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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 I: Campaign at room temperature

Adhesion &

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ABSTRACT

This paper focuses on an epoxy adhesive (LOCTITE 9466), which is particularly suitable for applications involving different materials and where a clearance is present between the adherents. The investigated subject is concerned with the effect of the Engagement Ratio (ER, coupling length over coupling diameter) on the shear strength of LOCTITE 9466 at room temperature. Motivations arise from the increasing interest in epoxy-adhesive joints in lightweight structures and from the consequent need for design data. Decoupling tests have been performed on pin-and-collar samples manufactured according to current Standards. The height has been adjusted in order to explore a sufficiently wide ER range at four different levels. The results have been processed by the tools of the Analysis of Variance and of the Fisher test to investigate the significance or the not significance of ER on the joint shear strength. The final outcome was that ER significantly affects resistance at a very high confidence level. This result has then been refined by the tool of orthogonality, in order to allocate the differences among the four levels of ER. This further analysis has shown that the joint strength is significantly enhanced, when ER exceeds 1 and assumes values around 1.3 or higher.

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1. Introduction

Adhesively bonded joints are used in many mechanical applications, because they offer several advantages, such as the reduction of weight, the increasing of strength and the improvement of fatigue and fretting corrosion. These advantages have been demonstrated to be effective in the case of interference fitted and adhesively bonded joints [\[1\],](#page-6-0) namely, hybrid joints, whatever is the production system. Many researches evaluated the strength of these joints in dependence of several variables, such as the assembly pressure level $[2]$, the type of materials in contact $[3-4]$ $[3-4]$, the curing methodology $[5]$, the operating temperature $[6-7]$ $[6-7]$, the loading type [\[8](#page-6-0)–[9\]](#page-6-0) and type of joining technique [\[10](#page-6-0)–[11\].](#page-6-0) In order to reduce the weight and the amount of the material, it is possible to reduce the Engagement Ratio (ER), which is the ratio between the coupling length over the coupling diameter. However, the reduction of the ER may lead to a reduction in the strength of the joint, therefore, its effect has been deeply investigated in a pinand-collar set of specimens in the case of anaerobic adhesive [\[12\].](#page-6-0)

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The anaerobic adhesive is really effective in the case of metal parts to be connected and, particularly, in the case of steel components, whereas in the case of aluminium alloy or, worse, of composite material, the adhesive strength is strongly reduced [\[4\].](#page-6-0) A more suitable adhesive in such cases is the epoxy one that is, normally, used in slip-fit joints, where a clearance is present between the adherents. An additional and particularly relevant advantage of epoxy adhesives is that they offer the opportunity of bonding materials with different physical and mechanical properties, without triggering detrimental variations of their chemical structure. An important outcome is that epoxy adhesives can be successfully used to bond protective coatings to structural parts, thus achieving simple repair, with many applications in petroleum, aviation and aerospace industries [\[13](#page-6-0)–[15\]](#page-6-0). A further application field is related to the development of joints between different materials, usually a steel shaft and a composite hub, mainly in automotive. According to [\[16\]](#page-6-0), composites are the most suitable materials for many suspension components in racing cars, where lightweight properties are essential for successful race participation. Examples of parts that are better suited to composites are pushrods, A-arms and steering arms, due to the tensile/compressive load they experience in operation conditions and as an effect of their good response in terms of strength and stiffness. Similar design strategies are likely to be followed even in high class car

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mass production. Epoxy adhesives, like LOCTITE 9466, are generally used for bonding composite tubes with steel shafts, thus obtaining mixed material joints, whose strength is often difficult to predict. Consequently, there is an increasing need for design data, involving in particular the effect of the length of the joint: an increased length is indeed able to positively enhance the joint strength, but with the outcome of an overall increase in dimensions and weight. The lack of studies investigating the effect of ER inspired the present work, whose subject consists in the experimental investigation of the effect of ER on the shear strength of LOCTITE 9466 epoxy adhesive. Tests on pin-and-collar samples have been conducted at room temperature, with the cylindrical geometry of specimens being justified by the aforementioned applications in the automotive field.

2. Materials and methods

First of all, the specimens have been designed and produced, following the Standard ISO 10123 [\[17\]](#page-6-0) for slip-fit joints. The sketch of the pin-and-collar samples produced and tested is reported in Fig. 1. Since the ISO 10123 suggests to choose an $ER = 0.9$ and since the dimensions investigated in [\[12\]](#page-6-0) are of four types (the half, the double and an intermediate value $ER = 1.3$), the ERs values were set at the same value for this work. The collars have been designed, so that their diameters were consistent with the recommended values in [\[17](#page-6-0)–[18\]](#page-6-0). Their heights have been adjusted, in order to meet the aforementioned values of ER, while the chamfer dimensions have been maintained unchanged, as well as the pin dimensions. The material of the samples is C40 UNI EN 10083-2 steel, whereas the adhesive type is the previously mentioned commercial LOCTITE[®] 9466, which is an epoxy glue with two components. In order to improve the statistical analysis and the definition of the significance of the ER parameter, a number of ten replications for each different ER has been chosen. The overall sample population consisted therefore of 40 Pin-and-collars for statistical evidence reasons. The whole set of specimens has been measured, in order to accurately determine the coupling diameters and to check their height values. For this purpose, a

Fig. 1. Pin-and-collar specimens with $ER = 0.4$ (a), $ER = 0.9$ (b), $ER = 1.3$ (c), $ER = 1.7$ (d) (all dimensions in mm).

Fig. 2. (a) Drawing of the bushing with inner PVC insert (all dimensions in mm), (b) two sets of self-alignment device, experimental setting in trial conditions shown by a section view (c) and a picture taken just after decoupling (d).

micrometre screw gauge, an inside micrometre, and a digital caliper, all with the resolution of 0.01 mm, have been used.

An important issue regards misalignments, which could arise during the pushing-out phase and may seriously affect results. In order to overcome this point, a self-aligning rig has been specifically developed. It consists of a steel punch with a Teflon bushing for its alignment and of a fixture for the pin-and collar samples. This has been worked out as a component consisting of two parts: a steel bushing and a PVC insert, where the samples were intended to be placed just before the pushing-out procedure. The goal of the here described research was to perform an experimental campaign at room temperature; however, a key issue of the fixture design was that it could be suitable even to tests at incremented temperature, to be performed at a subsequent stage of the study. Using the same device for testing has been essential to ensure the consistency of results. This is the reason why a PVC internal bushing was used: taking advantage of its low thermal conductivity coefficient made it possible to reduce heat dissipation. A drawing of the bushing with inner PVC insert and a photo of two sets of self-alignment devices are shown in Fig. 2. The experimental setting is also shown in the same figure by a section view of the described rig after the insertion of a sample and by a photo taken just after specimen decoupling.

The decoupling tests have been performed by a standing press, where the pushing-out force, F_{Ad} , was on-line measured. The shear strength τ_{Ad} , was finally computed as in Eq. (1), where A indicates the coupling area and D_c and L_c are the coupling diameter and length. D_c is computed as the average between the pin external diameter and the collar internal diameter. Finally, L_c is the collar length.

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

Fig. 3. Pin and collar samples with the assembly tools.

3. Experimental procedure

It has been remarked that the coupling diameters of the pins and of the collars were carefully measured. It was the opportunity to sort them out, in order to suitable combine the two parts with the aim of maintaining the level of clearance at an approximately constant value. Proceeding this way, the clearance average was in the order of 60 μ m with a standard deviation of 15 μ m, thus meeting the requirements of [\[17](#page-6-0)–[18\]](#page-6-0).

The assembly procedure was the following for the whole set of specimens:

- 1. The surfaces were cleaned by the LOCTITE $[®]$ 7063 cleaner and by</sup> a fine sandpaper;
- 2. The dual cartridge was inserted into the application gun and a light pressure was applied to the cylinders. The mixing nozzle provided by the manufacturer was mounted at the end of the cartridge to facilitate automatic mixing of resin and hardener. The two components were completely merged together, upon hand shaking for a few seconds until a uniform colour of the adhesive was obtained [\[19\].](#page-6-0)
- 3. The adhesive was spread on the mating surfaces of the male and female parts and, just after, the collar was shifted over the pin, with a helicoidal back-and-forth rapid movement.
- 4. The exceeding glue was removed, as it could have affected the specimen length and therefore the ER related results.
- 5. The adhesive was cured for seven days at room temperature, as recommended in [\[19\]](#page-6-0);

The samples collected before the joining operation and with the assembly tools described above, are reported in the picture of Fig. 3, whereas the joined specimens are reported in [Fig. 4.](#page-4-0)

The push-out tests have been run by a standing press equipped by two load cells (connected in series), having capacities of 25 kN and 250 kN. All the trials have been run in displacement controlled conditions, applying a ramp with 0.03 mm/s speed, thus meeting the recommendations of [\[17\]](#page-6-0). This speed has been increased to 0.5 mm/s after the maximum peak of force $(F_{Ad.})$, to complete the decoupling. Both the applied force and the actuator displacement were recorded during the pushing-out procedure at the sampling rate of 30 Hz.

A further issue consisted in the possibility that the order of trials may somehow affect the experimental results in terms of the retrieved pushing-out force and of the resulting strength. This question has been overcome, using two identical self-aligning rigs (visible in [Fig. 2](#page-2-0)), which have been alternatively mounted for decoupling tests, thus reducing the risk of their progressive deterioration. Moreover, the order of the tests involving the 40 samples with different ERs has been completely randomised, as suggested by Statistics Refs [\[20](#page-6-0)–[21\].](#page-6-0) The tested specimens with the identification labels (#), pin and collar dimensions, clearances and ERs are reported in [Table 1](#page-4-0) in the actual randomised order followed in the experimental campaign. It can be observed that samples with different ERs are all mixed up in the sequence.

4. Results

An example of the pushing-out diagram acquired by the system, is shown in [Fig. 5,](#page-4-0) with reference to a sample with $ER = 1.7$. The linearelastic behaviour of the glue, the stick-slip phenomenon, which occurred in almost all the tested couplings, and the plastic part up to the complete separation of the two sample parts are highlighted in the graph. The static strength of the adhesive (τ_{Ad}) has been calculated, according to Eq. [\(1\)](#page-2-0): the results, are reported in [Table 2](#page-4-0).

The adhesive strength values reported in [Table 2](#page-4-0) were then statistically analysed, in order to highlight if the ER parameter was significant.

5. Statistical analysis and discussion

As described in the previous Section, forty decoupling tests have been performed, involving pin-collar specimens, having four different ERs. Ten samples have been tested for each value of ER: the related results can be regarded as ten replications of the same measurement. The experimental outcomes, in terms of the shear strength of the adhesive, τ_{Ad} , are collected in [Table 3](#page-4-0), where the matrix columns correspond to the four levels of the ER, whereas the rows are related to the ten replicates RN. The results are also summarised in [Fig. 6,](#page-5-0) where each of the four bars stands for the mean shear strength determined for each of the considered ERs.

The four means are computed as averages of the values in the four columns of [Table 3.](#page-4-0) In order to have a full description of the experimental population, scatter bands, which account for the scattering from the minimum to the maximum values retrieved at each ER level, are added to each bar. As reported in the Introduction Section, one of the main goals of this research relies on the investigation of the effect of the ER on the adhesive response.

Especially from the point of view of structural design, it is important to discuss if the nominal adhesive strength may be affected, and, in case, enhanced, by a variation of the adherent length. The analysis of the bar graph in [Fig. 6](#page-5-0) suggests that the ER is likely to affect the adhesive response, since strength increases, as ER is incremented from 0.4 to 1.7. However, considering the related scatter bands, it can be easily observed that they are partially overlapped. Therefore, concluding that ER affects strength, would be questionable at this stage. In order to suitably tackle this question, the data in [Table 3](#page-4-0) have been processed by a one-factor Analysis of Variance (ANOVA) [\[20](#page-6-0)–[21\]](#page-6-0). This approach makes it possible to compare the outputs for different values of the considered factor (ER), accounting also for the experimental uncertainty, i.e. for the scattering of the data. The adoption of this methodology is supported by its successful application in several references, e.g. [\[12](#page-6-0), [22\].](#page-6-0)

The output of the ANOVA is summarised in [Table 4.](#page-5-0) The symbols in [Table 4](#page-5-0) have the meanings briefly described below, full details can be found in [\[20](#page-6-0)–[21\].](#page-6-0) Considering initially the data in the first two columns, SSQ is a generic term for Sum of Squares. SSBC stands for the Sum of Squares Between Columns and has the meaning of the amount of the total variance, being related to the effect of the studied factor. It can be computed according to Eq. (2) , where \overline{y} , is the mean of the j-th column in [Table 3](#page-4-0) (i.e.: the mean strength for the j-th value of ER, plotted in [Fig. 6\)](#page-5-0) and \overline{y} . is the overall mean of the data in [Table 3.](#page-4-0) Finally, R stands for the number of replications (ten in the present case), whereas C is the number of the considered levels for the factor ER (four).

$$
SSBC = R \cdot \sum_{j=1}^{C} (\overline{y}_j - \overline{y}_r)^2
$$
 (2)

SSW is commonly regarded as the Sum of Squares Within (Columns) and is related to the amount of variance depending on

Fig. 4. Pin and collar samples assembled before the pushing-out operation.

Table 2 Test sequence and result values.

Fig. 5. Example of Force vs. Displacement diagram.

Table 3 Adhesive shear strengths $\tau_{Ad.}$: [MPa] determined for different ERs.

RN	$ER = 0.4$	$ER = 0.9$	$ER = 1.3$	$ER = 1.7$
	29.6	28.5	33.1	37.3
2	27.6	27.1	29.8	34.1
3	28.3	28.9	29.9	31.1
4	21.3	28.3	31.2	36.5
5	26.0	36.4	33.3	32.0
6	30.6	25.5	33.3	36.8
7	24.2	24.0	30.3	31.4
8	23.1	27.7	33.8	32.5
9	28.2	28.8	33.1	28.6
10	24.9	29.1	29.1	32.6

Fig. 6. Bar graph summarizing the mean values of τ_{Ad} , for different levels of ER, along with scatter bands.

Table 4

One-factor ANOVA to investigate the effect of ER on $\tau_{\rm Ad}$.

the uncertainty of the experiment. It is computed in Eq. (3) , where y_{ij} is the yield in the i-th row and j-th column of [Table 3.](#page-4-0)

$$
SSW = \sum_{j=1}^{C} \left[\sum_{i=1}^{R} \left(y_{ij} - \overline{y}_{j} \right)^{2} \right]
$$
 (3)

TSS stands for the Total Sum of Squares and is the total amount of variance of the experiment. The latter is clearly related to the other terms by the following relationship: $TSS = SSBC + SSW$. The column referenced as DoF in Table 4 contains the degrees of freedom to be used to scale the SSQs and to finally retrieve the Mean Squares, generally indicated as MSQ. The Mean Square Between Columns (MSBC) and the Mean Square Within (Columns) (MSW) can be easily computed, dividing the SSQs by the corresponding degrees of freedom. The F_{calc} is then determined as a ratio between MSBC and MSW and has the meaning of the Calculated Fisher's ratio, to be used in the statistical Fisher's test. Finally, the $p-\nu$. is usually regarded as the $p-\nu$ value and retains the meaning of the probability of failing, when stating that the factor does affect the output. This coefficient can be easily computed by electronic sheets, based on the Fisher's distribution. The resulting p-v. is here very low, in the order of 10^{-6} , which indicates that there is a strong evidence that the ER has a significant influence on the adhesive strength τ_{Ad} .

This result indicates that significant differences are present between the yields in the columns of [Table 3](#page-4-0), obtained for different ERs. However, a possible drawback of the performed analysis is that it is not possible to directly allocate these differences. In other words, it is not possible to establish if the considered factor has an effect over the entire range, or if it is initially ineffective and acquires a significant impact only beyond a certain threshold of ER. The easiest option to tackle this question is to make use of a multiplecomparison test, to compare pairs of column means. Therefore, the Fisher's Least Significant Difference (LSD) test [\[20](#page-6-0)–[21\]](#page-6-0) has been used to compare the strengths determined for adjacent levels of ER. In particular, the strength for $ER = 0.4$ has been, initially, compared with the strength for $ER = 0.9$, while the further pairwise comparisons have involved level $ER = 0.9$ vs. level $ER = 1.3$ and level $ER = 1.3$ vs. level $ER = 1.7$. The application of the LSD criterion requires first the

Table 5

LSD Tests to investigate the significance of the differences between adjacent ER levels.

Table 6

Augmented ANOVA to deepen the analysis of the effect of ER on $\tau_{Ad.}$

Fig. 7. Partition of the SSBC.

computation of the Least Significant Difference term that gives the name to the test. Its calculation, based on [\[20](#page-6-0)–[21\],](#page-6-0) and considering a 95% confidence level, has yielded the threshold value of 2.51, to be compared to differences of mean pairs.

A resume of the results is shown in Table 5, where the LSD threshold is computed for a 95% confidence level of each pairwise test.

The differences are significant, when they overcome the LSD threshold. Therefore, the difference between the results for $ER = 0.9$ and $ER = 1.3$ is significant (at the 95% confidence level). The conclusion of the multiple comparison tests is that significance differences do exist between the second and the third level of ER. In other words, there is a significant enhancement between the adhesive response at low levels (0.4, 0.9) of ER and that at high levels (1.3, 1.7). Following this result, the analysis has been deepened, according to the approach of the orthogonal decomposition of the SSBC [\[20,](#page-6-0) [22\]](#page-6-0). This approach makes it possible to split the SSBC into three terms, which account for the possible sources of variability, finally determining whether they are significant or not. In the present case, the SSBC has been divided into:

- 1. One term depending on the differences of $\tau_{Ad.}$ at low levels (0.4, 0.9) of ER;
- 2. A second term related to the differences of τ_{Ad} : at high levels (1.3, 1.7) of ER;
- 3. A third final term depending on the differences between the average response at low levels of ER (results for $ER = 0.4$ and $ER = 0.9$ taken together) and the average response at high levels (results for $ER = 1.3$ and $ER = 1.7$ together).

The results are reported in the augmented ANOVA in Table 6.

The outputs of the Fisher tests indicate that the first and second terms are not significant at the 5% significance level. This threshold is usually the most suitable, according to several references, e.g. [20–21]. Whereas, the amount of variance related to the different response of the adhesive at low and high values of the ER is highly significant (probability of error, when accepting the significance hypothesis in the order of 10^{-6}). This outcome is consistent with those of the LSD comparison tests and is confirmed by the graphical sketch of the SSBC partition, shown in the cake diagram in [Fig. 7](#page-5-0). It can be remarked that almost 90% of the total amount of the SSBC is due the incremented strength when using high ERs.

6. Conclusions

The present paper provides some preliminary information concerning the influence of the Engagement Ratio on the strength of epoxy adhesive with two components, the commercial LOCTITE[®] 9466. The specimens are the standard pin-and-collar samples, specifically adapted in order to change their original Engagement Ratio. Four different Engagement Ratios have been studied; one is equal to the original dimension, one is smaller and two are greater. The results have been statistically analysed in order to highlight the significance of the Engagement Ratio parameter. The present analysis pointed out a strong dependence of the adhesive strength on the Engagement Ratio value, even if the significance is restricted between the two low and the two high levels. In conclusion, a threshold limit for the Engagement Ratio value, seems to be set equal to 1, so that it is advisable to use values at least equal to 1.3-1.5.

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