

This is a repository copy of *Development and implementation of direct electric cure of plain weave CFRP composites for aerospace*.

White Rose Research Online URL for this paper: <u>https://eprints.whiterose.ac.uk/199261/</u>

Version: Published Version

# Article:

Collinson, M.G., Swait, T.J. orcid.org/0000-0003-3199-7870, Bower, M.P. et al. (2 more authors) (2023) Development and implementation of direct electric cure of plain weave CFRP composites for aerospace. Composites Part A: Applied Science and Manufacturing, 172. 107615. ISSN 1359-835X

https://doi.org/10.1016/j.compositesa.2023.107615

# Reuse

This article is distributed under the terms of the Creative Commons Attribution (CC BY) licence. This licence allows you to distribute, remix, tweak, and build upon the work, even commercially, as long as you credit the authors for the original work. More information and the full terms of the licence here: https://creativecommons.org/licenses/

# Takedown

If you consider content in White Rose Research Online to be in breach of UK law, please notify us by emailing eprints@whiterose.ac.uk including the URL of the record and the reason for the withdrawal request.



Contents lists available at ScienceDirect

# Composites Part A

journal homepage: www.elsevier.com/locate/compositesa

# Development and implementation of direct electric cure of plain weave CFRP composites for aerospace

M.G. Collinson<sup>a,\*</sup>, T.J. Swait<sup>a</sup>, M.P. Bower<sup>a</sup>, B. Nuhiji<sup>a</sup>, S.A. Hayes<sup>b</sup>

<sup>a</sup> Advanced Manufacturing Research Centre, University of Sheffield, Wallis Way, Catcliffe, Rotherham S60 5TZ, UK
<sup>b</sup> Department of Multidisciplinary Engineering Education, University of Sheffield, 32 Leavygreave Road, Sheffield S3 7RD, UK

ARTICLE IN
------------

Keywords: Composite curing Out of autoclave Low power curing VARTM pre-preg Sustainable curing Joule heating

## ABSTRACT

Curing aerospace composites is time-consuming with high energy requirements, particularly when using methods such as autoclaves, ovens and heated presses. This study investigates direct electric cure (DEC), which uses the Joule effect to directly heat carbon fibre composites to their cure temperature. Its benefits include a significant increase in energy efficiency, control over the cure temperature and low void content compared to oven curing. Samples were manufactured with vacuum-assisted resin transfer moulding (VARTM) and prepreg, as well as curing a two-meter-long leading-edge component, to demonstrate and evaluate the curing method at scale. The average void content for VARTM DEC samples was lower by 0.82 % compared to the oven-cured panel, however, the average degree of cure (DoC) decreased by 6.25 %. Normalised energy consumption for the cure was significantly reduced for all DEC cures, with VARTM cures using 99 % less energy than oven-cured components.

# 1. Introduction

High-performance carbon fibre reinforced polymer (CFRP) composites for aerospace are traditionally cured in high-pressure autoclaves or ovens. This equipment can be prohibitively expensive, energy inefficient, time-intensive to operate and the components manufactured are limited by the size of the chamber [1]. However, they consistently produce parts with a void volume fraction (VVF) below 0.5% which is the critical VVF for high performance epoxy composites [2]. The rate of manufacture also needs to be addressed, with Boeing predicting that 171 aircraft a month will be manufactured from 2022 to 2041 [3]. To enable this throughput whilst minimising environmental impact, capital expenditure and energy consumption of current manufacturing methods needs to be reduced, or new materials and methods need to be introduced.

Direct electric cure (DEC) is the application of a current through conductive carbon fibres in a CFRP component, to heat the component to its cure temperature via the Joule effect. This has been used to cure panels on a small scale, showing the benefits in improvements to DoC, VVF, cure energy and time to cure [4–6]. It has significant energy-saving advantages over convection-based methods, up to 99 % [7,8]. These advantages show promise for DEC to be a future heating or curing method for composite materials [9].

Autoclave curing is the preferred method of manufacture of primary aerospace structures, as high pressure (6–20 bar) ensures voids are compressed or eliminated. Out of Autoclave (OoA) pre-preg systems have been developed to provide low VVF whilst using atmospheric pressures, allowing ovens or heated tools to be used. This reduces initial equipment and running costs, such as energy consumption. It is also possible to heat these material systems at rates of 10 °C/min or higher without degradation of final cure properties. This aligns well with novel cure methods such as DEC that are capable of reaching these heating rates [9].

A significant limitation of autoclave or oven curing is the that the cured component can't be larger than the heating chamber, limiting large component manufacture to those who can afford the high capital equipment expenditure. DEC has the potential to cure large components using comparatively low-cost power supplies, control equipment and consumables, significantly reducing the cost of increasing the manufacture rate of large CFRP components.

The resistivity of carbon fibres is low, with a 3 k tow being less than 25  $\Omega/m$  [10]. The matrix, commonly epoxy resin, is electrically insulating and used in high voltage applications with a breakdown voltage of 400 V/mm [11]. This contrast in conductivities results in issues when

https://doi.org/10.1016/j.compositesa.2023.107615

Received 16 January 2023; Received in revised form 15 March 2023; Accepted 7 May 2023 Available online 10 May 2023

1359-835X/© 2023 The Author(s). Published by Elsevier Ltd. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).





<sup>\*</sup> Corresponding author. E-mail address: m.collinson@amrc.co.uk (M.G. Collinson).

trying to electrically interface with the composite, with areas of high resistance causing excessive heat generation and uneven cure temperatures [12].

Copper electrodes have commonly been used to introduce current for DEC, either through copper clamps [13] or copper sheets [4,5,14–16] introduced in a vacuum bag. Previous investigations have shown the cure carbon tows [17,18] and pre-preg components, made up of unidirectional (UD) [5,14,15] and plain weave (PW) [4] plies. DoC has been shown to match or improve properties over oven-cured samples and be close to autoclave. VVF has also been compared, in some cases showing that it can be reduced over oven and autoclave cured samples, theorised to be enabled by the inside-out heating mode, as opposed to outside-in with conduction [15]. Power requirements for DEC are extremely low as there are very few losses in the process, compared to oven curing. As the component is the heating element, it can also be locally insulated for further energy efficiency gains, not possible in other curing methods.

In addition to component curing, Joule heating of composites has been previously researched in a wide variety of use cases and industries. Examples include it being used for foldable space applications [17,19], patch repairs [20], curing of multiple-zoned irregular shapes [14], heated tooling [21,22], preheating [12], anti-icing [23] and controlled curing of thick laminates [15]. The methods developed in this study will have multiple use cases and provide important data for future Joule heated composites. All of these studies share common characteristics and issues, such as contact resistance, temperature uniformity and control systems.

To cure a composite effectively, the temperature distribution needs to be uniform over the surface and through the thickness, which is a challenge with DEC [14]. UD plies can improve the uniformity of heating, however, it is important to consider universal compatibility with components, and not restrict the layup of a component to a certain curing method. As the scale of the component is increased, the temperature differentials can become larger and harder to control. Little research has been completed on compatibility with manufacturing methods and materials, such as dry fibre infusion, ply breaks, thickness changes and core materials.

This study builds on the pre-preg processes established in previous literature [4,5] of curing components with the DEC method and obtaining increased manufacturing and component characteristics. It develops the process for dry fibre vacuum-assisted resin transfer moulding (VARTM), and for larger laminates, up to 2 m in length. It also investigates other standard composite features such as ply breaks and core materials, increasing the methods compatibility with aerospace composite requirements. With these changes to the method, issues in laminate quality are encountered, however, suggestions for future research and development are suggested. If these were implemented, DEC would enable future large aerospace structures to be cured using this low power method, whilst retaining the benefits such as low void content and higher controllability over temperature and DoC.

## 2. Methods and materials

#### 2.1. Materials and composite layup

Pre-preg experiments used Cycom 5320–1 plain weave which contained 3 K Toray T650 fibres. The cure cycle used in this study is the recommended cycle from the manufacturer as follows: Ramp from room temperature (RT) at 3 °C min<sup>-1</sup> up to 120 °C min<sup>-1</sup>, hold for 2 h, then 2 °C min<sup>-1</sup> ramp up to 180 °C, hold for 2 h and then ramp down to RT at 2 °C min<sup>-1</sup>.

For the VARTM experiments, Chomarat plain weave 3 K fabric was used, containing Toray T300 fibres, matching the plain weave pattern used in the Cycom 5320–1 pre-preg material. This should ensure the conducting fibres should act similarly. The infusion resin system was Huntsman Araldite CY 179/ Aradur 917, which has a cure cycle defined as 100  $^{\circ}$ C for 2 h, and 160  $^{\circ}$ C for 6 h, with no defined ramp rates. The

layup sequences used were  $\left[0/90\right]_s$  for 4 ply, and  $\left[0/90/0/90\right]_s$  for 8 plies.

# 2.2. Tooling and ancillary materials

For all cures, vacuum was applied to the composite using vacuum bagging (Tygavac). For pre-preg cures breather fabric and release films were applied, and for VARTM peel ply and resin transfer mesh were applied (all obtained from Tygavac). During initial experimentation, the composites were cured on a ceramic tile covered in Polytetrafluoro-ethylene (PTFE). Later in development, glass fibre reinforced plastic (GRFP) tooling was used to ensure the composite was electrically insulated. Two layers of ¼ inch thick rock wool insulation (Morgan Advanced Materials) was used in experiments where thermal insulation is described.

# 2.3. Power supply, control, and data acquisition systems

An RMX-4124 (NI) power supply unit (PSU) was used to deliver power to the composite, at 0–30 V and up to 150 A, with a maximum power output of 1500 W. This was controlled by an PXI computer (NI) running a software PID controller programmed in LabVIEW. Up to 12 Ktype thermocouples (TC Direct) were used to monitor and control the cure using a PXIe 4353 (NI). A FLIR AC655 (Teledyne) was used to monitor the temperature of the surface of the vacuum bag, to provide a reading of the temperature distribution over the panel during initial experimentation.

#### 2.4. Component electric curing setup

A 0.035 mm thick copper sheet (CCI Eurolam) was used as the electrode material, which was laminated between plies at the edge of the composite. An overlap of the copper was left at the edge of the composite, which was hole-punched and bolted to crimped power cables, as seen on the left of Fig. 1. For VARTM, flat-ended bolts were used with penny washers and tacky tape to provide a vacuum seal, as seen in the right of Fig. 1. This was to ensure that air was not introduced into the bag through the multicore copper cable used.

## 2.5. Energy consumption measurements

The power output from the DEC power system was directly recorded for each component from the LabVIEW interface. Power usage data for oven curing was collected using a CUBE 400 (ND Metering Solutions) on a composite curing oven (Caltherm), measuring  $3000 \times 3000 \times 3000$ mm, with a 95 kW power output, and a smaller curing oven (Hedinair), measuring  $1000 \times 1000 \times 1000$  mm, with a 12 kW power output.

# 2.6. Characterisation

A Perkin Elmer DSC 4000 was used to determine the DoC. Sample weights were 20  $\mu$ g, and followed the heating cycle from 30 °C up to 300 °C at 10 °C min<sup>-1</sup>.

To determine fibre volume fraction (FVF) and VVF, samples were cut and cast in a potting resin, then polished (Struers). The samples were then imaged with a Zeiss LSM 800, then automatically processed using image recognition software to find VVF.

# 3. Results and discussion

#### 3.1. Development methodology

The aim of this work was to increase the scale of the components cured, whilst maintaining the benefits of electric cure such as low power requirements, high heating rates, high DoC and low VVF. The safety of the curing method, difficulty of setup, compatibility with common

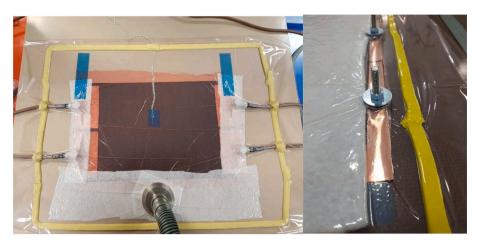


Fig. 1. Left: Example of a simple direct electric cure setup with reduced breather material to show layup sequence, Right: Example of through bag electrode setup, showing penny washers and tape fastened to the bag to ensure vacuum integrity of the bag.

composite layup techniques such as ply overlaps and the inclusion of core materials were also considered.

The initial work completed in this study was completed on pre-preg materials, building on previous literature, covered in sections 3.2 to 3.5. Once the fundamentals of the process had been understood, they were transferred and adapted for VARTM and to larger components, covered in sections 3.7 to 3.9. The culmination of these developments was demonstrated and tested with 2-meter-long leading-edge sections, manufactured with both pre-preg and VARTM, described in section 4.

# 3.2. Temperature distribution assessment

Achieving even temperature distribution over a CFRP component is a crucial step to implementing this technology as it directly correlates to DoC and the final mechanical properties. Previous research with woven CFRP produced an uneven temperature over the composite and with the optimal use of UD CFRP this temperature delta was improved, however the results were not fully optimal. These temperature differentials were expected to scale with the size of the component, so temperature distribution of the curing process was monitored.

A thermal imaging camera was used on a 200  $\times$  300 mm pre-preg panel being heated to 90 °C, as seen in Fig. 2. The heating appears in a band in the centre of the composite, with the edges of the composite

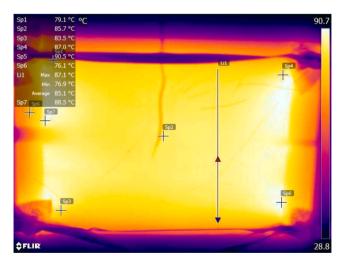


Fig. 2. Thermal image of a 200  $\times$  300 mm panel, showing the temperature differences over the panel. Set points (Sp) show temperature points over the surface, whereas the line (Li) shows the hottest and coolest part, which are the centre and the edge, indicated by up and down triangles respectively.

being considerably cooler, primarily due to higher thermal losses at the edges. This may be exacerbated by the negative resistance to temperature gradient of CFRP tows [24] Excessive heating is also observed around the electrodes, which is further discussed in section 3.6. Thermally insulating the tooling and surface of the composite reduces overall temperature differential. When using thermal insulation, line of sight is blocked to a thermal imaging camera, limiting the usefulness of this tool in this investigation. Therefore, in subsequent cures, an array of thermocouples was used to monitor temperature, particularly at edges and at the electrodes, monitoring for lower and higher temperatures respectively.

To investigate the cooling edge effect further, a  $200 \times 300$  mm prepreg panel was cured. A line of thermocouples was spaced 10 mm apart, from the centre to the edge, as seen in Fig. 3.

The central thermocouple was used as PID control input and was set to dwell temperatures of 120  $^\circ C$  and 180  $^\circ C$ , as seen in Fig. 4, without thermal insulation.

The temperature reduction from centre to edge for  $120 \,^{\circ}$ C and  $180 \,^{\circ}$ C set temperatures at the end of the dwell are  $50 \,^{\circ}$ C and  $77 \,^{\circ}$ C respectively. There is an increase in the temperature differential at the edge of the panel, meaning that the temperature is within  $20 \,^{\circ}$ C for  $70 \,^{\circ}$  of the width. For most components there is an edge trimming operation, which if designed carefully could allow for the under-cured sections to be removed. Temperatures at the start of the dwell are higher than at the end of the dwell end at both temperatures, which is immediately after a high-power input in order to raise the temperature.

Low frequency pulse width modulation (PWM) of the DC supply therefore could potentially provide more uniform heating. Zonal heating of UD CFRP sections has been used to uniformly heat complex geometries [25], which could also be applied to uniformly heat other layup scenarios, such as PW layups, or thickness changes. Selective insulation of the edges may be another means to improve temperature uniformity.

# 3.3. Degree of cure

Considering the temperature differentials seen in heating PW composites, seen in section 3.2, it is important to evaluate the DoC at different points on a component. A 575  $\times$  575 mm, 8 ply pre-preg panel was cured, of which the temperature profile can be seen in Fig. 11. Samples were taken in the centre and edge of the panel, and a point between the two, of which the results can be seen in Fig. 5.

These results align closely with the temperature trends seen in Fig. 4, the temperature reduction at the edge of the panel has directly resulted in a significant drop in DoC of approximately 36 %. This percentage drop is less than the temperature drop, due to this cure being thermally insulated, however it is unacceptable at present and requires further

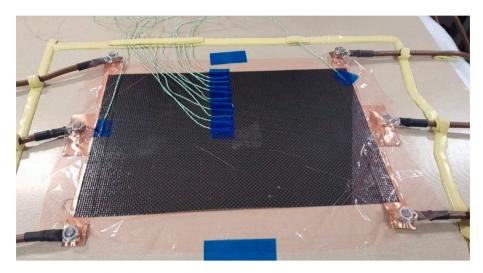
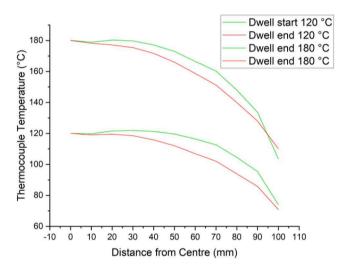


Fig. 3. Electric cured panel, showing the setup to monitor temperature drop off from centre to the edge.



**Fig. 4.** Temperature reduction from centre of a panel to the edge at 120  $^{\circ}$ C and 180  $^{\circ}$ C set temperatures. Note that this is without thermal insulation, so the reduction is exaggerated compared to other experiments detailed in this study.

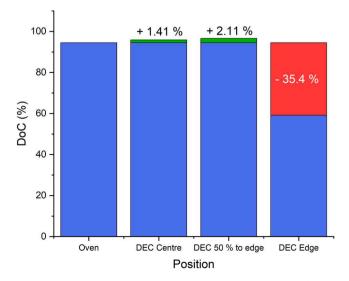


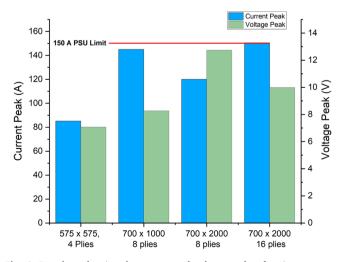
Fig. 5. Summary of DSC results of 575  $\times$  575 mm pre-preg panel which has been electrically cured.

development to address. Previous literature states that when using UD plies, temperature and therefore DoC over the surface is more uniform, therefore being similar to oven or autoclave cure, if the same cure cycle is used [5]. This assumption, however, is not possible when using plain weave plies as the main heating elements in the composite.

# 3.4. Safety considerations

There are multiple safety considerations to be made when using this method, due to the way high current is used to heat the component, and the associated risks with potential burning, and interactions that could occur with the tooling and consumables. Fig. 6 shows the current and voltages used to cure for various geometry samples.

The PSU used in this study was controlled in constant current mode. In this mode, there were slight setup inconsistencies with the electrodes, causing localised high resistance, resulting in hotspots around the electrodes. In rare cases, this would develop into burning when higher ramp rates were used, as the temperature increased beyond the thermal breakdown temperature of the epoxy. During a cure, these were identified by the user and the system powered down, to avoid further damage. Burning led to an increase in resistance of the component, and in constant current mode, would lead to spikes in power input to the panel seen in the left of Fig. 7. This power increase would compound the



**Fig. 6.** Bar chart showing the current and voltage peaks of various pre-preg panels manufactured using DEC. The peaks were all recorded during the ramp up to their set temperature.

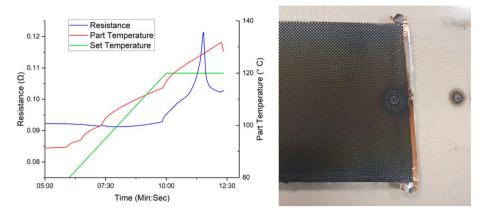


Fig. 7. Left: Graph showing the temperature runaway during a burning event. The behaviour of the part temperature is due to manual limits being applied to the output current. Note the sharp increase in resistance, leading to a 200 W spike in power input at the peak. Right: Component and PTFE plate burn damage after curing.

burning, which could lead to a dangerous runaway situation, as seen in the right of Fig. 7.

The control system was changed to control the calculated power reported by the PSU, which will reduce the severity of burning and allow for more time to intervene.

## 3.5. Ply breaks and core materials

The use of core materials and ply breaks are common in composite manufacturing, to increase component stiffness [26] and to change the thickness or join large sections together. Both features were tested for compatibility with the electric cure method. The following layup was used to test if core materials were compatible with DEC:  $[0/90/0/90]_s$ [GFRP/Nomex/GFRP]  $[0/90/0/90]_s$ . The carbon plies were heated to cure temperature, which through conduction cured the adhesive ply, bonding the Nomex to the adhesive ply. This was then repeated with GFRP ply laid up on top of the Nomex, then 8 plies joule heated, to form a sandwich structure. The cure cycle was completed as expected, and through visual inspection, the Nomex was successfully bonded within the sandwich structure.

Ply breaks were tested on a 2000  $\times$  700 mm, 8-ply pre-preg panel that had a single 10 mm overlap, alternating directions every ply. There was a slight increase in temperature of 2–5 °C on the overlap where

resistance increased. This overlap is too small to provide optimal mechanical strength [27], therefore could be increased to reduce temperature difference further. It was observed that the temperature increase was maintained along the width of the ply drop, rather than reducing in temperature towards the edge of the laminate, as seen in previous experiments in section 3.2.

#### 3.6. Contact arrangement

The electrodes were copper sheets applied along the full length of opposite edges of the uncured carbon fibre, or dry fibre for VARTM. Previous literature suggested that placing electrodes on the short edges of higher aspect ratio geometry had the best heating performance and efficiency [4] so this approach was adopted. As seen in Fig. 2, localised heating can occur at the electrode contact, caused by the contact resistance from the epoxy between the conductive copper and carbon fibres, as noted by Çelik et al [12]. If not controlled carefully, overheating can lead to damage to the component and its tooling, as seen in Fig. 7. Therefore, various layouts of electrodes were tested to reduce this effect, as seen in Fig. 8.

The main observations on the variation of contacts on temperature are:

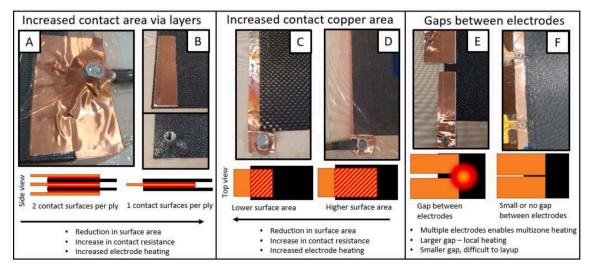


Fig. 8. Examples of copper electrodes used in electric cure: A & B: Demonstrating the use of more layers to sandwich a composite to increase contact area, C & D: Demonstrating the depth of the electrode within the composite to change contact area, E & F: Demonstrating the gaps between multiple electrodes and how these can cause unwanted localised heating.

- Efficient use of surface area in contact with the composite. Fig. 8A shows two electrodes with 2 contact surfaces, whereas Fig. 8B shows one contact with 2 contact surfaces, reducing contact resistance by half.
- Increasing depth into the composite from the edge, increasing the contact area. Fig. 8E generates more local heating compared to Fig. 8D. If the electrode is placed too deep into the layup, the current does not flow at the edge of the composite and is left unheated.
- Reducing gaps between multiple electrodes. When electrodes are placed with a gap between them as in Fig. 8E, the gap between the electrodes becomes a significant hotspot. Reducing this eliminates the hotspot as in Fig. 8F, however, this makes layup more difficult, particularly when this method is adapted for more complex geometries. Multiple electrodes will be essential for zonal heating, to be explored in future studies.

The peak power requirement during the cure cycle is the ramp, meaning heat is also generated at the electrode interface. Therefore, if the ramp rate was increased or lowered, there would be overheating of the electrodes or under-curing of the edges of the component respectively. Therefore, the electrode size and position need to be carefully considered for each cure of a different component geometry.

Full-length, single-piece electrodes were tested to enable a simpler layup, Fig. 8F and G. Multiple electrodes on the same edge had also been tested to enable current to be run through certain sections of the composite, to enable zonal heating. This was not compatible with PW causing excessive heating around the electrodes, however, which will be investigated in future work to modify the heating profile.

BigHead composite fasteners were used as electrodes, enabling the electrode to be fully integrated into the layup as seen in Fig. 8B, which gives composite fasteners dual use post-curing. Integrating a robust electrode within the surface, rather than at the edge, which allows for other joule heating use cases, such as anti-icing or heated composite tooling.

#### 3.7. Infusion development

Adaptations to the process were made to ensure that DEC was compatible with dry fibre, vacuum-assisted resin transfer moulding. The procedure for VARTM is as follows:

- 1. Apply the electrodes to the dry fibres and secure them with high-temperature tape.
- 2. Apply vacuum and do a vacuum drop test on the bag. Any leak can lead to significant localised heating around the electrodes.
- 3. Infuse the resin into the component. Once the infusion is complete, then start the cure cycle and apply current to the composite.

Multicore cable cannot be sufficiently vacuum-sealed, allowing air into the bag when the cable was passed through into the bag, using tacky tape to seal the insulation. The interface was replaced with a bolt, that provided a mechanical lock, power transfer and vacuum seal and enabled a quicker setup. This setup was used on all cures afterwards, including the large-scale components.

Using this method, it is possible to preheat the fibres and the tooling before infusion (heating the fibres without resin), which would decrease the viscosity of the resin during infusion. This may enable the infusion of high viscosity resins with thermoplastic hardeners or fillers to be infused using VARTM methods.

# 3.8. Cure kinetics and power delivery

DEC has high controllability of the power going into the component, compared to closed cavity curing, which offers novel opportunities for temperature control.

The first is to increase the ramp rates beyond 1-3 °C/min which is

standard for many pre-preg systems, including the ones used in this study. To test the potential of higher ramp rates, a small  $200 \times 300$  mm, 6 ply panel of pre-preg was laid up with the electrode arrangement of Fig. 8.

C was cured at 120 °C with a 10 °C/min ramp. The cure system was able to maintain the set temperature at this ramp rate, as seen in Fig. 9.

The peak power output of this ramp was 608 W, meaning that for curing larger components, the control system used in this study wouldn't be suitable for this heating rate. With increased power limits on the power supply, it would be possible to ramp cures at 10 °C/min or more.

Another opportunity is the low thermal mass of the system and how this can be used to further reduce the cure power consumption of the cure. Heat generated in the exothermic reaction of the epoxy can be controlled to further heat the part, without the danger of a runaway reaction. To test this, an infusion resin with a low activation and cure temperature (Gurit T-Prime 130) was infused into a 4-ply,  $1000 \times 350$  mm preform. To ensure a safe, self-sustaining exothermic reaction takes place, the temperature was raised up to 60 °C at 3 °C/min, which can be seen in Fig. 10.

The initial temperature increase set off the exothermic reaction, which continued beyond the cure temperature up to 66  $^{\circ}$ C, after the control system turned off the power. This exotherm lasted for 1 h in duration, ensuring that the composite was at the cure temperature without any external power input. With a more carefully controlled initial power input, the overshoot could have been reduced and time at zero power input increased. The average power after this section was 37 W, with the entire 2-hour 15-minute cure requiring 85.8 Wh.

The increased control over the temperature of components would allow for more custom cure cycles to be developed, which would be controlled by the cure kinetics of the resin. If the heat losses from the cure to the environment and resin cure kinetics could be calculated, then cure cycle time and energy could be minimised, whilst not having a runaway exothermic reaction. Unfortunately this is out of scope of this study, however, it provides another way of energy and cycle time reduction.

#### 3.9. Resistance observations during cure

In Fig. 11, the resistance of the component can be compared to the cure temperature, at the start and throughout the cure. The resistance could be used to detect irregularities in the layup, i.e., if there was a ply missing or an irregularity in the electrode setup. In this case it shows changes to the component during the cure, with electrical resistance increasing in respect to component temperature.

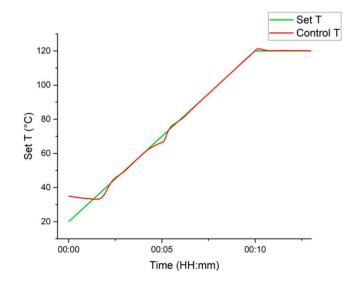
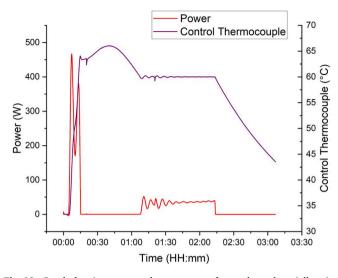


Fig. 9. Example of a sample being heated using DEC at 10 °C/min.



**Fig. 10.** Graph showing power and temperature of a panel cured partially using exothermic energy.

Thermal expansion during heating induces the piezoresistive effect, increasing the electrical resistance of the panel. A combination of these effects leads to an increase in resistance (0.0804  $\Omega$  to 0.0977  $\Omega$ , 18 % increase) from 120 °C to 180 °C. It is unlikely that this could be used as an accurate temperature measurement method, however, it could be used to monitor when the component reaches excessive temperatures. This could be useful to detect when sections of a component are over the glass transition (Tg) or thermal degradation temperature.

There is also an increase in resistance during the first isothermal dwell at 120 °C, where there is an increase from  $0.0772 \Omega$  to  $0.0804 \Omega$ , a 4 % increase. This also occurs at the beginning of the second dwell at 180 °C, from  $0.0910 \Omega$  to  $0.0977 \Omega$ , a 7 % increase. This trend is also seen in other pre-preg and VARTM panels. A physical change that occurs during the cure is the degree of polymerisation of the epoxy, which could account for the increase in the resistance. DC cure monitoring is a method to monitor the electrical resistance of epoxy resin, which can increase by a factor of  $10^4 M\Omega$  during a cure [28]. These resistance changes are also affected by the average temperature over the panel, (Right of Fig. 10) which shows a combination of these effects. During both dwells, the average temperature decreases by around 2–3 °C, whereas resistance increases during the first dwell, where the majority of the cure takes place, approximately 75 % [29]. During the second

dwell, there is a downtrend in resistance, which matches the average temperature trend, and the smaller amount of curing that happens in the post cure. In future experiments, this could be matched to the degree of cure more closely, and resistance changes to be used as an indicator to move to the post-cure.

# 4. Large component manufacturing and cure evaluation

The research and results described in the previous sections were used as the basis for the trial manufacture of two large scale components. Their cured material properties, energy efficiency and ease of manufacture were all evaluated. The two parts' geometries were identical, with one being manufactured with pre-preg and the other VARTM. The plies were  $2000 \times 700$  mm, including a Nomex core mid-ply of a 16-ply layup. This was cured in a GFRP tool that had the geometry of a NACA 2412 aerofoil, as seen in Fig. 12. The pre-preg cure was completed in one cure, whereas the VARTM cure had to be completed in multiple steps to ensure the Nomex core was not filled with resin during the infusion.

## 4.1. Heating uniformity and degree of cure

Curing effectiveness was evaluated by comparing the temperature uniformity and DoC with oven cured samples (following the manufacturers recommended cure cycle). This was done at six different positions on the component, as seen in Fig. 12. Based on previous work on DEC methods [5], it was assumed that if the temperature was matched to an oven cure, the DoC and consolidation of the component would also be the same. DoC for VARTM and pre-preg samples can be seen in Fig. 13.

For VARTM, the average DoC for the six locations is within 6.25 % of the oven cured samples. This includes sample position 2 which is almost 17 % under cured compared to the oven sample. Position 2 is edge of the part having the lowest temperature during the 3 cure stages, which is a result of the non-uniform temperature during the cures. The average is very close to the oven cured samples and shows the potential of the curing method to provide uniform DoC over a component. The range of thermocouple temperatures can be seen against the set temperature for the final cure stage, seen in Fig. 14.

After the ramp to dwell temperature, and the subsequent control system settling, the average range of temperatures is approximately 20 °C. A common standard for curing in aerospace ovens (SAE AMS 2750F Class 2 [30]) requires no more than  $\pm$  6 °C temperature uniformity tolerance. Whilst that has not been achieved in this work,  $\pm$  20 °C has been achieved with sub optimal materials and setup, at a significantly larger scale than that has been observed previously.

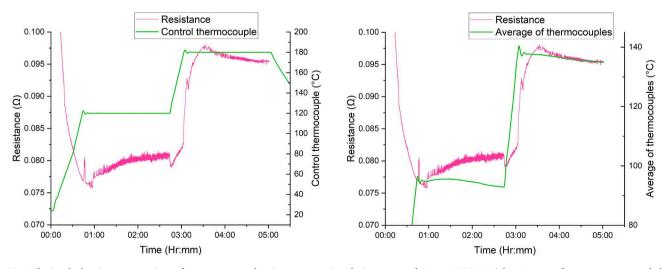


Fig. 11. Left: Graph showing a comparison of temperature and resistance over time during a cure of Cycom 5320–1, Right: Average of temperatures recorded and resistance over the same cure period.

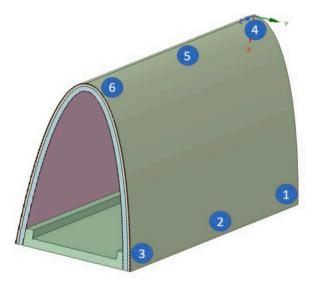


Fig. 12. Leading edge geometry used for large-scale cure evaluation, with numbers indicating sample positions for DoC and VVF evaluations.

For the pre-preg cure, due to power limitations of the PSU, the full 180 °C post-cure temperature was not possible, therefore a 160 °C post cure was used, see the 700  $\times$  2000 mm 16 ply sample in Fig. 6. During the cure, the temperature range was over 30 °C, meaning some areas reached only 90 °C during the cure and 130 °C during the post cure. These are significantly under the desired set temperatures leading to sections of the component being up to 35.77 % under-cured, compared to the 180 °C oven post-cured samples.

#### 4.2. Void volume fraction

VVF was tested for electric cure samples and compared to oven cured samples to assess if the cure method had any detrimental effect. The results can be found in Fig. 13.

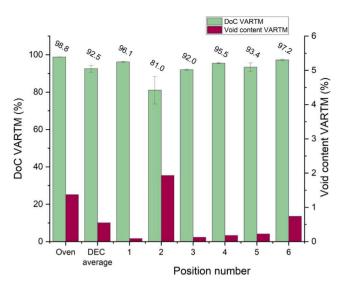
The VARTM component followed the cure cycle ensuring a DoC 6.25 % lower than oven, therefore it is expected the VVF would be similar. However, is it significantly lower, averaging 0.55 %, which is closer to that expected of autoclave cured samples. It is significant that the VVF is lower in the VARTM electric cured components, considering they were prepared similarly and followed the same cure cycles. If this is a consistent trend with DEC, it would be of interest to compare DEC components against autoclave cured ones.

The pre-preg VVF average and standard deviation were significantly higher than the oven cured samples, suggesting whist it is possible to achieve low VVF, it is not as consistent. This is likely due to the uneven temperature profiles being applied leading to void formation, of which examples can be seen in microscopy in Fig. 15.

#### 4.3. Energy consumption analysis

One of the key opportunities with DEC is the increases in energy efficiency of the process, compared to ovens and autoclaves. Energy consumption was compared by normalizing the energy used per hour of cure cycle, which gives an estimation of the efficiency of the curing methods for high temperature cures. Most significantly, the average normalised power usage was reduced by at least 99.15 %, as seen in Fig. 16.

It would be possible to cure more than one of these components in an oven at a time, increasing the efficiency as more are added. In the case of this comparison, even if 25 components could be cured in an oven, using DEC would still results in energy saving of greater than 95%.



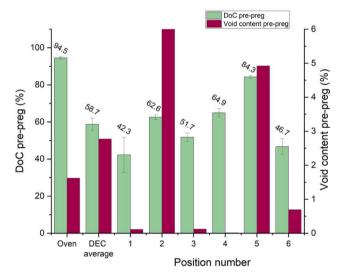


Fig. 13. DoC and void content for both VARTM (left) and pre-preg (right) large scale components.

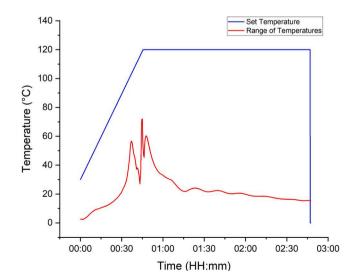


Fig. 14. Range in temperatures on the last stage of cure against set temperature for VARTM component.

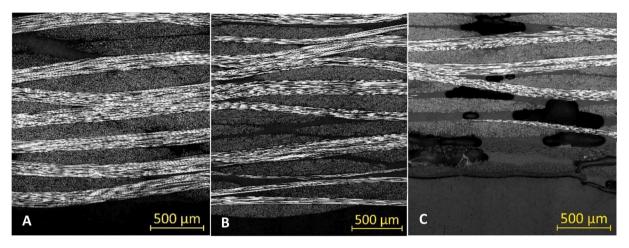


Fig. 15. Microscopy of cross section of final electrically cured components, A) VARTM with 0.09 % VVF, B) Pre-preg with 0.01 % VVF, C) Pre-preg with large voids, 10.81 % VVF.

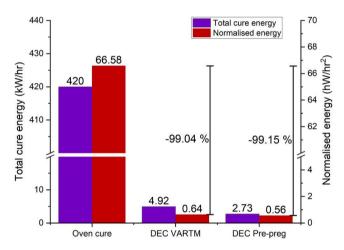


Fig. 16. Summary of cure energy and normalised cure energy of Oven, DEC VARTM and DEC Pre-preg, accounting for different cure lengths.

#### 5. Conclusion

This paper outlines the development of the DEC method, culminating in the manufacture of large-scale aerospace inspired components with plain weave VARTM and pre-preg materials. Safety and part quality was improved compared to previous trials through modifications to the control systems employed. Modifications to the electrodes were made to reduce hotspots, VARTM manufacturing was introduced and practicality of the setup was increased. Energy efficiency was significantly higher than oven curing, with 99 % less energy used when curing large components using the same cure cycle.

Pre-preg samples produced a lower DoC and higher void content, partially due to PW pre-preg composites performing poorer in producing uniform heating, as well as problems in reaching the 180  $^{\circ}$ C post cure temperature due to limitations in the available PSU.

The use of PW reduced the heating uniformity compared to studies that used UD, however in the case of VARTM, it produced samples within an average DoC within 6.25 % of an oven cured part, and 0.82 % lower void content. This shows that it is possible to cure components using DEC, achieving a DoC close to oven-cured samples with comparable or improved void content, whilst achieving a significant energy saving.

Through testing the limits of PW cured with DEC, it increases the knowledgebase on what range of materials it is compatible with, and how best to apply it to new and existing layups. Future research must address the temperature uniformity and resulting DoC, which is essential to application in curing aerospace structures. Another significant boundary to adoption is the added complexity of the layup to enable DEC. For small structures, the benefits of this method may not outweigh the time and cost required to apply electrodes. For larger structures, like the ones demonstrated in the last section of this study, the benefits start to outweigh the negatives. When these issues related to the cure are resolved, the cost reduction in manufacturing large structures will be significant, whilst reducing overall environmental impact. There are also opportunities to use a cure kinetic modelling system coupled with the ability to rapidly heat and closely control the temperature and exotherms to reduce cure times and input energies further. In the meantime research will be of use to other Joule heating applications such as anti-icing or heated tooling, which can be exploited in a shorter timeframe.

# **Funding sources**

Funding: This work was supported by the European Union's Horizon 2020 research programme under grant agreement No 760940, under the project titled MASTRO.

# CRediT authorship contribution statement

M.G. Collinson: Conceptualization, Methodology, Software, Investigation, Writing – original draft. T.J. Swait: Conceptualization, Methodology, Writing – review & editing, Supervision, Funding acquisition. M.P. Bower: Methodology, Investigation, Writing – review & editing. B. Nuhiji: Writing – review & editing, Supervision, Project administration. S.A. Hayes: Conceptualization, Methodology, Investigation, Writing – review & editing, Supervision, Funding acquisition.

# **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

Data will be made available on request.

# References

 Liddell HPH, Brueske SB, Carpenter AC, Cresko JW. Manufacturing energy intensity and opportunity analysis for fiber-reinforced polymer composites and other lightweight materials. In: Proceedings of the American Society for Composites - 31st Technical Conference, ASC 2016; 2016.

- [2] Yoshida H, Ogasa T, Hayashi R. Statistical approach to the relationship between ILSS and void content of CFRP. Compos Sci Technol 1986;25:3–18. https://doi. org/10.1016/0266-3538(86)90018-7.
- [3] Boeing. Commercial Market Outlook. 2022.
- [4] Hayes SA, Lafferty AD, Altinkurt G, Wilson PR, Collinson M, Duchene P. Direct electrical cure of carbon fiber composites. Adv Manuf Polym Compos Sci 2015;1: 112–9. https://doi.org/10.1179/2055035915Y.0000000001.
- [5] Liu S, Li Y, Shen Y, Lu Y. Mechanical performance of carbon fiber/epoxy composites cured by self-resistance electric heating method. Int J Adv Manuf Technol 2019;103(9-12):3479–93.
- [6] Yue C, Zhang Y, Lu W, Zhang Y, Wang P, Li Y, et al. Realizing the curing of polymer composite materials by using electrical resistance heating: a review. Compos Part A Appl Sci Manuf 2022;163:107181.
- [7] Lee J, Ni X, Daso F, Xiao X, King D, Gómez JS, et al. Advanced carbon fiber composite out-of-autoclave laminate manufacture via nanostructured out-of-oven conductive curing. Compos Sci Technol 2018;166:150–9.
- [8] Lee J, Ni X, Daso F, Xiao X, King D, Gómez JS, et al. Supplementary materials: advanced carbon fiber composite out-of-autoclave laminate manufacture via nanostructured out-of-oven conductive curing. Composite Sci Technol 2018;166: 150–9.
- [9] Collinson M, Bower M, Swait TJ, Atkins C, Hayes S, Nuhiji B. Novel composite curing methods for sustainable manufacture: a review. Composites Part C: Open Access 2022;9:100293. https://doi.org/10.1016/j.jcomc.2022.100293.
- [10] Wentzel D, Sevostianov I. Electrical conductivity of unidirectional carbon fiber composites with epoxy-graphene matrix. Int J Eng Sci 2018;130:129–35.
- Dakin TW. Application of epoxy resins in electrical apparatus. IEEE Trans Electr Insul 1974;9:121–8. https://doi.org/10.1109/TEI.1974.299321.
- [12] Çelik M, Noble T, Haseeb A, Maguire J, Robert C, Conchúr, et al. Contact resistance heating of unidirectional carbon fibre tows in a powder-epoxy towpregging line. Plastics Rubber Composites 2022:1–10. doi: 10.1080/14658011.2022.2108982.
- [13] Joseph C, Viney C. Electrical resistance curing of carbon-fibre/epoxy composites. Compos Sci Technol 2000;60(2):315–9.
- [14] Liu S, Li Y, Shen Y, Goh YM. A multi-zoned self-resistance electric heating method for curing irregular fiber reinforced composite parts. vol. 0, 2021, p. 1–6. doi: 10.3233/ATDE210028.
- [15] Zhang B, Li Y, Liu S, Shen Y, Hao X. Layered self-resistance electric heating to cure thick carbon fiber reinforced epoxy laminates. Polym Compos 2021;42:2469–83. https://doi.org/10.1002/PC.25992.
- [16] Fukuda H. Processing of carbon fiber reinforced plastics by means of Joule heating. Adv Compos Mater 1994;3(3):153–61.

- [17] Naskar AK, Edie DD. Consolidation of reactive Ultem®Powder-coated carbon fiber tow for space structure composites by resistive heating. J Compos Mater 2006;40 (20):1871–83.
- [18] Sarles SA. Controlled resistive heating of carbon fiber composites. Virgin Tech 2006:18–60.
- [19] Smith BP, Tuttle ME, Devasia S. Investigation of embedded resistive heating for high strength adhesive bonding of modular space structures 2017. doi: 10.2514/ 6.2017-5228.
- [20] Liu S, Li Y, Xiao S, Wu T. Self-resistive electrical heating for rapid repairing of carbon fiber reinforced composite parts. J Reinf Plast Compos 2019;38:495–505. https://doi.org/10.1177/0731684419832793.
- [21] Weiland JS, Hartmann MP, Hinterhölzl RM. Cure simulation with resistively in situ heated CFRP molds: implementation and validation. Compos Part A Appl Sci Manuf 2016;80:171–81. https://doi.org/10.1016/j.compositesa.2015.10.020.
- [22] Athanasopoulos N, Koutsoukis G, Vlachos D, Kostopoulos V. Temperature uniformity analysis and development of open lightweight composite molds using carbon fibers as heating elements. Compos B Eng 2013;50:279–89. https://doi.org/ 10.1016/j.compositesb.2013.02.038.
- [23] Idris MK, Qiu J, Melenka GW, Grau G. Printing electronics directly onto carbon fiber composites: unmanned aerial vehicle (UAV) wings with integrated heater for de-icing. Eng Res Express 2020;2:025022. https://doi.org/10.1088/2631-8695/ AB8E24.
- [24] Forintos N, Czigany T. Reinforcing carbon fibers as sensors: the effect of temperature and humidity. Compos Part A Appl Sci Manuf 2020;131:105819. https://doi.org/10.1016/J.COMPOSITESA.2020.105819.
- [25] Shen Y, Lu Y, Liu S, Liu Q, Tao S, Hao X. Self-resistance electric heating of shaped CFRP laminates: temperature distribution optimization and validation. Int J Adv Manuf Technol 2022;121(3-4):1755–68.
- [26] Vinson JR. The Behavior of Sandwich Structures of Isotropic and Composite Materials. The Behavior of Sandwich Structures of Isotropic and Composite Materials 2018. doi: 10.1201/9780203737101.
- [27] Jin H, Nelson K, Werner B, Briggs T. Mechanical strength of composites with different overlap lengths. Livermore, CA; 2018.
- [28] Pantelelis NG, Efthymios Bistekos. Process monitoring and control for the production of CFRP components. In: Proceedings of Conference SAMPE'10; 2010.
- [29] Kratz J, Hsiao K, Fernlund G, Hubert P. Thermal models for MTM45-1 and Cycom 5320 out-of-autoclave prepreg resins. J Compos Mater 2013;47:341–52. https:// doi.org/10.1177/0021998312440131.
- [30] SAE International. AMS2750F Aerospace Material Specification Pyrometry. 2020.