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Fracture toughness of structural adhesives for the automotive industry

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Abstract

Adhesive bonding is currently employed by automotive manufacturers to complement (or replace) welding in joining dissimilar materials. In order to reduce the impact on the existing manufacturing infrastructures, structural adhesives are deployed in the body shop but hardening is accomplished in the paint cure oven. Various adhesive formulations have been specifically developed for the implementation in the automotive manufacturing chain. However, it is very important to assess the mechanical behaviour of the joints which results from the peculiar curing strategy. In the present work, automotive grade single component epoxy and two component epoxy modified acrylic adhesives were evaluated. T-joints were fabricated using a cold rolled galvanized steel (FeP04) employed in the production of car body parts. The fracture toughness of the joints was determined using the test protocol proposed by the European Structural Integrity Society (ESIS). Optical microscopy was employed to ascertain the mechanisms of failure. The results indicated that both adhesives were able to provide a fairly good mechanical response with minimum preparation of the mating substrates. Moreover, the obtained values of fracture toughness were shown to be essentially independent of the adhesive layer thickness.

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Keywords: automotive, adhesives, fracture toughness, T-joint

1. Introduction

Cars are responsible for around 12% of total EU emissions of carbon dioxide (CO_2). To improve the fuel economy of cars sold on the European market the EU legislation established mandatory emission reduction targets, such as those recently disclosed in the Climate Action EU no. 333 (2014). In order to meet these requirements, automotive manufacturers are currently increasing the share of lightweight materials and high strength steels in car body manu-

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facturing, see D'Aiuto (2016). The strategy pursued is to place the right materials with the right properties at the right place. This approach led to the compelling need of joining materials with dissimilar properties. From this standpoint, adhesive bonding emerges as a suitable technique able to replace -or complement- fusion or spot welding (Chiodo et al. (2015); Rotella et al. (2015)). However, at present time it is highly desirable that the introduction of structural adhesives in car body manufacturing is made with minimum impact on the manufacturing infrastructures. For this reason, adhesives are commonly applied in the body-in-white stage (*i.e*. assembly of frame and panels) while final curing is performed in the paint shop. Tailored structural adhesives have been developed to accommodate the implementation in the manufacturing chain. The scope of the present work is to assess the fracture properties of two types of automotive grade structural adhesives deployed in modern car body manufacturing. T-joints were fabricated following the curing cycle employed in the paint shop according to the classical process chain of automobile production. Adhesive joints featuring cold rolled galvanized steel substrates (FeP04) were bonded with either a single component epoxy (DOW Betamate 1060S) or a two component epoxy modified acrylic adhesive (LORD Versilok 265/254). The results of mechanical tests were post-processed using the ESIS Test Protocol (2010) proposed by the European Structural Integrity Society. The fracture surfaces were finally analyzed by means of optical microscopy to ascertain the mechanisms of failure.

2. Materials and methods

2.1. Materials

The steel employed herein for the fabrication of T-joints is a cold rolled galvanized steel (FeP04) deployed in the production of body components in the automotive industry. Two selected substrate thickness have been considered in mechanical tests, *i.e*., 1.2 mm and 1.5 mm. The chemical composition of the FeP04 steel is reported in Tab. 1 while the corresponding stress-strain curve obtained through tensile tests is reported in Fig. 1(a).

Table 1: Chemical composition of the FeP04 cold rolled galvanized steel

Material	C	Fe	Mn	P	S
FeP04	≤ 0,08%	≥ 98%	≤ 0,4%	≤ 0,03%	≤ 0,03%

Two selected structural adhesives have been used to fabricate the joints, namely the Betamate 1060S (DOW Chemical Company, USA) and the Versilok 265/254 (LORD Corporation, USA). The Betamate 1060S is a one component, heat curing, epoxy based adhesive. It is especially tailored for the body shop because it features excellent adhesion to automotive steels (including coated steels and pre-treated aluminium). Moreover, it is compatible with the e-coat process and it is wash off resistant. Typical applications include bonding of the vehicle body structures. No details are disclosed concerning the curing conditions, *e.g*., curing temperature and duration. On the other hand, the adhesive Versilok 265/254 is a two component epoxy-modified acrylic adhesive used to bond a variety of automotive sheet metals. Glass beads are included in the adhesive system to prevent shifting of mating substrates panels. The manufacturer claims that the adhesive bonds well through various stamping lubricants and eliminate the need for advanced cleaning or surface preparation. The data sheet suggests low temperature cure conditions for getting high strength joints, while induction heating is recommended at 110°C. Here we use the curing conditions employed in the paint shop for both adhesive types and which consists in thermal heating at 180°C for 30' followed by slow curing down to room temperature (*i.e*., 25°C).

2.2. Sample fabrication and determination of fracture toughness

T-joints were fabricated and tested according to the procedures and recommendations reported in the standard ISO 11339 (2010) and ASTM 1876-08 (2015). Joints geometry and boundary conditions are reported in schematic of Fig. 1(b). FeP04 plates were cut down to 25×200 mm^2 strips from larger sheet metals. Surface degreasing was carried out as standalone surface pre-treatment in order to remove dust and contaminations due to substrate cutting and handling.

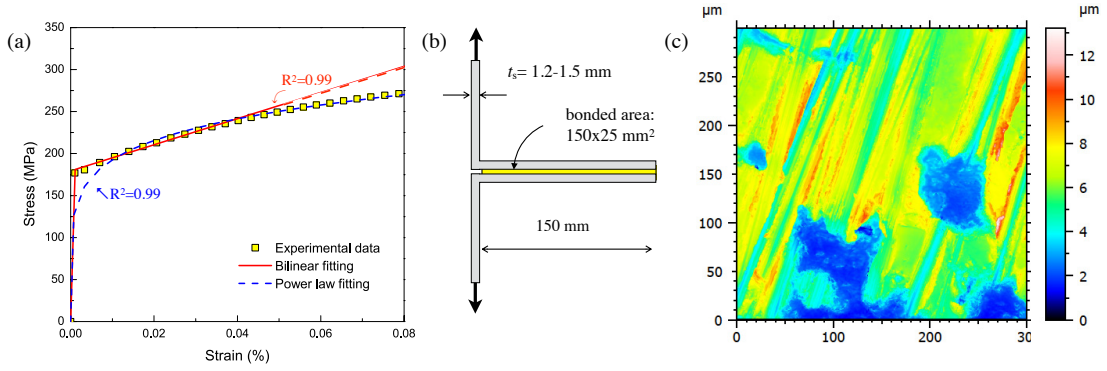

Fig. 1: (a) Tensile stress-strain curve of the FeP04 steel. (b) Schematic depiction of the T-joints employed for the determination of fracture toughness. (c) Surface height map of the as produced FeP04.

Surface analyses were carried out to ascertain the morphology of the mating substrates before bonding. A non contact 3D Optical Profiler was deployed (CCI HD Taylor-Hobson, UK) with a resolution of 340 nm on the longitudinal plane and 1 nm on the vertical axis. A typical surface scan of the FeP04 substrates is reported in Fig. 1(c). The average surface roughness was evaluated according to the ISO 25178-2:2012 and was found to be equal to (1.46±0.15) μm. Two adhesive thicknesses have been examined, that is 200 μm and 350 μm, and nylon wires were employed as spacers to ensure the consistency of the adhesive thickness. The overall bonded area was set equal to 25×150 mm^2. Substrates have been clamped before curing to prevent unwanted sliding along the overlap area that could lead to improperly fabricated joints. To confer the T-shape, the un-bonded portion was gently bent by wedge splitting after curing. Mechanical tests were carried out by using an electromechanical testing machine (MTS Criterion, model 42) and the displacement rate was set equal to 100 mm/min. The ESIS Test Protocol (2010) was used to determine the adhesive fracture toughness from the results of mechanical tests. The adhesive fracture energy was obtained from an energy balance in which the input energy to the peel test is resolved into the various contributions as follows:

$$G_c = \frac{dU_{ext}}{bda} - \frac{dU_s}{bda} - \frac{dU_{dt}}{bda} - \frac{dU_{db}}{bda} = G - W_p, \tag{1}$$

where U_{ext} is the external energy supplied by the load, U_s is the stored strain energy, U_{dt} is the elastic and/or plastic energy dissipated in tension, U_{db} is the energy dissipated through plastic bending (*i.e.*, the main contribution), G is total energy input after correction for tensile elastic and plastic deformation and W_p is the plastic work dissipated in bending. The test protocol requires given inputs such as the stress-strain response of the substrates, the average peel load recorded experimentally and sample dimensions. The spreadsheet provided by the ESIS is then used to segregate the fracture toughness of the adhesive (G_c) from the bulk plasticity coming from the steel substrates (W_p). The plastic work is determined trough an analytical formulation in which the peel tests are modelled using large-displacement beam theory with modifications for plastic bending. After that Eq. 1 is invoked to determine the fracture toughness of the adhesive. In order to perform the iterations of the method the stress-strain response can be given in bilinear form, *i.e.*,

$$\sigma = \sigma_y + \alpha E(\epsilon - \epsilon_y), \tag{2}$$

or power-law form, *i.e.*,

$$\sigma = \sigma_y \left(\frac{\epsilon}{\epsilon_y}\right)^N. \tag{3}$$

Alternatively, the stress-strain data obtained through tensile tests can be used. The toughness predicted by the three approaches was essentially similar. The results quoted herein are those obtained using the experimentally determined stress-strain response.

3. Results and discussion

The fracture toughness (G_c) obtained through the ESIS protocol is reported in Figs. 2(a) and 2(b). Focusing on the results pertaining to the single component adhesive, it is noted that the toughness is approximately equal to (1.45±0.26) kJ/m^2 and was independent of the adhesive layer thickness. Also, G_c did not display any relevant dependency on

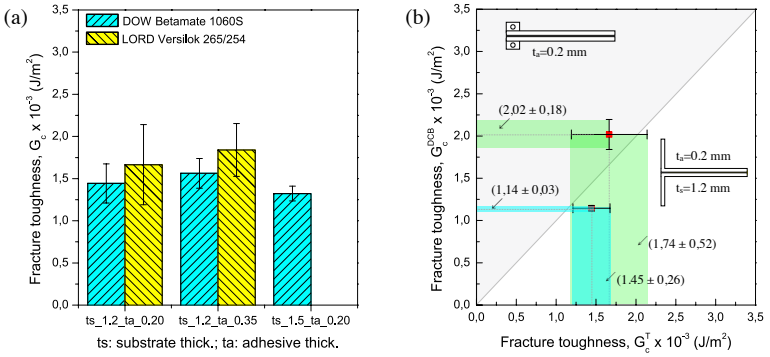

Fig. 2: (a) Fracture toughness of T-joints bonded with DOW Betamate 1060S and LORD Versilok 265/254 adhesives. (b) Comparison between the results obtained using T-joints and Double Cantilever Beam samples. (G_c^{T}: fracture toughness as determined in T-joint tests. G_c^{DCB}: fracture toughness as determined in DCB tests.)

the substrate thickness and therefore the obtained value is deemed objective.The epoxy modified acrylic adhesive displayed similar trends. However, the fracture toughness was higher and equal to (1.74±0.52) kJ/m^2. The results have been compared with those obtained on the very same kind of adhesives using a different test geometry, *i.e.* the Double Cantilever Beam (DCB). In particular, DCB tests were carried out according to the procedures and the recommendations reported in ASTM Standard D3433 (2012). A fairly good correlation between the two sets of results can be observed in Fig. 2(b), therefore it is concluded that the so obtained fracture toughness is quite consistent and independent of the test geometry. The fracture surfaces of the samples were analyzed using optical microscopy and are reported in Fig. 3.

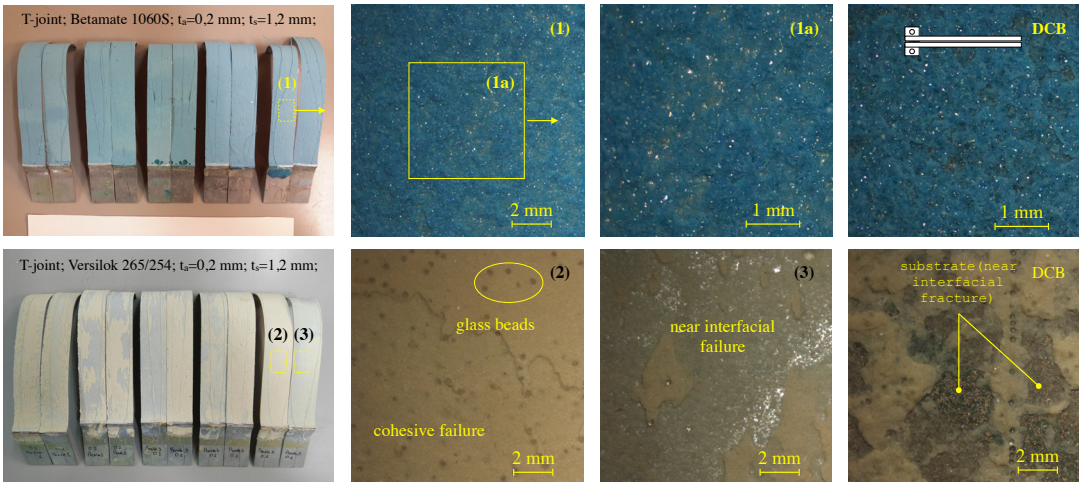

Fig. 3: Post-failure visual inspection and optical microscopy images of fracture surfaces.

It is apparent that, for both adhesives, failure of T-joints was essentially cohesive within the adhesive layer (*i.e.*, cohesive failure). Occasionally most of the adhesive remained on one of the mating substrates, especially for the

epoxy-modified acrylic adhesive as shown in Fig. 3. The fracture surfaces of the DCB joints displayed cohesive failure, quite similarly to T-joints.

4. Conclusions

In this work the fracture toughness of structural adhesives typically used in the automotive industry was evaluated. Sample curing was carried out following the conditions dictated by the automotive manufacturing chain in terms of both surface pre-treatment and curing cycles. The data obtained in mechanical tests were processed using the ESIS Test Protocol (2010) and the results were compared with the fracture toughness determined using the DCB test coupon. The comparison indicated a fairly good agreement and the fracture surfaces, which were assessed by visual inspection and optical microscopy, did not show significant differences between DCB and T-Peel joints. Moreover, no significant dependence of the fracture toughness upon the adhesive layer thickness was observed in the investigated range. High resolution imaging will be carried out in the follow-up work to unravel the mechanisms of failure at a lower scale. In addition, the effect of advanced surface pre-treatment will be also explored.

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References

Chiodo, G., Alfano, M., Pini, S., Pirondi, A., Furgiuele, F., Groppetti, R., 2015. On the effect of pulsed laser ablation on shear strength and mode I fracture toughness of Al/epoxy adhesive joints. Journal of Adhesion Science and Technology 29 (17), 1820-1830.

D'Aiuto, F., 2016. Innovative materials and solutions for automotive components, CRF-GML-Metals Department, ANFIA 2016.

Da Silva, L.F.M., Dillard, D.A., Adams, R.D., Blackman, B., 2012. Testing Adhesive Joints, Best Practices. Wiley-VCH.

European Parliament, Reducing CO_2 emissions from passenger cars, Regulation (EU) No 333/2014 (Climate Action), March 2014

Moore, D.R., Williams, J. G., 2010. A protocol for the determination of the adhesive fracture toughness of flexible laminates by peel testing: fixed arm and T-peel methods. ESIS Protocol, 2010.

Rotella, G., Alfano, M., Candamano, S., 2015. Surface modification of Ti6Al4V alloy by pulsed Yb-laser irradiation for enhanced adhesive bonding. CIRP Annals - Manufacturing Technology 64 (1), 527-530.

Standard Test Method for Peel Resistance of Adhesives (T-Peel Test), ASTM D1876-08(2015)e1.

Adhesives T-peel test for flexible-to-flexible bond assemblies (german version) - EN ISO 11339:2010.

Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Metal Joints. D3433-99 (Reapproved 2012).