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SENSING CAPABILITIES OF MULTIFUNCTIONAL COMPOSITE MATERIALS USING CARBON NANOTUBES

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Multifunctional composite materials are a rising trend of the past decade in the aerospace world, as they can offer mass reduction to the final design. Nanotechnology, on the other hand, has helped significantly towards the development of such materials by providing nano-scale materials with exceptional property combinations (e.g. carbon nano-tubes (CNT)) that can greatly affect the macro-scale properties and enable new functionalities when incorporated inside a composite material. One of these functionalities is sensing. Self-sensing, being a sub-field of sensing, is one of the functionalities with a high potential of being integrated into future structures. Until now self-sensing concepts in composites have been able to address issues related to the existence and evolution of the damage. A remaining challenge is the localization of damage and even further the evaluation of its shape and extent. To address this limitation, this study proposes the use of the Electric Resistance Tomography (ERT) technique. To implement this technique, we developed an ERT system for the measurement of voltage at multiple locations of the materials. In this work, the technique was applied to inherently conductive CNT reinforced Glass Fiber Reinforced Polymers (GFRP). ERT measurements were collected before and after the damage was introduced. Differences in the measurements due to the damage were recorded. These measurements were used for the estimation of conductivity maps. A correlation between the damage location and its size with the estimated conductivity maps was attempted. The results are very encouraging for further evaluation as the technique was capable of locating a defect less than 0.1% of total area with a relatively small error. Throughout this work, we report on the developments and challenges as well as on the prospects of the electrical tomography concept.

I. INTRODUCTION & REVIEW

I.I Multi-functionality of structures

The incorporation of multiple functionalities into structures has been a trend in research due to the demand for lighter and more advanced structures.[1] Sensing is one of the functionalities commonly investigated for incorporation in structures to avoid the addition of external sensors to the systems.

Sensing refers to an inherent capability of a system to identify a stimulus or change in state and "reflect" it consistently in another measurable property (e.g. temperature sensing through change in electrical resistance). To achieve this, novel materials and technologies need to be developed in parallel, to develop a smart system. [2]

Complementary to the above, Non-Destructive Testing (NDT) and Inspection (NDI) are concerned with the detection of flaws in a material or structure without damaging or interfering with its basic functionality. Various types of structural damage may occur, including crack initiation, crack propagation, delaminations etc due to fatigue, impact damage, debris, micrometeorite impact and others. In some space configurations, manual inspection is not feasible over the operating life of the structure; therefore an on-line monitoring system would make sense. Where it is feasible to inspect, the availability of information from different systems is very important for detecting and evaluating a damage.

Combining these ideas, structural materials with inherent sensing-NDI capabilities seem to be a reasonable approach and therefore have been a field of research interest within the past years. However, relevant works have been limited on coupon level and works on components have not been reported to the knowledge of the authors.

One possible application of such smart systems is in Reusable Launch Vehicles (RLV).[3] For these applications, the overall aims are to ensure safety, reduce turn-round times between flights, focus maintenance operations to reduce costs, and allow reusability. With these systems in-flight, real time monitoring of a structure could be achieved while on-ground verification could be supported.

Throughout this work sensing is used to describe the identification of a material's health state. Sensing of other stimuli has not been considered. Additionally, any reference to the term damage is considered as anything that disrupts the continuity of the material and can effectively decrease the load bearing capability of the structure (even though the structural part is not assessed in this work).

I.II Carbon NanoTubes for Sensing & NDI

The discovery of carbon nanotubes and the developments in nanotechnology have helped toward the achievement of novel materials with potentially tailorable material properties.

The use of conductive carbon fibre composite materials as self-sensors has been proposed nearly two decades ago[4,5] and the developments in the field were reviewed recently.[6] The concept is based on the assumption of a direct relation between conductivity and health or loading state of a specimen. Any change in the material's health/functional state (e.g. cracking) will affect the apparent conductivity/resistivity of the system. The conductivity value as well as its evolution with time can give important information about the system under monitoring.

The same concept was evaluated in various studies, in which conductive composites have been developed, either through dispersion of conductive fillers[7,8,9] or impregnation of nano-filler preforms[10,11]. These studies indicated the potential use of these materials as sensors for structural health monitoring with high potential to tailor their sensing performance. In addition, the developed materials exhibited enhancement in the mechanical performance of the polymer matrix (e.g. increase in Young's Modulus).

Ultimately, nano-polymers targeting structural applications are to be used as matrices for fibrous composites. When this idea was established and tested, apart from the change in the mechanical and thermal properties CNT would offer, they enabled new integrated functionalities. It has been shown that CNT can enable load and health monitoring of previously non-conductive systems (such as glass-fibre composites) under different loading conditions, both dynamic[12] and static[13,14]. Improvement of damage identification sensitivity of CFRP which were already conductive before the addition of CNT has also been reported[15].

I.III Motivation for Electrical Tomography (ET)

All the aforementioned works and others with CNT-Polymers or CNT-FRP have dealt with a 1D measurement approach, which was to monitor an electrical value (mostly resistance) as expressed between two points or several points in linear positioning. Any variation in the measured value was attempted to be correlated with the input/stimulus of the system. All these works targeted the detection of any damage. Some of them could also provide a rough prediction of where the damage was or of remaining life or extend of damage. However, for locating the damage they required the electrodes to be covering the damage area. Therefore the challenge still exists to locate the damage and assess it in terms of size, severity etc in a more global and structured way.

Locating the damage in a composite structure using electrical fields gave the motivation to apply the Electrical Tomography (ET). It has been proposed in an initial work[16], but has not been fully applied and elaborated in composites. Driven by simplicity in development and applicability, we decided to apply Electrical Resistance Tomography (ERT). The basis and background of ET are discussed in the following paragraph.

I.IV Description of Work

In this paper, we report on the application of ERT for detecting and locating damage in composite materials. We assessed the feasibility to apply the method to structural materials and try to identify the critical parameters that enable its application. We applied the method to inherently conductive Glass Fiber Reinforced Carbon Nanotube Polymers (CNT-GFRP). Implementation issues and difficulties are discussed throughout, along with challenges and development recommendations.

II. ET: THEORY AND APPLICATIONS

The scientific origin of the technique is in geophysics. ERT has been employed for year to evaluate the presence of subsurface water and oil. Other applications of electrical tomography techniques are found lately in the medical sector (e.g. pulmonary monitoring).

A conceptual diagram of ET process can be seen in **Fig. 1.** Three major parts can be identified; the system under test, the measurement-current unit and the software/handling unit.

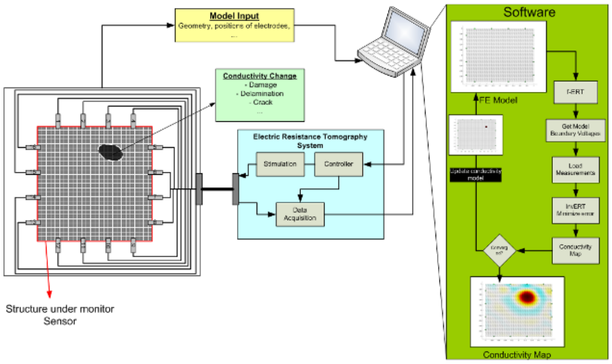

Figure 1 - Electrical Tomography concept: the material-structure under test (left), the measurement unit (middle-blue) and the software/handling unit (right-green).

The idea of ET is to non-invasively assess a system and thus to take measurements on the boundary of it and estimate the internal distribution of its electrical properties. To achieve this, electrodes should be placed on the periphery of the system under test. Current is injected between two electrodes and voltage is measured on the rest of the electrodes. Then the current is injected between another couple of electrodes and the voltage is measured etc following a specific strategy (see section III.III).

For a system with known internal distribution of properties (e.g. conductivity), by injecting a current between two points, one can calculate the voltages expected on the electrodes. This is referred to as the "Forward Problem". Any change in the internal distribution of the material's electrical properties, will be expressed through a change in the voltage measurements.

The Forward Problem can be described by the following formulation[17,18] referred to as complete electrode model:

$$\nabla \cdot (\sigma \nabla u) = 0 \, , \, \bar{r} \in \Omega \quad \textbf{[1]}$$

With the following boundary conditions:

$$\int_{e_\ell} \sigma \frac{\partial u}{\partial \bar{n}} dS = I_\ell \, , \bar{r} \in e_\ell \, , \ell = 1,..,L \quad \textbf{[2]}$$

$$\sigma \frac{\partial u}{\partial \bar{n}} = 0 \, , \, r \in \partial\Omega \setminus U_{\ell=1}^{L} e_\ell \quad \textbf{[3]}$$

$$u + \zeta_\ell \, \sigma \frac{\partial u}{\partial \bar{n}} = U_\ell \, , \, \bar{r} \in e_\ell \, , \ell = 1,..,L \quad \textbf{[4]}$$

Where: σ-conductivity, r-spatial coordinates, u-electr.potential inside the body, U_i-electr.potential under electrode I, ζ_i-contact impedance of electrode i, n-outward unit normal.

In addition, conservation of charge and potential reference level equal to zero also applies.

The estimation of the internal distribution based on the expressed changes in voltage measurements is the "Inverse Problem". Utilizing the experimental measurements and specialized mathematical techniques, one can estimate the distribution of the system's properties. Thus it is possible to identify where the changes in conductivity are, with a respective level of confidence. Although this might seem trivial, it is a big challenge and it is not always guaranteed that there can be a solution, as the problem is an ill-posed inverse problem. The solution of the inverse therefore required calculation techniques available by EIDORS[19].

III. EXPERIMENTAL

III.I Manufacturing of CNT-GFRP and specimen preparation

The material used in this study was a glass fibre composite with a 0.5%wt MWCNT reinforced epoxy matrix. The addition of CNT was done to achieve a conductive FRP. Details on the manufacturing process can be found in works[7,15].

For the quality assessment of the plate, ultrasonic scanning (C-scan) was used. No flaws were detected. The final composite had a fibre volume fraction of 42%. The final thickness of the manufactured plate was 2.5mm. From the plate, square 10cm specimens were cut.

The specimens were prepared for ERT by attaching electrodes on its periphery. For this process, a preparation protocol was established. Holes of 1mm diameter (20 in total) were drilled at evenly spaced points at 3mm distance from the boundary. The inside of each hole was painted with silver-paint to provide a good conductive interface. A commercially available two part conductive epoxy (supplied by Circuit Works) was used to position and hold the electrode cables in place. The epoxy was cured for 4hrs at 55^{o}C. In total, 20 electrodes were attached on each plate, as shown in the close-up of Fig. 2.

III.II ERT Setup development

To apply the process, a tomography system was developed. Based on a bibliographic review, a simple system was built. The final setup, shown in Fig. 2 together with a specimen close-up, consists of a direct current source, a switching unit (multiplexer) with an internal digital multi-metre and a controlling and post-processing laptop unit.

For this study, opposite current drive was chosen. Voltage on each electrode was measured with reference to the ground electrode of each pattern.

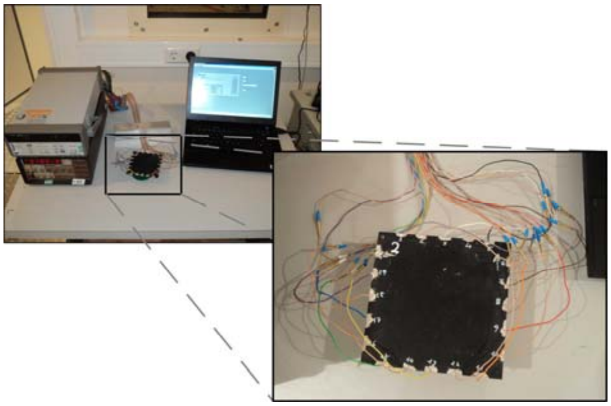

Figure 2 - Photo of the ERT setup and close-up of the specimen under test.

On the application side, setting different measurement parameters with the current system the duration of a full scan (200 measurements) can be from 40sec to 6min. This can be minimized based on the various strategies of measurement available (e.g. taking directly the 160 measurements and not excluding them later). For the present study, the speed of measurement was not a guiding parameter as the goal was the comparison of two different states and not the progressive monitoring of a phenomenon or process.

<u>III.III Work Approach</u>

The work approach followed was a parallel one between ERT system setup and simulation implementation. A calibration of the two was performed at a later stage.

On the experimental part, once the specimens were prepared, a characterization of the assembled system took place by performing a noise analysis. This was performed in order to assess the level of noise in the measurements and its statistical behaviour. The result of the analysis was the normality evaluation of the measurements of each electrode. All electrode measurements followed a Gaussian distribution. The mean standard deviation of the non-current bearing electrodes was in the range of 10^{-4}V.

On the simulation part, a FE Model of the specimen was developed using NetGen and MATLAB. An electrically isotropic and homogeneous material was assumed. Initially, two models were formulated; a 2D and a 3D. The equivalence of the two was evaluated and it was decided that the 2D was to be used for this work due to less computational demands.

A calibration process took place in order to deliver a representative FE model of the system. This process is described in paragraph IV.I.

Once the model and the experimental setup were in agreement, the experimental procedure started.

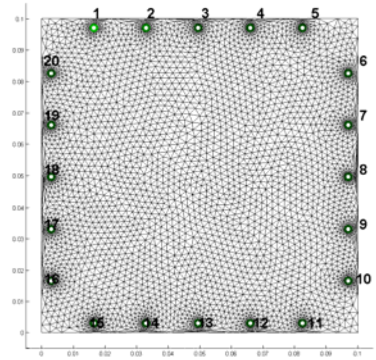

Figure 3 - Finite Element Model: 4154 Nodes, 7982 Elements, Electrodes are in green and are numbered clockwise.

The ET procedure used in the study is as follows: current is injected into one electrode (e.g. electrode 1) and leaves the system from the opposite one (e.g. electrode 11) which is grounded; this is referred to as a current pattern (i.e. 1-11). While the current is injected, voltage measurements are taken for all the electrodes (i.e. 1,2,3,...). Once all measurements are taken, the next current pattern is applied (i.e. 2-12) and the respective measurements are taken. Ideally, this continues until all 20 current patterns have been applied and the 20x20=400 measurements have been taken. However, not all measurements are useful, in the sense that they do not contribute new information. Due to reciprocity, half of them are redundant, leaving 200 available. It is common practise to exclude any measurements taken from the current carrying electrodes due to uncertainties in the measurement. This leaves 18 measurements per current pattern, but because adjacent differential measurements are finally used, in the end 16 measurements are available. This leaves a total of 16x10=160measurements to be used for the inverse calculation.

For the damage, it was important to know the position, size and shape of the change introduced to the material. This would allow us to evaluate the performance of the technique. For this reason, it was decided that a drilled hole was to be the discontinuity and act as the simulated "damage".

Holes of different sizes (having diameters: 1, 1.8 and 3mm) were drilled successively to the specimen at a chosen point (X_{damage},Y_{damage})=(6cm, 6cm). Between each change in the material, a set of 30 frames was taken. At a later stage another hole of 3mm was drilled at a different location with coordinates (4cm, 2.2cm).

For each state, a total of 6000 measurements were taken (30 frames each having 200 measurements). From these, the mean values and standard deviations were calculated for each electrode. The mean values were in turn used to calculate the adjacent voltage differences. At the final stage of the measurement processing, the selection of the "useful" values was performed to form the final vector to be used for the inverse calculation.

Before performing any attempt for an inverse calculation, the gathered data were evaluated to see if any change or indication could already be identified. As a first step the differences between the voltage profiles for the undamaged and damaged state were calculated. Then the adjacent voltages were compared.

Having the experimental measurements of both the un-damaged and the damaged specimen, a series of approaches to solve the Inverse Problem took place utilizing different available techniques[20].

The forward and inverse problem was solved using EIDORS routines. Visualization of the models and the results were performed using MATLAB and MayaVi.

IV. RESULTS AND DISCUSSION

IV.I Forward problem and measurements

The first part of the work was to solve the Forward Problem; inject current and assuming a given conductivity, try to estimate/calculate the experimental measurements. For this, the FE model was used and experimental measurements were taken.

For the model, we began by defining the "given" conductivity. From previous studies, such materials exhibited conductivities in the 10^{-4}S/m. The exact value of it had to be determined in order to be used in the forward model simulations. For this, a two way approach was followed; an experimental and a numerical.

For the experimental, a series of measurements was performed to produce V-I curves for the material using different electrode sets, under controlled current. To account for and evaluate any anisotropic behaviour of the material (in terms of electrical properties), the electrode sets were chosen to be in different directions. Four sets, indicated in parentheses, were chosen to express the basic angles, indicated in brackets; (3-13)-[0°], (6-16)-[-45°], (8-18)-[90°], (10-20)-[+45°]. From the curves we could extract the apparent conductivity of the material. Apart from the set 3-13, the values obtained did not exhibit large deviation. The deviation of set 3-13 is attributed to material inhomogeneity (e.g. locally non-uniform CNT dispersion) induced during manufacturing. Nevertheless the value is in the same range (10^{-4}S/m) and the difference to the other values is in the level 18%. The mean value was calculated to be 4.89e-4 S/m.

For the numerical approach, a least square error fit between experimental measurements and simulations for unit conductivity was performed. Through this we calculated a value with which the unit conductivity was to be multiplied to best fit the experimental results. The optimal conductivity value was calculated to be 4.479e-4 S/m. This value is very close to the observations made in the experimental approach described before.

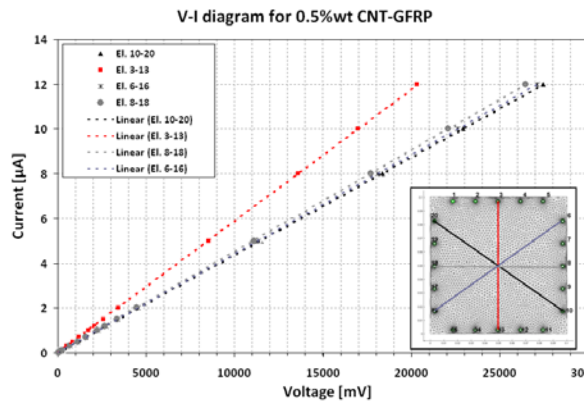

Figure 4 – Experimental I-V curves of the specimen for different electrode sets.

Having these values in hand, the forward solution could be performed and a vector of simulated measurements calculated. The differences in the simulated measurements were negligible between the two derived values. Fig. 5 shows a simulated voltage distribution inside the specimen for a random stimulation set (current injected in electrode 4, ground connected to electrode 14).

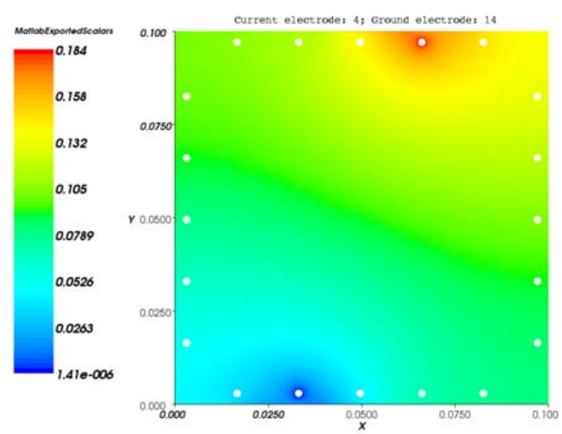

Figure 5 – Voltage distribution of CNT-GFRP: FE model results (CUR:4 – GND:14).

From this calculation a voltage profile corresponding to the simulated measurements can be extracted. The voltage profile consists of the measurements at all the electrodes and can be imagined as the voltage curve along a clockwise line connecting the electrodes. This was directly compared to the experimental measurements taken and are shown in Fig.6.As it can be seen, differences between the numerical model and the experimental measurements exist. These deviations are attributed to material inhomogeneity; regions having locally different conductivity. This is contrary to the assumption of an isotropic and homogeneous material for the model.

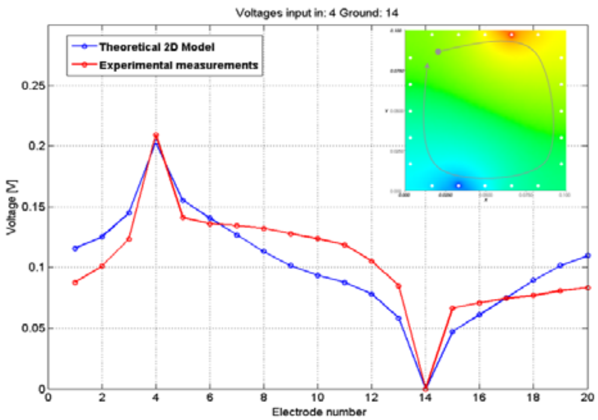

Figure 6 –Voltage profile of CNT-GFRP: Theory Vs. Experimental (CUR:4 – GND:14).

Electrical homogeneity of nano-polymers may be assumed, but it cannot be verified by using some established experimental technique. Manufacturing parameters affect the formation of the CNT network and can lead to local differences in apparent macro-scale conductivity.

The observations made vitiate the homogeneity assumption. Nevertheless, this information can already be used to get indications for our system (e.g. to locate the area of the in-homogeneity). Additionally, having a first "signature" profile of the real material is important as it will be used as reference for the inverse problem.

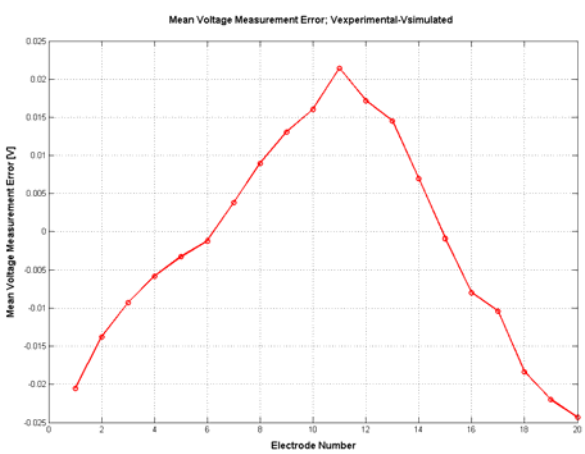

Figure 7 – Mean voltage measurement error for each electrode.

From the plot of the mean measurement error for each electrode (experimental minus the simulated) in Fig.7, it can be seen that the model underestimates the measurements from electrodes 6-15 while it overestimates the measurements for the rest of the electrodes. These differences cannot be attributed to preparation parameters (e.g. contact resistance from conductive glue) as the influence would be more stochastic and not as the one shown in Fig.7. This behaviour might give an indication of where the inhomogeneity extends in the specimen. The inhomogeneity most probably is located at the lower part of the specimen as defined by a straight line connecting electrodes 6 and 15 (Fig.3). As the experimental voltage values are higher, this area should have lower conductivity (higher resistivity). This would result in inhomogeneous current distribution through the specimen area; less current passing through this low conductivity area and more being directed to the rest of the specimen.

Following this as described before, the differences between adjacent electrodes were calculated (Fig.8). These values, excluding the current and ground electrode values, are used for the inverse solution.

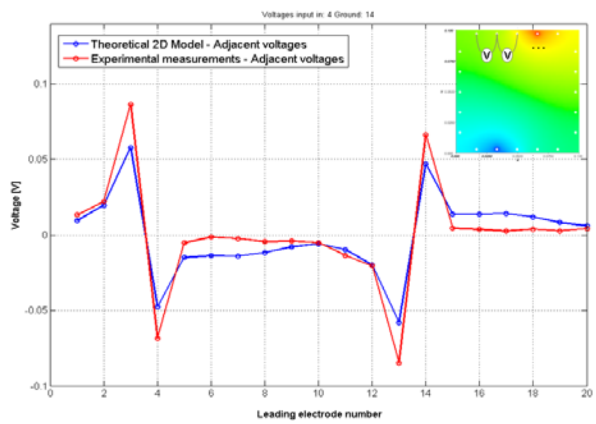

Figure 8 – The profile of fig. 6 expressed in adjacent voltage measurements of CNT-GFRP: Theory Vs. Experimental (CUR:4 – GND:14).

<u>IV.II Measurements on damaged materials</u>

In Figure 9, the difference between the simulated measurements vector of the undamaged and the damaged case (1mm hole@(6cm,6cm)) is presented. These are the differences expected to be recorded by the measurement system for an accurate reconstruction. For this specific case, the differences are in the range of 10^{-4}V. This gives an indication of the required accuracy and precision that the measuring system should have.

As all experimental measurements are subject to some level of noise, this should be taken into consideration for the selection and design of the whole setup. The level of noise plays an important role in the confidence of the reconstruction as well as the resolution of the estimation.

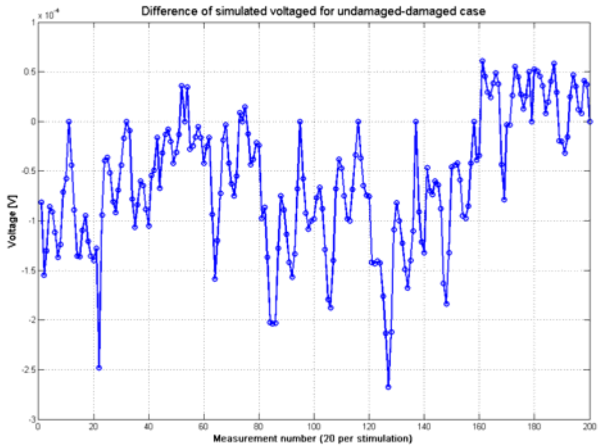

Figure 9 – Differences in simulated voltage measurements of CNT-GFRP: Undamaged – Damaged; for a hole D=1mm corresponding to 0.0079% of the total specimen area.

Figure 10 presents the difference between the adjacent experimental measurement vectors of the undamaged and the damaged case. The damage in this case is a 3mm hole at the (4cm, 2.2cm) position. Measurements from electrodes 4-10 show no change. The differences are notable and more expressed for the measurements recorded by the electrodes closer to the damage (11-16).

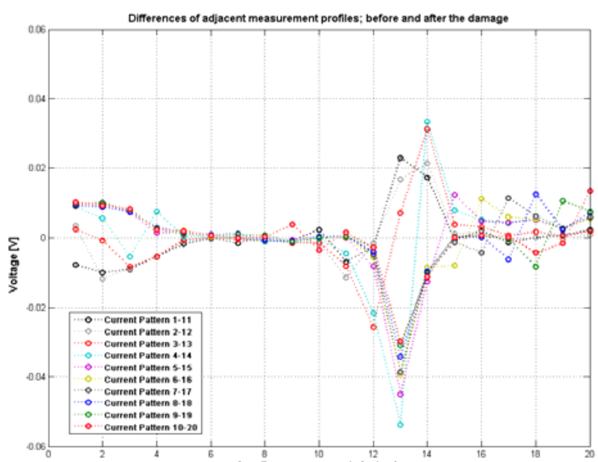

Figure 10 – Differences in experimental voltage measurements of CNT-GFRP: Undamaged – Damaged; for a hole D=3mm at (4cm, 2.2cm) corresponding to 0.0707% of the total specimen area.

IV.III Conductivity maps: the estimations of the inverse solution

With the conductivity estimation maps, we aim at gaining a qualitative assessment of the system rather than a quantitative. Therefore we will focus on the evaluation of the maps in terms of increase/decrease indication and the location and extend of the change estimation.

In principle, it could be possible to identify any inhomogeneity in the virgin-undamaged material using the theoretical measurement against the experimental measurements acquired for the undamaged system. For this reconstruction however, no information is available on where the inhomogeneity is, even though some preliminary observations were made (see paragraph IV.I). Thus the reconstruction could not have been evaluated. Therefore it was decided not to perform any attempt for the undamaged system.

The experimental measurements, before and after the damage was introduced, were used as input for the inverse solution. With this, estimating the location of the damage was targeted.

For the holes drilled at a centrally located point (6cm, 6cm), the estimation maps did not give any clear pattern and therefore correlation with the real position of the hole were not evaluated further.

When the hole was drilled in a non-central zone, changes were more evident in terms of estimation maps. For the 3mm off-centre hole at (4cm, 2.2cm), the estimation map is shown in Fig.11.

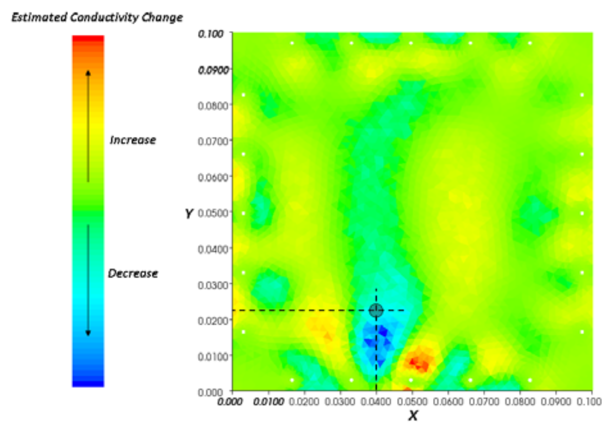

Figure 11 – Estimated conductivity change map for the CNT-GFRP specimen with a hole drilled @(0.04m, 0.022m) .

From observing this map, the reader's interest is attracted in the box area between X=(0.02, 0.06) and Y=(0, 0.03). Small variations of conductivity are estimated throughout the area of the specimen. However these are in the range of the mean change which is very small. Therefore our focus is set on the large estimated magnitude changes located in the aforementioned box area. According to the map, a peak decrease at (4.1cm, 1.4cm) should be expected. The shape of this decrease is elliptical in the Y axis, with a 1cm major axis and 0.6cm minor axis. Additionally, a peak increase with a circular shape is also expected at (5.2cm, 0.83cm).

With the real hole being located at (4cm, 2.2cm), in terms of anomaly detection a general agreement between the real specimen and the map exists.

Comparing the real defect to the estimated map, correlation on the location of the negative peak, especially on the X component, is evident (real: 4cm, estimated: 4.1cm). For the Y component, the real value is 2.2cm while the estimated is 1.4cm. The error is around 8% (0.8cm off in a 10cm overall specimen dimension).

From inverse solution theory[21], the reconstruction of a hole in ERT should appear as a dipole source. The combination of the aforementioned closely located opposite peaks indicates such a dipole source behaviour. This dipole source can be located at (0.045, 0.01), between the two peaks. When comparing the real defect position to the dipole source position, the error is 15%; 1.5cm in a 10cm.

It can be concluded that with the current design and parameters, the technique is capable of locating relatively small defects (large conductivity change less than 0.1% of the total area). It is more sensitive to defects located closer to an electrode. Thus centrally located defects may not be identifiable.

V. CONCLUSIONS

An online health/damage monitoring technique for conductive composites has been proposed, developed and presented in this paper. The technique utilizes electrical fields to sense variations in the system under measurement. It does not require access to the whole systems; electrodes are only placed on the periphery. Conceptually, using mathematical techniques and the experimental measurements, one can estimate the existence and location of conductivity changes, which in turn may be correlated to damage.

The proposed technique was applied to inherently conductive CNT-GFRP. A detailed preparation procedure for composites was developed. For the first time, a structured tomographic measurement protocol is applied to this type of composites. Electrical tomographic measurements on CNT doped fibrous composites are collected and reported. A FE model of the material system has been developed to predict its forward electrical behaviour. The agreement between the computational model and the experimental values is encouraging. Differences between the measurements of the undamaged and the damaged case were notable. Estimations of conductivity maps were successful in indicating the area where the damage was introduced. The errors of these maps are relatively small, considering that it is the first application of the technique and no optimization (e.g. current injection strategy) took place. The technique showed sensitivity to damage closer to the electrodes.

It has been identified that among the important parameters for the application of ERT is the noise of the measurements and the current-measurement strategy. With the available mathematical tools for analysis, a second more mature design of the application can be established.

Several issues of ET technology for composite structures remain to be assessed further. On the other hand, several other "tools" and techniques (both experimental and mathematical) are available for dealing with these issues. We believe that with this work we set the grounds and report on the challenges that arise for further applications of ET in composites and other structural materials.

VI. FUTURE OUTLOOK

After having established an ERT setup and a protocol for the preparation of the specimens, the first actions will focus on the optimization of the measurement setup. Noise minimization and precision are key parameters for further progress and applications. Furthermore, a new design of the current injection and measurement strategy could provide more information for a more accurate reconstruction.

Targeting other materials, the evaluation of carbon fibre reinforced polymers (CFRP), which are inherently conductive, is among the first steps. Additionally, a more detailed search of correlations between damage modes and ERT findings will be performed, together with different localization algorithms and techniques.

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