Design and Manufacture of Reference and Natural Defect Artefacts for the Evaluation of NDE Techniques for Fibre Reinforced Plastic (FRP) Composites in Energy Applications

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Abstract. The excellent mechanical properties, low weight, fatigue and corrosion resistance of fibre reinforced plastic (FRP) composites gives them considerable advantages in renewable energy, oil and gas and transport applications. The use of FRP composites has the potential to reduce fossil fuel reliance, consumption and greenhouse gas emissions. However, full exploitation is hindered by the diverse range of defects that can reduce the strength, stiffness and life of FRP structures. One of the challenges facing accurate and repeatable defect detection in FRP composites is the multitude of defect types that exist, each with characteristics that present different challenges to the NDE practitioner. In order for a particular NDE technique to achieve broad acceptance by industry, it is desirable for the technique to be able to detect a range of defect types with a high level of confidence. This paper details the design and manufacture of several FRP composite defect artefacts that have been produced to evaluate the detection capabilities of phased array and air-coupled ultrasonic, microwave, active thermographic and laser shearography inspection techniques. This work was undertaken within EMRP project ENG57 Validated Inspection Techniques for Composites in Energy Applications VITCEA [1].

1. Introduction

Defects may be introduced during the processing and fabrication of composite components and can initiate or grow in-service. In the context of this paper, the term ‘defect’ refers to imperfections introduced during manufacture/processing and/or secondary machining operations, as well as damage sustained during a component’s service life. One of the challenges facing accurate and repeatable defect detection in FRP composites is the...
multitude of defect types that exist, each with characteristics that present different challenges to the NDE practitioner. In order for a particular NDE technique to achieve broad acceptance by industry, it is desirable for the technique to be able to detect a range of defect types with a high level of confidence. A simple classification of defect types can be made between those arising from the manufacturing process and those occurring subsequently in-service (e.g. load, handling, environment etc.). The main defect types in FRP composite material systems can be categorised as delaminations, de-bonds in bonded joints, cracking, porosity, voids, fibre misalignment and waviness, and wear/erosion. Within each of these categories, there are subsets of individual defect types. For example much attention has been focused on transverse ply cracking in composite systems; a form of multiple matrix cracking occurring in off-axis plies. Transverse cracking reduces the stiffness of the laminate and is often a precursor to delamination. Important factors that influence defect detection using NDE include: (i) material type, fibre format and thickness, (ii) defect location (e.g. internal radius), (iii) defect distribution (e.g. discrete (void) and dispersed (porosity), (iv) defect origins (e.g. natural (impact damage) vs. artificial (peel-ply inclusion)) and (v) architecture of component (e.g. sandwich construction, ply-drops, thick bond-lines etc.).

When a component is in service, defects may be introduced by a variety of mechanisms including overloading, impact, heat, erosion, fatigue etc. Defects will manifest themselves as changes in the condition of certain properties or physical parameters such as, in the instance of erosion, thinning of the component. Setting of acceptance criteria for each of these defect types requires a formal defect assessment based on material considerations and an analysis of the stresses that the component will experience when in service. NDE techniques must be sensitive enough to detect defects of a sub-critical size and any subsequent defect growth that is likely to reduce the life expectancy of the component. The relative importance of each defect type will depend on the design/failure criterion and application. For example in a stiffness-based design, delamination or significant de-bonding could lead to failure by buckling. Such defects may be more tolerable in a strength-based design. Delaminations often have a limited impact on integrity, but are important as initiation sites or precursors for more severe damage. The relative importance of manufacturing versus in-service inspection will depend on the energy application.

This paper details the design and manufacture of several FRP composite defect artefacts that have been produced to evaluate the detection capabilities of phased array and air-coupled ultrasonic, microwave, active thermographic and laser shearography inspection techniques. A series of reference defect artefacts (RDAs), in which the defect sizes and locations are well defined and controlled, have been designed and manufactured. Defect types featured within the RDAs include artificial delaminations, in-plane fibre misalignment, back-face drilled holes (to represent voids and porosity) and kissing bonds. In addition, a number of natural defect artefacts (NDAs) were produced with defects (e.g. matrix cracking, delamination) created via controlled loading mechanisms (tensile loading and low velocity impact). The NDAs are more representative of real defects than the RDAs, as they are generated by natural processes and comprised of multiple defects of unknown size, specific location and nature. Both the RDAs and NDAs were produced using the same range of FRP composite materials including thermoset and thermoplastic matrix systems reinforced with glass and/or carbon unidirectional and multi-directional tape and fabric formats.

2. Design of reference and natural defect artefacts (RDAs and NDAs)

The approach to the development of NDE techniques and procedures adopted within VITCEA [1] in terms of the selection of material systems, defect types and locations to be studied is two-fold. Firstly, at the simplest level, well characterised material systems that
contain defects that are routinely required to be inspected, and that are positioned in accessible locations have been chosen be used to develop the various NDE operational procedures. Secondly, additional trials are being undertaken on more complex cases where either the inherent nature of the material, the defect or its location present significant difficulties in detection, e.g. stitched fabrics, kissing bonds or back-face skin delaminations within a sandwich construction. This latter activity is designed to refine NDE procedures, extending their application to more complex inspections where feasible and ascertaining the limitations of the NDE techniques under investigation.

A key objective of the VITCEA project was to ensure that the choice of artefacts that have been designed, manufactured and inspected are representative of the defects, materials, processing routes and structural elements that are of concern to a range of renewable energy (wind, wave and tidal), oil and gas, and transport applications in which FRPs are used. As such, close collaboration with industry was undertaken to define the required sensitivity and accuracy of the methods for defect detection and characterisation and hence inform the choice of defects, defect sizes and materials. A range of FRP composites was chosen to cover applications in the lightweight transport, wind/marine and oil and gas sectors. Details of these materials, associated processing routes and nominal fibre volume fractions \( (V_f) \) are provided in Table 1.

### Table 1. Details of materials used in construction of RDAs and NDAs

<table>
<thead>
<tr>
<th>Material</th>
<th>Fibre</th>
<th>Resin</th>
<th>Process route</th>
<th>Ply format/thickness</th>
<th>( V_f ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gurit SE84LV (transport)</td>
<td>Carbon T700</td>
<td>Epoxy</td>
<td>Hand lay-up + autoclaved (120°C)</td>
<td>UD prepreg/ ~0.30 mm</td>
<td>~55</td>
</tr>
<tr>
<td>Hexcel 913G (transport)</td>
<td>E-glass</td>
<td>Epoxy</td>
<td>Hand lay-up + autoclaved (120°C)</td>
<td>UD prepreg/ ~0.13 mm</td>
<td>~53</td>
</tr>
<tr>
<td>Resin infused quadraxial glass fabric (wind/marine)</td>
<td>E-glass</td>
<td>Prime 20LV epoxy</td>
<td>Resin infusion (post-cured at 60°C)</td>
<td>Quadraxial fabric (Formax FGE111) /~</td>
<td>~53</td>
</tr>
<tr>
<td>Celstran®CFR-TP PA12 GF60 (automotive)</td>
<td>E-glass</td>
<td>Polyamide-12</td>
<td>Spot solder laminated and autoclaved (220°C)</td>
<td>UD prepreg/0.33 mm</td>
<td>~37</td>
</tr>
<tr>
<td>Cytec MTM®28 (oil and gas)</td>
<td>E-glass</td>
<td>Epoxy</td>
<td>Hand lay-up + oven cured (120°C)</td>
<td>UD prepreg/~0.25 mm</td>
<td>~52</td>
</tr>
</tbody>
</table>

2.1 Design and construction of reference defect artefacts (RDAs)

The following sections detail the designs and construction of RDAs. A summary of the RDAs fabricated within the VITCEA project are detailed in Table 2.

2.1.1 Monolithic flat panel RDAs

Three monolithic flat panel RDAs (RDA_1a, RDA_2a and RDA_5) were fabricated from Gurit SE84LV (carbon fibre), Hexcel 913G (glass fibre) and Celstran® PA12 (glass fibre) pre-impregnated tape materials, respectively, to the design shown in the schematic of Figure 1.
<table>
<thead>
<tr>
<th>Material/sector</th>
<th>Name</th>
<th>Description</th>
</tr>
</thead>
</table>
| SE84LV         | 2 off | - UD lay-up ([0]_{16}) ~ 5 mm thick
- artificial delaminations of \(\varnothing 3, 6, 12 & 25 \text{ mm}\)
- individual drilled holes \(\varnothing 1, 2 & 3 \text{ mm}, 3 \times 3 \text{ arrays of } \varnothing 1 \text{ mm holes} 
- 15° in-plane fibre misalignment (6, 12 & 25 mm square) |
|                | RDA_1a| - kissing bonds of \(\varnothing 3, 6, 12 & 25 \text{ mm}\)
- two UD laminates ([0]_{8}) bonded with Redux 312 film adhesive |
|                | NDA_imp| - QI: ([+45/0/-45/90]_5 & ([+45/0/-45/90]_2) for ~3 & 5 mm thick laminates
- low velocity impact of 240 x 100 mm coupons |
|                | NDA_ten | - notch machined to mid-thickness & near back-face
- 240 x 50 mm coupons loaded in tension |
| 913G           | RDA_2a| - cross-ply ([0/90]_{10}) ~ 5 mm thick
- artificial delaminations; \(\varnothing 3, 6, 12 & 25 \text{ mm}\)
- individual drilled holes \(\varnothing 1, 2 & 3 \text{ mm}, 3 \times 3 \text{ arrays of } \varnothing 1 \text{ mm holes} 
- 15° in-plane fibre misalignment (6, 12 & 25 mm square) |
|                | RDA_2b| - kissing bonds of \(\varnothing 3, 6, 12 & 25 \text{ mm}\)
- two laminates bonded with Redux 312 film adhesive |
| Resin infused quadraxial glass fabric | RDA_3 | - sandwich construction 450 x 650 mm
- resin infused GFRP skins with PVC foam core; two flat regions of different core thickness (10 mm & 40 mm thick core) with smoothly varying transitions
- top skin \(\sim 3 \text{ mm thick} \) & bottom skin \(\sim 10 \text{ mm thick} \)
- artificial delaminations of \(\varnothing 3, 6, 12 & 25 \text{ mm in skins} 
- holes in core of \(\varnothing 3, 5 & 10 \text{ mm/} {\frac{1}{4}, \frac{1}{2} \text{ and } \frac{3}{4} \text{ core thickness from back-face.}} |
| Marine/wind    | NDA_imp| - 5 & 8 layers of quadraxial fabric for ~3 and 5 mm thick laminates
- low velocity impact of 240 x 100 mm coupons |
|                | NDA_ten | - notch machined to mid-thickness & near back-face
- 240 x 50 mm coupons loaded in tension |
| MTM®28         | RDA_4 | - two 10 mm thick pipe sections (partial circumference) of MTM®28 abutted & overwrapped with 5 mm thick repair laminate (resin infused quadraxial GFRP) at joint over 124 mm length
- interface kissing bonds under repair of \(\varnothing 3, 6, 12 & 25 \text{ mm} 
- artificial delaminations of \(\varnothing 3, 6, 12 & 25 \text{ mm in monolithic pipe section} 
- drilled holes \(\varnothing 5, 10 & 25 \text{ mm in pipe section; } 1.5, 3 & 5 \text{ mm deep} |
| Oil and gas    | NDA_imp| - QI: ([+45/0/-45/90]_5 ~ 5 mm thick
- low velocity impact of 240 x 100 mm coupons |
|                | NDA_ten | - notch machined to mid-thickness & near back-face
- 240 x 50 mm coupons loaded in tension |
| PA12 GF60      | RDA_5 | - UD lay-up ([0]_{10}) ~ 5 mm thick
- artificial delaminations of \(\varnothing 3, 6, 12 & 25 \text{ mm} 
- individual drilled holes \(\varnothing 1, 2 & 3 \text{ mm, 3 } \times 3 \text{ arrays of } \varnothing 1 \text{ mm holes} 
- 15° in-plane fibre misalignment (6, 12 & 25 mm square) |
| Automotive     | NDA_imp| - QI: ([+45/0/-45/90]_5 ~ 5 mm thick
- low velocity impact of 240 x 100 mm coupons |
|                | NDA_ten | - notch machined to mid-thickness & near back-face
- 240 x 50 mm coupons loaded in tension |
The defects incorporated within these RDAs include artificial delaminations (3, 6, 12 and 25 mm diameter), individual flat bottom, back-face drilled holes (1, 2 and 3 mm diameter) and 3 x 3 arrays of 1 mm diameter back-face drilled holes and square regions of 15° in-plane fibre misalignment.

![Fig. 1. Generic design of monolithic, flat panel RDAs 1a, 2a and 5](image)

All defects were included at 3 different depth locations; near front-face (~1/4 laminate thickness cover), mid-thickness and near back-face (~3/4 laminate thickness cover). Artificial delaminations were constructed as per the method detailed in [2-3] and shown in Figure 2. Two sheets of 0.05 mm thick PTFE film were encapsulated in oversized layers of high-temperature adhesive tape such that a PTFE ‘pocket’ was sealed around its edge. It has been shown [3] that the combined effect of high attenuation (from an ultrasonics perspective) defect material and the entrapped air between the PTFE sheets produces a well-defined acoustic barrier almost entirely preventing the transmission of an ultrasonic signal.

![Fig. 2. Construction of artificial delaminations](image)

Regions of fibre misalignment were created by removal of squares of material within the desired ply and replacement with the same size of square but misaligned by 15°. Flat bottom back-face holes were drilled using a CNC drilling machine in the National Physical Laboratory (NPL) Engineering workshop. The process route for each material type is detailed in Table 2.
2.1.2 Secondary bonded kissing bond RDAs

Two secondary bonded flat kissing bond RDAs (RDA_1b and RDA_2b) were fabricated from Gurit SE84LV (carbon) and Hexcel 913G (glass), respectively. For each material, two sub-laminates (UD for SE84LV and cross-ply for 913G) were autoclave cured and bonded together using Redux 312 film adhesive. Prior to bonding, one laminate for each RDA was surface treated in the circular regions shown in Figure 3 by creating a ‘mask’ from adhesive backed PTFE sheet. The surface treatment applied was four coats of Tygacote® SP441 release agent, baked after each application, and followed by four coats of PTFE dry lubricant spray. This surface treatment procedure was developed in previous work [4] and found to be effective in creating a ‘near zero’ bond strength. Indeed, trial tests showed that the treated sections of bonded laminate came apart on the application of light finger pressure whilst non-treated regions exceeded 16 MPa tensile strength when tested in flatwise tension. Once the surface treatment had been applied, the PTFE film mask was removed and the two sub-laminates were bonded together in an autoclave at 120°C.

![Arrangement of kissing bond regions in RDAs 1b and 2b](image)

2.1.3 Resin infused quadraxial glass fabric skin – PVC foam core sandwich RDA

Figure 4 shows the design of RDA_3 which is a sandwich construction comprising resin infused quadraxial glass fibre fabric skins and a PVC foam core. This RDA was designed to be representative of a generic wind turbine blade or marine structure and contains embedded artificial delaminations (see Section 2.1.1) at three depth locations in the ~10 mm thick bottom skin and at mid-depth in the top ~3 mm thick skin. Holes of 3, 5 and 10 mm diameter were drilled into the foam core to three depths (through-the-thickness from the back-face) to represent core damage. The entire construction was resin infused in one process and to prevent the drilled holes in the foam core filling up during the infusion process, thin pre-cured resin ‘cover-slips’ were recessed into the foam over each drilled hole.
2.1.4 Composite over-wrap pipe repair RDA

Figure 5 shows the design of RDA_4 which represents the repair of an ageing GFRP pipe with a resin infused GFRP repair patch.
This RDA contains a series of back-face drilled holes and artificial delaminations in the pipe substrate as well as kissing bonds, created using the same surface treatment described in Section 2.1.2, situated in the interface between pipe substrate and resin infused over-wrap.

2.2 Designs for natural defect artefacts (NDAs)

The following section briefly details the production of NDAs, a summary of which is detailed in Table 2.

Two types of NDA, those created by low velocity impact and those in which damage was initiated by tensile loading of coupons containing machined notches, were designed and created for all material types with the exception of the Hexcel 913G system. For both types of NDA, monolithic laminates (300 x 600 mm and ~3 and 5 mm thickness) were fabricated using the process route detailed for each material type in Table 1. The lay-ups for all materials were quasi-isotropic in nature i.e. reinforcement was present in +45°, 0°, -45° and 90° directions.

For the NDAs created by low velocity impact, 240 x 100 mm rectangular coupons of material were impacted using a drop-weight tower fitted with a 12 mm diameter hemispherical steel indentor. Coupons were clamped on a support frame as shown in Figure 6 and impacted at energy levels of between 1 and 16 J depending on the material type and coupon thickness. The degree of damage showed a good level repeatability for impact tests undertaken at nominally the same impact energy level for a given material.

Fig. 6. Low velocity impact arrangement for creation of NDAs and typical image damage created in the resin infused quadraxial glass fabric material (N.B. figure shows two repeat impacts at 13.5 J)

Fig. 7. Notched tensile NDA and distribution of delamination created
NDAs were also created by tensile loading of coupons with square profile notches machined across the width (Figure 7). On tensile loading, delamination initiates and propagates from the route of the notch in a repeatable manner and is visible from the edge of the coupon and from thermograms on tested coupons (Figure 7(c)).

3. Selected NDE inspection results

Selected initial NDE inspection results are given in Figure 8-10. Figures 8 and 9 show initial results of microwave inspections undertaken at 10 and 34 GHz on glass fibre based RDAs 2a and 5, respectively (see Figure 1 for defect arrangements). The artificial delaminations and back face drilled holes (individual and arrays) are detected well at 34 GHz and a scanning index increment of 0.5 mm, but the detection at 10 GHz is generally poor due to the large wavelength at this frequency and increased reflections from the panel edges.

Fig. 8. Microwave inspection results for RDA_2a at 10 GHz (left) and 34 GHz (right)

Fig. 9. Initial microwave inspection results for RDA_5 at 10 GHz (left) and 34 GHz (right)

Figure 10 shows a comparison of scans for RDA_2a obtained using immersion pulse-echo ultrasonic C-scan (5 MHz 3” focussed spherical probe), microwave (34 GHz) and active thermography. The results highlight the relative detection capability of the techniques and that as expected none of the techniques evaluated to date are capable of detecting the in-plane fibre misalignment. In general, only ultrasonic C-scan was capable of detecting all sizes of artificial delamination and all three techniques were incapable of detecting the deepest 3 x 3 array of 1 mm diameter holes.
Conclusions and further work

To date, the work within the VITCEA project has focussed on the design and fabrication of a series of RDA and NDA reference samples fabricated from a range of materials relevant to FRP composites used in a number of applications across several energy sectors. Artificial defects have been successfully created, including delaminations, kissing bonds and individual and arrays of small diameter holes to represent discrete voids and regions of porosity, respectively. Some initial non-destructive inspections have been undertaken using ultrasonic, microwave and thermographic techniques, with selected results reported in this paper. During the remainder of the VITCEA project, all of the RDAs and NDAs will be used to thoroughly evaluate the suitability of the phased array and air-coupled ultrasonic techniques, variations of active thermography, microwave and laser shearography techniques. In addition, once all inspections have been undertaken, the NDAs will be destructively inspected using optical and scanning electron microscopy (SEM) techniques to characterise the nature and extent of damage created more accurately and for comparison to non-destructive results.

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References