ULTRASONIC JOINING OF THROUGH-THE-THICKNESS REINFORCED TI-4AL-6V AND POLYETHERIMIDE HYBRID JOINTS

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Abstract

Ultrasonic joining is an alternative direct-assembly joining technology to produce through-the-thickness reinforced hybrid joints between surface-structured metals and unreinforced or fiber-reinforced thermoplastics. As a result, joint damage tolerance can be improved. This paper presents a preliminary evaluation on the influence of joining energy on the joint formation, microstructure and mechanical performance of Ti-6Al-4V-Polyetherimide hybrid joints. Process-related microstructural changes and mechanical performance of optimized were assessed. The ultimate lap shear force of hybrid joints was six times higher (1860 ± 260 N) than the non-reinforced reference joints (292 ± 7 N). A considerable increase of ten times in displacement at break for ultrasonic joints was also achieved in comparison to reference joints. This is an indication that joint damage tolerance was increased due to an efficient load transfer by pin interlocking between the metal and polymer parts. Initial joint failure was by bearing – a non-catastrophic failure type – while shearing of the metallic pins was responsible for the final parts’ separation during lap shear testing.

Introduction

To enable the manufacture of fuel-efficient and low-emission vehicles, designers and engineers are increasingly compelled to select lighter and high performance materials. The substitution of conventional steels or cast iron by lighter hybrid materials, such as lightweight alloys and high-performance or fiber-reinforced polymers, is an immediate solution to optimize specific strength of transport systems [1–3].

Although promising, the transition from similar to hybrid lightweight structures usually requires new joining concepts, since the traditional welding and joining techniques are not directly transferrable to such material combination. This is mostly due to their physical and chemical material dissimilarities, which hinder or limit parts’ miscibility during joining [4, 5]. Consequently, the joining of hybrid materials is usually accomplished by a third element, such as adhesives or fasteners. However, these joining approaches normally present several drawbacks. The stress concentration caused by the through hole in bolted and riveted joints, the limited load transfer capability and long curing times of adhesively bonded joints are examples of these limitations.

In order to overcome these issues, novel direct-assembly concepts have been proposed. These new joining methods seek to produce high-performance metal-composite hybrid joints with higher out-of-plane strength by incorporating through-the-thickness reinforcements [6]. Therefore, damage tolerant hybrid structures can be produced by improving load-transfer efficiency.

The manufacture of hybrid structures by direct-assembly can be divided into two stages: 1) surface structuring and 2) assembly method. During the surface structuring phase, reinforcement elements – e.g. pins and columns - are integrated in the surface of the metallic part. Examples of surface structuring techniques are additive layer manufacturing (ALM) [7], Surfi-Sculpt™ (electron and laser beam structuring) [8,9] Cold-Metal Transfer (CMT) [10] and metal injection molding (MIMStruct) [11]. In Stage 2 (Assembly), the composite is either assembled layer by layer onto the structured metallic surface, or for instance by co-curing or vacuum infusion. All these concepts are still in their exploratory development phases. Therefore, one might expect a time-consuming assembling phase due to the resin cure or consolidation (the later in case of thermoplastic composites).

More recently, an innovative assembly methodology based on ultrasonic energy - the ultrasonic joining, U-Joining- has been introduced [12] as an alternative solution to assemble surface-structured metallic parts with polymers or composites. This new assembly approach considerably shortens the assembling phase, since the joints are manufactured usually in less than 2 seconds [12].
In this work, the U-Joining process was applied to produce hybrid joints between surface structured Ti-6Al-4V and polyetherimide (PEI), those being lightweight materials of particular interest for the aircraft industry. The influence of joining energy on joint formation was systemically evaluated by a one-factor-at-time parameter study. Additionally, selected microstructural characteristics, local (microhardness testing) and global (lap shear testing) quasi-static mechanical properties, as well as failure mechanisms were addressed to explain the correlation between joining process, microstructure and mechanical performance for the case-study hybrid joints.

**Ultrasonic joining**

Ultrasonic joining (U-Joining) is a new joining technique developed by Helmholtz Zentrum Geesthacht (patent application EP 3 078 480 A1 [13]) to produce damage-tolerant and crash-resistant metal-polymer (or composite) hybrid joints. This new direct-assembly concept joins a pre-structured metallic part with unreinforced or fiber-reinforced thermoplastics by ultrasonic energy. As a result, through-the-thickness reinforced hybrid joints with improved out-of-plane strength are obtained [12].

The joining process can be schematically divided into five steps, as shown in Fig. 1 for the metal (horizontal motion) ultrasonic joining variant [12]. Initially, the parts are fixed between the anvil and sonotrode, whereby the reinforcement elements of the metallic part are placed into contact with the polymeric part’s surface (Fig 1-1). In the second phase (Fig 1-2), the sonotrode approaches the metallic part`s surface and applies the clamping pressure. At this time, ultrasonic vibration is started in a back-and-forth motion. As the upper part of the joint has close contact with the sonotrode, ultrasonic vibration is transmitted to the interface between the metallic reinforcement and the polymer, where frictional heat is created. Due to the combination of high vibration frequency and pressure at this interface, the temperature locally increases and softens a small volume of the polymeric part. Consequently, the metal reinforcements start to be inserted into the polymer (Fig 1-3). While the reinforcement penetrates in the polymeric part, compressed molten/softened polymer is expelled outside the joint area, wetting the metal part’s surface. After complete penetration of the reinforcement elements, the sonotrode vibration is stopped and the cooling phase starts (Fig 1-4). During this phase, pressure may be applied in order to compensate polymer shrinkage and consolidated the joint. Finally, the sonotrode is retracted (Fig 1-5) from the metal-polymer hybrid joint.

**Materials and methods**

The 15.5 x 35 x 3 mm metallic parts (Fig. 2a) were produced with Ti-6Al-4V alloy by a modified metal injection molding structuring process (Helmholtz-Zentrum Geesthacht, patent EP 2 468 436 B1 [11]). The MIM-structured parts have six 3 mm high round-tip conical pins (Fig. 2b). The fabrication of the metallic parts was previously discussed into details in [12]. Parts present a typical Widmanstätten microstructure [14] with lamellar colonies of alpha-phase (grey) and beta-phase (white) (Fig. 3). MIM-structured parts had an intrinsic residual porosity of 2.0 ± 0.6%.

The polymeric parts consisted of extruded polyetherimide plates (PEI 1000 Arthur Krüger, GmbH, Germany). The extruded plates were cut to create joining
samples of 15.5 x 35 x 6.8 mm (Fig 2a). A 15.5 x 21 mm rectangular overlap area was set for the hybrid joints.

Joining was performed using a commercially available metal ultrasonic welding system (Ultraweld L20, Branson Ultrasonics). The joining equipment operates with a fixed sonotrode oscillation frequency of 20 kHz and joining power can be increased up to 4 kW. When the ultrasonic system is controlled by the so-called energy mode - i.e. the sonotrode vibration is kept constant until a pre-set energy value is delivered - the following joining parameters can be adjusted: joining energy (E_J), clamping and joining pressure (C_P and P, respectively), and sonotrode oscillation amplitude (A_0). Under this control mode, the joining system will operate in a close-loop. The joining cycle will be automatically adjusted to deliver the pre-set value of energy (E_J) according to Equation 1[15],

\[ E_J = P \times t \]  

where \( P \) is the power and \( t \) is the joining cycle time. The joining system integrates the used power in intervals of 5 milliseconds and adjusts the energy delivered. Power is a function of force and velocity. Force is determined by multiplying the surface area of the cylinder by the sonotrode manometric pressure (i.e. the joining pressure, P), while velocity is derived from the frequency and amplitude (A_0)[15].

In this work, an exploratory experimental study based on a one-factor-at-time design of experiments (OFAT) was chosen to analyze the \( E_J \) influence on the joint formation, pins’ penetration, \( \Delta d \), (obtained from the sonotrode displacement) and joining cycle (t). Table 1 presents the set of joining parameters and the responses investigated in this work. Three levels of \( E_J \) were tested 1800, 2000 and 2200 J and three replicates for each energy level were produced. Based on preliminary studies [12], joining pressure and amplitude were set constant at 15 psi and 42 µm, respectively.

Table 1. Joining parameters and investigated responses.

<table>
<thead>
<tr>
<th>Joint condition</th>
<th>Joining parameters</th>
<th>Responses</th>
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<tr>
<td></td>
<td>( E_J ) [J]</td>
<td>( A_0 ) [µm]</td>
</tr>
<tr>
<td>J1</td>
<td>1800</td>
<td>42</td>
</tr>
<tr>
<td>J2</td>
<td>2000</td>
<td></td>
</tr>
<tr>
<td>J3</td>
<td>2200</td>
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The joint produced with the best set of OFAT joining parameters was further characterized in terms of process temperature development, microstructural features and quasi-static mechanical performance. The temperature during the U-Joining was evaluated at the interface between pins and PEI, as schematically shown in Fig 4. A high-speed infrared thermo-camera (model ImageIR® 8300 hp, Infratec, Germany) was used to assess the process temperature.

The microstructure and fracture surface of the joints were evaluated by digital photography (macro-scale) and scanning electron microscopy, SEM (micro-scale), (Quanta™ FEG 650, USA). The SEM analyses were carried out with a voltage of 10 kV and spot size of 2.5 and work distances of approximately 10 mm.

Process-related changes in local mechanical properties were evaluated by Vickers microhardness. The used equipment was a ZHV microindenter from Zwick Roell, Germany. Tests were performed in accordance with ASTM E384-10 on materialography polished specimens using an indentation load of 0.495 N and holding time of 15 s. A microhardness map containing six horizontal lines with 48 indentation points were obtained, resulting in a measurement area of 3.0 x 9.5 mm

Prior to mechanical testing, non-destructive micro-computed tomography was selected to assess the pin integrity after U-Joining. The equipment used was a Y. Cougar from XYLON International (Germany), and the 3D models were reconstructed and analyzed using the software VGStudio 30. A customized lap shear test specimen was used to evaluate the joint mechanical strength (Fig. 2a). The tests were performed using a universal mechanical testing machine model 1478, Zwick Roell, Germany. Three replicates of the selected best joining condition were tested at room temperature with a traverse speed of 2 mm/min.

**Results and discussion**

Fig. 5 presents the effect of joining energy on the joint formation, evaluated by a cross-sectional cut at the joining area (Fig. 5a), sonotrode displacement – a quantitative response for the pins’ penetration (Fig. 5b) - and joining cycle (Fig. 5c). Considering that U-Joining was controlled by energy in this work, a certain threshold of energy is expected to ensure the complete penetration of the through-the-thickness reinforcement (pins) in the polymeric part. According to the results in Fig. 5a and Fig. 5b the energy required to ensure the complete pins...
penetration was determined to be 2000 J, when keeping the sonotrode amplitude and pressure at 42 µm and 15 psi, respectively. Therefore, it is expected that further increases in EJ will not produce any gain in joint mechanical properties. Indeed, it is well known from other friction-based joining process that increases in energy or heat input, usually lead to higher process temperatures in PEI-Aluminum hybrid joints [16], which may exceed the onset of thermal degradation leading to defective joints [17]. Joints with volumetric flaws in the polymer part usually have lower quasi-static strengths [18]. Therefore, one may expect that the increase of 200J for joining condition J3 may induce higher levels of thermal degradation near the inserted pins, without additionally increasing pins’ penetration (see Fig. 5b). Detailed studies adopting more complex design of experiments approaches are being performed to better understand these correlations and support this assumption for U-Joining.

One of the main advantages of the U-Joining for the assembly of through-the-thickness-reinforced hybrid joints is its very short joining cycles. The full penetration of the pins was achieved within very short joining cycles (e.g. 1.26 ± 0.04 s for condition J2, Table 1). Moreover, the increase in EJ resulted in a slight increase in the joining cycle (Fig 5c), since the sonotrode has to keep its vibration for longer times to deliver the higher pre-set levels of EJ.

Figure 5. Effect of joining energy on the joint formation (a), sonotrode displacement (b) and joining cycle (c).

Based on the previous discussion, the joining condition J2 was selected for further characterization, considering that it resulted in the full penetration of the pins (the highest sonotrode displacement) with the smallest energy amongst the three investigated joining conditions. The maximum temperature measured by infrared thermography for this condition was 362 ± 32°C. However, the cut-off point for the measurement was defined by the complete insertion of pins (i.e. when the surface of the metal part touches the polymeric part). As a result, the recorded temperature value may not refer to the maximum temperature reached during U-joining. Nonetheless, this temperature level is already inside of the degradation temperature range of polyetherimide, which occurs in two stages [17,19]: crosslinking from 320 to 380 °C and chain scission above 400 °C.

Despite the fact that the degradation temperature was achieved during joining, one should take into consideration that the joining cycles were very short (1.26 ± 0.04 s), as previously given in Table 1. Therefore, an extensive degradation of the polymeric part is not expected. Moreover, the temperature developed during the U-Joining is restricted to small volumes along the interface between tip of the pins and polymer. In this way, only a small volume of process-affected polymer will be formed.

Fig. 6 presents the results of the micro-CT evaluation for a J2 joint replicate. The side-view (Fig. 6a) and top-view (Fig. 6b) 2D X-ray tomograms indicate that the pins’ integrity was preserved after joining. Furthermore, complete pin penetration could be accomplished confirming the results from the sonotrode displacement (Fig. 5b). Besides, the detailed 3D-reconstruction of the Ti-6Al-4V part (Fig. 6c) confirmed that no pin damage or deflection by plastic deformation was induced during pin plunging. This may imply that the molten polymer viscosity was low enough to allow a smooth insertion of the pins in this joining condition.

Although no apparent damage was induced in the metallic part, the combination of pin feeding and frictional heat at the pin-polymer interface, locally affected the properties of the polymeric part. The microstructure of the joint produced with the joining condition J2 is shown in Fig. 7. As it was expected, no microstructural changes were observed on the Ti-6Al-4V pins in comparison to the as-produced MIMStruct parts (see Fig. 3). This is mainly due to the relatively low process temperature, well below the phase transformation ranges of Ti-6Al-4V alloy (T(α→β))

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transition) = 995 ± 15 °C [20]). As it is shown in Fig. 7b the pin microstructure after joining was unchanged, displaying a typical Widmanstätten microstructure consisting of lamellar colonies of alpha-phase and beta-phase (white).

Our previous study on the U-Joining [12] showed the formation of a polymer thermomechanically-affected zone (PTMAZ) due to the pins feeding and increase in process temperature. Although in this study the PTMAZ extension could not be clearly defined by SEM (Fig. 7), volumetric thermal flaws, such as pores, were found around the pins, as shown in Figs. 7c and 7d. We have reported that such volumetric flaws (pores) are typically found in the PTMAZ of ultrasonically joined hybrid joints [12]. These usually present smooth internal walls, as shown into detail in Fig 7e. Formed pores may be a result either of moisture evolution or evolution of gaseous products from partial thermal degradation [16, 17].

During joining, the molten polymer layer tends to flow upwards due to the pin insertion. After joint consolidation, a close contact of the molten polymer was observed at the pin-polymer interface (Fig. 7d and Fig. 7e). Furthermore, the designed geometrical undercut - conceived to decrease the stress concentration at the pin base around the pins - were completely filled with polymer (Fig. 7e). However, localized regions with a poor interface were also observed (Fig. 7f), which may be caused by polymer deconsolidation, because of the differential shrinkage related to the large difference in coefficient of thermal expansion between metal and polymer.

The evaluation of the local mechanical properties was reserved for the polymeric part, as the process temperatures were not high enough to cause changes in the microhardness of the Ti-6Al-4V part. The microhardness distribution (colored map superposed on the left-hand side of the joint cross-section view, Fig. 8), revealed the formation of the PTMAZ, where a decrease of about 9 % in local mechanical strength (or Vickers microhardness) can be observed in comparison to the base material. At this zone, competitive phenomena such as chain reorientation, changes on molecular weight and partial polymer degradation may influence its mechanical properties [17].

Due to the pins’ penetration, squeeze flow of molten polymer takes place, whereby squeezed polymer remains entrapped at the interface formed between the surfaces of the metallic and polymeric parts. Nevertheless, part of the molten polymer remains around the inserted pins. The polymer chains of this consolidate polymer volume was probably reoriented following the squeeze flow direction. The chain reorientation and material flow at high temperatures may also increase free volume (i.e. packing density). These phenomena combined with the decreases in molecular weight due to chain scission may collaborate to the decrease of microhardness in this region [16, 17]. Further physicochemical characterization of the PTMAZ is in progress to confirm this assumption.
interlocking effect increased significantly the energy absorbed until joint final failure (toughness), as observed from the force-displacement curves in Fig. 9. As a result, the displacement at break increased from 0.15 ± 0.02 to 1.59 ± 0.16 mm. This is an indication of a more damage-tolerant joint in comparison to the reference joints, which fractured by adhesive failure.

Table 2. Ultimate-lap-shear force and displacement at break for the non-reinforced reference and joining condition J2 specimens.

<table>
<thead>
<tr>
<th>Joining condition</th>
<th>ULSF [N]</th>
<th>Displacement at break [mm]</th>
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<tr>
<td>Non-reinforced reference</td>
<td>292 ± 7</td>
<td>0.15 ± 0.02</td>
</tr>
<tr>
<td>J2</td>
<td>1860 ± 260</td>
<td>1.59 ± 0.16</td>
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Figure 9. Example of the force-displacement curves for a hybrid joint produced with the joining condition J2 and a non-reinforced reference joint.

The lap-shear specimens of J2 joints failed through a combination of shear (at the base of the metallic pin) and a mixed cohesive-adhesive failure in the consolidated polymer molten layer entrapped at the interface between upper and lower parts (squeeze flow polymer). Figs. 10a (polymeric part) and 10b (metallic part) present the overview of the fracture surfaces for a J2-joint after lap-shear testing.

The polymeric volume around the pins appears to have undergone considerable plastic deformation during the lap-shear testing, as shown Fig. 10c, suggesting that the reinforcements successfully contributed to bear and redistribute external load. Therefore, through-the-thickness reinforcement appears to contribute to induce non-catastrophic bearing failure around the pins in the initial stage of the lap-shear test. In the final stage of the mechanical testing, the pins experience considerable plastic deformation, prior to final failure by shearing. This is supported by the high magnification picture (Fig. 10d) where the fracture surface of sheared pin revealed a great amount of dimples spread over the pin fracture surface. This is an indication of local ductile fracture. After shear fracture, the pins remain embedded in the polymeric part.

Figure 10. Evaluation of the fracture surface for a joint produced with the J2 joining condition: (a) polymer part; (b) metal part; (c) detailed SEM micrograph at the pin region marked in (a); (d) high-magnification image showing the fracture surface of the region marked in (c); (e) detailed SEM micrograph of the attached polymeric molten layer marked in (b) and (f) its corresponding high-magnification image (area marked in (e)).

Furthermore, a considerable amount of consolidated polymer (a result of the squeeze flow) can be observed at the interface between upper and lower parts (Figs 10b and 10e). This indicates a strong interaction between metal and consolidated polymer, corroborating to the assumption of mixed cohesive-adhesive failure mechanism. Tearing of the polymer attached to the titanium surface can be clearly observed in Fig. 10f. It suggests that this polymeric layer underwent considerable plastic deformation before failure, which indicates a localized ductile fracture mechanism.

Conclusions

The U-Joining process has been applied to join PEI to surface-structured Ti-6Al-4V alloy, produced by metal injection moulding (MIMStruct). Hybrid joints with through-the-thickness reinforcements (conical pins) were successfully produced. The influence of the joining energy on joint formation was investigated according to a one-factor-at-time parameter study. Complete penetration of the reinforcement in the polymeric part of the joint was
accomplished with 2000 J of joining energy, 42 μm of amplitude and 15 psi of joining pressure.

The measured process temperature (362 ± 32°C) was within the degradation range of PEI; however, the joining cycles were very fast (1.26 ± 0.04 s). Thus, extensive polymer degradation is not expected. The Vickers microhardness evaluation showed that the process-affected volume of materials is restricted to a small volume around the pins. A microhardness decrease of about 9% was measured in comparison to the base material, due to process-related physicochemical changes in the polymer (e.g. initial chain scission inducing decreases in molecular weight, chain reorientation by squeeze flow, increase of free volume, etc.). The microstructural characterization revealed that a close contact between polymer and metal was created while few thermal flaws (pores) were found sparsely located at the PTMAZ.

The through-the-thickness reinforcement significantly increased the mechanical performance of the joints under quasi-static loading. The average ultimate lap shear force for the hybrid reinforced joints was six times higher (1860 ± 260 N) in comparison to non-reinforced reference joints (292 ± 7 N). Toughness – qualitatively obtained from the displacement at break – was also higher (1860 ± 260 N) in comparison to non-reinforced reference joints (292 ± 7 N). Toughness – qualitatively obtained from the displacement at break - was also higher for the ultrasonic joints (1.59 ± 0.16 mm) in comparison to the reference joints (0.15 ± 0.02 mm). This is an indication that ultrasonic joints have higher damage tolerance due to the additional pin mechanical interlocking. The fracture mechanisms in the hybrid joint combined shearing of the metallic pins and mixed cohesive-adhesive failure of the consolidated polymeric volume. However, joint failure was initiated by bearing, a non-catastrophic and detectable failure type usually required in aircraft structures.

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