Fatigue Crack Growth Identification in Bonded Joints by Using Carbon Nanotube Doped Adhesive Films

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ABSTRACT

An investigation on fatigue crack growth monitoring of bonded joints has been conducted employing a novel carbon nanotube (CNT) adhesive film, chosen for their proven exceptional properties for sensing purposes. Single lap shear fatigue tests have been carried out at a 7 kN load, load ratio 0.1 and 10 Hz frequency. A correlation between the electrical response of the adhesive joints and the fatigue crack growth has been established by means of voltage acquisition through the thickness in combination with crack length monitoring by microscopy analysis. Three phases can be distinguished during the fatigue tests: phase one without crack initiation with a steady electrical response; phase two in which cracks start propagating, with a slight increase of the electrical resistance, and phase three, corresponding to final propagation to failure with a drastic increase of the electrical resistance. A correlation between the electrical response, the estimated crack area and the measured edge-crack size is achieved, allowing to get a deeper understanding of crack growth phenomenon as a function of the number of cycles. Thus, the potential and the applicability of the proposed technique for monitoring crack growth in lap joint fatigue tests have been demonstrated.

1. INTRODUCTION

Nowadays, composite materials are attracting the interest of many researchers due to their exceptional mechanical properties and their corrosion resistance, which make them very competitive in comparison to conventional metallic alloys [1,2]. Research on composite structures is particularly active in the aerospace industry, where the light-weight design is a driving requirement, thus justifying the effort in developing new

materials with increasing specific properties, sometimes applied to complex mechanical systems. However, this results in complex or unknown failure modes, which can often only be identified and predicted with difficulty, especially when stress concentrations occur, e.g. in correspondence of mechanical joints. Therefore, it is necessary to develop efficient assembly techniques with a higher reliability.

When joining composites, care must be taken not to damage them nor to induce damage, by letting stresses flow smoothly across the parts to join. In the case of mechanical joints like bolts, the contact between the bolt and the inner surface of the hole can induce delamination if the contact pressure exceeds some limit values [3]. Thus, metal inserts have to be placed into the laminate and co-cured. This allows the creation of a joint that can be easily disassembled and inspected. However, the presence of the bolt and the insert contribute to increase the weight of the structure. In this context, bonded joints present many advantages over bolted connections as they avoid stress concentrations at bolt holes and do not increase the overall mass, thus increasing the potential for weight savings [4,5].

However, a specific program of off-line non-destructive inspections is mandatory in order to ensure their reliability, e.g. by means of either ultrasonic or eddy current tests, to mention a few. More recently, Structural Health Monitoring (SHM) as a technology offers the promise of reduced cost-of-ownership and increased safety of operation by a real-time inspection of critical engineering structures. SHM, in its general form, combines either local or distributed measures with damage identification algorithms in order to provide real-time anomaly detection and assessment. Local monitoring is often based on fiber optic strain sensors, leveraging on the multiplexing capabilities of the local Bragg grating sensors [6,7] and on their reduced size, which favors sensor embedding in the composite material [8] with minor effects on the mechanical performances. However, they only

provide a punctual information about the damage, making the damage inference strongly dependent on the availability of a robust process for feature identification and extraction from the raw sensor data. A quasi-distributed measure is also possible based on fiber optic sensors, exploiting the Rayleigh backscatter [9]. Differently, piezoelectric transducers are widely used for distributed SHM, e.g. for guided Lamb Waves monitoring [10-13] and acoustic emission sensing [14,15] to mention a few applications, but the interpretation of the acquired signal is often very complex, especially if realistic structures are considered, owing to the boundary wave reflections and to environmental influences.

In this framework, carbon nanoparticles such as carbon nanotubes (CNTs), showing excellent mechanical and electrical properties, are now attracting the interest of many researchers [16-19]. Their addition as reinforcement into a resin matrix leads to an enhancement of its mechanical and, especially, electrical properties [20-24]. The improvement of the electrical properties is explained by the formation of percolating networks inside the material [25-28], which causes an increase of the electrical conductivity of several orders of magnitude. This fact, their piezoresistive behaviour and the tunneling effect between adjacent nanoparticles make them very useful for SHM applications [29-37], showing much more sensitivity than other conventional SHM techniques [38,39].

In this paper, the excellent sensing characteristics of CNTs are exploited for bonded joint monitoring, specifically concerning adhesive films. Some studies are available in the literature using CNT-reinforced paste adhesives [40-43], proving the huge potential of this technology for detecting defects. In addition, it has been observed that the mechanical properties of the bonding layer are not compromised, due to a CNT-bridging effect on the adhesive [44-46]. However, if compared to paste adhesives, which require the adoption of more complex dispersion techniques such as three roll milling or toroidal

stirring [47,48], the use of adhesive films, such as those adopted in the current study, implies a higher thickness control and a generally lower distributed porosity. The effect of CNT addition on the electrical and mechanical properties of adhesive films has been rarely investigated in the literature [49]. It has been observed in previous studies [50] that CNTs provide good conductive pathways without being detrimental to the mechanical properties. Crack propagation monitoring in adhesive films has been performed by comparing the mechanical and electrical response of the joints [51] at specific instants during the tests with a static and constant load application, noticing that the electrical resistance increases with the crack length and identifying a trend during fatigue crack growth. However, to date, no studies are available in the literature regarding real-time monitoring of CNT-doped adhesive films by continuous acquisition of the CNT signals for each load cycle during the fatigue tests.

This study, thus, aims to investigate the real-time SHM capabilities of the CNT-doped adhesive films during fatigue testing. To achieve this purpose, single lap shear (SLS) specimens have been subjected to fatigue loading while their electrical response is continuously measured by means of a voltage acquisition system. In addition, the crack length has been also measured by using an optical microscope camera and the crack shape estimated after testing, based on the analysis of the fracture surface. The final target is to find a correlation between some features of the real-time electrical response of the CNT-circuit and the fatigue damage.

2. METHODS

2.1 Manufacturing of bonded joints

Adhesive joints were manufactured by secondary bonding of unidirectional CFRP substrates, supplied by Fundación para la Investigación, Desarrollo y Aplicación de Materiales Compuestos (FIDAMC-AIRBUS), by using a Multi-Walled CNT (MWCNT) reinforced adhesive film. This adhesive film is an epoxy based FM300K, supplied by Cytec, with the highest mechanical performance over the FM300 series. It has a wide open knit tricot carrier, which allows a better thickness control during curing. MWCNTs are NC7000, supplied by Nanocyl, with an average diameter of 11 nm and a length up to 2 μm .

0.1 %wt. CNTs were dispersed in distilled water. CNT dispersion was achieved by means of ultrasonication for 20 min at 80 % of amplitude and a radiation time of 0.5 s using a horn sonicator *UP400S* supplied by Hielscher. In order to induce a higher CNT disaggregation, a surfactant called sodium dodecyl sulfate (SDS) was added, in the amount of 0.25 %wt. Then, CNT dispersion was sprayed over the adhesive surface by using an airbrush at a pressure of 1 bar and at a distance of 40 cm for 0.5 s in order to achieve a high CNT homogenization into the adhesive. The curing cycle of both CNT-doped and neat adhesive joints was set as shown in Table 1 by using a hot-press. Here, pressure and temperature were applied simultaneously. In this case, the maximum pressure is applied before reaching the final curing temperature in order to promote a good adhesive flow and remove the entrapped air while curing. After curing, the CNTs were embedded through the adhesive joint as shown in the microstructural studies of previous works [51] and confirmed in FEG-SEM images of Figure 1, making the creation of electrical pathways through the joint possible.

Parameter	First Stage	Second stage
pressure	Ramp from 0 to 0.6 MPa for 15 min	0.6 MPa for 90 min

temperature

Ramp from 25 to 175 °C
for 45 min

175 °C for 60 min

Table 1: curing cycle parameters of secondary bonding.

(a)

(b)

Figure 1: FEG-SEM images from a previous work [51] (not published) showing (a) the presence of CNTs over the carrier and (b) inside the adhesive film once cured.

2.2 Electromechanical tests

Fatigue tests were carried out for the neat adhesive and CNT reinforced bonded joints using an *MTS 810* test machine. The tests were carried out under force control and the mechanical response (in terms of force and displacements) was measured simultaneously with the electrical monitoring. The peak load was set to 7 kN with a load ratio R of 0.1 at a test frequency of 10 Hz. These parameters were selected in order to obtain a **desired finite fatigue life**, according to S-N curves in the literature for similar adhesives and substrates [52] **and knowing the maximum shear force in quasi-static tests, around 20 kN according to a previous study by some of the authors [53].**

The dimensions of the bonded joints are according to the standard SLS coupons. Each substrate is $104 \times 25.6 \times 2.5 \text{ mm}^3$ with an overlapping area of $25.6 \times 25.6 \text{ mm}^2$.

The monitoring of adhesive joints was carried out by means of voltage measurement between two points through the thickness as shown in the set-up schematics of Figure 2. The electrodes, placed in the midpoint of the overlap area, were made of copper wire attached to the CFRP substrate with silver ink and sealed with an adhesive layer to protect them from the environment. The proposed electrode configuration is valid due to the large difference between the CFRP substrate and the CNT adhesive layer, in terms of electrical conductivity. According to our previous study [53], the electrical conductivity of the CFRP substrate is in the range of 2 S/m, whereas those of the CNT adhesive layer is around 10^{-3} S/m. Therefore, the contribution of the CFRP substrate to the total resistance of the joint can be considered negligible. The current was generated by using a power supply *STAB AR60* connected with an integrated circuit in order to keep the current (1 mA approximately) constant. Voltage acquisition was carried out by using a *NI 9234* module from National Instruments with a sampling frequency of 5 kHz. This set up implies a great effort for noise reduction by grounding all the connections and insulating the CFRP substrates with GFRP tabs, in order to avoid electrical interference with the testing machine. This configuration of insulating tabs and electrodes on the substrate surface is similar to that used in other studies, which analyzed sensing capabilities and crack propagation monitoring in SLS joints [54,55]. Although in a realistic scenario the noise would be inherently present in the monitored system, due to connection with adjacent components, this could hamper the joint monitoring strategy if multiple and varying electrical paths will exist, due to modification of the baseline reference signal. A possible solution to this problem can be the adoption of an insulating layer (i.e. GFRP) to avoid the interferences with other equipment, but these aspects require a deeper analysis and dedicated research. However, this is considered outside the scopes of the present activity, this being the first attempt of monitoring lap joints under fatigue by CNT

networks. Future research must be dedicated in finding electrical isolating solutions for practical implementation of the sensing technology at larger scale.

As not sufficiently precise damage identification was obtained with ultrasonic scans of the overlapping area, crack edge length was measured by optical microscopy (also adopted in the literature [56]) at different load cycles during the tests, always referring to the same reference maximum load to control crack opening. Specifically, portable optical microscope Dino-Lite AM413ZTA was used as an aid to visual testing [52] by scanning the edges of the lap joint, collecting images of the edge crack, then measuring the edge crack length by using a calibrated image-processing software with an approximated resolution of 0.5mm and correlating this dimension with the electrical resistance variation. Figure 3 shows an example of edge crack length measured on a specimen after failure of the joint. As highlighted in green, the edge crack length is intended hereafter as the perimeter of the crack that is visible from the edges of the lap joint. A direct measure of the crack area was not available during the test. Yet, an equivalent preliminary estimation is proposed later in this study, based on a correlation with the electrical resistance variation.

Four fatigue tests were carried out for the neat adhesive and for the CNT reinforced bonded joints, respectively, for a preliminary assessment of the effect of the CNT embedding on the mechanical performance of the joint, specifically concerning fatigue resistance, as reported in Section 2.3. Then, two specimens with CNT reinforced bonding were selected, named Specimen 1 and 2, and analyzed in the Results and Discussion section. They were selected in order to show two typical types of fatigue behaviour of SLS joints under repeated fatigue load and to demonstrate how their behaviour is reflected in the CNT monitoring signal. The other 2 specimens showed a similar electromechanical behaviour and relative results were omitted for the sake of brevity.

Figure 2: (a) Schematics of fatigue test set-up and (b) image of the actual configuration.

Figure 3: optical image of the fractured surface after joint failure, including the schematics of the measured edge crack length (green-line) and the crack area at joint failure (highlighted in red).

2.3 Microstructural study

Fractured surfaces were characterized by means of Scanning Electron Microscopy (SEM), using a *S-3400N* apparatus from Hitachi in order to identify the main failure mechanisms. For that purpose, lap joint fractured surfaces were cut and gold coated with a layer of approximately 10 nm thickness.

3. RESULTS AND DISCUSSION

In this section, the electromechanical behaviour of two kinds of specimens is shown and analyzed. The first one, named Specimen 1, showed a more uniform crack propagation whereas in the second one, Specimen 2, a stick-slip effect, which is an indication of unstable crack growth [51], was observed. Firstly, CNT effect on fatigue resistance has been discussed. Then, a detailed analysis of the Specimen 1 was carried out in order to understand and characterize the electrical behaviour of the joints subjected to fatigue testing. Finally, the stick-slip phenomenon was analysed in Specimen 2 to get a better understanding of the crack propagation mechanisms and to show the sensitivity of the proposed monitoring method.

3.1 Effect of CNT embedding on fatigue resistance

A preliminary investigation on the influence of CNT addition to SLS joint fatigue resistance was performed to verify any potential detrimental effect on the bonding mechanical resistance. Figure 4 shows the total number of cycles to failure and to crack initiation of both CNT doped and neat adhesives for the four specimens tested. Specifically, crack initiation is intended as the number of cycles at which the crack is first detected during the visual inspection of the joint. The average fatigue life of the CNT doped adhesive was found to be slightly higher than the neat adhesive. However, the scattering was also higher, probably due to heterogeneities in the CNT distribution along the adhesive, as reported in previous studies [50]. In fact, these heterogeneities counteract the bridging effect of the CNTs and can lead to differences in the mechanical properties of the joint. Similar results were found when analyzing the number of cycles to crack initiation. An ANOVA single factor analysis was carried out for the two adhesive

conditions in order to statistically infer the influence of the CNT on the fatigue resistance. The results suggest the addition of CNTs does not significantly affect the cycles to failure of the SLS joints, while a potential benefit exists on the number of cycles to crack initiation. It is necessary to point out that this statistical analysis is only a rough estimation of how the CNT addition potentially affects the mechanical properties of SLS joints during fatigue testing. More test repetitions are required in order to provide a more quantitative analysis.

Figure 4: (a) Number of cycles to failure and (b) crack initiation for neat and CNT doped adhesive joints.

3.2 Electrical measurements

Figure 5 shows the electrical response of the Specimen 1 during fatigue tests. Test time and number of cycles were correlated to the frequency of the load during the tests, 10 Hz. A current generator was used to keep the current constant. The changes in the normalized resistance were obtained from the voltage measurements by applying the Ohm's Law:

$$\frac{\Delta R}{R_0} = \frac{R - R_0}{R_0} = \frac{\frac{V - V_0}{I}}{\frac{V_0}{I}} = \frac{V - V_0}{V_0} \quad (1)$$

Being R_0 , V_0 , R and V the resistance and the voltage at the initial stage and at a generic time, respectively.

The electrical response of the specimens during SLS joint fatigue testing was affected by two factors. First, when a sinusoidal load is applied to the substrate, the adhesive layer is experiencing a shear deformation, which varies along the length and through the adhesive thickness. The local strain variations in a load cycle generate an average global cyclic modification of the electrical resistance, as observed in Figure 5(b), due to tunneling resistance variation. This is due to the CNT piezoresistive behaviour and the tunneling effect between adjacent particles, which leads to an approximately exponential increase of the electrical resistance with the applied strain, as previously observed for quasi-static SLS tests [51]. The non-linearity of the strain in the joint section as a function of the applied force, coupled with the non-linearity between the strain and the tunneling resistance, cause a non-sinusoidal signal from the CNT network. The signal anomaly in the valley region of Figure 5(b) can be associated to the occurrence of plastic deformation at the lap fillets (Figure 3), which reveals as some strain increases during unloading. Although this assumption is corroborated in Figure 6(c) by the progressive disappearing of the signal anomaly when the crack further evolves outside the fillets, additional research is needed to verify this hypothesis.

(a)

(b)

Figure 5: Variation of the electrical resistance as a function of time (a) for the entire fatigue test and (b) detailed at 10 consecutive cycles.

Second, the crack evolution during fatigue tests induces a variation of the electrical response, as analyzed in detail below. Figure 6 shows a detailed analysis of the electromechanical behaviour of the joints. The electrical response was correlated with the total crack edge length measured by the microscope camera through the top, bottom and lateral sides of the joint, as shown in the schematics of Figure 3, hereafter referred to as **perimeter crack length**. For that purpose, the peak and the valley trends were presented (Figure 6(a)), defined as the value of the normalized resistance at maximum and minimum force on each fatigue load cycle. A sensitivity parameter was also defined as the difference between the peak and valley within a single cycle (Figure 6(b)), provided a constant load cycle was applied by the test machine as a boundary condition. A more detailed graph of the acquired electrical signal can be observed for three different stages of the fatigue test (Figure 6(c)). In this regard, significant changes in the shape of the signal can be noticed. For this purpose, the cross-correlation, $xcorr$, of a 10-cycle signal window with respect to a baseline signal window taken at the start of the test was used in Figure 6(d) to highlight modifications of the signal shape within one load cycle. In this context, three regions were visible.

The first region reflects the steady behaviour of the electrical response. A small trend was observed in the normalized resistance as a function of the number of cycles and the sensitivity was almost constant, as shown in the region I of Figures 6(a) and (b). At this stage, no nucleation of the fatigue damage was detectable by optical microscopy (Figure 6(c)) and the sensitivity only took into account the tunneling effect caused by adhesive

deformation. The small variations of the normalized resistance and the sensitivity were caused by the presence of microcavities, which usually appear at the interface between the nano-reinforcement and the matrix before the crack starts to propagate, as stated in previous studies on nanocomposites [57], also causing the crack nucleation. Figure 6(d) showed a general decrease of the cross-correlation factor with the number of cycles due to the presence of the aforementioned microcavities.

The second region is associated to a significant variation of the electrical response. An increase of the normalized resistance was observed as a function of the number of cycles (region II of Figure 6(a)) as well as a slight variation of the sensitivity (Figure 6(b)), associated to crack initiation and propagation (Figure 6(c)). In fact, the electrical resistance increased with the **perimeter** crack length, due to the breakage of the conductive pathways. The variation of the sensitivity can be explained by observing the schematics of Figure 7. At the maximum force, the crack was totally open, while closure occurred in correspondence of the minimum load due to a lower applied stress and strain. This crack opening differences induced a higher variation of the electrical resistance within a single load cycle, leading to higher sensitivities. Therefore, the higher the crack length, the higher the variations of the electrical resistance and, thus, the sensitivity. However, the analysis of the *xcorr* evolution was unclear at this point, as shown in Figure 6(d), where the presence of peaks and valleys (region II) make the crack growth identification less straightforward. Depending on the crack propagation inside the material, this effect can lead to a partial recovery of the initial shape of the electrical curve at some points.

Finally, the third region is associated to a drastic increase of both the normalized resistance and the sensitivity as a function of the number of cycles. This is associated to the last stages of fatigue testing in which crack propagates very rapidly until final brittle failure, inducing drastic variations of the electrical response. This can be also reflected in

a sudden decrease of the *xcorr* factor in the last stages of region III, which is associated to a significant modification of the signal shape after one load cycle.

(a)

(b)

(c)

Figure 6: (a) Detailed analysis of the electromechanical response during fatigue tests in which red and blue lines correspond to the peak and the valley trends of the normalized resistance, (b) analysis of the electrical sensitivity, (c) correlation with the perimeter crack length measured by optical microscopy and (d) graph of the x_{corr} parameter as a function of the number of cycles.

Figure 7: The effect of crack opening during a single cycle of a fatigue SLS test.

3.3 Analysis of debonding area evolution

A more detailed analysis was performed to analyze the crack propagation behavior during the fatigue test, leveraging on an equivalent estimated debonding area. In practice, although not directly measured, the size of the crack area is hereafter estimated as a function of the number of cycles, based on the measure of the normalized resistance, as done in previous studies [51,58]. The bonded joint was modeled as an electrical circuit, supposing that CNTs were homogeneously distributed along the adhesive, that the current flow through the adhesive was homogeneous and assuming that the electrical conductivity of the CFRP substrates was much higher. The electrical resistance can be expressed by the second Ohm's law:

$$R = \rho \cdot \frac{l}{A} \quad (2)$$

where ρ , l and A are the electrical resistivity, the thickness and the overlap area of the bonded joint, respectively. The normalized resistance at a generic time can thus be expressed in terms of the bonded area:

$$\frac{\Delta R}{R_0} = \frac{R}{R_0} - 1 = \frac{A_0}{A} - 1 \quad (3)$$

where A_0 and A are the initial (undamaged) and actual (cracked) bonded areas, respectively. The crack area, A_d , can finally be estimated as:

$$A_d = A_0 - A \quad (4)$$

We remark that A_d has to be intended as an estimation of an equivalent area, rather than a quantification approach, due to non-homogeneity of the CNT dispersion and, thus, of the current flow through the adhesive.

Figure 8(a) shows the measured crack edge length and the estimated crack area as a function of the number of cycles. At the initial stage, previously identified as region I in Figure 6, there was a slight increase in the estimated crack area although the measured perimeter crack length remained constantly zero, as no edge crack was visible from the

microscope camera. This is explained by the presence and coalescence of microcavities inside the adhesive joints. As commented on above, these microcavities usually appear at the interfaces between the matrix and the carbon nanoparticles [57], globally affecting the electrical resistance, though without inducing visible edge cracks.

Some additional information about the crack shape was obtained by analyzing the estimated crack area as a function of the **perimeter** crack length measured at the edges of the joint (Figure 8(b)). The initial value of the estimated crack area (point 1 in the schematics of Figure 8(b) and (c)) corresponded to the total size of the internal microcavities at the end of phase I. Then, a minor increase of the estimated crack area was observed, although a significant increase of the **perimeter** crack length was measured at the edges of the joint. This means that the evolution of the debonded area took mainly place at the lap joint edges (points 2 and 3 of schematics of Figure 8(b) and (c)).

Figure 8: (a) Measured perimeter crack length and calculated crack area as a function of the number of cycles, (b) correlation between them with region 1 corresponding to the initial condition, region 2 to the first stages of crack propagation through the edges, region 3 to the propagation in both edges and inside the lap joint and region 4 to the final propagation to failure, with a crack propagation mainly inside the joint, as highlighted with the schematics in (c) (here, the red regions correspond to cracked area (A_d) and green ones correspond to actual bonded area at an specific moment of fatigue test (A)).

At higher edge-crack length levels (4), the estimated crack area increased faster, which means that the debonding was propagating inside the lap joint, as shown in the region 4 of the schematics of Figure 8(c) and also confirmed by Bernasconi et al. [59]. Finally, in the last stages of the fatigue test, the crack propagated quickly along the entire surface of the lap joint until the final failure. This reflected in a sudden increase of the damage area vs. the perimeter crack length. The fractured surface after the joint failure of Specimen 1 is visible in Figure 3, where the final crack evolution in the central region of the bonded area is clearly highlighted. These results are in good agreement with the numerical

investigation shown in other studies [60], demonstrating that higher stresses are concentrated at the edges of the lap joint where crack initiates. However, although a symmetric damage growth morphology is shown in the schematics of Figure 8(c), the non-perfectly homogeneous adhesive properties in the upper and lower fillets and the presence of randomly distributed porosity inevitably translate in a delay of the crack nucleation at the lower fillet, which can be considered limited if compared with the test duration.

3.4 Fatigue assessment based on crosshead displacement

As detailed in the Methods section, the fatigue test was executed under force control and the crosshead displacement was measured synchronously to the CNT signal. Although rigorously an extensometer is required to exactly measure the displacement of the specimen during testing, neglecting the test machine deformability, it was not applicable in this case due to the presence of electrical cables and the need for the specimen electrical isolation. However, though the measure of the crosshead displacement includes a bias related to the deformability of the test machine, it is possible to correlate any deviation from the initial condition to the presence of cracks and debonding in the joint area. In fact, by analyzing the mechanical response in Figure 9, the crosshead displacement followed a similar trend to the electrical resistance through the joint thickness, with a more rapid increase in the last stages of the fatigue test, just before failure. This is explained by the presence of fatigue damage implying a reduction of the lap joint stiffness, thus leading to higher displacement ranges for the same applied load cycle. However, by comparing the electrical response from Figures 5 and 6 to the

monitored displacement of Figure 9, the electrical resistance acquisition was evidently much more sensitive to crack growth.

Figure 9: Displacement of the specimen grips as a function of the number of cycles.

3.5 Analysis of other crack propagation mechanisms: Stick-slip case

The electromechanical behaviour of the SLS joints can significantly change depending on the crack propagation mechanisms. Figure 10 shows the electromechanical response of Specimen 2. In this case, the edge crack length followed a stick slip pattern as a function of the number of cycles. This means an unstable crack growth with sudden and softer increases, usually correlated to high test rates [61,62]. The electrical response presented clearer differences among the regions previously defined, that is, region one (I) with no crack nucleation, region two (II) in which crack starts propagating, and finally region three (III) with rapid crack propagation until final failure.

Figure 10: (a) Electromechanical response and (b) sensitivity of the stick-slip pattern specimen. Red and blue lines denote peak and valley trends.

By observing the estimated debonded area as a function of the measured perimeter crack length, the phases with reduced crack growth rate were noticeably associated to the damage evolution inside the lap joint (point 3 of Figures 11(b) and (c)), as the crack area increased rapidly. On the other hand, sharp increases of perimeter crack length were correlated to faster crack propagation through the joint edges (points 2 and 4 of Figures 11(b) and (c)). This anomalous behavior could be associated to some manufacturing defects, such as overflowing or an irregular pressure distribution during curing, as commented on in previous studies [51]. In fact, these irregularities could lead to the presence of areas with a different adhesive distribution and, thus, a non-uniform stress distribution, as detailed in the next paragraph. Therefore, a microstructural characterization of the fractured surfaces was carried out in order to have a more detailed knowledge of the fracture mechanics and the adhesive distribution. This could be correlated to the CNT signal, in order to validate the previous statements about the electrical and mechanical response of lap joints.

Figure 11: (a) Crack area and perimeter crack length as a function of the number of cycles, (b) correlation between them, and (c) schematics of the debonding evolution.

3.6 SEM analysis of specimens 1 and 2

Figure 12 shows SEM images of the fractured surfaces after the fatigue test. An irregular distribution of adhesive was observed in the case of Specimen 1, showing areas with higher (Figure 12(a)) and lower (Figure 12(b)) presence of adhesive. This effect was more prevalent in the Specimen 2, due to the stick-slip effect (Figures 12(c) and (d), respectively). As reported in a previous study [51], the regions with a lower adhesive content can be correlated to a faster crack propagation, due to manufacturing defects such

as overflowing caused by an irregular pressure distribution during the hot press compaction. In addition, a more prevalent adhesive failure was observed on the edges of the lap joint (Figures 12(e) and (f)), due to a higher stress concentration in these areas, as commented on above. Moreover, these variations in the crack growth can also be associated to the variation of the strain energy release rate with the crack length. Therefore, the analysis of the fractured surfaces is in good agreement with the measurements and theoretical analysis previously described. In this context, Figure 12(g) shows a global overview of the fractured area where the irregular adhesive distribution can be observed.

(e)

(f)

(g)

Figure 12: SEM images of the fractured areas with a higher (left) and lower (right) presence of adhesive for (a), (b) Specimen 1 and (c), (d) Specimen 2 and (e) and (f) images of the fractured surface near the edges showing the prevalent presence of substrate, indicating an adhesive failure and (g) optical image of the fractured surface with red and green areas indicating regions with lower and higher presence of adhesive, respectively, while blue areas indicate regions with adhesive failure.

4. CONCLUSIONS

Fatigue tests on SLS bonded joints using CNT reinforced adhesive films were carried out while their electrical response was measured by means of voltage acquisition. The proposed technology was proven to be able to properly monitor the crack propagation without having a detrimental effect of the mechanical properties of the joint. A clear relationship between the electrical and the mechanical response was observed. Three distinguishable regions were identified during fatigue tests: i) region one without crack nucleation and thus, approximately constant electrical resistance; ii) region two, with a slight increase of the electrical resistance associated to the first stages of crack propagation; and iii) region three, exhibiting a sudden increase of the electrical resistance due to a rapid crack propagation. In addition, by carefully analyzing the electrical response, the crack evolution inside the joint could be monitored. Cracks were shown to propagate through the edges and then inside the joint, in agreement with previous experimental and numerical studies. Stick-slip behavior was also detected by sharp increases and arrest phases and it was found to be associated to an irregular adhesive distribution along the joint. The latter was further supported by the SEM analysis of the fractured areas, which exhibited zones with higher and lower presence of adhesive.

In this paper, we demonstrated the applicability of CNT-doped adhesive films for monitoring fatigue crack growth in SLS joint. The proposed approach not only provides detailed information about crack evolution but also allows one to identify different mechanical behaviour types without having a detrimental effect on the mechanical performance, thus, showing a huge potential for SHM applications.

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