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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].

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

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