Accepted Manuscript

Real-time electrical impedance monitoring of carbon fiber–reinforced polymer laminates undergoing quasi-static indentation

Khaled Almuhammadi, Arief Yudhanto, Gilles Lubineau

PII: S0263-8223(18)32749-1

DOI: <https://doi.org/10.1016/j.compstruct.2018.09.030>

Reference: COST 10176

To appear in: *Composite Structures*

Please cite this article as: Almuhammadi, K., Yudhanto, A., Lubineau, G., Real-time electrical impedance monitoring of carbon fiber–reinforced polymer laminates undergoing quasi-static indentation, *Composite Structures* (2018), doi: <https://doi.org/10.1016/j.compstruct.2018.09.030>

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

Real-time electrical impedance monitoring of carbon fiber-reinforced polymer laminates undergoing quasi-static indentation

Khaled Almuhammadi^a, Arief Yudhanto^a, Gilles Lubineau^{a,*}

^a*King Abdullah University of Science and Technology (KAUST),
Physical Sciences and Engineering Division (PSE), COHMAS Laboratory,
Thuwal 23955-6900, Saudi Arabia*

Abstract

Laminated composites are vulnerable to damage from out-of-plane loading, particularly impact loading, and the incurred damage is often only detected by evaluating the post-impact condition of the composites. Real-time monitoring techniques are desirable for early detection of damage. Utilizing changes in the electrical properties of composites to track incurred damage is promising, but the interpretation of such measurements is still challenging. Here, an electro-mechanical system is introduced to understand how well we could detect mechanical degradation in carbon-fiber-reinforced polymer (CFRP) plates undergoing a quasi-static indentation (QSI) test, which is representative of an impact load. The system measures the in-situ, real-time changes in impedance and phase angle along the specified conductivity paths. Two different electrode configurations are proposed and tested. In all studied cases, the system effectively detected severe damage, characterized by an immediate reduction in strength, in CFRP. Using our proposed electrode configurations, we discovered that the early detection of barely visible damage strongly depends on two factors: (i) the location of the injection-measurement points with respect to the damage,

*Email address: gilles.lubineau@kaust.edu.sa

and (ii) the orientation of the measurement paths with respect to the fibers orientation in the laminated CFRP surface.

Keywords: CFRP, Electrical properties, Non-destructive testing, Structural health monitoring, Electrical impedance tomography, Quasi-static indentation.

1. Introduction

Laminated composites are used to make large structures in the aerospace, automotive, oil and gas, and civil construction industries, as well as other fields. These composite structures are inevitably subjected to impact loading. Depending on the energy level of the impact, the resulting degradation may be visible (such as penetration or perforation damage) or almost undetectable (barely-visible impact damage, BVID). In all cases, the degradation is immediately detrimental to the compressive strength and, eventually, the overall structural integrity [1]. For this reason, a structural health monitoring (SHM) method that determines the real-time damage level to the composites in-situ is highly desirable. Monitoring methods such as an ultrasonic C-scan [2, 3, 4], X-radiography, or micro-computed tomography [5, 6, 7, 8, 9] are commonly used to support SHM schemes for measuring impact damage in composites. However, these methods incur additional costs and time because they require the structures to be offline (system shut-down) and highly skilled personnel to physically inspect the structures and systems. Therefore, a user-friendly SHM method for monitoring the composite structures is still required to improve maintenance and repair procedures and to avoid unexpected catastrophic failure due to undetected degradation.

Electrical-based SHM methods have received great attention and are promising for use in composites (e.g., carbon fiber-reinforced polymer, CFRP) [10, 11, 12, 13, 14]. Since the carbon fibers in CFRP are naturally conductive and act as sensors, the change in

impedance or conductivity that occurs in CFRP can be measured to monitor the material state [10]. To this end, electrical impedance tomography (EIT) systems have been proposed to reconstruct the induced damage in CFRP by monitoring changes in conductivity using electrodes attached to a specimen [11, 15]. An EIT system was also proposed for detecting impact damage, although the signal reception was strongly influenced by the sensor location [16]. Thus far, the use of EIT systems has been limited to determining the existence or location of damage in CFRP post-mortem and invasive electrode attachment (i.e., drilling the specimen is required) [11, 12, 15, 17, 18]. In light of these limitations, there is still a need for an effective EIT system that is capable of detecting real-time damage progression, as well as operational on real structure and non-invasive for CFRP (e.g., utilizing surface electrodes, which do not require drilling of the CFRP plate).

In this work, we first developed an electro-mechanical system capable of observing changes in the electrical properties of CFRP laminate subjected to out-of-plane loading, e.g., quasi-static indentation (QSI). The system can be used in real time (it can acquire the in situ electrical parameters as the laminate is subjected to mechanical load) and offline (before and after mechanical loading) conditions. We electrically isolated the CFRP laminate from the surroundings using non-conductive plates and a ceramic indenter to apply the load. Second, we explored the feasibility of utilizing the real-time measurements of electrical impedance and phase angle from a CFRP laminate undergoing QSI loading to assess its internal damage. To achieve this purpose, we designed two electrode configurations on the surface of the CFRP specimen, a four-by-four grid pattern named Configuration-A and a more dense five-by-four pattern named Configuration-B. These configurations were specifically designed to probe the effect of the localization with respect to the damaged area covered by the injection and measurement electrodes. We also recommend best practices for the placement of the electrodes and for the interpretation of the measured signals.

2. Experiments

2.1. Materials and manufacturing

We used CFRP with toughened epoxy resin (HexPly M21/35%/268/T700GC from Hexcel Composites). The ply thickness was 0.125 mm, and the nominal fiber volume fraction was 56.9%. The lay-up was quasi-isotropic, i.e., $[0/+45/90/-45]_s$. We manufactured the laminate from compression molding (static press method, Pinette Emidecau Industries PEI 15T) as follows: (i) full vacuum was applied to the laminate stack at 1 bar to reduce voids; (ii) the applied pressure was increased to 7 bar while the temperature was simultaneously increased to 180°C and maintained for 120 min; and finally (iii) the temperature was reduced to 25°C at a 2°C/min cooling rate. The thickness of the resulting plates was 2 mm. We cut the plate to obtain 150 mm $\times$ 150 mm specimens.

2.2. Electrode preparation

The two electrode configurations, Configuration-A and Configuration-B, are shown in Fig. 1. Configuration-A contained 16 electrodes arranged in a four-by-four pattern, where the spacing between electrodes (in both horizontal and vertical directions) was 27 mm and each of the electrodes were 7 mm $\times$ 7 mm. Configuration-B was an optimized version of Configuration-A with a higher density (five-by-four) of smaller electrodes (6 mm $\times$ 6 mm) for better coverage of damage; spacing between the electrodes was 28 mm and 14 mm in the horizontal and vertical directions, respectively. Following the procedures outlined by the authors in Ref. [19], we then removed the resin from CFRP surface using Yb-fiber laser irradiation, cleaned the surface, masked it with vinyl tape and used silver paste (Electron Microscopy Sciences, USA) on the unmasked regions as the electrode material. Wires were bonded to the silver paste using a conductive silver epoxy adhesive. Upon curing of the

adhesive, the mask was removed and the sample was cleaned again using acetone. Finally, we applied epoxy resin on the electrodes as a protective layer.

Figure 1: Schematics of the electrode arrangements and actual CFRP plates for (a) Configuration-A with four-by-four electrodes, and (b) the optimized Configuration-B with five-by-four electrodes.

2.3. Quasi-static indentation test

The QSI test was performed using an electrically insulated experimental setup consisting of (i) a fixture fit into an Instron 5882 (100 kN load cell) (Fig. 2a); (ii) a clamping

system with steel and plexiglass plates (Figs. 2b-c); and (iii) a ceramic (Macor) indenter with a hemispherical tip with a diameter of 16 mm for loading and real-time electrical measurement (Fig. 2d). We also used a steel indenter to perform the QSI test on non-instrumented samples for damage analysis and to validate the QSI test results using the ceramic indenter. First, we clamped the specimen into place by tightening eight screws and applied the load at a 1.25 mm/min loading speed (ASTM D6264). We simultaneously recorded the time, force, and displacement using Instron's Bluehill software, and measured the electrical parameters described in the next section.

Figure 2: (a) Quasi-static indentation setup, (b) schematic of clamping system with metal and isolation plates (dimension in mm), (c) non-conductive, isolation plates made of plexiglass to electrically insulate the specimen from the fixture during the QSI test, and (d) ceramic (Macor) and steel indenters.

2.4. Real-time data acquisition system and measurement procedures

We performed the QSI test while measuring the impedance and phase angle using an LCR-meter (Agilent-E4980A Precision LCR meter) following the diagram shown in Fig. 3. We based the real-time data-acquisition system (DAS) on a high-speed electrical switching module and controlled the system (e.g., current injection and switching between electrodes) using LabView software. We used a four-probe method to measure the impedance. We applied alternating current (2.5 kHz, 50 mA) to the outer electrodes, and measured the electrical impedance from the inner electrodes (we prescribed a delay time of 250 ms between the current injection and the actual impedance measurement to ensure that the system reached a steady-state condition).

Figure 3: Diagram for real-time electrical monitoring system.

We followed the measurement patterns shown in Figs. 4a and b for the two configurations, Configuration-A and Configuration-B, respectively, which are also summarized below.

- Configuration-A was designed to capture the damage originating from the center in-

dentation point (cross sign) shown in Fig. 4a. Using this configuration, we performed four impedance measurements along the 0° -direction (Patterns 1-4), one along the -45° -direction (Pattern 5), four along the 90° -direction (Patterns 6-9), and one along the $+45^\circ$ -direction (Pattern 10).

- The arrangement of the electrodes was then optimized to obtain Configuration-B (a more dense mesh). We aimed at identifying the best pattern for measuring the progression of damage throughout the QSI test. We measured the impedance along the 90° -direction following Patterns B1, B2, and B3, which respectively represented three cases (Fig. 4b): (i) the damage was outside the injection and measurement points; (ii) the damage was underneath the injection points; and (iii) the damage was underneath the measurement points. Patterns B1, B2, and B3 are not aligned with the surface ply and drove the electrical current to probe internal damage deeper within the laminate [20] than when following Patterns 1-10. Finally, we also measured the impedance along the 0° -direction (Pattern B4), which represented the case where the damage was underneath the injection and measurement points.

Specimen quantity, electrode configuration, and corresponding QSI test type (monotonic or load-unload) are given in Table 1. We performed load-unload QSI tests at three displacement levels of 3 mm, 5 mm, and 7 mm. At the displacement of 7 mm, the specimen was penetrated, but not fully perforated where we could focus on the damage right after the major load drop in the force-displacement curve. In monotonic QSI test, we performed the test up to a 14 mm displacement to ensure that the specimen was completely perforated.

Figure 4: (a) Measurement patterns for Configuration-A, covering the 0° (Pattern 1-4), -45° (Pattern 5), 90° (Pattern 6-9), and +45° directions (Pattern 10). (b) Measurement patterns for Configuration-B, covering the 90° direction (B1, B2, B3) and the 0° direction (B4).

Sample type	Configuration	QSI type	Displacement (mm)	Sample quantity
Non-instrumented samples	-	Load-unload	3	3
	-	Load-unload	5	3
	-	Load-unload	7	3
	-	Monotonic	14	3
Instrumented samples	Configuration-A	Load-unload	3	3
		Load-unload	5	3
		Load-unload	7	4
		Monotonic	14	5
Instrumented samples	Configuration-B	Monotonic	14	4

Table 1: Summary of the tested samples.

2.5. Damage characterization techniques

The damaged area in the CFRP specimen was characterized using micro-computed X-ray tomography (micro-CT, Nikon XTH 225). Before performing micro-CT, we applied a diodo-methane dye penetrant (Sigma Aldrich) to the indented region and allowed 30 min for the penetrant to infiltrate the internal damage. A scanning electron microscope (SEM, Quanta 600 by FEI) was also used to observe the damage to the specimen's cross-section. We prepared the cross-section of the specimens after they were subjected to 3 mm, 5 mm, and 7 mm displacements. The specimen was cut along the longitudinal (0°), transverse (90°), and bias ($+45^\circ$, -45°) directions.

3. Results and discussion

3.1. Force-displacement curves

The representative force-displacement curves obtained from monotonic QSI tests up to $d = 14$ mm are shown in Fig. 5. The curves obtained from tests with steel and ceramic indenters are identical, suggesting that introducing the ceramic indenter instead of the conventional steel indenter during the QSI test did not modify the mechanical behavior of CFRP plates. The maximum force reached around 6000 N, after which the load dropped suddenly at a displacement of around 6 mm. The next section unveils the degradation mechanisms associated with several stages in this global loading curve.

Figure 5: Force-displacement curves of CFRP specimens loaded using either a steel or a ceramic indenter.

3.2. Damage characteristics

Fig. 6a shows the degradation (damage) in the CFRP plate as revealed by X-ray corresponding to the indenter displacements of 3 mm (A), 5 mm (B), and 7 mm (C). The

black dots displayed in Fig. 6a represent the electrode arrangement in Configuration-A. We were able to reconstruct the sequence of damage development based on SEM and X-ray; the damage sequence is summarized in Fig. 7 (for the longitudinal L-L' and the transverse T-T' cross-sections) and Fig. 8 (for bias cuts).

Figure 6: (a) Force-displacement curve and corresponding damage observed by X-ray in CFRP specimens at 3 mm, 5 mm, and 7 mm displacements. (b) SEM image of damage along the longitudinal cross-section after being indented up to 7 mm.

In the two stages of displacement before the load drop (A, $d = 3$ mm; B, $d = 5$ mm), transverse cracks appeared over a small area bounded by the four inner electrodes. Some of these transverse cracks were connected to local delaminations. Since we did not observe fiber breakage in any of the plies, the observed damage might not be directly critical for the structure. A limited number of delaminations does not necessarily result in compressive strength reduction, which is usually the main concern in impacted composite

panels. Nonetheless, detecting such barely-visible damage is still important since it is the seed of the forthcoming severe damage.

After the load drop (C, $d = 7$ mm), the damage became larger and was characterized by a much higher density of transverse cracks and extended delamination. In addition, the SEM image depicted in Fig. 6b shows that fiber breakage also occurred internally, especially in the 45° plies, which is clearly seen in the bias cuts in Fig. 8. This is the reason for the large, sudden drop in the force-displacement curve. The observed damage, i.e. extended delamination, was immediately critical for the structure as it would reduce the compressive strength of the laminate. We do not qualify such degradation as “barely-visible damage” since it could be observed during a thorough visual inspection. However, a monitoring system capable of identifying the existence of such damage in a large structure is needed for safety considerations. This is precisely a degradation mechanism where the distributed structural health monitoring is desirable.

Figure 7: Reconstruction of internal damage sequence in CFRP specimens based on SEM images under quasi-static indentation at 3 mm, 5 mm, and 7 mm displacements. The specimens were cut along the longitudinal (L-L' cross-section) and transverse (T-T' cross-section).

Figure 8: Reconstruction of internal damage sequence in CFRP specimens based on SEM images under quasi-static indentation at 7 mm displacement. The specimens were cut across the bias directions ($+45^\circ$ and -45°).

3.3. Real-time electrical measurement using Configuration-A

Configuration-A (using all prescribed patterns) did not capture any changes in the impedance or phase angle at 3 mm and 5 mm displacements (Figs. 9a and b, respectively). A change in the impedance was finally observed at 7 mm (after the load drop; Fig. 9c)

and 14 mm (after perforation; Fig. 9d), specifically using off-axis patterns (i.e. Patterns 5 to 10). Patterns 1 to 4, which were aligned with the surface ply (0°), were not able to detect changes in the impedance or phase angle over the course of loading because the damage was located outside the conductivity pathways of the surface ply (0°), and it did not intersect with the measurement patterns.

We can draw some major conclusions from the results of testing with Configuration-A. The early stages of laminates degradation were not detected using Configuration-A. Nonetheless, Configuration-A was able to monitor the sub-surface fiber failure occurring from $d = 7$ mm onward using off-axis measurement paths (not aligned with the surface-ply fiber orientation). Detecting the sub-surface fiber failure is deemed important as it may significantly reduce the structural strength. The results of present off-axis measurement using Configuration-A is consistent with the shadowing effect described in Ref. [21]. The shadowing effect occurs when the anisotropy of the electrical properties in CFRP restricts the current to flow through the surface-ply if the measurement path is parallel with the orientation of the surface-ply fibers.

Figure 9: Real-time electrical impedance and phase angle of a CFRP specimen with Configuration-A during QSI loading: (a) loading/unloading up to $d = 3$ mm, (b) loading/unloading up to $d = 5$ mm, (c) loading/unloading up to $d = 7$ mm, and (d) monotonic loading up to $d = 14$ mm.

3.4. Real-time electrical measurement using Configuration-B

The optimized Configuration-B with a higher density of electrodes improved the coverage of damage in the CFRP specimens. Results from Configuration-B (Patterns B1, B2, and B3, which measured the change in impedance and phase angle along the 90° direction) are shown in Figs. 10a, b, and c, respectively. Similar to the results from Configuration-A (Patterns 5-10, see Figs. 9c-d), Patterns B1, B2, and B3 also effectively detected the damage development, i.e., fiber breakage of the -45° plies and the associated delamination after the load drop.

Different results were achieved using these patterns depending upon the relative position of the inspection points (current injection or voltage measurement electrodes) with respect to the damage. Pattern B1 detected an increase in impedance (Fig. 10a) similar to the Configuration-A off-axis patterns, while Patterns B2 and B3 showed a branching of the impedance curves (Figs. 10b-c) when the damage was below the injection or measurement electrodes. We clarified this branching by measuring the strain utilizing an optical fiber (fiber Bragg gratings) attached to the laminate surface, and we found that the branching suggests that B2 and B3 were able to detect asymmetrical damage, as evidenced by a schematic reconstructed based on SEM analysis (Fig. 8). Such asymmetrical damage alters the trend of conductivity and causes the change in the electrical impedance.

Then, we used Pattern B4 along the 0° direction to observe changes in the impedance and phase angle. We detected an increase in the impedance before the load drop (Fig. 10d), though we did not observe a change in the phase angle during the test. The impedance started to increase when the displacement reached around 5 mm, when only transverse cracks and local delamination appeared. Pattern B4 was effective enough to capture fiber breakage localized below the indenter during out-of-plane loading.

Figure 10: Real-time electrical impedance and phase angle of a CFRP specimen with Configuration-B during QSI loading: (a) Pattern B1, (b) Pattern B2, (c) Pattern B3, and (d) Pattern B4.

4. Conclusions

We developed a real-time monitoring system for characterizing the impedance and phase angle of carbon fiber-reinforced polymer (CFRP) under quasi-static indentation (QSI) loading. The CFRP specimens were fully isolated from the environment during testing by an insulating clamping system and a ceramic indenter. We studied two configurations of electrodes in order to understand the detection capability of the developed system with respect to the real-time damage progression. We reconstructed the damage sequence in CFRP composites under QSI loading using images obtained from micro-computed tomography and a scanning electron microscope. Our investigation shows that the system effectively detected sub-surface damage provided, firstly, that the measurement electrodes cover the damaged region and, secondly, that the orientation of the monitoring path is not oriented along the direction of the surface-ply fibers. The second condition is necessary to ensure that sufficient excitation signal penetration occurs in the laminates, so that the damaged plies can be effectively monitored in detail. In addition, asymmetrical damage was detected using our proposed electrode configuration, as indicated by the branching in the impedance results. Future work is still necessary to pursue a systematic analysis of the impedance data.

5. Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

Acknowledgments

This research was supported by King Abdullah University of Science and Technology (KAUST) Baseline Research Funds under award number BAS/1/1315-01-01, and by Col-

laborative Research Grants under award number URF/1/2281-01-01. The authors would like to thank Mr. Meshal Abdulkareem (KAUST Core Lab) for his assistance in building the data acquisition system; Ms. Ohoud Alharbi (KAUST Core Lab) and Mr. Ran Tao (COHMAS) for their assistance in SEM imaging.

References

- [1] M. O. W. Richardson and M. J. Wisheart. Review of low-velocity impact properties of composite materials. *Composites Part A: Applied Science and Manufacturing*, 27A:1123–1131, 1996.
- [2] C. Scarponi, G. Briotti. Ultrasonic technique for the evaluation of delamination on cfrp, gfrp, kfrp composite materials. *Composites Part B: Engineering*, 31:237–243, 2000.
- [3] M. Perez, L. Gil, S. Oller. Non-destructive testing evaluation of low velocity impact damage in carbon fiber reinforced laminated composites. *Ultragarsas*, 66(2), 2011.
- [4] F. Aymerich, S. Meili. Ultrasonic evaluation of matrix damage in impacted composite laminates. *Composites Part B: Engineering*, 31:1–6, 2000.
- [5] W. Cantwell, J. Morton. Detection of impact damage in cfrp laminates. *Composite Structures*, 3:241–257, 1985.
- [6] D. J. Bull, L. Helfen, I. Sinclair, S. M. Spearing, and T. Baumbach. A comparison of multi-scale 3D X-ray tomographic inspection techniques for assessing carbon fibre composite impact damage. *Composites Science and Technology*, 75:55–61, 2013.
- [7] D. J. Bull, S. M. Spearing, I. Sinclair, and L. Helfen. Three-dimensional assessment of low velocity impact damage in particle toughened composite laminates using

- micro-focus X-ray computed tomography and synchrotron radiation laminography. *Composites Part A*, 52:62–69, 2013.
- [8] V. Crupi, G. Epasto, and E. Guglielmino. Internal Damage Investigation of Composites Subjected to Low-Velocity Impact. *Experimental Techniques, SEM*, pages 1–14, 2014.
- [9] S. Garcea, Y. Wang, P. Withers. X-ray computed tomography of polymer composites. *Composites Science and Technology*, 156:305–319, 2018.
- [10] K. Schulte and Ch. Baron. Load and failure analyses of cfrp laminates by means of electrical resistivity measurements. *Composites Science and Technology*, 36(1):63 – 76, 1989.
- [11] R. Schueler, S. Joshia, K. Schulte. Damage detection in cfrp by electrical conductivity mapping. *Composites Science and Technology*, 61:921–930, 2001.
- [12] N. Angelidis, N. Khemiri, P. Irving. Experimental and finite element study of the electrical potential technique for damage detection in CFRP laminates. *Smart Materials and Structures*, 14(1):147–154, 2005.
- [13] A. Todoroki, K. Omagari. Detection of Matrix Crack Density of CFRP using an Electrical Potential Change Method with Multiple Probes. *Journal of Solid Mechanics and Materials Engineering*, 2(6):718–729, 2008.
- [14] T. Swait, F. Jones, S. Hayes. A practical structural health monitoring system for carbon fibre reinforced composite based on electrical resistance. *Composites Science and Technology*, 72(13):1515–1523, 2012.

- [15] A. Baltopoulos, A. Vavouliotis, V. Kostopoulos, N. Polydorides, L. Pambaguian. *Emerging Technologies in Non-destructive Testing V*, chapter Electrical tomography as a tool for non-destructive assessment of composite structures, pages 389–394. CRC Press, 2012.
- [16] J. Pohl, S. Herold, G. Mook, F. Michel. Damage detection in smart cfrp composites using impedance spectroscopy. *Smart Materials and Structures*, 10(4):834–842, 2001.
- [17] S. Nonn, M. Schagerl, Y. Zhao, S. Gschossmann, C. Kralovec. Application of electrical impedance tomography to an anisotropic carbon fiber-reinforced polymer composite laminate for damage localization. *Composites Science and Technology*, 160:231–236, 2018.
- [18] S. Wang, D.D.L. Chung, J. Chung. Impact damage of carbon fiber polymer–matrix composites, studied by electrical resistance measurement. *Composites Part A: Applied Science and Manufacturing*, 36(12):1707–1715, 2005.
- [19] K. Almuhammadi, L. Selvakumaran, M. Alfano, Y. Yang, T. K. Bera, G. Lubineau. Laser-based surface preparation of composite laminates leads to improved electrodes for electrical measurements. *Applied Surface Science*, 359:388–397, 2015.
- [20] K. Almuhammadi, T. K. Bera, G. Lubineau. Electrical impedance spectroscopy for measuring the impedance response of carbon-fiber-reinforced polymer composite laminates. *Composite Structures*, 168:510–521, 2017.
- [21] L. Selvakumaran, Q. Long, S. Prudhomme, G. Lubineau. On the detectability of transverse cracks in laminated composites using electrical potential change measurements. *Composite Structures*, 2014.