	1.31	662.9	11.0
32	12.68	12.78	16.77	0.10	1.31	670.7	11.9
33	12.66	12.73	11.07	0.07	0.87	441.5	6.6
34	12.66	12.72	16.71	0.06	1.31	666.2	11.6
35	12.64	12.67	22.21	0.03	1.75	883.0	13.8
36	12.66	12.73	11.09	0.07	0.87	442.3	7.3
37	12.64	12.67	16.70	0.03	1.32	663.9	11.2
38	12.64	12.68	22.25	0.04	1.75	884.9	15.2
39	12.64	12.67	22.23	0.03	1.75	883.8	11.4
40	12.66	12.72	5.60	0.06	0.44	223.3	3.8

5–8 °C, than the nominal temperatures of the campaign, mainly due the time elapsed between the actual end of decoupling and the measurement task, with sample rapid cooling following its exposition to fresh air. The higher the temperature level, the higher delay, up to 30–60 s, due to steel dilatation inside the fixture and to the need for using insulated gloves, when handling the samples.

After the tests all the mating surfaces have been analyzed to assess the correct adhesive polymerization in the samples with different sizes. In particular, it has been checked that the traces of the adhesive were spread over the entire involved heights both of the pins and of the collars.

The same procedure was repeated for the campaigns at the three temperature levels. The specimen arrangement in pairs after dimensional measurement has been generally changed to fulfill the maximum uniformity of clearance over the three campaigns.

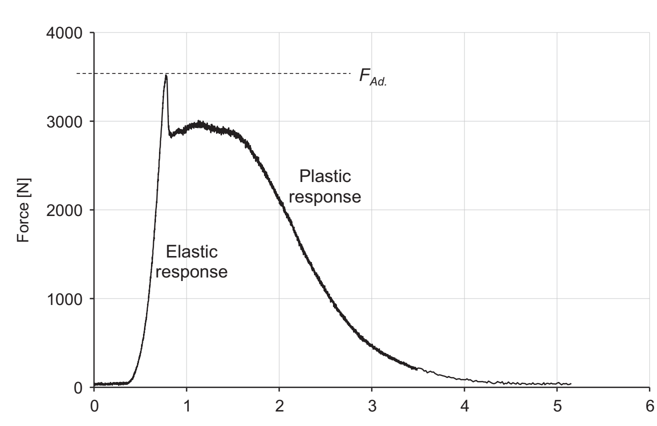

Fig. 3. A recorded force-displacement curve with indication of the peak value $F_{Ad.}$.

Fig. 4. Some specimens with different *ERs* after decoupling at the temperature of 80 °C.

Table 3
Shear strengths $\tau_{Ad.}$ [MPa] retrieved for different values of *ER* at the temperature of 40 °C.

Replication	$ER=0.4$	$ER=0.9$	$ER=1.3$	$ER=1.7$
1	23.4	25.8	23.2	25.7
2	20.7	26.9	31.1	25.6
3	23.5	26.7	29.5	25.2
4	25.3	23.9	25.6	27.9
5	19.5	15.8	28.0	24.3
6	22.0	22.7	26.7	28.8
7	23.3	25.4	31.9	28.3
8	23.9	24.7	28.4	27.3
9	24.0	25.3	28.8	28.2
10	22.7	23.9	27.2	26.1
Means	22.8	24.1	28.0	26.7

4. Results

The outcomes of the three experimental campaigns are collected in Table 2, for the temperatures of 40 °C, 60 °C and 80 °C. The tested samples and related results, in terms of the maximum pushing-out force, $F_{Ad.}$, are shown in the randomized order followed in the decoupling tests.

An example of a recorded force vs. displacement curve is shown in Fig. 3, where the peak of force $F_{Ad.}$, used for further processing, and elastic and plastic parts are highlighted. This graph refers to the pushing-out of a sample with $ER=0.4$ at the maximum temperature of 80 °C.

The analysis of the mating surfaces after decoupling indicated that a proper polymerization was likely to have occurred, as the traces of the adhesive appeared to be spread both on the pins and on the collars for the full heights. Some photos of samples with the four investigated *ERs* decoupled at the highest temperature of 80 °C are collected in Fig. 4.

5. Discussion

The first step consisted in the computation of the shear strength $\tau_{Ad.}$ of the Pin-and-collar joints: Eq. (1) was applied, considering the decoupling forces and the mating areas listed in Table 2. The results of the three experimental campaigns at the three temperature levels are going to be considered separately. Subsequently, the interaction between *ER* and temperature and the overall effect of heating is discussed.

The adhesive shear strengths determined in the first campaign at 40 °C are collected in Table 3, where the results are arranged, as recommended for one-factor ANOVA [22,23]. The matrix columns (number of columns$=4=C$) refer to results at different levels of *ER*, whereas the ten rows (number of replications$=10=R$) are corresponding to the ten replications. The same results are also summarized in the bar graph in Fig. 5(a), where the bar extensions correspond to the column means, i.e., to the average strengths at the four *ER* values. Scatter bands that indicate the variation from minimum to maximum values at each *ER* level are also included in the bar graph, to indicate data scattering.

The discussion regarding the impact of *ER* may start from the analysis of the bar graph: considering averaged values, the shear strength increases following an increase of *ER*, even if there is a slight drop between $ER=1.3$ and $ER=1.7$. The described outcomes are quite consistent with those at room temperature, except for the occurrence that in that case the mean values of strength kept increasing for ascending *ER*. As well as in [9,12], the tool of one-way ANOVA has been applied to determine whether *ER* affects strength at the studied temperature, comparing the outputs at different levels to experimental uncertainty.

The results of *ANOVA* are summarized in Table 4. The acronym *SSQ* generically indicates Sum of Squares: within this category, the Sum of Squares Between Columns (*SSBC*), the Sum of Squares Within Columns (*SSW*) and the Total Sum of Squares (*TSS*) are calculated and listed. *TSS* is the total amount of variance to be split between *SSBC* and *SSW*. *SSBC* refers to the portion depending on the effect of the studied factor, *ER*, whereas *SSW* considers experimental uncertainty. The Mean Squares (*MSQ*) are computed, dividing the *SSQs* by the related degrees of freedom (*DoF*). Finally, the Fisher ratio, $F_{calc.}$, is estimated as the ratio between *MSBC* and the uncertainty dependent term *MSW*. Further details can be found in [12,22–23].

The final output of the analysis, the *p*-Value, *p–v*, indicates that *ER* reasonably affects the output with a probability of failing in the order of 10^{-5}.

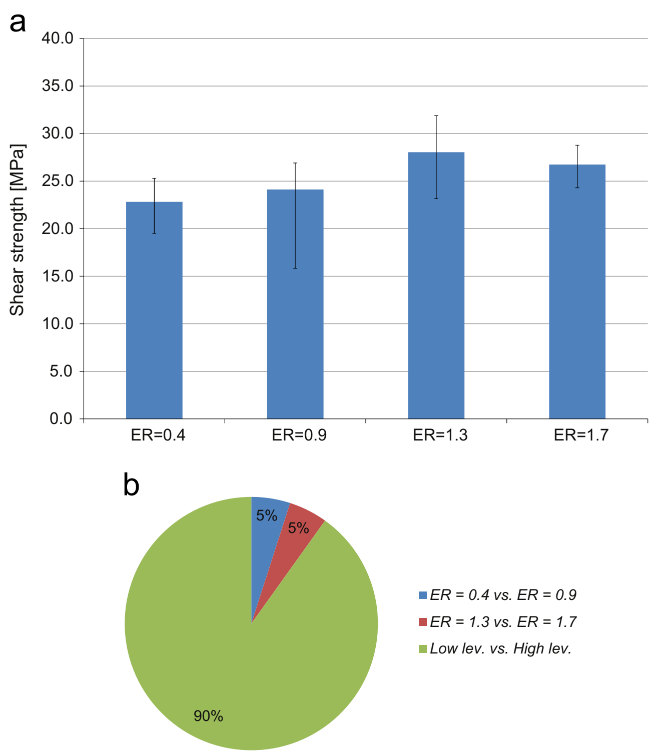

Fig. 5. (a) Shear strengths at different *ER*s at the temperature of 40 °C, (b) sources of variance.

Table 4
One-factor *ANOVA*, considering the results at the temperature of 40 °C.

SSQ		*DoF*	*MSQ*	$F_{calc.}$	*p–v.*
SSBC	170.17	3	56.72	10.26	$5 \cdot 10^{-5}$
SSW	199.09	36	5.53		
TSS	369.26	39			

Following the procedure in [12], this result, consistent with that obtained at room temperature, has been refined in order to allocate the differences between Table 3 columns, i.e., between the *ER* levels. Fisher's Least Significant Difference (*LSD*) and orthogonality approaches has been followed for this purpose.

The *LSD* threshold value according to [22–23] is 2.13, considering a 95% confidence level. This value must be compared to the differences of pairs of column means. Ordering the four means of each *ER* in increasing order and considering adjacent values, the only significant difference is that between the average strengths for *ER*=0.9 and *ER*=1.7. Whereas, the differences between *ER*=0.4 and *ER*=0.9, *ER*=1.3 and *ER*=1.7 are below the significance threshold. This outcome is consistent with that obtained at room temperature [12].

The tool of orthogonality [33] has been used to spilt the *TSS* into three terms, the first two depending on the differences between strengths at low values of *ER* (*ER*=0.4 and 0.9), and those between strengths at the highest levels of *ER* (*ER*=1.3 and 1.7). Considering that the aforementioned differences are not significant at the 5% significance level [22–23], the results at low and at high levels of *ER* have been merged together to evaluate the different performance for *ER* lower and higher than 1. Conversely, this difference is highly significant (*p–v.* in the order of 10^{-6}). All the results are collected in the augmented *ANOVA* in Table 5. A cake diagram, reporting the total variance decomposition along with component percentage rates, is shown in Fig. 5(b).

Table 5
Augmented *ANOVA*, to allocate the differences between the levels of *ER* at the temperature of 40 °C.

SSQ		*DoF*	*MSQ*	$F_{calc.}$	*p–v.*
SSBC	170.17	3	56.72		
ER=0.4 vs. ER=0.9	*8.44*	*1*	*8.44*	*1.53*	*22.5%*
ER=1.3 vs. ER=1.7	*8.40*	*1*	*8.40*	*1.52*	*22.6%*
Low levels vs. High levels	*153.33*	*1*	*153.33*	*27.7*	$7 \cdot 10^{-6}$
SSW	199.09	36	5.53		
TSS	369.26	39			

Table 6
Shear strengths $\tau_{Ad.}$ [MPa] retrieved for different values of *ER* at the temperature of 60 °C.

Replication	*ER*=0.4	*ER*=0.9	*ER*=1.3	*ER*=1.7
1	19.9	14.4	19.2	21.3
2	22.5	19.0	19.4	10.1
3	19.0	22.4	16.9	20.2
4	19.9	18.4	19.7	20.6
5	20.3	17.7	19.5	19.0
6	20.8	17.7	19.4	16.5
7	24.9	19.8	13.5	16.1
8	19.8	20.9	19.6	18.8
9	19.4	19.8	19.7	16.2
10	17.5	17.2	18.8	19.1
Means	20.4	18.7	18.6	17.8

The conclusion is that at the temperature of 40 °C the adhesive exhibits the same performance described in [12] at room temperature, with strength enhancement for *ER* > 1.

The results of the experimental campaign performed at the temperature of 60 °C are reported in Table 6. A summary of column mean values, with related minimum to maximum intervals, is shown in the bar graph of Fig. 6(a). It can be easily observed that this result is completely different from those at the previous temperature level and at room temperature. The highest mean strength has been retrieved for the lowest value of *ER* (0.4), whereas the other means are all very close with overlapped intervals.

The tool of one-way *ANOVA*, applied to these outputs has led to the conclusion that *ER* does not significantly affect the strength, i.e., result values are statistically the same. The results of the *ANOVA* are summarized in Table 7, where the symbols retain the previously specified meanings. The *p–v.* of 11.9% must be interpreted in the light of a 5% significance threshold, as commonly accepted for similar analyses [1, 12, 33]. The variance decomposition has therefore been stopped at this stage without the need of a further result refinement.

Finally, the outputs of the campaign at 80 °C are summarized in Table 8 and Fig. 6(b). The analysis of the bar graph suggests that result values at this temperature level are again different from those at room temperature and at 40 °C and are conversely consistent with those at 60 °C. The application of one-way *ANOVA* (the summary of the outputs is collected in Table 9) confirms that *ER* does not significantly affect strength.

The outcomes of the paragraphs above provide an experimental and statistical evidence that the effect of *ER* is different, depending on the actual working temperature of the joint.

At the temperature of 40 °C a sufficiently high value of *ER* improves resistance: this result is consistent with the outcomes of the study at room temperature [12]. The recommended value for *ER* is greater than 1, but not too high, since a saturation occurs when *ER* increases up to 1.7. A value around 1.3-1.5 can therefore be regarded as a good compromise. Conversely, between the temperatures of 40 °C and 60 °C the adhesive response experiences an abrupt change: at the levels of 60 °C and 80 °C *ER* is no longer significant at

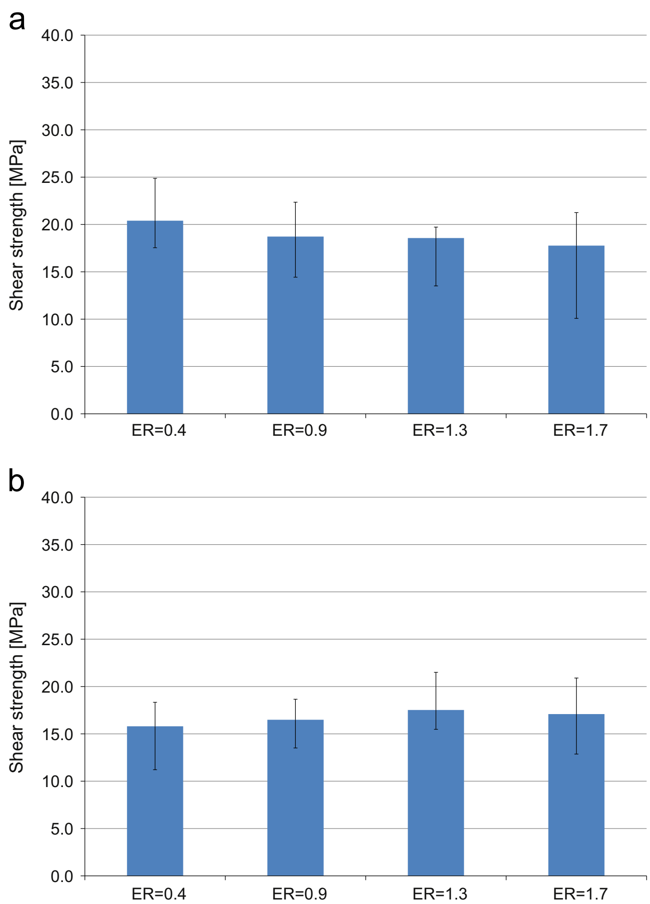

Fig. 6. (a) Shear strengths at different *ERs* at the temperature of 60 °C and (b) at the temperature of 80 °C.

Table 7
One-factor *ANOVA*, considering the results at the temperature of 60 °C.

SSQ		*DoF*	*MSQ*	$F_{calc.}$	*p–v.*
SSBC	36.61	3	12.20	2.09	11.9%
SSW	210.51	36	5.85		
TSS	247.12	39			

Table 8
Shear strengths $\tau_{Ad.}$ [MPa] retrieved for different values of *ER* at the temperature of 80 °C.

Replication	*ER* = 0.4	*ER* = 0.9	*ER* = 1.3	*ER* = 1.7
1	17.1	15.0	15.6	17.5
2	18.0	15.8	17.7	17.3
3	13.5	16.5	17.0	20.9
4	11.2	17.8	16.6	17.0
5	17.2	13.5	17.4	15.6
6	14.4	16.8	19.0	17.6
7	18.3	18.6	21.5	17.7
8	15.8	15.7	18.2	12.9
9	16.4	18.7	15.5	17.1
10	16.2	16.5	16.9	17.3
Means	15.8	16.5	17.5	17.1

enhancing the joint strength. It is interesting to remark that this change of behavior occurs at the aforementioned temperature threshold for glass transition, which is 62 °C for the studied adhesive. Therefore, it can be stated that the adhesive exhibits a

Table 9
One-factor *ANOVA*, considering the results at the temperature of 80 °C.

SSQ		*DoF*	*MSQ*	$F_{calc.}$	*p–v.*
SSBC	16.74	3	5.58	1.55	21.9%
SSW	129.80	36	3.61		
TSS	146.54	39			

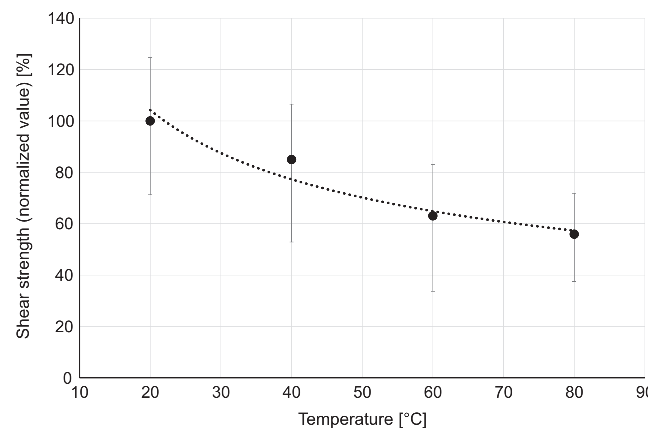

Fig. 7. Shear strength (normalized with respect to the response at room temperature [12]) plotted vs. temperature.

temperature threshold for the strength response vs. *ER* that can be related to the glass transition temperature.

The described outcome must be interpreted in the light of the general response of the adhesive vs. temperature. The characterization of the adhesive joint at increased temperature was performed by the manufacturer, considering lap-joints, according to ISO 4587 [34]. The results retrieved by the manufacturer, available in [27], indicate that the joint strength abruptly drops to 20% and 10% of the strength at room temperature at respectively 60 °C and 80 °C.

Considering the here-described campaigns on Pin-and-collars, the trend of the shear strength vs. temperature curve is shown in Fig. 7. In order to be able to compare this trend to that provided by the manufacturer, the strength data retrieved at each temperature level for the four different *ERs* have been averaged, i.e. grand mean values have been considered. Moreover, the grand means computed for the tests at 40 °C, 60 °C and 80 °C have been normalized with respect to the average strength determined at room temperature (29.9 MPa) [12], which has therefore been assigned a 100% value. Furthermore, scatter bands are also appended to each point, to account for the overall range of variation retrieved at each temperature level, considering all the tested specimens, regardless of their sizes. Two issues need to be observed: first of all, the curve that interpolates the points in Fig. 7 follows a hyperbolic decreasing trend, consistently with the Manufacturer's results [27]. Secondly, the results of the current campaign indicate that temperature has indeed a detrimental effect on the joint strength. However, its decrease for axisymmetric joints is much lower than that for lap joints. Considerably high strengths, around the 60% of the maximum considering average values, around 40% at the lower boundaries of the variation intervals, are still available at the two top temperature levels. This is a particularly important outcome for the many applications in mechanics, where axisymmetric joints are used, e.g., those pointed out in [35].

Back to the combined effect of temperature and *ER*, temperature increase implies a drop of the adhesive shear strength: at 40 °C it can still be compensated by an increase of *ER* exceeding 1. At higher temperatures, the strength decrease is more consistent, and consequently it levels out the differences between the

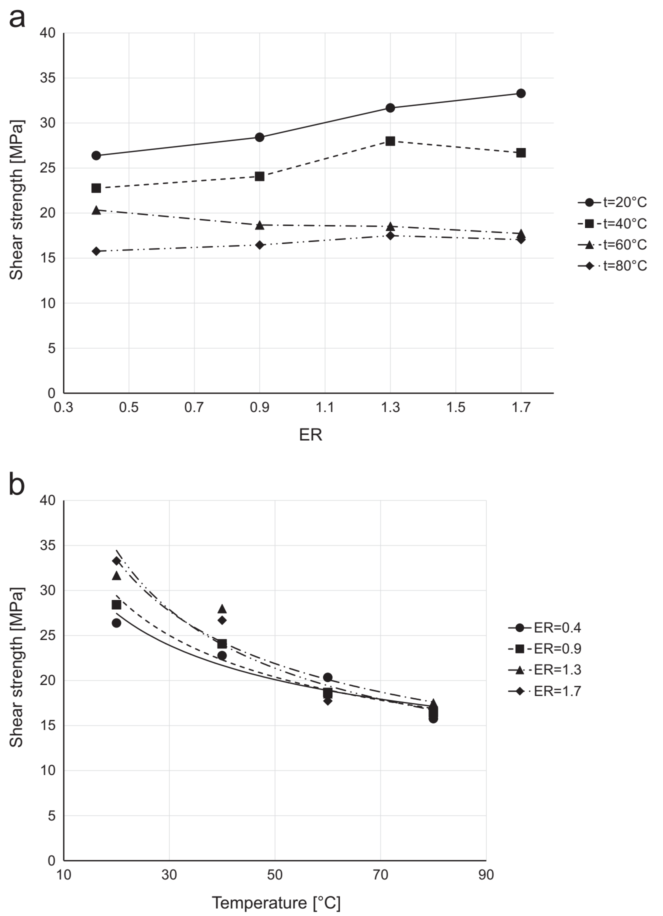

Fig. 8. Interaction at (a) different levels of temperature and (b) different levels of *ER*.

responses at different values of *ER*. Therefore, compensation is no longer possible: in other words, the effect of *ER* is shadowed by temperature and the adhesive response is made insensitive to the different proportioning of the joint.

The described behavior indicates that for increasing temperature, the effect of *ER* is more and more decreased from being initially significant to finally negligible. It implies that a negative interaction occurs between the two factors involved in this study: *ER* and temperature. The presence of a significant interaction has therefore been checked by a two-factor *ANOVA*, considering simultaneously the effects of temperature and *ER* and including the results at room and at increased temperatures. This further analysis has confirmed that a significant interaction takes place between the two factors, at a very high confidence level (*p–v.* in the order of 10^{-7}).

The effects of negative interaction are visible in the diagrams sketched in Fig. 8. In the first one (a) the trends of shear strengths vs. *ER* are plotted for each of the investigated temperature levels. The column means are used for this purpose. The decreasing rate of the distributions, as a result of a decreasing *ER* effect, can be easily observed. The trends of averaged shear strengths vs. temperature are depicted in the second graph (b) for the four investigated values of *ER*. In this case, it can be remarked that the higher *ER*, the steeper the related curve and therefore more negative and detrimental the effect of temperature. An interesting outcome of this behavior is that the effect of temperature, reducing the joint strength, is less detrimental at low values of *ER* (decrease by 40%) rather than at high levels (decrease by 49%). In other words, short joints are less sensitive to temperature increase.

In the Introduction Section it has been pointed out that previous studies are focused on the separate effects of joint proportioning and of temperature on the mechanical response of epoxy adhesive joints, even if these two factors had never been studied in combination. The outcome of this study indicates that the simultaneous analysis of these two factors is a required approach, to be able to appreciate their high interaction that significantly affects the joint response.

6. Conclusions

Epoxy adhesives have wide applications in the automotive industry and in the construction of lightweight structures, often in the aeronautic or avionic fields. Two important factors, potentially affecting the shear strength of the joint, are its proportioning, in particular its engagement ratio (coupling length over coupling diameter), and the temperature in use conditions. Based on the current literature, the combined effect of these factors has never been investigated. This issue has been tackled here with reference to the bi-component adhesive LOCTITE 9466.

Pin-and-collar samples with four different engagement ratios have been prepared and tested at the temperature of 40 °C, 60 °C and 80 °C, thus covering the temperature range suggested by the adhesive manufacturer. Tests conducted under a standing press in the displacement control mode have been performed to measure the peak decoupling force and therefore the joint strength.

Statistical tools, in particular one-way and two-way *ANOVA* and orthogonality, have been applied to process the strength data. They led to the conclusion that the engagement ratio significantly affects strength, which is enhanced for a value around 1.3, up to a temperature of 40 °C. At the higher temperatures, the adhesive strength experiences a generalized drop, so that the differences due to different geometry are levelled out and the effect of proportioning is therefore shadowed. The found threshold for the adhesive behavior vs. the Engagement Ratio can be correlated to its glass transition temperature.

The decrease of the maximum strength, around 44%, when the temperature increases, is much lower than the values retrieved by the manufacturer by tests on lap joints.

Finally, it has been remarked that studies dealing with the simultaneous effect of the joint proportioning and of the temperature are missing. However, considering them in combination is the key approach to estimate the actual response regarding strength. An interesting outcome is that the detrimental sensitivity of the adhesive strength to temperature is lower (up to 9%, according to the investigated range for the engagement ratio) for short joints rather than for long ones.

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