The development of a high temperature tensile testing rig for composite laminates

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ARTICLE INFO

Article info
Received 15 November 2012
Received in revised form 30 January 2013
Accepted 15 April 2013
Available online 23 April 2013

Keywords:
A. Ceramic–matrix composites (CMCs)
B. Mechanical properties
C. High-temperature properties
D. Mechanical testing

ABSTRACT

This study aimed to develop a high temperature tensile test capable of testing fibre reinforced composites up to 1000 °C, in order to understand the behaviour of certain composites at these temperatures and produce data suitable in the design of high temperature structures. The test design was assessed using finite element analysis before validating at ambient temperatures against conventional tensile test equipment. The chosen design achieved reliable tensile results and high temperature testing was then successfully conducted, using polysialate composite materials, establishing high temperature mechanical data which was previously unknown. The test setup and data achieved in this study are vital in the design of next generation high temperature structures.

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1. Introduction

The recent resurgence and growing interest in high temperature structures to maximise design space and performance in aerospace and motorsport applications, has led to the need for greater understanding of high temperature composite materials. Fibre reinforced composite materials are typically required for their low weight and high mechanical performance, properties which are necessary for structural application. In addition to this, temperatures up to 1000 °C can be encountered, and as such, ceramic matrix composites are often sought [1]. However, the suitability of materials to perform in these environments is difficult to ascertain due to a lack of high temperature design data.

Mechanical property data is needed to understand the behaviour and limitations of composite materials at high temperatures, as well as allow accurate modelling to take place. Several mechanical tests are available to assess the mechanical properties of a material, of which tensile and flexural testing are the most common. Tensile testing is generally preferred to flexural testing, as flexural testing produces maximum stresses only in small regions at the test specimen surface, which can lead to localised effects. In addition to this, several failure mechanisms can be seen in flexural testing; tensile, compression and shear, which can allow more than one mechanism to dominate results. Tensile testing, however, produces maximum stresses throughout the test specimen as a whole, and the data achieved is more established and appropriate regarding the design and modelling of structures.

There are several components and aspects to tensile testing; the test coupon, grips and strain measurement are all required [2]. Typically tensile testing of composite materials uses hydraulic grips on flat rectangular samples, equipped with bonded end tabs to reduce stress concentrations and through thickness crushing. This is especially important with composite materials as they tend to be weak in the through thickness direction. However, the adhesives used to bond end tabs to composite coupons are only suitable up to a maximum of 300 °C. In addition to this, at temperatures up to 1000 °C attention is needed to both the gripping mechanism and material that the grips are constructed from. Strain gauges are usually employed to measure tensile strain, although these are typically bonded to the coupon, and the same problems with end tab adhesives arise. Therefore, high temperature tensile testing requires unique gripping, heating and strain measuring devices as well as an optimised specimen geometry, in order to successfully test tensile properties of composite coupons at high temperatures.

Current high temperature tensile test equipment is hard to source and typically impractical for use with thin composite laminates. Most available tensile test facilities with the ability to reach 1000 °C are designed for metallics with the use of cylindrical test coupons. Strain measurements are typically taken through the use of clip gauges, which can require notches in the coupon and can impart thermal stress concentrations. This study aims to provide a high temperature tensile test, suitable for thin, flat fibre
reinforced composite laminates. The use of thin coupons, approximately 1 mm, is required as it is representative of those used in structural applications and of the material thicknesses readily available for high temperature composites. In addition to this, there are several requirements that are sought to expand the ease-of-use of the test setup and drive the design. These include a simple, quick and cost effective high temperature tensile test, which makes use of small coupons to reduce cost, and where possible, integration into existing tensile equipment. The ability to add a high temperature capability to existing test equipment will add novelty to the test design as the majority of high temperature testing requires significant investment in both cost and time.

2. High temperature test method

The methods discussed for conducting high temperature tensile testing of fibre reinforced composite materials have highlighted the different approaches that can be taken. The requirements for this study stated that a simple, quick and cost effective high temperature tensile test should be chosen. In addition to this, thin, small coupons are needed due to the cost and availability of ceramic matrix composites, which are a focus of this test method. It is also advantageous for time and resource considerations to make use of existing and available test equipment where possible. With these requirements the most appropriate high temperature tensile test method was developed.

2.1. Grip design and test setup

After an extensive review of high temperature testing methods [2–6], hot passive grips, which grip the coupon through a wedge interference fit and apply edge loading, were selected as the most appropriate gripping technique. The coupon geometry is simplified and less manufacturing is required, as opposed to other methods such as pin grips, which require the drilling of several holes to the test coupon. This grip design can also be easily used with conventional servohydraulic tensile test machines available to this study. The grips will run hot, again to simplify the test procedure but also to remove thermal gradient problems associated with cold grips. In addition to this, the initial cost of the grips is lower than cold grips. This is because less manufacturing is required, as internal coolant networks are no longer necessary within the grip body, which also removes the need for ancillary pump systems to circulate the coolant. The use of hot grips demands that the grips be made from materials which retain sufficient strength at test temperatures. Nickel alloys, specifically Inconel 718 grade, have been selected as the most appropriate grip material for this reason. Although coupon test temperatures of 1000 °C were sought, only localised surface heating of the grips was observed at test temperatures during testing. This was due to the speed of coupon heating and the thermal mass of the grips themselves.

The grip design is similar to that used by Holmes [3] in the testing of SiC reinforced ceramic composites at 1200 °C. The grips comprise of the main grip attachment with an oversized cut-out for the coupon to be inserted, centreing inserts placed either side of the coupon once in the grip, retaining plates either side of the centreing inserts to reduce associated stress concentrations at the root from the coupon cut-out, and finally blinds to prevent the light produced by the rear emitter interfering with the optical strain measurement equipment. Misalignment of the coupon can cause significant bending stresses which can induce failure outside of the gauge section [3]. The centreing inserts reduce coupon misalignment and twist, keeping the coupon aligned between opposing grips, whilst also allowing lateral forces to be imparted on the coupon from the retaining plates. The use of the retaining plates to exert a degree of pressure laterally onto the centreing inserts and coupon face is important when testing thin coupons, as this mitigates any edge crushing effects on the coupon as the inserts are of the same material as the grip body. The grip assembly has been illustrated in Fig. 1, which shows the individual components discussed. Initial finite element analysis (FEA) of the grip setup indicated that significant stresses were developing at the root edges of the coupon cut-out. To reduce these effects the retaining plates were incorporated into the grip setup to reduce splaying of the cut-out forward edges, and as such, stresses at the root edges.

Heating of the coupon is conducted through an indirect heating approach, specifically infrared (IR) emitters. Several indirect heat sources could be used but short-wave IR emitters produced by Heraeus Noblelight Ltd. were chosen due to their small size and ability to reach temperatures as high as 2500 °C. In addition to this, a gold coating is specified on the back surface of the emitter so heat is typically radiated in one direction, minimising heating of other components and maximising the efficiency of the emitter. Initial testing of the test rig indicated that even at maximum emitter power the temperature of the rig and extended grip ends does not exceed 80 °C. In addition to this, the time required to reach uniform coupon test temperatures is typically between 1 and 2 min depending on the temperature sought. This is significant when comparing against conventional testing and heating methods which can require more than 60 min to achieve uniform temperatures. The thermal conductivity of the material tested also has an influence on the rate of heating, however, the use of thin coupons reduces this significantly, highlighting the effectiveness of the IR emitters and their placement within the setup. As heating of the coupon is through radiation, the high temperature capability of the emitters allows us to vary the power or distance of the emitters from the coupon and still achieve the test temperatures required. This extra flexibility in emitter placement is useful in the positioning of strain and temperature measurement equipment. Three emitters are used, placed either side of the coupon faces, allowing the test coupon to be seen from one side. The emitter setup is demonstrated in Figs. 1 and 2. The power of the emitters allows the setup to be unenclosed, which again provides easier access for strain and temperature measurement. Control of the emitters is by varying the power supplied to the emitters in order to achieve a specific temperature. Calibration is required to evaluate the appropriate power and the distance of the emitters to the coupon, to achieve the required coupon test temperature.

Fig. 1. Final grip assembly and test setup; coupon (1), three IR emitters (2), grips and rig (3), blinds (4), thermocouple (5) and bolts (6). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
Several strain measurement techniques are available, however, with consideration given to the test requirements, video extensometry has been chosen. Video extensometers are non-contact strain measurement devices, which reduce external influence on the test coupon [7]. Speed of testing is greatly increased and minimal coupon preparation is required, as opposed to high temperature strain gauges, which require significant sample preparation time and consumables. To conduct video extensometry, a speckle pattern is needed on the coupon to achieve the necessary contrast required for accurate video strain measurements. Coupons were speckled using Dykem® a white high temperature paint produced by ITW Dymon. Blinds were included on either side of the test coupon to remove issues of excessive light caused by the rear emitter. Light produced when the rear emitter is in operation is otherwise directed down the video extensometer camera, which has the effect of over exposing the video and silhouetting the test coupon, resulting in inaccurate strain measurements. The blinds were made from the same composite to be tested, which also provided additional regions for temperature measurement. In addition to this, neutral density (ND) filters were incorporated into the camera to mitigate the levels of light produced from the front emitters reflecting off the test coupon and entering the camera during testing.

Temperature measurement is important not only to determine the test temperature of the coupon but also as part of a feedback loop to calibrate and eventually control the IR emitters. High temperature type-K thermocouples are used as they offer the simplest solution and are easily integrated into a future control setup. To avoid influencing the test coupon, thermocouples can be placed on the blinds either side of the coupon. These are made from the same composite being tested and sit under the exact same thermal conditions. Initially several will be used to determine the temperature distribution, but once calibrated to ensure uniform heating, the number could be reduced.

The complete test rig is illustrated as a CAD model in Fig. 1, where the base plate includes support holes for attachment to the test machines. Fig. 2 illustrates the positioning of the camera viewing port, where strain measurements of the gauge section will be made. The grips are oversized for safety reasons, to ensure reduced heating of the load cell and surrounding tensile test machine. Holes included in the rig are required for attachment of cables and other ancillary components required to operate the emitters, as well as provide access for high temperature thermocouples.

Tensile testing was conducted using an Instron 1342 mechanical tester. Tests were performed over a temperature range of 20–800 °C, with a cross-head displacement rate of 1 mm/min used. At least 4 coupons were tested at each temperature increment. ASTM specifications C1366 [8] and C1359 [9] were followed at all times. Composite tensile strength was calculated using Eq. (1), where $F$ and $A$ are the applied force at breaking point and area of the coupon respectively. Failure strain was measured using a video extensometer supplied by Imetrum Ltd. and combined with the composite tensile stress in Eq. (2) to yield the tensile modulus.

$$\sigma = \frac{F}{A}$$  \hspace{1cm} (1)

$$E = \frac{\sigma}{\epsilon}$$  \hspace{1cm} (2)

2.2. Coupon design

Several high temperature composite tensile testing reviews were consulted and it was found that for passive grips little change in the fundamental specimen design. Tapering of the specimen width between the tab end and gauge length is a consistent feature across studies and was sought in the final design. Worthem [10] investigated how best to homogeneously stress material volumes to initiate failure in the heated gauge section of composite coupons, using tapered widths. This showed that a gradual taper is necessary in achieving reliable tensile failure of composite materials, a feature which was emulated in the design of the coupon used here. Ultimately a coupon design from ASTM standards [8,9,11], echoed by Grande [12] and Holmes [3], for tensile testing of ceramic composites at elevated temperatures was chosen. This provided a simple flat geometry with a suitably gradual taper, as illustrated in Fig. 3, and has been successfully implemented with SiC–SiC composites at temperatures up to 1200 °C [3]. Thicknesses of 3 mm are suggested by Holmes [3], however, the specimen thickness can be decreased or increased as required. The decision to use 1 mm thick material was taken due to the requirements of the study.

2.3. Materials

High temperature composite materials suitable for testing up to 1000 °C needed to be selected in order to verify the test design. In addition to this, the material should be representative of those to be used in high temperature structures in order to maximise the reach of the research. Typically, traditional engineering ceramic matrix composites (CMCs) would be envisioned for these applications, however, one material which has shown potential for application in high temperature structures is polysialate matrix composite.

Polysialates are ceramics derived from inorganic polymers and processed through a polymerisation chemical activation, rather than the extreme temperature processing synonymous with traditional engineering ceramics. This gives them a number of advantages over typical CMC materials such as low production times,
environmental friendliness and low density [13]. High temperature resistance, up to 1200 °C, and an incombustible nature, with no harmful smoke or fumes being released [14–16] are also retained. Polysialates can be reinforced with several different reinforcements, although the most common are carbon or silicon carbide (SiC) fibres, typically in a woven architecture. Some of the most comprehensive research in this field was conducted by Davidovits [17], who first applied the term geopolymer to these materials, with several terms now being used, such as inorganic polymers, or in this case, polysialates. Very little thermal or mechanical data exists for polysialate composites, with the majority of data available limited to testing conducted at ambient temperatures. This study aims both to develop the testing method and to generate useful high temperature data for this class of composites.

The polysialate composite materials used in this study were produced by Pyromeral Systems, as they produce the most developed and utilised polysialate-type materials commercially available. Composites are available with both carbon and silicon carbide reinforcement, but given the temperature range explored in this study, silicon carbide reinforcement was selected. This material is referred to as PyroSic® and is a polysialate matrix composite, reinforced with Tyranno silicon carbide fibres in a plain weave architecture. Due to quality control concerns all materials were produced in-house by Pyromeral Systems. Test coupons made use of 1 mm thick material constructed from 4 woven plies using a 0/90 layup, as this is representative of the material likely to be employed in high temperature structures. Coupons were cut using computer numerically controlled (CNC) equipment with the 0 fibre direction orientated along the coupon length, to achieve the geometry and tolerances required for use in the passive gripping mechanism.

3. Finite element validation

The grip design was modelled using FEA to establish the loading distribution on the coupon and determine any problems with the design. Three dimensional (3D) quarter models of the grip setup were created and analysed using Abaqus CAE. The grip and coupon were modelled, with the influence of centreing inserts, retaining plates and bolts represented using relevant boundary conditions and loads.

The geometry of the grip meant that solid elements were needed in order to effectively capture the behaviour of the setup. The grip body made use of C3D10 10-node quadratic tetrahedrons as the complex geometry, including holes and curvatures, proved difficult to mesh with hexagonal elements. The coupon was modelled using C3D20 20-node quadratic hexagonal bricks and incorporated a more refined mesh than that used in the grip, as stress within the coupon is of primary interest. The final mesh used is the outcome of a mesh refinement study, which optimised the mesh against the stresses achieved and the required computational time. The model is illustrated in Fig. 4, where the equivalent mesh densities are visible. Symmetry boundary conditions were used to represent the complete geometry, with surface interactions used to simulate contact and friction between the coupon and relevant components. Load was applied to the end of the coupon in the form of a ramped uniform tensile stress, which ensured uniform loading of the coupon and simulated tensile test conditions. The applied pressure of 275 MPa is representative of the room temperature tensile failure stress of PyroSic, as given in the datasheet [18]. In addition to this, thermal boundary conditions simulating a temperature rise from 20 °C to 1000 °C were implemented on the coupon, in order to study the effect that the coefficient of thermal expansion (CTE) has on stresses within the grip and coupon. Temperature dependant CTE data for both PyroSic and Inconel alloys were taken from previous thermal characterisation and manufacturers data-sheets respectively.

The temperature conditions are in all probability an over estimation. The coupon is unlikely to achieve 1000 °C over the whole geometry due to contact with the grips, which act as a thermal sink. Also, the grips will most probably achieve more local temperature concentrations, closer to the emitter heat sources, rather than a
uniform temperature distribution. However, these conditions produce a worst case scenario which should always be designed for. Boundary conditions and interactions within the model are more complex than first appears, as the coupon interacts against the grip and centreing insert surfaces with a degree of friction. Assumptions are made to simulate these effects, which could induce some error in the results achieved. These conditions and the quarter model design permitted suitable run times, whilst still providing adequate information on the stress state within the grip and coupon.

3.1. Results

Fig. 5 shows the longitudinal S11 coupon tensile stresses present in the coupon at 25 °C and 1000 °C. The results show that tensile stress across the coupon simulated at 25 °C reduces from 288 MPa in the gauge section to approximately 0 MPa at the tab end. Similarly, at 1000 °C, the stress reduces in comparative manner, but with slightly higher magnitude, from 290 MPa to becoming slightly compressive at approximately −13 MPa. In both instances the stress is higher in the gauge section, promoting tensile failure in this region as desired. The taper in the coupon produces this effect, where suitable transition in tensile load is observed, and with it, confidence in the accurate high temperature tensile testing of composite coupons.

Small stress concentrations are noted on the edges of the gauge section. This is probably caused by ‘pinching’ of the coupon by the edge of the coupon cut-out, as the grip expands due to CTE effects and is constrained by the retaining plates. In addition to this, greater compressive stress is observed in the 1000 °C simulation due to compression of the grip on the coupon, again in all likelihood due to CTE expansion, although the magnitudes are very low. It is also possible that these effects could be due to limitations in the modelling technique. It is worth noting that at this stage in the design process the tensile strength of PyroSic at elevated temperatures was unknown. In reality the strength could be higher or lower at elevated temperatures, which would have an impact on failure, although FEA results indicate that tensile failure should still dominate, due to the gradual taper in the coupon geometry.

Transverse S22 stresses across the coupon, perpendicular to the tensile stress, and S33 stresses through the thickness, are less significant. Fig. 6 shows that compressive contact stresses are observed near the coupon edge in both the ambient and high temperature simulations, although this is expected due to the nature of the gripping mechanism used. Localised contact stresses of −50 MPa are observed in the 25 °C simulation, which gives rise to maximum stress of −40 MPa across the coupon width, but applied away from the gauge section. A similar trend is observed with the 1000 °C simulation, where contact stress is now higher at −150 MPa, but reduces to a maximum or −100 MPa across the coupon width. Compressive contact stresses are in both cases below the −170 MPa compressive resistance of PyroSic [18]. This increase in stress magnitude over the low temperature simulation, is coupled with a shift in area of influence closer to the gauge region. This is again due to thermal expansion effects, with the influence area shifting as the grip profile in this region is more constrained due to the retaining plate and associated components, encouraging the profile to expand preferentially in the direction of the coupon.

S33 stresses through the coupon thickness are negligible with only the clamping force from the centreing insert and retaining plate evident.

The compressive gripping stresses perpendicular to the tensile load could cause some localised edge crushing. However, the stress should not affect tensile results as they are away from the gauge region and of significantly lower magnitude. In addition to this,
the centreing inserts will act to mitigate any edge crushing effects and stop any out of plane deformation from occurring, whilst ensuring true tensile failure of the coupon. A small but significant compressive stress concentration can be observed in the high temperature simulation, in the rear corner of the coupon tab region. This is thought to be unrepresentative of reality given its location and magnitude, and is probably due to boundary effects, over constraint or singularities. However, it serves to highlight the complexity of the interactions and, in any case, has no effect on the performance of the test in performing accurate tensile loading.

Stresses in the grips never exceed the high temperature strength of Inconel alloys, which retain a yield strength of 275 MPa at 900 °C [19]. The main stress regions are as anticipated, located in contact areas between the coupon and grip in the S22 and S33 directions. Similarly as with the coupon, small regions of stress concentrations are prominent at the edge of the model, however, this is most probably due to over constraint and boundary effects.

Strain distribution in the coupon follows a similar pattern as that for the tensile stress. Maximum elongation of 0.34 mm is confined to the gauge section, with very little change in the strain magnitude or location with the change in temperature. Total strain achieved mimics the 0.9% quoted in the datasheet, which confirms material properties are well simulated by the model.

Modelling was conducted to determine if the grips would be properly loaded and to ascertain if any unexpected effects were occurring. Full confidence in the results is difficult given the complexity of boundary conditions implemented, however, it serves to indicate tensile failure is most probable even at high temperatures, although practical testing of the setup was required to validate the analysis and confirm this.

4. Results and discussion

4.1. Test validation

Testing of the final design and setup was undertaken to verify the choices made and FEA predictions. This included establishing whether the coupons were being correctly loaded, in order to ensure accurate tensile properties can be evaluated from the results. In addition to this, the region of failure was noted in order to determine the primary failure mechanism. The passive grips were validated by testing PyroSic coupons against control tests of coupons tested in conventional hydraulic grips, at room temperature. In-stron tensile machines were used in both tests. The results achieved for both the passive grips and control tests are given in Table 1, which includes the standard deviation present in the test data.

The results show that the passive grips compare extremely well with the control tests, highlighting that the grips can produce data similar to traditional testing methods. In addition to this, all test coupons tested in the passive grips failed in the gauge section, as predicted in FEA and should guarantee a tensile failure mode and accurate tensile data, even at elevated temperatures. The average strength results for the passive grip are similar to those achieved in the control tests at 288 MPa. In addition to this, a standard deviation of 5% highlights the reproducibility of the passive grip and test setup. Modulus results for the passive grips are also very reliable at 32 GPa with a standard deviation of 8%. This is slightly lower than the modulus of 33 GPa achieved in the control tests, but neither the stresses nor strains measured are significantly different from those measured using the conventional test methodology.

Strain results were slightly higher for the passive grip, at 0.99%, than that achieved for the control tests at 0.89%, again with an associated standard deviation of 9%. All test coupons tested in both the passive and hydraulic grips demonstrated a linear stress strain behaviour.

Further confidence in the test is gained when data is compared against data sheet values for PyroSic materials [18]. The strength and strain data from both the passive grip setup and control tests agree well with each other, and data available in the PyroSic data sheet, which gives values of 275 MPa and 0.9% for tensile strength and strain to failure respectively.

4.2. High temperature tests

Tensile strain, strength and modulus were measured using the high temperature tensile test described. Maximum test temperatures were restricted to 760 °C due to restrictions with the IR emitter placement and material performance. This was due to the available proximity of the IR emitters to the test coupon, which were impeded by direct interference from the grips. An increase in coupon length would remove this interference and allow the front two emitters greater access with regard to heating the test coupon, which in turn would result in higher available test temperatures. All test coupons failed in the gauge section and all tests

Table 1

| Grip type | Strain (%) | Strength (MPa) | Modulus (GPa) |
|-----------|------------|----------------|---------------|
| Passive   | 0.99 ± 0.09 | 288 ± 14.0     | 32.0 ± 2.5    |
| Hydraulic | 0.89 ± 0.08 | 266 ± 18.5     | 33.4 ± 3.9    |

Fig. 7. Strain performance of polysialate composites at elevated temperatures, error bars represent ± one standard deviation. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 8. Strength and stiffness performance of polysialate composites at elevated temperatures, error bars represent ± one standard deviation. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
produced linear stress strain relationships, with uniform heating exhibited in the coupons at each test temperature.

The tensile properties of the polysialate composite tested undergo significant change over the test temperature range. Polysialate composite failure strain increases from approximately 1.0–1.3%, as shown in Fig. 7. Conversely, strength decreases from 288 MPa to 213 MPa, which ultimately reduces the stiffness of the material from 32.0 GPa to 17.3 GPa, as illustrated in Fig. 8. The error bars shown in the test results highlight the reproducibility of the test method. Error experienced in the higher temperature results is comparable to that in the room temperature and hydraulic control tests. This gives confidence in the data and test design, where the error present is therefore more likely to be present due to bulk material inconsistencies in a brittle material. Standard deviation in the vast majority of results is less than 10%.

5. Conclusion

The results show the test procedure is capable of giving reliable results in the high temperature testing of thin composite coupons. Grip design and the IR emitters provided uniform tensile loading and rapid heating of coupons respectively, with test run and turn around times significantly faster than conventional high temperature methods. Results strongly suggest that accurate high temperature tensile testing can be performed with this grip design and test setup, with the data achieved accurate and offering a good representation of how composite materials behave under tensile loading at elevated temperatures. Temperatures of 760 °C were achieved during testing but this could easily be increased with an adjustment in coupon size.

The test rig heating design is suitable for both ceramic and polymer matrix composites, and it may be possible to adapt it for compression and bending tests. The inclusion of control mechanism for temperature control would be a useful addition to future test changes and to ensure accurate and reproducible temperature and heating rates. The rapid heating and cooling achievable with this design of test rig allows it to be used to efficiently investigate important engineering properties, such as the impact of thermal cycling and thermal shock under load.

Acknowledgements

The author thanks McLaren Racing Ltd. for the supply of materials used and financial contribution throughout the study. The financial contribution of the ESPRC in sponsoring this work as part of the ACCIS DTC (Advanced Composite Centre for Innovation and Science Doctoral Training Centre) at the University of Bristol, Grant Number EP/G036772/1, is also gratefully acknowledged.

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