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Characterization of the effect of an epoxy adhesive in hybrid FSW-bonding aluminium-steel joints for naval application

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ABSTRACT

This study focused on the characterization of an epoxy resin-based adhesive used in the hybrid friction stir welding-adhesive bonding (FSW-AB) technology which involves the use of polymeric adhesives to join aluminium and steel elements giving significant advantages as well as uniform distribution of bond-joint stress, protection from corrosion, and reduced noise and vibration. Thermo-analytical techniques such as differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were applied to study the curing rate of the structural epoxy adhesive. Infrared (IR) spectroscopy was used to examine the material before and after cure, scanning electron microscopy (SEM) to perform for the morphological analysis and microcalorimetry to evaluate the fire resistance properties. The mechanical performance of metal joints bonded by epoxy resin were studied as well. The joint strength was determined by utilizing single-lap shear test. The experimental results showed that single lap joints (SLJs) in the presence of the epoxy adhesive increased shear strength compared to SLJs of neat metals.

1. Introduction

The use of a hybrid joining technology, which involves both welding and adhesive bonding processes, intends to maximize the advantages of both while minimizing their drawbacks [1].

In specific we will make use of friction stir welding (FSW) technique, firstly developed at The Welding Institute (TWI, Cambridge, UK), since it is an efficient and versatile solid-state (or semi-solid) joining process that can take place at temperatures lower than the materials melting points [2].

This aspect makes possible to produce welded joints of metal alloys, difficult to weld with conventional techniques, that have superior mechanical properties, low distortions, good surface finish and high automation potential. FSW is an environmentally friendly process due to advantages such as low material waste and lack of radiation and/or harmful gas emissions that are normally associated to fusion welding processes [3–6].

The FSW technology uses a rotating tool, constituted of a pin/probe and shoulder, which is pressed onto the edges of the work pieces that are to be joined, generating a heat that allows the material to undergo plastic deformation and consequently to transfer the tool along the welding line, creating a weld seam. This tool, pin plus shoulder, has two main functions: (i) heating the work piece and (ii) creating a material flow that will result in a joint [2]. Problems arise for welding in lap-joint configuration, because, in this case, the sheets to be welded overlap and, therefore, the stirred material must be moved from top to bottom and no longer from right to left (or vice versa) [7,8]. The overcome of employing a limited mixing zone in the plane separating the sheets causes the formation of a hook shape. This zone can be also a trigger for the propagation of cracks causing localized corrosion phenomena and additionally affecting the static strength of the welded seam and limits its resistance to fatigue stress [9,10].

Adhesive bonding (AB), another joining technique for solid materials that has been proven to be efficient, consists in joining materials placing an adhesive, in liquid or semi-liquid form, between two work pieces, with following solidification, resulting in a strong joint. There are several advantages in using the adhesive-bonded joints compared to traditional joining technology, like the reduction of distortions, especially in the junction of very thin and precision parts, the possibility of manufacturing heterogeneous joints, the non-need of holes, screws and

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Table 1
Chemical composition (%) of Aluminium Alloy AA 5083 H321 and Steel 355J2.

Material	Fe	Al	C	Mn	P	S
AA 5083 H321	0.33	bal.	–	0.55	–	–
S355J2	bal.	0.034	0.17	1.18	0.012	0.004
	Cu	**Mg**	**Si**	**N**	**Ti**	**Zn**
AA 5083 H321	0.07	4.79	0.13	–	0.01	0.13
S355J2	0.01	–	0.01	–	–	–

marks of welding and the reduction of weight. Like any other process, AB presents also some disadvantages as low resistance to low and high temperatures, short working and shelf life, low resistance to peeling strength and the necessity of preparations of surfaces [11,12].

The use of hybrid joining technology, which combines AB and FSW, (from now on FSW-AB), appears very good to retain the advantages and overcome the problems of each technique. It is an optimal candidate for joining stainless steel and other similar materials which is a complex process due to the high melting points and poor joint properties associated with them, moreover its low resistance to loads creates shear stresses and affects the fatigue life.

The addition of adhesive system improves the fatigue life of the joint by damping vibrations; moreover, the adhesive could also work as a sealant of the joint solving the corrosion associated to FSW [13].

The interest in hybrid technique is becoming an emerging subject in the welding field as testified by the presence of several works as weld-bonded, rivets and bolts with adhesive, laser weld bonding and even friction stir spot welding (FSSW) with adhesive [14–17].

Its development present different challenges and more in details:

- the process temperature range is between 300 °C and 470 °C [18];
- the chemical nature of the polymeric adhesives used is relevant, in particular with reference to their thermal resistance (during the process) and chemical resistance (during the use in corrosion environment);
- the avoidance as much as possible of solvents, or non-environmentally friendly additives, in order to reduce the environmental impact of the process.

Taking into consideration the factors above, it is mandatory the use of suitable polymeric adhesives with high thermal resistance, good wetting and flow characteristics in order to obtain a good quality bond of the metal surfaces. The best candidates for this purpose appear to be epoxy-based adhesives (mono- or bi-component) [19,20] and polyimide-based adhesives [20] (already in use in the automotive and aerospace industries).

Exhaustive investigations on the mechanical behavior of hybrid FSW-AB joints considering both static and dynamic behavior are still lacking. Static strength and distortion levels of the manufactured hybrid joints were assessed and compared to FSW-only and AB-only joints by Braga et al. [21]. The results established that hybrid FSW-AB produced higher strength and more ductile lap joints than FSW lap joints, although not as strong or ductile as adhesive-bonded joints. In another study, Fortunato et al. [22] found the application of ultrasonic testing to friction stir weld-bonded joint inspection a successful method to evaluate joint quality.

In the present study, in order to clarify the mechanism involved in FSW-AB, we investigated its use in the assembly of aluminium and steel elements.

Differential Scanning Calorimetry (DSC), Thermogravimetric analysis (TGA) and Fourier-transform Infrared Spectroscopy (FTI-IR) were used to study and characterize the curing rate and the mechanical behaviour of the adhesive. Mechanical tests were also employed to study the mechanical resistance of the system in exam.

Table 2
Mechanical properties of Aluminium Alloy AA 5083 H321 and Steel 355J2.

Material	Yield Strength [MPa]	Tensile Strength [MPa]	Elongation at break [%]
AA 5083 H321	271.3	362.5	16.4
S355J2	454	543	27.3

Table 3
Mechanical properties of Betamate™ 1496S [from producer data sheet].

Adhesive	E Modulus [MPa]	Tensile Strength [MPa]	Elongation at Break [%]	Lap Shear Strength [MPa]
Betamate™ 1496S	1450	30	13	28

2. Experimental

2.1. Materials

The substrates used were plates made from AA 5083 H321 Aluminium alloy (melting point range 591–638 °C, manufacturer Aviometal) and S355J2 steel (melting temperature liquidus 1460 °C, manufacturer Rolla, Traverso & Storace S.p.A.). The nominal composition of both alloys is reported in Table 1 (obtained from the material certificate according to UNI EN 573-3 for Aluminium and UNI EN 10025-2 for steel) and the mechanical properties are presented in Table 1 (according to UNI EN 485-2 for Aluminium and UNI EN 10025-2 for steel) (see Table 2).

The adhesive chosen for the FSW-AB experiments was the commercially available epoxy adhesive Betamate™ 1496S, supplied by Dow Automotive, referred as Betamate from now on. It is a mono-component structural adhesive of new generation used in the automotive industry, it combines high strength with large ductility and presents good resistance and durability. According to the manufacturers, the optimum curing regime for Betamate adhesive is about 30 min at 180 °C. The mechanical properties of this adhesive are presented in Table 3.

2.1.1. Epoxy adhesive characterization

The chemical structure of the epoxy resin-based adhesive was analyzed before and after the curing reaction (conducted in an oven at 160 °C for 1 h) by using a PerkinElmer Spectrum Two™ FTIR-ATR spectrometer recording the absorbance spectra in the range 4000–400 cm^{-1}.

The study of the curing reaction of Betamate sample was performed with a DSC 821^e Mettler Toledo calorimeter. For the preliminary DSC studies the sample was heated (cured) from room temperature up to 250 °C with a heating rate of 10 °C/min under a N_2 flow rate of 40 mL/min. For the determination of the initial T_g, the uncured sample was cooled at a rate of 40 °C/min from room temperature to −120 °C, kept at this temperature for 3 min, then heated to room temperature at 5 °C/min (all analyses performed in N_2 flux). Finally, the material was cured in an oven at 180 °C for 30 min and subsequently thermally characterized with DSC.

The epoxy resin was subjected to microcalorimetric combustion analysis to determine its fire resistance using a Fire Testing Technology FAA microcalorimeter. The Total Heat Release (THR), Heat Release Capacity (HRC), and Peak Heat Release Rate (PHRR) were recorded. The measurements were carried out heating the sample from 150 up to 750 °C at 1 °C/min under N_2 flux. During pyrolysis, the volatilized decomposition products were transferred in a stream of N_2 to a high-temperature (900 °C) combustion furnace where pure O2 was added and the decomposition products completely combusted.

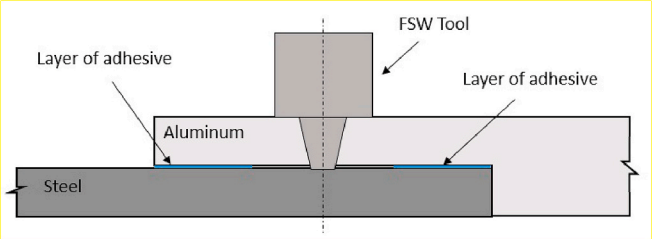


Fig. 1. Schematic of SLJ plus FSW tool based on [2].

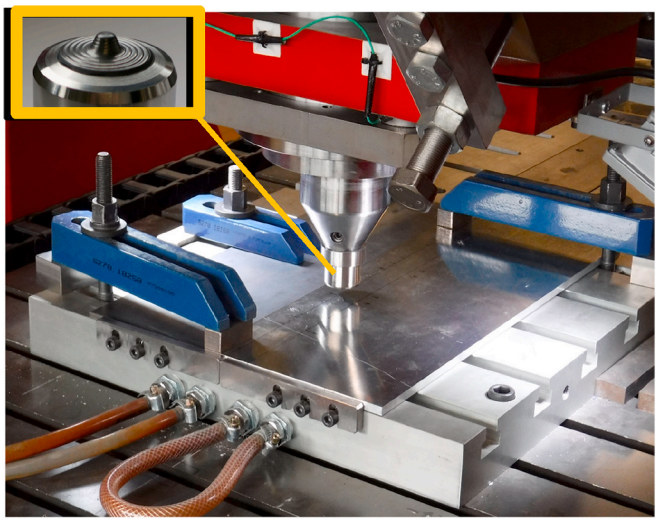


Fig. 2. FSW gantry machine and inset of the welding tool.

2.2. Joint manufacture

The friction stir welds were produced by using a gantry machine (H. Loitz – Robotik). Welding was performed under vertical downward force control. The best results were obtained with force control values comprise between 21 and 30 kN and with feed speed in a range of 1.7 ÷ 1.9 mm/s. The optimal welding parameters were assessed checking the quality of the joints through metallographic analysis and non-destructive tests (i.e. visual and radiographic tests). These parameters were subsequently optimized in order to realize hybrid joints with the pre-applied adhesive layer(s) in-between the interface of the two metal sheets. The values of the process parameters that allow obtaining satisfactory results for FSW steel-aluminium lap joints were also adopted for hybrid technology FSW-AB.

Fig. 1 shows a schematization of the FSW-AB process. The size of the metal sheets used was 500 × 250 × 4 mm. The adhesive was previously applied on both aluminium and steel substrates, in-between the over-lapped area. The viscosity according to the technical sheet is 75 Pa s [23].

To avoid adhesive degradation due to the high temperature reached during welding, the adhesive was not applied in the area of action of the FSW tool but on the side (about 30 mm on each side, the overlap was 80 mm). Prior to bonding, the metal substrates were surface treated with acetone in order to clean the surface of contaminants, sandblasted to increase surface roughness and then treated by plasma.

The aluminium and steel sheets were then positioned in the welding jigs and fixed together. Aluminium was positioned over the steel.

After the welding process, the adhesive was cured in oven according to the thermal cycle suggested by the adhesive manufacturer (180 °C for 30 min).

The FSW tool used in this study were manufactured by MegaStir Inc. from a composite ceramic material: refractory metal designed Q70. This material contains 70% by volume of polycrystalline boron nitride in a tungsten-rhenium binder (pin lenght: 4.5 mm). The tool designed comprises of a stepped spiral probe and scroll shoulder mechanically held in a metal shank. Proper geometrical features are present on the surface of the tool to enhance the stirring effect of the tool in contact with the welded material. Fig. 2 shows a picture of the FSW gantry machine with a detail of the welding tool (inset).

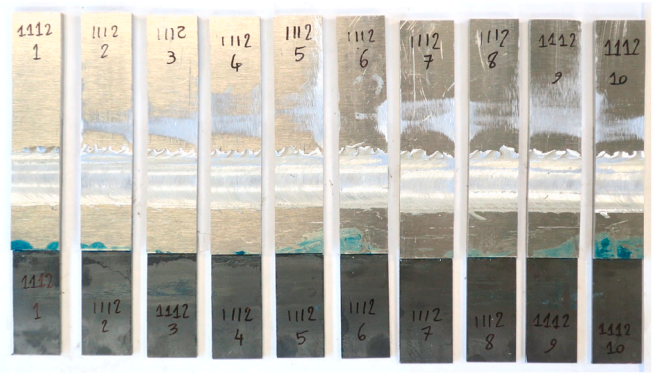
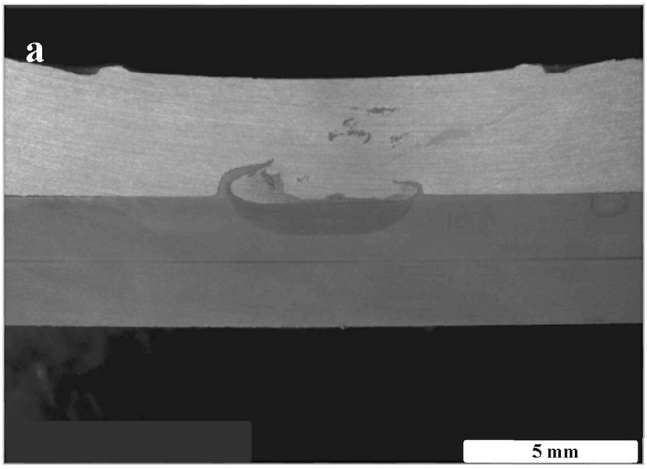


Fig. 3. Specimens for tensile tests.



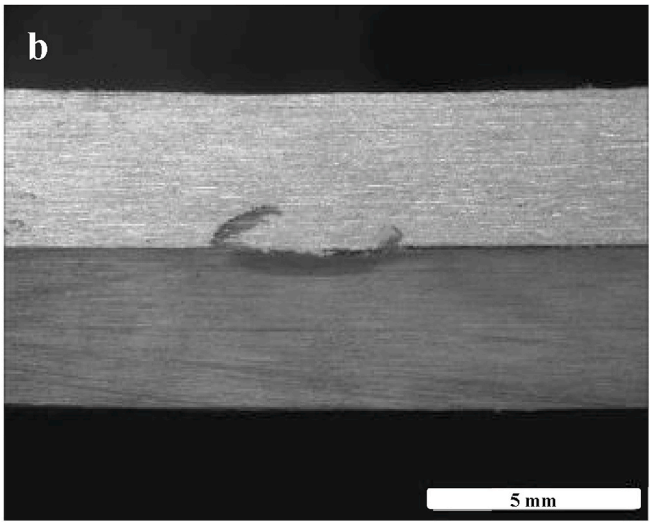


Fig. 4. Macrographic inspection of FSW joint (a) and FSW-AB joint (b).

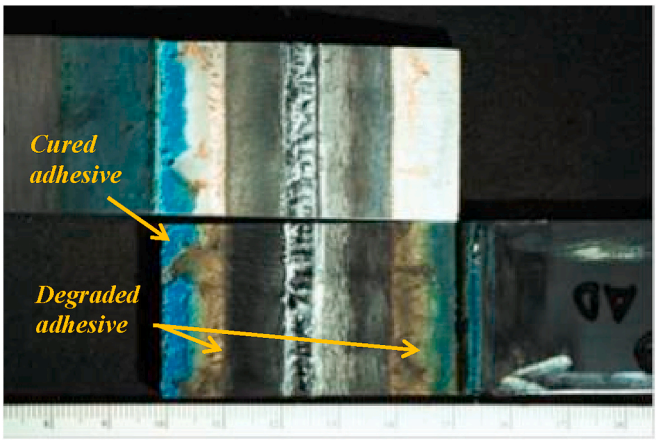


Fig. 5. Macrographic analysis of the fracture surface of an AB joint.

2.3. Macrographic analysis

Specimens for macrographic investigation were collected from the transverse section of welded and weld-bonded joints. Optical inspection was performed using a stereoscopic microscope (Zeiss Stemi 2000-C) operating at 6.5–50× magnification. The samples were cut in rectangular shape then polished and finally etched.

2.4. Mechanical characterization of welded and adhesive SLJ's

2.4.1. Single lap shear test

The single lap tests were made with a Zwick tensile test machine at a load capacity of 50 kN (according to ASTM D1002 standards [24]). Tensile tests were carried out with crosshead speed of 2 mm/min. Specimens taken from both FSW lap joints and FSW-AB lap joints were tested. Specifically, the dimensions of the specimens (Fig. 3) are as follow: length 210 mm, width 30 mm.

The thickness of the applied adhesive was between 0.1 and 0.3 mm (after curing).

3. Results and discussion

3.1. Macrographic analysis

In order to validate the weld-bonding process, the first step was to carry out a macrographic investigation of the joints. Fig. 4 presents the macrostructures of the front view of an FSW and FSW-AB joints. As reported in the literature [9,10], at a first observation the macrographic analysis revealed the presence of a typical evident hook defect inside both joints which is due to the vertical movement of the material during the FSW process and strongly reduces the resistant section to increment the adhesion and the tensile strength of the joint;

Fig. 5 displays the transverse section of the fracture surface of the AB joints where the phenomenon of chemical deterioration suffered by the adhesive during welding is clearly evident. The heat generated during the welding process affected the adhesive, which degraded in the part in-between the plates directly under the tool shoulder. Furthermore, an evident lack of mixing caused by the presence of the adhesive, which acts as an insulant and opposes the passage of heat from the upper sheet to the lower one during welding, is present. These two phenomena, the presence of the hook defect and the chemical deterioration of the adhesive, contribute to compromising the mechanical response.

3.2. Adhesive bonding (AB) joints characterization

3.2.1. FTIR-ATR analysis

ATR-FTIR spectra were obtained from the Betamate sampled before the curing reaction conducted in an oven and also on specimen collected from welded joints on the cured and degraded adhesive (see Fig. 5).

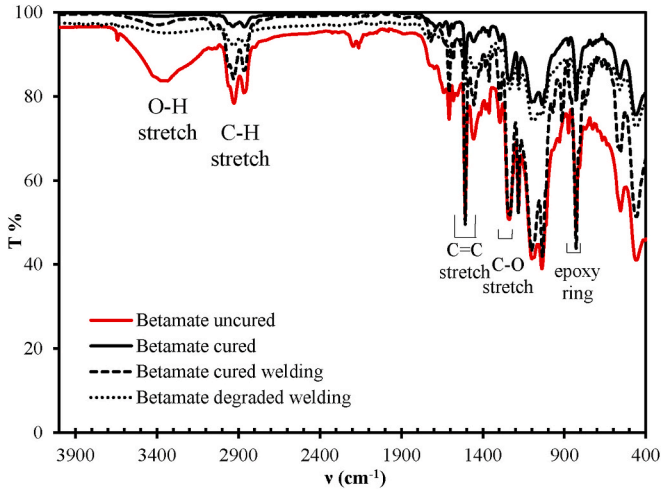


Fig. 6. ATR-FTIR spectra of uncured Betamate sample (red line) and after curing (black line) and the layer of Betamate adhesive in the AB joint. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

The sample were studied after curing them in oven in order to simulate the overall behavior of the adhesive at the suggested curing temperature (reported in the technical sheet).

Additional measurements were performed in order to study the behavior of the epoxy during the welding process, considering that in the "joint zone" there is a wide distribution of temperatures according to the distance from the tool [18].

For the uncured Betamate sample (red curve of Fig. 6), the most significant signals are assigned to the O–H stretching from 3400 to 3100 cm^{-1}, the ether C–O vibration at 1240 cm^{-1} and a band at about 850 cm^{-1} typical of the epoxy ring; after the curing reaction (black curve of Fig. 6) the band of the ether bond is still observed and the spectral features above 3100 and at 850 cm^{-1} (hydroxyl groups and epoxy ring) are still present even though with much lower intensity, as expected. The epoxy resin also shows a series of bands related to its aromatic portion (the C–H stretching from 3100 to 3000 cm^{-1}, the C=C stretching at 1590 and 1490 cm^{-1}, and the C–H bending bands of para disubstituted benzene derivatives at 830 cm^{-1}) due to the presence of

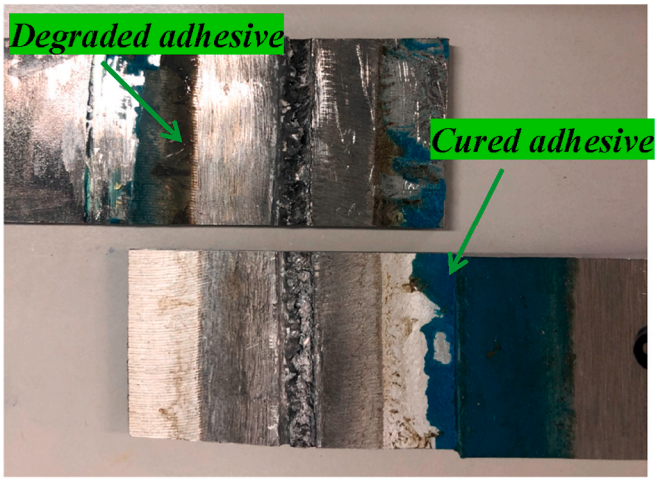


Fig. 7. Image of the FSW-AB joint. The internal welding zone is distinguished from the external one due to the presence of a thin layer of degraded Betamate adhesive.

Table 4
DSC data of the Betamate adhesive.

Sample	T_g (°C)	T_C (°C)	ΔH_{TOT} (kJ/mol)	$\Delta H_{TOT\ nor}$ (kJ/mol)
Betamate	−29	179	194	237

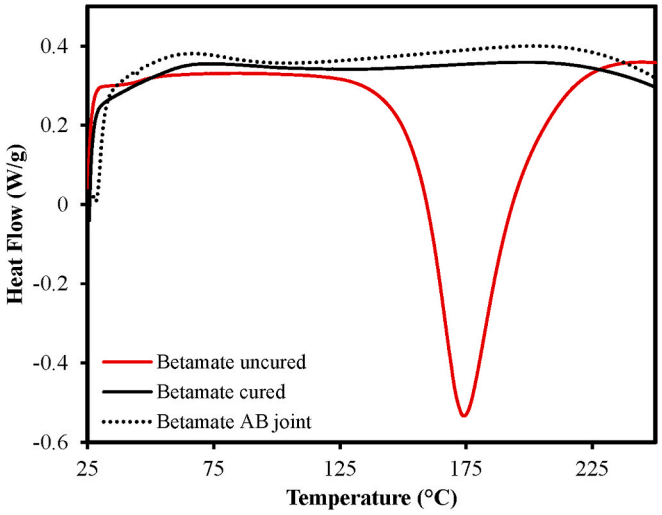


Fig. 8. DSC curves of the Betamate uncured and respective curves of the sample during and after curing.

bisphenol A moieties in the formulation. Fig. 6 also shows the IR spectra of the Betamate adhesive taken directly from the less degraded (dotted black) and more degraded (grey curve) part the adhesive bonded joint that were respectively more distant and closer to the zone were the process of FSW took place.

Both cured welding and degraded welding present a higher transmittance in the O–H region in comparison with the uncured epoxy that confirms the curing of the adhesive during the welding.

It also to notice that the cured welding presents a lower transmittance in the epoxy ring zone that suggests the difficulties in obtaining a uniform cured zone under the welding process as already reported above. This is confirmed also by the lower value of the transmittance for the frequencies near 950-1050 cm^{-1} ascribed both to the bending of C═C and eventually to the presence of CO_2 or other anhydrides generally added to these kinds of resins as curing agents. The uncured sample spectra presents also the characteristic peaks of CO_2 that are located at 2300 cm^{-1} corresponding to the asymmetric stretching mode of CO_2 and small peaks in the range 3600–3800 cm^{-1} corresponding to combination bands [25].

The image of the FSW-AB joint provided in Fig. 7 shows the internal and external area of the weld from which part of the Betamate sample is removed for performing FTIR-ATR analysis.

3.2.2. DSC analysis

Betamate was thermally studied by means of DSC and in particular its glass transition temperature, Tg, which indicates the passage between glassy and rubbery state, and its temperature of curing, T_C as reported in Table 4.

Specimens were sampled from the joint taking care of selecting a non-degraded zone as previously determined by the FTIR analysis.

Fig. 8 shows the DSC curves of the Betamate untreated in heating from the low temperatures. The values of T_g of the Betamate™ 1496S uncured is equal to −29 °C.

Fig. 8 presents the DSC thermograms of the Betamate on heating up to 250 °C, during which the polymerization reaction (curing) takes place (curves in red, T_c 179 °C), and the subsequent heating of the cured samples (curves in red). As can be seen from the red curves profile of the sample in Fig. 8, the polymerization reaction in practice takes place completely during the first heating.

The DSC curve (grey dotted line) related to the sample withdrawn from AB joint shows that upon the FSW-AB process the adhesive curing is completed, as confirmed by its profile which is almost identical to the Betamate cured in oven. The parameter of ΔH_{TOT} and $\Delta H_{TOT\ nor}$ are reported in Table 4.

Table 5
Flammability results and relative peak temperatures for Betamate sample.

Sample	HRC (J/g·K)	Peak HRR (W/g)	THR (kJ/g)	T_{peak} (°C)
Betamate	265.0 ± 2.7	263.8 ± 3.0	21.08 ± 0.10	407.6 ± 3.3

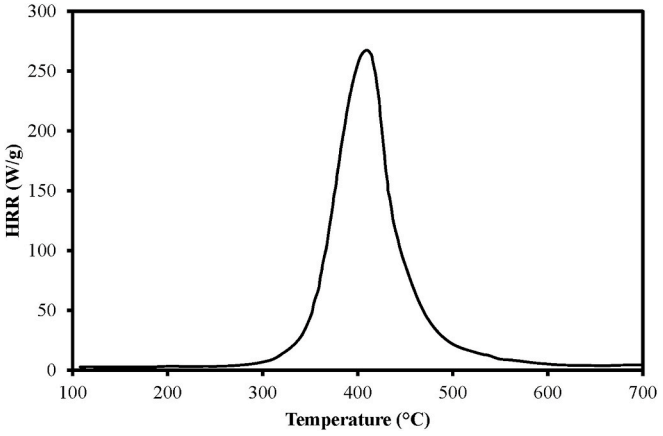


Fig. 9. Heat release rate measured for Betamate adhesive.

Table 6
Values of tensile strength (kN) and displacement (mm) for FSW joints and FSW-AB joints.

Specimen	Tensile Strength (kN)	Displacement (mm)
FSW-AB_1	17.1	0.53
FSW-AB_2	23.2	0.46
FSW-AB_3	21.0	0.35
FSW_1	12.2	0.38
FSW_2	13.6	0.41
FSW_3	13.0	0.36

3.2.3. Microcalorimetric combustion analysis

A pyrolysis-combustion flow microcalorimeter was used as test to study fire behavior of the Betamate adhesive. The microcalorimetric data of the materials analyzed in terms of mean value and standard deviation are summarized in Table 5. Four tests were carried out for Betamate sample.

The heat release curves of the Betamate resin is shown in Fig. 9.

From the results shown in Table 5 and from the thermograms of Fig. 9 it can be observed that Betamate sample shows a single degradation peak centered at about 410 °C.

From microcalorimetric combustion analysis it can be observed that the HRC, HRR and THR values related to the mass unit, even if we consider a normalization of these values which takes into account the organic portion of the sample (about 80% for Betamate) are elevated. The Betamate sample quickly releases a large amount of combustion heat. This result is therefore unfavorable for the properties of fire resistance, although in any case, as already noted in other analysis, in its favor the Betamate begins to decomposition at higher temperatures of about 500 °C. The configuration tested (which was already optimized after preliminary test to obtain an optimal joint process) indicates that at least 30% of the adhesive undergoes to a degradation.

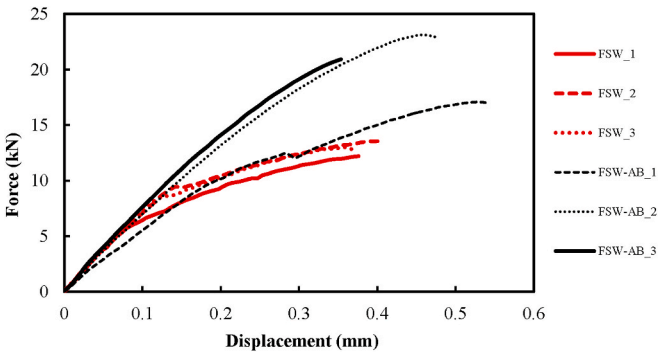


Fig. 10. Force-Displacement plot for FSW and FSW-AB joints.

3.3. Single lap-shear tests

The single lap shear test of the joints was performed in order to know their strength and predict their behavior under extension loads. Table 6 compares the tensile strength values of the FSW and FSW-AB joints and report the displacement values (each series of measurement was repeated on at least 3 samples). The samples obtained by the FSW (without adhesive) with conditions of 800 rpm and a welding speed of 1.7 mm/s show an average lap strength of 13±1 kN. The analogous system with the contribute of the adhesive shows a significant difference in the average lap strength with a value of 20 ± 3 kN (56% average increase). The values of displacement (at maximum load) for the FSW are 0.38 ± 0.02 mm while for the FSW-AB we have 0.45 ± 0.10 mm (33% average increase). Both results confirm the contribute to the mechanical properties of the added epoxy. Fig. 10 reports the plot for the load displacement curves obtained for the condition for the FSW and FSW-AB.

4. Conclusions

In this work we presented the effect of adding an epoxy adhesive during the process of Friction stir welding, in order to assess its contribute to the efficient joining of aluminium and steel elements. The results of the experiments performed showed that:

- Optical microscopy imaging confirmed the completed curing of the adhesive in the FSW-AB process and its partial degradation localized in the internal zone of the joint;
- FTIR spectroscopy highlighted that the process of FSW developed enough heat to make the curing process of the added epoxy resin possible with a not uniform distribution in the welding area showing the presence of degraded and cured adhesive zones in dependence of the distance between the tool and the epoxy.
- Calorimetric analysis confirmed the results obtained by FTIR and OM about the curing and degradation process that the adhesive undergoes during FSW-AB.
- Pyrolysis-combustion analysis confirmed that the cured adhesive begins to decompose at temperatures higher than 500 °C.
- The mechanical properties investigated (tensile strength and displacement) show increased values for the samples obtained via FSW-AB techniques thanks to the combined effect of the FSW and the presence of the adhesive.
- Notwithstanding this, the effective contribution of the “preserved” (the non-degraded) adhesive was confirmed by the lap shear test results.

All the results presented confirm that the effect of the addition of the adhesive gives a positive contribution to the application of this technique and of the subsequent modifications designed to increase the performance of the hybrid system.

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