Surface machining of Ti6Al4V by means of Micro-Electrical Discharging to improve adhesive joining

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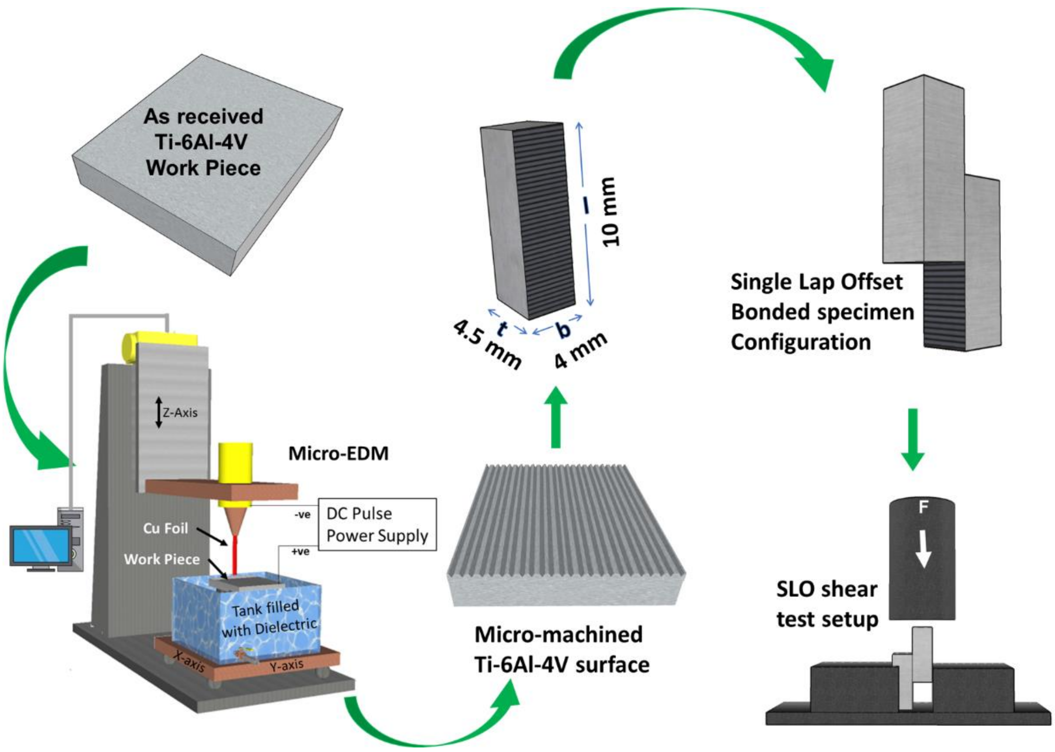
Abstract

The Micro-Electrical Discharge Machining (Micro-EDM) technique has been employed to machine micro-patterns with shaped micro-slots on Ti6Al4V surfaces. Ti6Al4V substrates, with and without micro-slots, were bonded using a commercial epoxy adhesive. Optical microscopy and SEM were used to observe the micro-patterned Ti6Al4V surfaces before and after joining and to analyse the fracture surfaces after mechanical tests. The joints were mechanically characterised, with and without micro-patterns, by means of Single Lap Offset (SLO) shear tests under compression to understand the effect of differently shaped micro-slots. The effects of the shape of the micro-slots, their interlocking or overlapping, and their orientation, with respect to the applied load, are presented and discussed in terms of mechanical performance of the joints.

Graphical Abstract



Keywords

surface machining; joining; mechanical shear test; adhesive bonding.

1 Introduction

Joining and assembling is of vital importance for engineering structures. Today, there is a growing demand for highly reliable joints for structural applications in several fields, including the automotive and aerospace industries.

Adhesives are materials that are widely used to join metals to each other or to dissimilar materials. Unlike polymeric materials, metals and metal oxides usually have higher free surface energies in the ultra-clean state, which are generally greater than 500 mJm^2. Kinloch (1980) underlined the importance of the environmental conditions during the bonding operation, especially for metallic substrates, which are usually covered by an oxide layer. He found that metals in an ultra-clean state and in a controlled environment are readily wetted by organic adhesives that have lower free surface energies. However, when metallic components are not in a controlled environment, preparation of the metal surface is needed to obtain good adhesion.

Several reviews, such as the one published by Kinloch (1980), have shown that this issue was studied intensively in the past. Kinloch (1980) identified the chemical and surface properties of metallic substrates as being critical factors in determining the strength of adhesive joints. Venables (1984) discovered that, as a consequence of certain etching or anodisation pre-treatments, aluminium and titanium surfaces produce oxide films that lead to surface porosity and microscopic roughness. He subsequently demonstrated that these features enhance mechanical interlocking when a polymeric adhesive is used and may result in much stronger bonds than when the surfaces are smooth and untreated. For these reasons, the bonding of metals with epoxy resins usually requires conversion coatings or a bond primer to enhance the adhesion between the substrate and the epoxy resin. None of these coupling agents were used in this work.

Several attempts were made in the past to enhance the strength of adhesive joints, acting on both the properties of the adhesive and on the properties of the surface. Mention can be made of the work of Jojibabu et al. (2016) , who reported an improvement in joint lap shear strength as a result of adding carbon nano-fillers to the joining material; they described the effect of fillers in epoxy adhesives, but limited their study to aluminium alloy substrates. Hernandez et al. (2014) examined the effects of drilled holes on ceramic composite surfaces on joint strength; the machining of the composites resulted in a six-fold increase in the shear strength, compared to non–machined joints. Demir et al. (2017) experimented with the laser etching of surfaces; they showed the possibility of using a pulse fibre laser to generate texture patterns on Ti6Al4V, in order to improve the adhesion strength. Ahmmed et al. (2014) proposed the advanced femtosecond laser micromachining technique, and produced regular laser-inscribed patterns on metal surfaces.

Other examples reported in literature refer to plasma modifications of metallic substrates: Aliasghari et al. (2016) reported an improvement in adhesive joint strength, by means of a plasma pretreatment of Al adherent surfaces. They carried out a detailed analysis of different plasma electrolytic oxidation pre-treatments which significantly influenced the failure mode (adhesive or cohesive) of the joints.

Increasing the surface area as a result of the micro-machining of the facing components is a well-known technique that is used to improve the adhesive bonding of metallic joints. A considerable amount of literature has recently been produced on the theme of surface modifications by means of micromachining on several kinds of metals (copper, brass, nickel, Ti alloys). Kim and Loh (2010) produced precise micro-grooves and pyramidal patterns on the surfaces of materials using elliptical vibration cutting (EVC).

The objective of this work has been to study the influence of surface structuring, by means of the Micro-Electrical Discharge Machining (Micro-EDM) technique, on the adhesive joints of a commercial Ti6Al4V alloy. Ti6Al4V alloy is an important structural material that is often used for the manufacturing of critical components for aerospace vehicles and high-end automotive devices, due to its lower density combined with enhanced high-temperature mechanical properties. It shows good corrosion and oxidation resistance, together with higher specific strength properties, as highlighted by Lino Alves et al. (2016). The surface machining of Ti6Al4V alloys by means of conventional methods is difficult, but in recent years micromachining has produced promising results to improve joint strength.

Rajukar et al. (2013) suggested Electrical Discharge Machining (EDM) and Electrochemical Machining (ECM) as a solution to produce micro and nano machined features on the surfaces of difficult-to-machine materials. Li et al. (2016), using a sodium nitrate solution, successfully produced micro-patterns on Ti64AlV; these authors pointed out that it is possible to obtain an array of micro-holes using optimised parameters, but also that further work is required to reduce issues related to the loss of solution and the elimination of by-products.

Sjöström and Su (2011) successfully produced micropatterns on pure Ti surfaces utilising ethanol glycol electrolytes. Lu and Leng (2005), employing a Jet-Electrochemical Micromachining (Jet-EMM) method, produced micro-hole patterns on a Ti6Al4V surface by combining jet flow impingement and electrochemical reactions. Aliasghari et.al (2016) modified a titanium surface, by means of Electrolytic Plasma Oxidation (EPO) to improve adhesive bonding, using specific pre-treatments, such as aluminate-phosphate and silicate- phosphate electrolytes. Zimmermann et al. (2012) demonstrated that a significant improvement in the adhesive forces between Ti alloy and an epoxy adhesive can be achieved by modifying the titanium surfaces via laser-induced surface oxidation and roughening.

Laser-assisted techniques generally offer contactless surface micro-machining together with higher precision and production rates than other conventional techniques. Moreover, they can also be applied to a wider range of materials, due to their larger wavelength range, extended pulse (from 10^{-12} seconds to 10^{-6} seconds) and iterative rates, as has recently been shown in review format by Dongre et al. (2016). Palmieri et al. (2013) observed improved adhesion when adopting laser ablation surface machining to modify a Ti6Al4V surface for adhesive bonding. The laser ablation process is a cold cutting process in which atoms, or molecules, on the surface are supplied with enough energy to make them leave the surface. The duration of the energy supply is very short, and there is hardly sufficient time to transmit the energy to the adjacent atom or molecule. The laser treatment of the surface leads to the formation of a nanostructured layer, which helps to increase the bonding performance. Vorobyev and Guo (2007) developed the first study on the femtosecond laser surface treatment of titanium; they observed the production of several nanostructures on a Ti surface. However, they only analysed the optimal conditions for manufacturing periodic groove patterns on a surface, and did not highlight the benefit of this technique, that is, of increasing adhesion at the interface.

Among the surface modification techniques that are available for titanium alloys, the micro-ECM and micro-EDM techniques are considered to be cost effective and they can easily produce different macro- to-micro patterns. However, the micro-ECM process suffers from some drawbacks pertaining to the production of accurate patterns, including over cut and taper cut effects, the necessity of selecting appropriate electrolytes for alloys with different compositions, etc. The micro-EDM technique can instead produce patterns more accurately and with a higher repeatability, as the spark gap can easily be controlled using appropriate parameter settings. Furthermore, Micro-EDM is an economic and a very versatile process: it can be used to machine different micro-texturing features, which can be modelled to optimise the effectiveness of joints; the process can be used for both conductive and non-conductive materials.

Finally, several methods have been introduced to establish the mechanical characteristics of adhesively bonded joints. Matsuzaki and Suzuki (2010) adopted a tensile adhesive test and a compressive shear adhesive test to investigate the effects of surface modifications on the joint strength of composites, while Yukimoto et al. (2015) studied failure modes and crack-propagation by submitting carbon-fibre-reinforced, micro pattern surface plastic/adhesive bonded specimens to a transverse load in a Single Leg Bending (SLB) configuration. Da Silva et al.(2010) performed the tensile testing of single lap joints to observe the effects of micro-slots on the strength of aluminium adhesively bonded joints.

ASTM D905-08 (Standard Test Method for the Strength Properties of Adhesive Bonds in Shear by Compression Loading) describes the standard test method used to determine the comparative shear strength properties of adhesive bonds by means of compression loading. As stated by Casalegno et al. (2010), lap tests do not provide a suitable shear strength value for design purposes, but the use of samples with the same size and shape can offer a first approximation.

In this work, a novel approach is presented to increase the adhesive joint strength of Ti6Al4V components, by means of surface machining, using an in-house developed Micro Electrical Discharge Machining (Micro-EDM) procedure. Micro-EDM has here been used for the manufacturing of surface patterned samples with simple geometries, such as V-shaped, U-shaped and semi-circle slots. Joints, with and without micro-patterns, were mechanically characterised by means of Single Lap Offset (SLO) shear tests under compression to understand the effects of differently shaped micro-slots.

2 Materials and Methods

Ti6Al4V alloy sheets (100 mm x 100 mm x 4.5 mm) were supplied by M/s Aich Enterprise, Kolkata-6, India. A commercial, two-part thixotropic epoxy paste adhesive (LOCTITE EA 9321 AERO, commercial name Hysol-EA9321), supplied by the Henkel Corporation, USA, was used as a joining material. Its constituents are Triglycidyl-p-aminophenol (30-60 %), Aluminum (10-30 %), Epoxy Resin (10-30%) and a filler (1-5%), as described in the supplier's datasheets. Part A of the commercial adhesive was grey in colour, and had a density of 1.24 g/ml and viscosity of 290 - 710 Pa·S at 25°C, while Part B was off-white in colour and had a density of 1.22 g/ml and viscosity of 20 - 80 Pa·s at 25°C. The two parts of the adhesive were mixed, prior to application, in a 100 (Part A) to 50 (Part B) by weight ratio, at room temperature. The mixture, which was grey in colour, with a density of 1.23 g/ml, showed a lap shear strength of 27MPa (provided by the supplier) after measurement by means of the standard ASTM D1002 (Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading Metal-to-Metal) procedure. This epoxy adhesive cures in 5-7 days at room temperature, but the curing can be accelerated by increasing the temperature. According to the technical datasheets, it takes only 1 hour to cure at 85°C. This thixotropic adhesive was selected to join the previously mentioned metal substrates, as a representative joining material for the integration of components in the aerospace industry. Ti6Al4V substrates, with and without modified surfaces, were bonded and mechanically tested.

The apparent shear strength of the joined samples was evaluated using the single lap offset (SLO) test under compression at room temperature and 65% relative humidity (universal testing machine SINTEC D/10, equipped with a 5kN load cell), according to a method adapted from ASTM D905-08; the crosshead speed was 1 mm/min. The maximum force was recorded, and the apparent shear strength was calculated by dividing the maximum force by the joining area. The size of each single component for the lap off-set shear tests was 10 mm × 4 mm × 4.5 mm, with an average joined area of 24 mm^2 (60% of the total facing surface area).

The transverse and cross-sectional morphology of the as received and micro-machined Ti6Al4V and the bonded cross-sections were characterised by means of optical microscopy and electronic microscopy using Field Emission Scanning Electron Microscopy (FESEM- ZEISS Supra 40) with an Energy Dispersive Spectroscopy (EDS- SW9100 EDAX) detector.

2.1 Ti6Al4V Surface Modification

A Micro-electrical discharge machining (micro-EDM) setup, developed in-house at the Indian Institute of Technology (ISM), Dhanbad, India, was used to produce micro-slots on the Ti6Al4V workpiece. The Micro-EDM setup is comprised of a rotating spindle and X-Y-Z CNC (Computer Numerical Control) stages. Figure 1 shows a schematic diagram of the Micro-EDM setup which was used for the experimental work in this study. The spindle is mounted onto the Z-stage and the workpiece is merged in a dielectric tank, which is mounted onto the X-Y stage.

A flat copper 100 µm thick foil was initially used as a tool to machine parallel slots, and a sinking micro-EDM operation was performed. However, it was difficult to produce uniform and parallel slots with this process. Moreover, the repeatability of the process was poor when the straight slot was more than 3 mm long. To overcome this problem, the spindle assembly of the machine was replaced with a guided travelling wire mechanism (Figure 2 a), which was mounted onto the Z-stage of the machine. A travelling 100 µm diameter wire was used as the tool. Through this modified setup, it was possible to machine V-shaped, U-shaped and semi-circle micro-slots on the Ti6Al4V work surface with better repeatability.

The Ti6Al4V block was mounted onto a fixture and immersed in a dielectric medium (deionized water). The different profile movements (V, U and semi-circle) were introduced to the work piece through computer numerical control programmes, but the position of the travelling wire was kept fixed. Several trial experiments were conducted to find the best width of the spark gap (Figure 2 b), which is essential to achieve dimensional accuracy of the machined slots. The different parameters used for the machining of different slots were: pulse on time: 8µs, machining voltage: 30V, Peak current: 2A and duty factor: 30%. The average machining time of one slot was approximately 3 minutes for the aforementioned conditions. However, this time may vary according to the dielectric flashing condition, the length of the slot that has to be machined, the thermal properties of the work material and the properties of the dielectric medium. The average values of the specifications of the produced micro-slots, the average dimensions (width and depth) and the average distance between slots are reported in Table 1, together with the number of micro-slots per centimetre and the relative increase in the surface area for each micro-slot shape. The produced micro-slot dimensions on average deviated by about 7 % from the pre-set values fed to the machine.

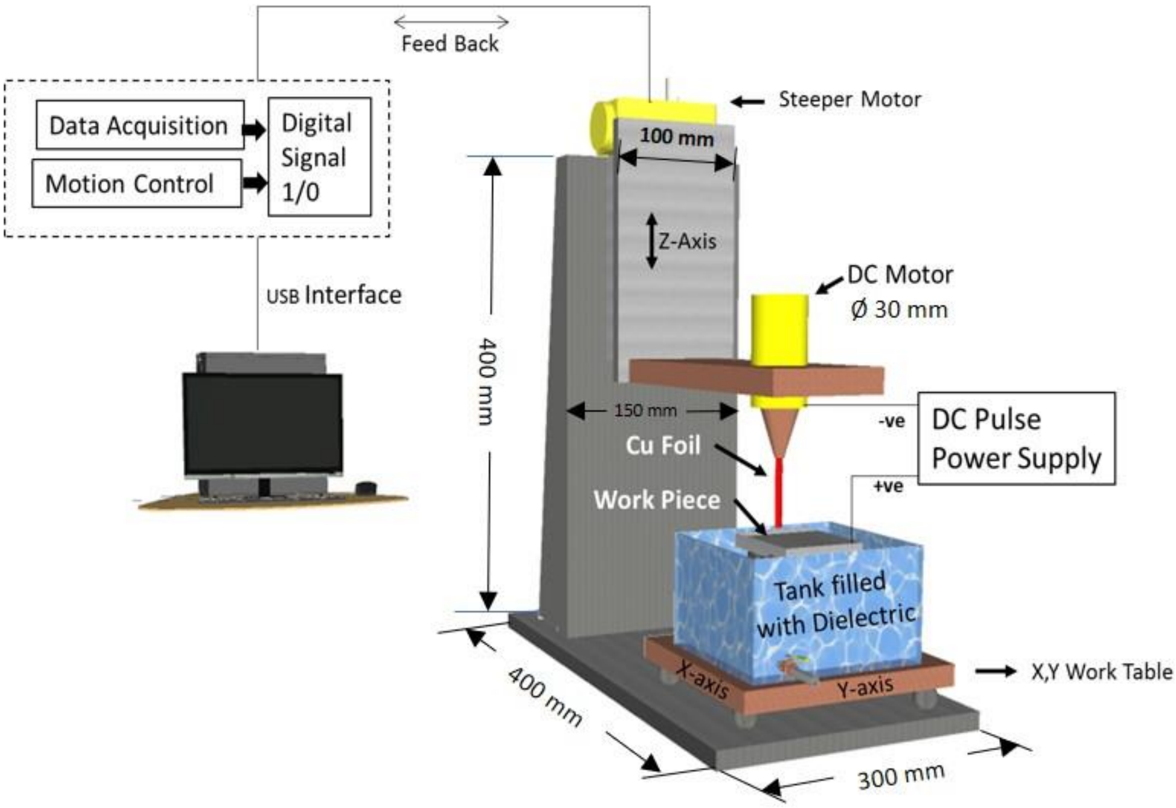


Figure 1 *Design of the Micro-EDM setup.*

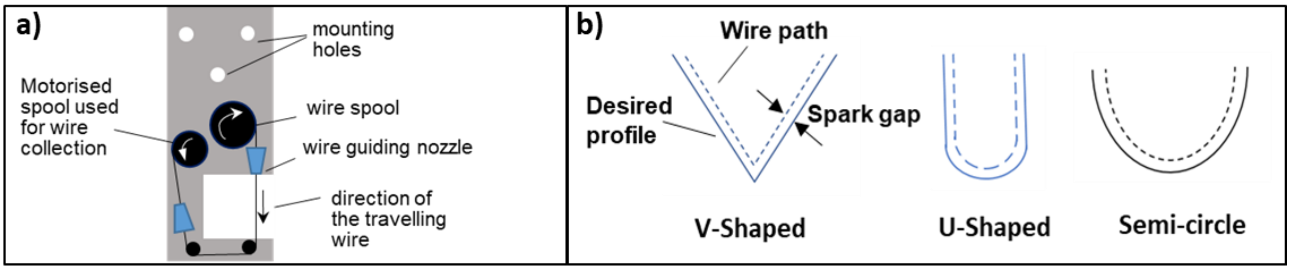


Figure 2 *(a) Guided travelling wire mechanism; (b) Wire paths for the machining of different profiles*

Table 1 *Average dimensions of the micro-slots produced on Ti6Al4V surfaces and different slot configurations; slots oriented perpendicular to the load direction, unless otherwise indicated*

Surface	Width [μm]	Depth [μm]	Spacing [μm]	Slots [cm]	Surface area [cm^2]	Slot configuration
As Received	-	-	-	-	1	NA
V-Shaped	330	280	550	11.36	+27%	Semi-overlapped
						Semi-overlapped (*Slots ‖ to the load direction)*
Semi-circle	500	197	345	11.83	+23%	interlocked
U-Shaped	258	230	628	12.04	+38%	Overlapped
						Semi-overlapped
						misaligned

A pulsed DC power supply is used to control the spark energy. A spark develops during the pulse-on-time and the molten material in the pole cools down quickly during the pulse-off-time. This phenomenon generates a high-pressure shock wave in the liquid dielectric medium, which in turn causes the material to leak from the molten metal pole into the dielectric medium in the form of debris, thereby leaving a micro-crater on both the tool and on the surface of the workpiece. The accumulation of the craters on the workpiece attributes removal effects to the material, but the accumulation of craters on the tool surface leads to tool erosion. Material transfer occurs between the tool and the workpiece as a result of material melting during the sparking process.

2.2 Preparation and characterisation of the specimens

Specimens of 10 mm (l) x 4 mm (b) x 4.5 mm (t) were cut from the micro-machined and as received Ti6Al4V sheets. Only as received Ti6Al4V substrates were polished with SiC-paper (P600) prior to joining to remove surface scratches. The polished substrates and the substrates with micro-machined surfaces were cleaned with acetone in an ultrasonic bath for 10 min at 60˚C to degrease the surface and remove the dirt/dust particles. The commercial epoxy adhesive mixture was applied to the dried surfaces of the substrates, which were stacked in an offset configuration, as shown in Figure 3. The bonded pair of substrates were shimmed together to ensure that the adhesive filled the micro-slots before the curing process. The heat treatment was carried out at 85°C for 1 hour in an oven without applying any pressure.

The micro-slots machined on the facing surfaces were combined in different configurations, according to the data reported in Table 1 (semi-overlapped for V-shaped specimens; semi-overlapped, overlapped and unaligned for U-shaped specimens; interlocked for semi-circle specimens). Only overlapped, semi-overlapped and misaligned configurations were tested on the U-shaped samples. The overlapping of V-shaped samples, during adhesive joining, was much more difficult than in the case of rounded shapes (such as U-shaped or semi-circle shapes), because of the squeeze effect of the joining materials at the edge of the overlapping, which can be better accommodated with a rounded geometry. Moreover, it was observed, during the experimental activity, that the V-shaped and semi-circle slot specimens easily resulted in a slight misalignment, which was unavoidable during joining and curing. It was much easier to obtain overlapped, misaligned and semi-overlapped configurations for the U-shaped samples.

Bonded substrates with as received surfaces, with V-shaped micro-slots parallel to the direction of the load and with V-shaped micro-slots perpendicular to the direction of the load were produced to determine the effect of adhesion, as shown in Figure 3 (b, c and d). The offset specimens were placed in the fixture, shown in Figure 3 (e), and subjected to a compression load; the loading was stopped when joint failure occurred. The maximum load values at failure were recorded for each test event. The apparent shear strength of the joined samples was calculated by dividing the maximum force by the joining area. Each reported value is the average of at least five tests.

Fracture surface analysis was carried out using both an optical microscope and a scanning electron microscope. U-shaped and semi-circle shaped joined samples, with micro-slots perpendicular to the load, were tested.

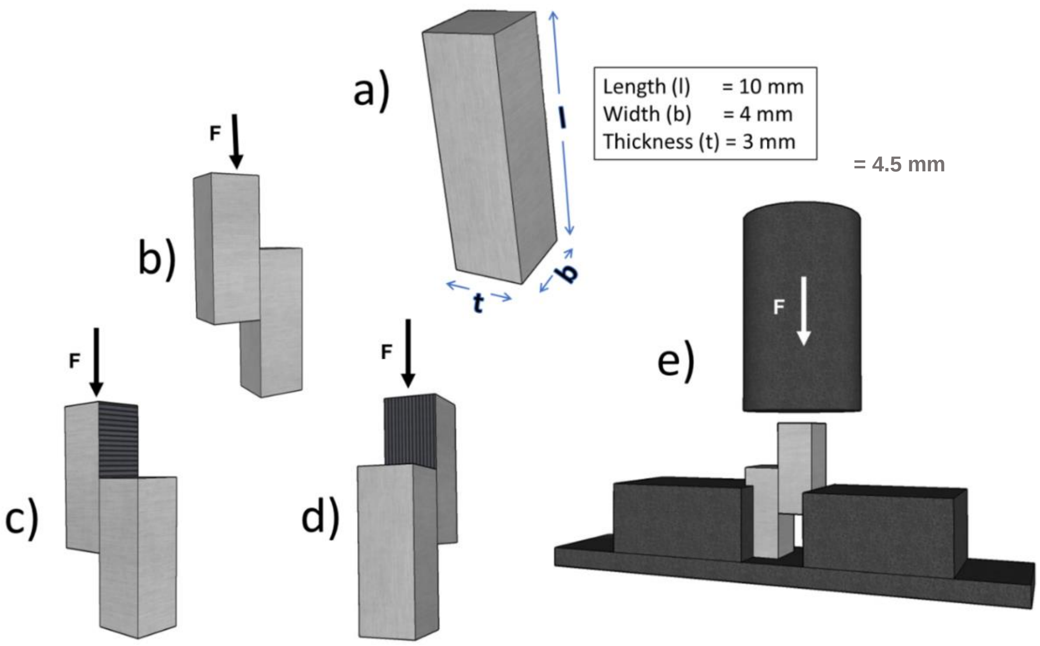


Figure 3 *The Single Lap Offset (SLO) shear test specimens and test configuration. (a) Ti6Al4Vsubstrate dimensions, (b) bonded sample with as received surfaces, (c) bonded sample with micro-slots oriented perpendicular to the load, (d) sample with micro-slots parallel to the load, (e) SLO test setup.*

3 Results and Discussion

3.1 Microscopy Analysis

The Micro-EDM machine was set to produce different geometries on the Ti6Al4V surface: V-shaped, U-shaped and semi-circle micro-slots were produced according to Table 1. Figure 4 shows, as an example of a micromachined surface, an optical microscope image of the Ti6Al4V surface before and after V-shaped slot machining; the micro-slots (parallel to each other) have a pitch of approximately 11 micro-slots per centimetre. Material removal takes place in the micro-EDM as a result of the electrical sparks that develop between the tool and the sample through the dielectric liquid when a threshold electrical potential is applied across them. When the applied voltage crosses the threshold potential, dielectric breakdown takes place, and a spark is produced between the two closest points on the tool and the workpiece that offers the lowest resistance to the current flow. The temperature of the points can reach as much as 10000°C, which melts the surrounding material. The coloured surface of the machined sample shown in Figure 4 (b) indicates material transfer from the Cu electrode to the processed surface.

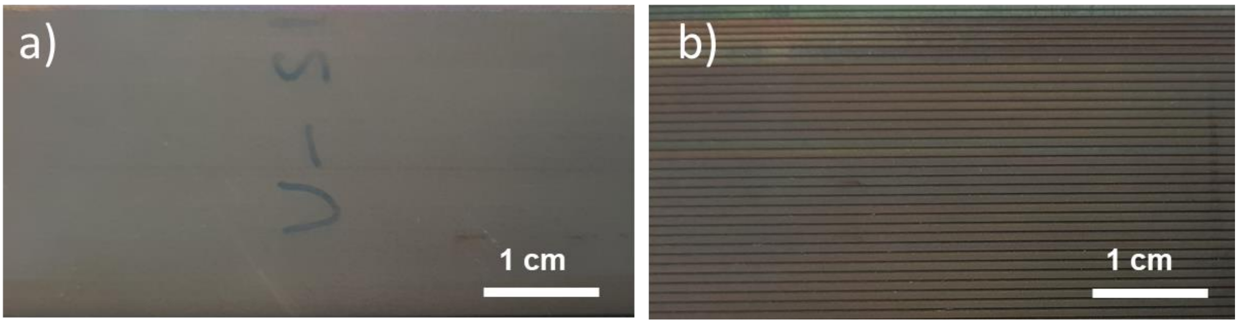


Figure 4 *Macro Images of the Ti6Al4V surfaces, (a) As-received surface, (b) slotted (micro-machined) surface.*

The V-shaped geometry of a micro-machined Ti-alloy surface is shown in Figure 5, as a representative example. The Micro-EDM process resulted in an uneven surface in the machined area with protruding micro-structures (Figure 5 d), which could further enhance the adhesive/metal joining interface strength. Surface roughening, coupled with machining, can be expected to lead to a beneficial effect on adhesion at the interface. It was calculated that micro-machining increased the surface area of Ti6Al4V by 27%, 38% and 23% for the V-shaped, U-shaped and semi-circle slots, respectively, compared to the as received alloy.

Figure 6 shows the obtained joining seams. Continuous joined seams were observed at the interface between each substrate, and porosity was also noted in the joined region, due to the presence of gaseous products released during adhesive curing.

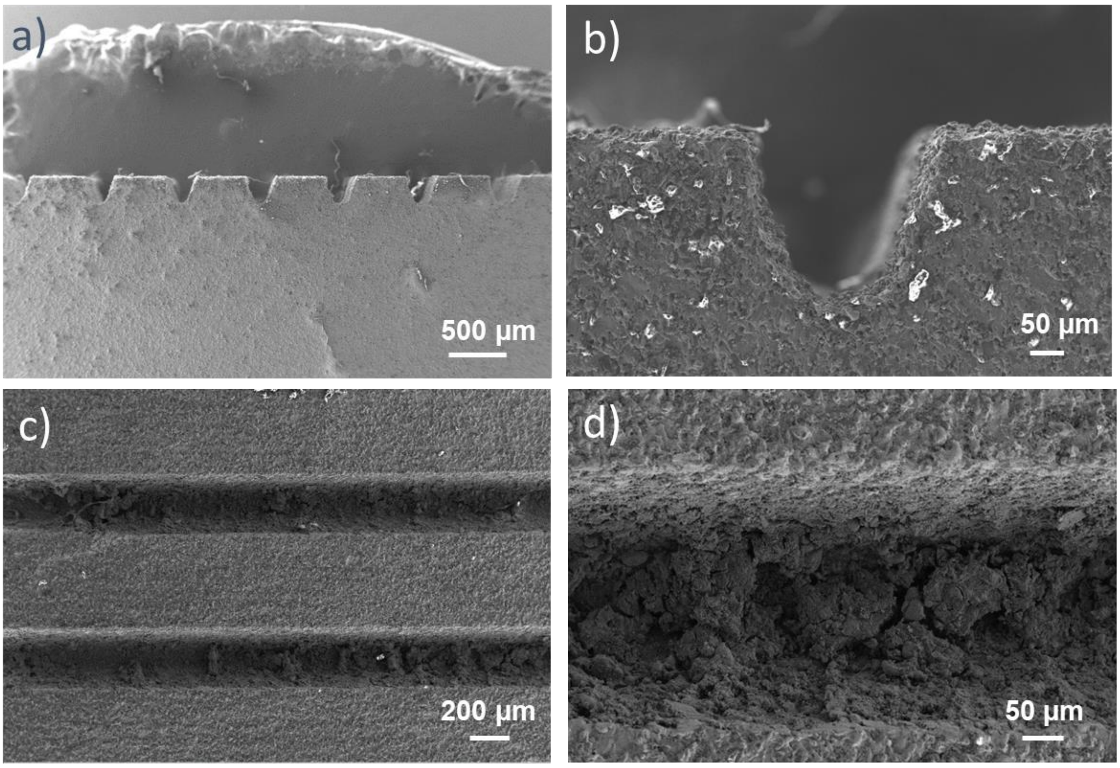


Figure 5 *Micrographs of the V-micro-machined Ti6Al4V sample. (a) and (b) side views, (c) and (d) top views.*

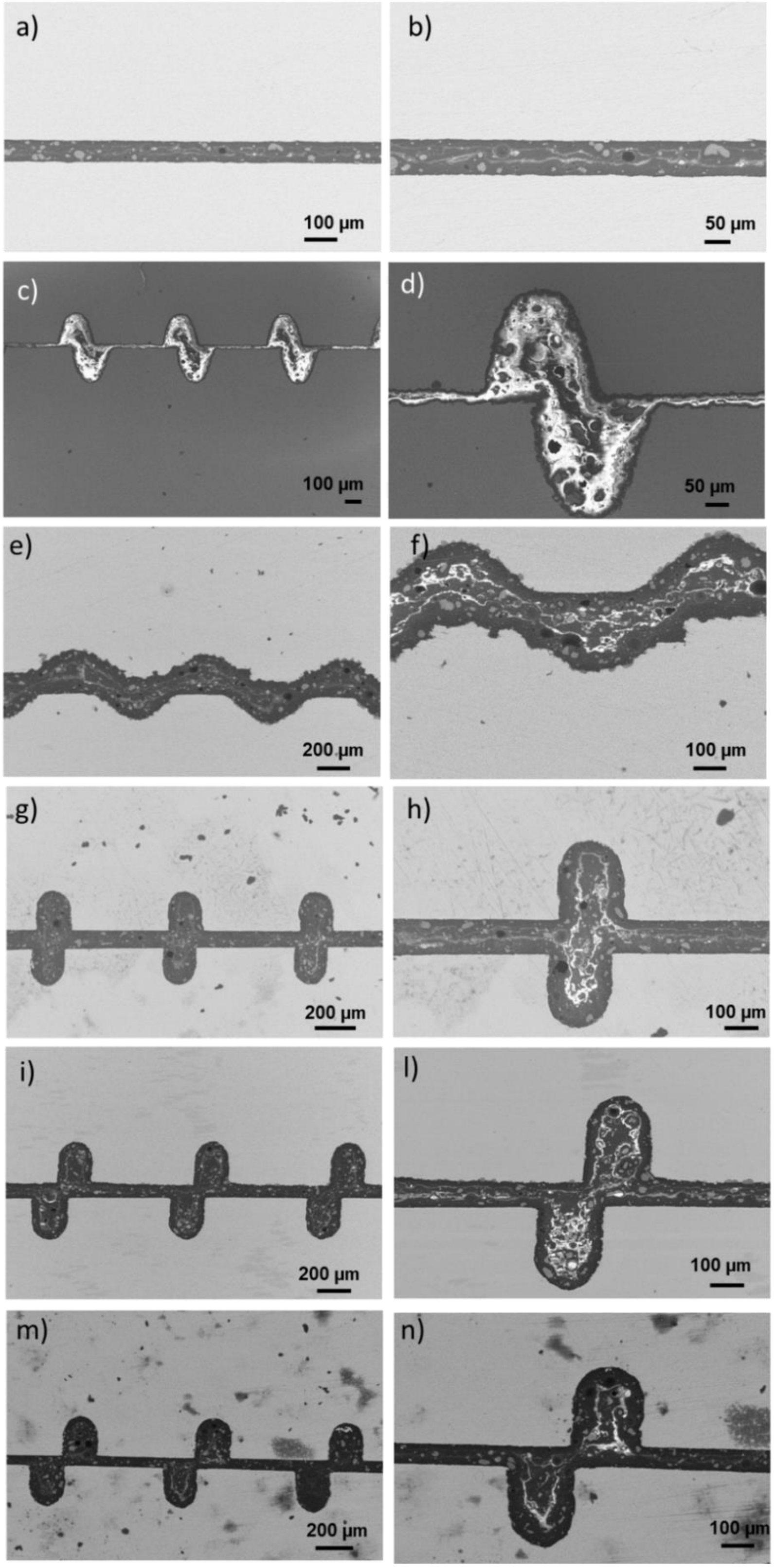


Figure 6 *Cross-sections of the adhesive bonded Ti6Al4V samples. (a) and (b) as received surfaces, (c) and (d) semi-overlapped V-shaped, (e) and (f) interlocked semi-circle, (g) and (h) overlapped U-shaped, (i) and (l) semi-overlapped U-shaped, (m) and (n) misaligned U-shaped micro-slots*

The semi-overlapped V-shaped micro-slots on Ti6Al4V (Figures 6 c and d) exhibit a continuous interface, both inside and outside the micro-slots. The semi-circle micro-slots were produced with curved edges to facilitate the interlocking at the joining interface, as can be seen in Figures 6 (e) and (f). Mechanical interlocking is evident between the adhesive and the adherent surface in the micro-machined area for the semi-circle micro-slots.

Substrates with U-shaped micro-machined surfaces were bonded in three different alignments. Figure 6 (g) and Figure 6 (h) show the cross-section of a typical U-shaped micro-machined bonded specimen, where the micro-slots overlapped each other. Figure 6 (i) and Figure 6 (l) show the U-shaped micro-slots that semi-overlap each other in the bonded sample. A third option for the U-shaped micro-machined samples was a misalignment of the micro-slots, as shown in Figure 6 (m) and Figure 6 (n).

The surface modification of each bonded sample increases the bonded surface with respect to the as received Ti4Al4V. The joining material completely filled the micro-slots, regardless of the micro-pattern geometry (U or V-shaped), and no porosity or unfilled volumes were detected in the manufactured samples.

The average joint thickness of the specimens with unmodified joining surfaces, for a similar amount of joining material (adhesive), was 65 µm. The specimens with micro-machined joined surfaces showed a joint thickness of about 35-40 µm in the areas where unmodified surfaces were in contact. This difference can be explained by considering the accumulation of adhesive material in the cavities of the micro-slots, which leads to less adhesive being available outside the unmodified surfaces. For these reasons, it was impossible to produce micro-machined joints with the same joint thickness as the unmodified ones.

3.2 Mechanical Characterisation

The average lap shear test values for each set of bonded specimens are reported in Table 2. Surface machining, by means of the Micro-EDM technique, showed a significant effect on the joint strength. The orientation of the micro-slots, with respect to the direction of the applied load, is known to be a critical factor for the joining configuration. The orientation was only studied for the V-shaped geometry at this stage of the work, but an encouraging average increase of about 12% in the joint strength was observed when the samples with micro-machined slots were aligned *perpendicular* to the applied load direction, while a decrease of about 11% in the joint strength was measured when the micro-slots in the bonded specimen were aligned *parallel* to the applied load direction. The micro-slots seem to play an important role in preventing cracks and increasing the ultimate failure when they are *perpendicular* to the applied load: they also help sustain the applied pressure by distributing the load over the machined areas. A certain resistance, provided by the edge surface of the grooves, was not possible for the specimens with micro-slots parallel to the applied load. Moreover, it seems that the fracture propagation was relatively swift when the micro-channels were parallel to the applied load. In principle, it is possible to hypothesise that this result could also apply for other geometries, but further investigations are needed to study the effect of loading configuration on the shear strength of joints with different slotted surfaces. However, considering the results of the V-shaped slots perpendicular to the load direction, all the other geometries were tested with slots perpendicular to the load direction.

***Table 2**. The average results of the Single Lap Offset (SLO) test for all the bonded specimen sets; the values in percentage represent the increase in joint strength in relation to the substrate with no pattern; slots oriented perpendicular to the load direction, unless otherwise indicated*

Joint Specifications	Average lap shear strength, [MPa]	Effect of micromachining on the joint strength (%)
 As received polished surfaces	34.6 ± 1.7	Reference
 V- shaped micro-machined (semi-overlapped)	38.7 ± 1.5	+11.8
 V- shaped micro-machined (semi-overlapped) *(Micro-slots oriented parallel to the load direction)*	30.7 ± 2.5	-11.2
 Semi-circle shaped micro-machined (interlocked)	23.6 ± 2.8	-31.8
 U-shaped micro-machined (overlapped)	32.2 ± 3.2	-6.7
 U-shaped micro-machined (semi-overlapped)	45.5 ± 5.0	+31.5
 U-shaped micro-machined (misaligned)	36.6 ± 4.0	+5.8

The specimens with interlocked semi-circle micro-slots on the surface displayed a decrease of about 32 % in the joint shear strength, compared to the as received polished specimens. The non-edged, rounded walls of the grooves in the semi-circle micro-slots, compared to the more edged V-shaped ones, are possibly the reason for the lower joint shear strength, which led to lower resistance to load and fracture propagation.

The micro-slot alignment of the specimens bonded to each other was also found to be a factor of influence on their joint shear strength. It was observed that when the U-shaped micro slots overlapped, they showed a decrease in the joint shear strength, compared to the misaligned configuration, which displayed an increase of approximately 6 %, with respect to the as-received specimens. However, when the U-shaped micro-slots were bonded together in a semi-overlapped configuration, a remarkable increase in the joint strength, of around 31 %, was observed, while the effect of micromachining on the joint strength in the case of the misaligned U-shaped micro-slots led to a less significant increase.

The fracture surface analyses of the bonded specimens (Figure 7) showed the following features after the SLO test: substrates with unmodified joining surfaces showed adhesive failure (Figure 7 a) , while all the bonded substrates with micro-machined surfaces displayed mixed mode failure (Figures 7 b-g), where “mixed mode” failure is here intended as fractures that occurred partially at the adhesive/Ti6Al4V interface and partially inside the adhesive itself, i.e. cohesive *and* adhesive failure, with the adhesive being partially present on both surfaces. In the case of micro-slots parallel to the applied load (Figure 7 c), the adhesive is present on both fracture surfaces, thus the mechanical strength was mainly due to cohesive forces within the adhesive, without any contribution from the micro-slots. Furthermore, the fracture propagation in the parallel orientation was apparently favoured by the parallel alignment of the micro-slots to the load direction, which resulted in a lower joint strength of the bonded substrates with respect to the as received samples. Da Silva et al. (2010) observed similar behaviour, and reported a decrease of around 6 % in joint strength when the micro-slots in the bonded area were oriented parallel to the applied load. In this work, when the micro-slots are parallel to the applied load, the fracture propagation path is again rather straight, compared to the micro-slots perpendicular to the applied load (Figure 7 b), where the fracture propagation path can be seen as being sinusoidal.

However, the activity described in the paper does not consider linear grooves of different orientation (i.e. 0°, 45°, 90°), as described by Da Silva et al. (2010) for aluminium substrates. The orientation of the slots is surely of extreme importance for an improvement in joint strength, as is the geometry pattern, which is the focus of this work.

The mixed mode failure observed in the micro-machined samples is evidence of an improvement in adhesion as a result of the micro-EDM process, compared to the adhesive failure mode observed in the samples with unmodified surfaces, and is also in agreement with similar observations of Zimmerman et al (2012). Similar failure modes have been observed and reported in literature when modified titanium surfaces were bonded using adhesives, e.g. Palmieri et al (2013) observed an increase in bond strength for laser ablated Ti6Al4V samples and a predominantly cohesive failure mode in the adhesive. Figure 7 shows the fracture surfaces of specimens without any pattern and with various micro-slot geometries. The failure mode was completely adhesive for the reference joints (no machined surfaces) at the adhesive/metal interface (Figure 7 a); all the other fracture surfaces instead indicated a mixed adhesive/cohesive failure mode, (Figure 7 d, for semi-circle shaped, interlocked micro-slots). Mostly cohesive failure can be observed when the fractures in Figure 7 b are compared with those in Figure 7 c, which is a direct sign of high joint strength for a set of bonded substrates in which the micro-slots were oriented perpendicular to the applied load direction.

Micrographs of the fractured joints of representative bonded substrate specimens are shown in Figure 8; the cross-sectional image of each fracture surface is also given to highlight the adhesive failure mode of the joint. Complete adhesive joint failure (Figure 8 a) means poor adhesion of the joining material to the substrate.

The adhesive forces increased and overcame the cohesive forces, because of the increased surface area resulting from micro-slotting on the Ti6Al4Vsurfaces, and in turn led to the mixed failures shown in Figures 8 b, c and d. Mixed failure can easily be detected in the cross section of the fractured joint shown in Figure 8 d, where the adhesive fills the micro-slots, but is partially detached from the metallic surface. Figure 8d also refers to joints that were obtained using a semi-overlapped U-shaped micro-slot surface; the mixed failure mode confirms a remarkable increase in lap shear strength (+31,5%), as reported in Table 2.

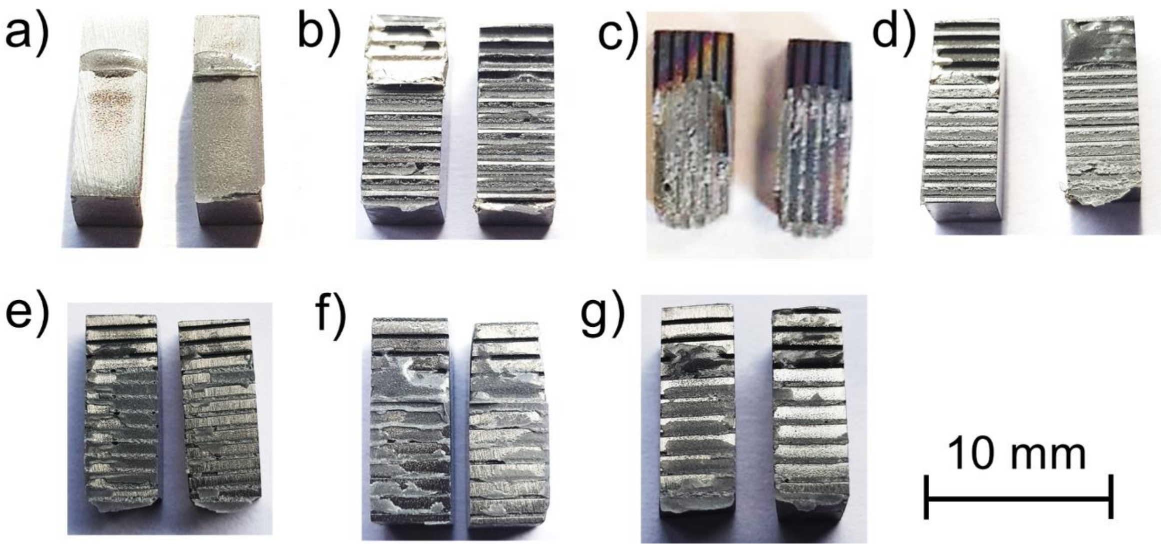


Figure 7 *Fracture surfaces of specimens bonded with adhesive; the facing surfaces were machined with different micro-slots, a) Unmodified surface joint, (b) V-shaped micro-slots, (c) V-shaped micro-slots, oriented ║to the load, (d) Interlocked semi-circle shaped micro-slots, (e) Overlapped U-shaped micro-slots, (f) Semi-overlapped U-shaped micro-slots, (g) Misaligned U-shaped micro-slots; slots oriented perpendicular to the load direction, unless otherwise indicated*

In short, it seems evident, from an observation of all the fracture modes, that the transition of pure adhesive failure mode (Figures 7 a and Figure 8 a) to mixed adhesive/cohesive mode (Figures 7 b-g and Figures 8 b-d) is due to the rough surface morphology of the micro craters within the micro-slots, which improved adhesion bonding at the metal/adhesive interface. Yukimoto et al. (2015) also found cohesive failure of micro-slotted CFRP joined by means of an epoxy adhesive, although they only observed adhesive failure in unmodified bonded surfaces.

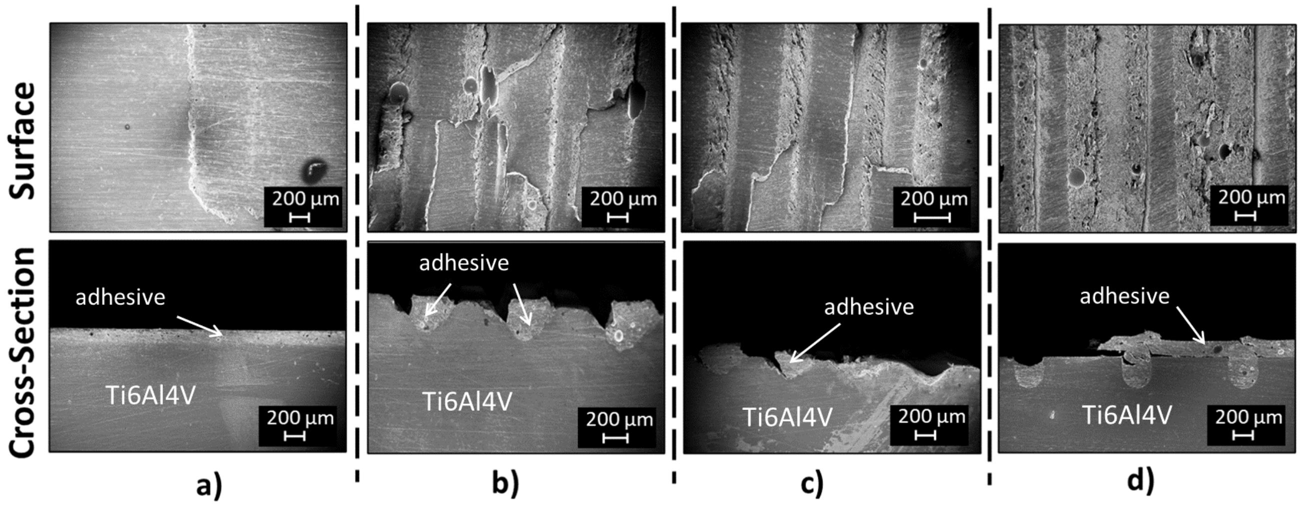


Figure 8 *Joint fracture analysis at higher magnification: top view and cross – sectional micrographs (a) Unmodified surface, (b) Semi-overlapped V-shaped micro-slot surface, (c) Interlocked semi- circle shaped micro-slot surface, (d) Semi-overlapped U-shaped micro-slot surface; all the tested samples were oriented perpendicular to the load.*

As is worth noting in the fracture surface analysis shown in Figure 8, the fracture mode is adhesive in the case of a non-machined surface (Figure 8 a), while it is mixed, i.e. adhesive and cohesive, for all the micro-machined joined samples (Figure 8 b-d). As a result of the evident improvement in bonding performance, the use of coupling agents may not be necessary after suitable micro-machining, and sample manufacturing might be simplified.

Finally, it is well known that lap tests can only be used as comparative tests for a set of samples that have the same size and test mode. However, they can offer a first approximation of the effect of joining materials and interface variation on the joining strength. The single Lap Offset results obtained in this study can be compared, in a qualitative way, with other lap shear results reported in the literature.

The method dealt with in this paper (micro-EDM) has been shown to be efficacious in increasing the mechanical strength of joints, and it can be compared with or is even better than other techniques that are normally used for surface machining purposes. Even better results are envisaged when a specific slot geometry is adopted after a suitable modelling: work is in progress in this direction.

4 Conclusions

Ti6Al4V surface machining has been carried out, by means of the Micro-EDM method, to improve the adhesion and joint strength of Ti-6Al-4V. Moreover, V-shaped, U-shaped and semi-circle micro-slots were successfully produced on Ti6Al4V surfaces using an in-house developed Micro-EDM setup. The mechanical shear test results showed an improvement or a decrease in joint strength for bonded samples with micro-slots manufactured in different ways, compared to reference (unmodified surface) specimens.

Micro-patterning of the facing surfaces has been found to be beneficial, in terms of increased contact area, but, interestingly, the mechanical test results have proved sensitive to the design, the alignment of the micro-slots with respect to the each other in a joint and to the orientation of the micro-slots with respect to the load direction, that is, perpendicular or parallel. The fracture analysis confirmed an improvement in adhesive bonding, as a result of an increase in the bonded surface area, and a mixed adhesive-cohesive fracture mode was observed for the bonded samples with micro-machined surfaces.

Further improvement to the in-house built Micro-EDM micromachining setup may lead to a cost effective and alternative solution for industries in which surface machining is employed. This technique has shown to be easily reproducible and may be easily implemented in automatic and production machinery.

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