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Thermal and tensile properties of polysialate composites ☆

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Abstract

Polysialate, or geopolymer, composites have gained interest due to their inherent high temperature resistance, low density and ease of manufacturing. These characteristics also suggest that polysialate composites have significant potential as materials in high temperature structures, although little is known about their thermal and mechanical properties. This study aimed to determine relevant thermal and mechanical properties over a representative temperature range. The results show that polysialate composites can exhibit stable thermal properties up to 1000 °C. Tensile properties up to 760 °C highlight a significant reduction in stiffness, but a retention of strength, at these temperatures. The thermal and mechanical results achieved provide strong evidence that polysialate composites can be suitable for use in high temperature structures, whilst subsequently providing an understanding of their limitations. In addition to this, the values ascertained also provide the data required for the design and modelling of next generation high temperature structures.

Keywords: C. Thermal properties; C. Mechanical properties; E. Structural applications; E. Thermal applications

1. Introduction

Improvements in engine efficiencies, compaction of components closer to heat sources to reduce mass, and performance gains associated with use of high temperature exhaust flows has produced a demand for high temperature, light weight structures for both aerospace and motorsport applications. The key requirements for these applications are a demand for high temperature resistance up to 1000 °C, low density and good mechanical properties. Secondary requirements include the ability to form complex geometries at low lead times. The demand within industry for high temperature structures is significant and developments in this field have the potential for immediate implementation.

Polymer matrix composites (PMCs) have traditionally been exploited to produce light weight structures. Typically, carbon fibre epoxy, polyimide, bismaleimide or cyanate ester composites are used in these applications, however, they can only withstand maximum operating temperatures up to 300 °C. Ceramic matrix composite (CMC) materials are often used in higher temperature applications, as these can withstand elevated temperatures in excess of 1600 °C, although concerns still remain regarding their structural performance. The cost, and more specifically processing times, of CMC's can also be prohibitive when considering application in high temperature structures. In addition to this, the mass penalty compared to traditional PMC materials can be significant and as such their use has been limited. There is, therefore, a need for materials which bridge this gap to aid in the development of high temperature structures.

One such material which meets the initial requirements for high temperature structures are polysialate composites. Polysialates are ceramics derived from inorganic polymers and processed through a polymerisation chemical activation, rather than the extreme temperature processing synonymous with ceramic materials. The polysialate matrix is based on polymineral resins which contain alumino-silicate binders, allowing them to cure at temperatures below 150 °C. This allows them

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to be manufactured using conventional polymer composite lay-up and bagging techniques [1,2]. Not only does this allow conventional composite laminating and curing equipment to be used, but it also allows for complex geometries to be fabricated, offering a unique advantage over traditional ceramic composites. This includes low lead times, as parts are fabricated using similar cycle times to traditional prepreg PMC's. In addition to this, they possess significant high temperature resistance, environmental friendliness and low density, where density is typically less than other ceramic composite systems and comparable to PMC's at 1800 kg/m^3 [3]. Polysialates also retain the oxidation resistance of ceramic materials, which is important as high temperature environments are typically oxidising in nature. The advantage over polymer matrix composites is that although they have a low cure temperature they still maintain a high temperature resistance, up to 1200 °C, and an incombustible nature, with no harmful smoke or fumes being released [2,4,5]. Polysialates can be reinforced with several different reinforcements [6,7], 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 [8], who first applied the term geopolymer to these alkali activated alumino-silicates, with several terms now being used such as inorganic polymers, or, as 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. Mechanical data gathered by Papakonstantinou et al. [1] shows that polysialate composites are comparable with conventional CMC's and can be stiffer, if not stronger, than conventional PMC's. These properties make them an ideal candidate material for high temperature structures, with previous studies identifying their potential in these applications [9]. Laminates, sandwich panels and beam structures have all been constructed using polysialate composites [6,7,10,11], although only the compressive or flexural response at ambient temperatures has been documented. Very little is known about their high temperature thermal or mechanical performance, data which is critical in evaluating their high temperature structural suitability. In terms of high temperature mechanical properties, current work has only evaluated the flexural and shear properties of polysialates at high temperatures [2,5,12–14], data which is difficult to implement from a design perspective. This study aims to correct this absence whilst simultaneously understanding the suitability of polysialate composites as materials for high temperature, low weight structural solutions.

This will include characterisation of the thermal and mechanical properties of polysialate composites up to temperatures of 1000 °C, in order to fully evaluate their high temperature structural potential. The thermal properties include thermal conductivity, specific heat capacity and coefficient of thermal expansion (CTE), all of which are necessary in the evaluation of heat transfer in structures under thermal loads. Mechanical properties will be evaluated as ultimate tensile strength, strain and Young's modulus, as tensile properties are much more applicable than flexural properties in the design of structures. In addition to this, mass loss over the specified temperature range is considered, as any changes could be significant in the performance and limitation of the material. The construction of composite materials and reinforcement results in an orthotropic nature, where the material can have different properties in all three principal directions. This is considered where necessary in the evaluation of properties, in order to provide the full spectrum of design data. In addition to understanding the behaviour and suitability of polysialate composites as materials for high temperature structures, the data is also required in the modelling of heat transfer and stress response of structures constructed from polysialate composites, a step which is critical in the design and development of future high temperate structures.

Only commercially available polysialate composites will be considered, so as to assess the capability of present technology in developing high temperature structures. This also satisfies the demand for immediate application of these concepts within the aerospace and motorsport industries. At present polysialate composites have only been used in heat shield applications, it is hoped that this study will allow exploitation of their properties to other, more structural, applications.

2. Experimental

2.1. Materials

Polysialate composite materials produced by Pyromeral Systems were chosen as the material to investigate, as it is one of the most developed and widely used polysialate materials commercially available. Composites are available with both carbon and silicon carbide reinforcement, although given the temperature range explored in this study, silicon carbide reinforcement was selected. This material is referred to as PyroSic® and utilises a potassium polysialate matrix, reinforced with Tyranno silicon carbide fibres in a plain weave architecture, with an average fibre diameter of 11 µm. This produces a composite with a 30% fibre volume fraction. Due to quality control concerns all materials were produced in-house by Pyromeral Systems.

2.2. Sample preparation

Coupons for thermal and tensile property evaluation were autoclave cured in a controlled environment before post-curing at higher temperatures in order to stabilise the matrix. The exact cure conditions are considered confidential by the manufacturer, however, typical autoclave conditions use a temperature of less than 150 °C and a required pressure of less than 9 bar.

Thermal property analysis was conducted using material with a plain weave 0/90° layup and 3 mm thickness, in order to fully evaluate properties representative of the bulk composite rather than the constituent elements, and in order to meet minimum thickness requirements in the thermal analysis equipment. Coupons for thermal property analysis were created using a wet saw, with dimensions suitable for the test equipment used. Coupons for tensile testing made use of 1 mm thick material, constructed from 4 woven plies using a 0/90° layup, as this is representative of the material thickness likely to be employed in high temperature structures. The use of thinner material will not have an influence on the results achieved due to the relationship between force and the cross-sectional area that the force is applied to, which is necessary in determining tensile properties. Coupons for tensile testing were cut using computer numerically controlled (CNC) equipment as the test requires complex coupon geometry and tight tolerances, due to the gripping mechanism employed.

2.3. *Mass loss analysis*

Mass loss analysis was performed using a simultaneous thermal analyser (Netzsch STA 449 F3 Jupiter®). Experiments were conducted in a dynamic argon atmosphere with a gas flow rate of 70 ml/min and a heating rate of 20 °C/min. Samples were held in graphite crucibles.

2.4. *Thermal conductivity analysis*

Thermal conductivity was measured in the in-plane and through thickness fibre directions using laser flash apparatus (Netzsch LFA 457 F3 MicroFlash™). Measurements were conducted in a dynamic argon atmosphere at a flow rate of 100 ml/min.

2.5. *Specific heat capacity analysis*

Specific heat measurements were performed using differential scanning calorimetry (Netzsch DSC 404 F1 Pegasus®). Experiments were conducted in a dynamic argon atmosphere with a gas flow rate of 70 ml/min and a heating rate of 20 °C/min. Samples were held in graphite crucibles.

2.6. *Thermal expansion analysis*

CTE measurements were conducted in the in-plane fibre direction using a high temperature dilatometer (Netzsch DIL 402 C). The dilatometer uses much larger test coupons than other CTE measurement methods, such as thermal mechanical analyser (TMA), which makes it more representative of the bulk material. Experiments were conducted with a heating rate of 3 °C/min.

2.7. *Tensile testing*

Tensile properties were evaluated using an Instron 1342 mechanical tester. Tests were performed over a temperature range of 20–760 °C using a previously developed high temperature test setup [15], which built upon work conducted by Holmes [16]. A cross-head displacement rate of 1 mm/min was used, with at least 5 coupons tested per temperature point. ASTM specifications C1366 [17] and C1359 [18] were followed at all times, using the coupon geometry given in Fig. 1.

Composite ultimate strength was calculated using Eq. (1), where F and A are the applied force at breaking point and cross-sectional area of the coupon gauge section respectively. Ultimate strain was measured using a video extensometer supplied by Imetrum Ltd and combined with the composite tensile stress in Eq. (2) to yield the Young's modulus.

$$\sigma = \frac{F}{A} \tag{1}$$

$$E = \frac{\sigma}{\epsilon} \tag{2}$$

3. Results and discussion

3.1. *Mass loss*

Results for the mass loss of PyroSic composite over a temperature range of 20–1000 °C are given in Fig. 2. Mass loss was minimal and restricted to a total of 0.7%, the majority of which takes place before 500 °C. Although the amount of mass loss was minimal, there is a significant kink in the mass loss data. Between room temperature and 400 °C a mass loss of 0.5% occurred with a relatively shallow gradient. However, between approximately 400 °C and 500 °C the rate of mass loss increased substantially, with a further 0.2% decrease in mass over the 100 °C temperature difference. This increased rate then stabilises, with no further mass loss up to 1000 °C.

This behaviour and loss of thermal history could be due to the release of moisture or decomposition of impurities within the composite. Due to the chemical curing process of polysialate materials, the mass loss could also be due to the curing of unreacted elements, which would also suggest insufficient post-cure temperatures were achieved during composite manufacture. Ultimately the mechanisms behind this phenomenon require additional dedicated investigation to fully understand the processes behind the exhibited mass loss. The same polysialate composite sample was tested again over the same temperature range, with no further mass loss exhibited.

3.2. *Thermal conductivity and specific heat capacity*

Thermal conductivity and specific heat capacity of PyroSic composite is presented in Fig. 3. Data for the conductivity is given in-plane and through the thickness, as the woven nature of the material dictates that these should be different enough to warrant investigation. Specific heat capacity was determined with a preheated sample to remove any discrepancies associated with mass loss.

Through thickness thermal conductivity was fairly static over the 20–600 °C range at approximately 0.7 W/mK. This gradually increased to 0.8 and 0.9 W/mK for 800 °C and 1000 °C respectively. A similar trend was observed in the in-plane thermal conductivity, albeit at a higher starting value. Between 20 and 600 °C temperature range the thermal conductivity is

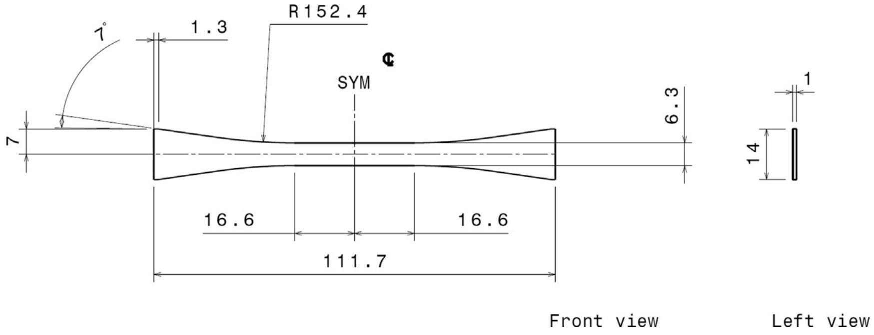

Fig. 1. Tensile coupon geometry.

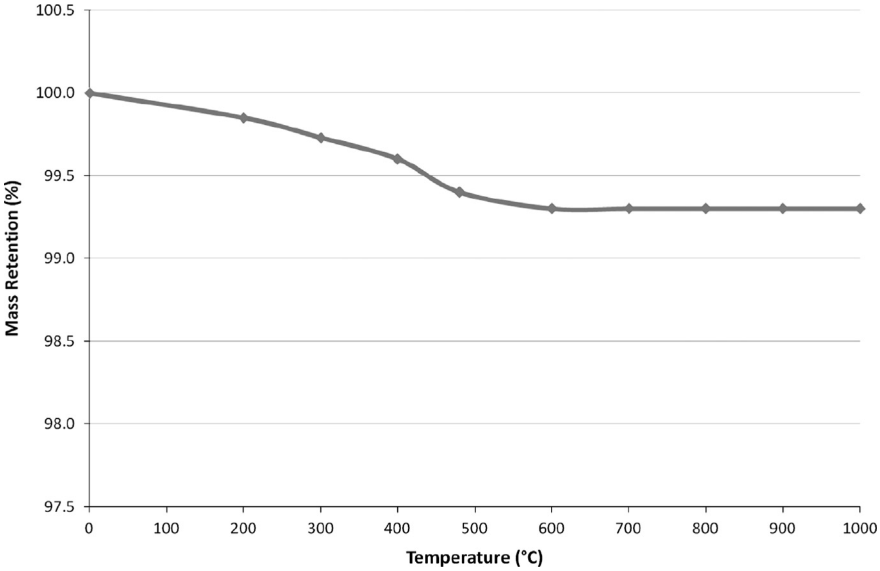

Fig. 2. Mass loss of polysialate composite over 1000 °C range.

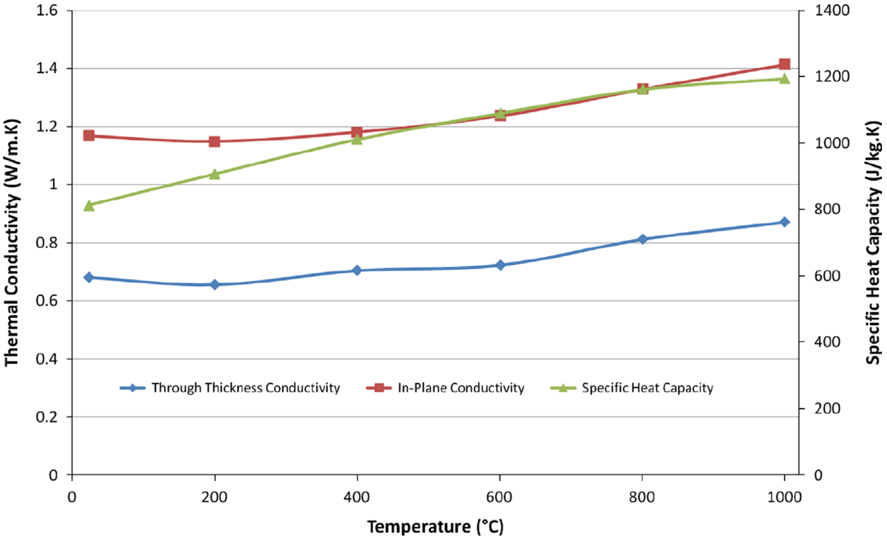

Fig. 3. Polysialate composite thermal conductivity and heat capacity.

approximately 1.2 W/mK, which again increases to 1.3 and 1.4 W/mK between 800 °C and 1000 °C. The thermal conductivity of the SiC reinforcing fibres suggests that the in-plane results are reasonable. The Tyranno fibres used in PyroSic composites have a thermal conductivity of approximately 2.5 W/mK [19]. The thermal conductivity of the polysialate matrix is as yet unknown, but as an engineering ceramic material it can be assumed to be low. In-plane the fibres have significantly more influence in the overall properties of the composite, both thermal and mechanical, because they are orientated in this direction. The in-plane thermal conductivity is therefore greater than the through thickness due to the higher thermal conductivity of the fibres, in comparison to the polysialate matrix, and the influence that they have in the in-plane directions. The assumption of the polysialate matrix having a lower thermal conductivity than the reinforcement is given weight with the through thickness results. The matrix properties are dominant in the through thickness direction and a lower conductivity of approximately 40% was observed. The woven nature of the composite ensures that the in-plane conductivity is the same in both the 0 ° and 90 ° orientations. Specific heat capacity rises almost linearly from 800 to 1200 J/kgK over the temperature range.

3.3. Thermal expansion

The in-plane thermal expansion measurements are given in Fig. 4. Measurements were made on samples already exposed to 1000 °C in order to remove any effects from the previously documented mass loss. The results indicated an initial rapid increase in CTE over the first 180 °C, from 3.0×10^{-6}/°C to 6.9×10^{-6}/°C, after which CTE reduced steadily to 5.2×10^{-6}/°C until the test end.

In-plane thermal expansion is dominated by the reinforcing fibres. The Tyranno fibres used in reinforcing this polysialate composite have a CTE between 3×10^{-6}/°C and 4×10^{-6}/°C [1,19]. The CTE of the polysialate matrix used in the composite is unknown, as they vary depending upon the specific composition used. However, it is generally higher than the reinforcing fibres, with an average CTE of approximately 15×10^{-6}/°C over a similar temperature range suggested by He et al. [12]. The individual thermal expansion of the composite elements supports the CTE measurements exhibited in the data, especially given the dominance of the reinforcing fibres in the in-plane direction.

The initial variation in CTE could be due to water loss within the material, curing of unpolymerised components in the matrix, or crystallisation of the polysialate as suggested by Barbosa and MacKenzie [20]. However, all of these are unlikely as the material has already been exposed to 1000 °C in order to remove any effects due to mass loss, and as such would already experienced any of the suggested mechanisms. The reduction in CTE is not due to melting or creep as the data shows the material becomes dimensionally stable again after 800 °C. Porosity will affect the CTE of the material. As a ceramic material, some porosity is likely and this could in part be responsible for the results observed.

The relatively small magnitude of CTE means that an average value for in-plane CTE of 5×10^{-6}/°C is an acceptable approximation over the majority of the 1000 °C temperature range. The likelihood of matrix cracking due to thermal shock in ceramic composites is directly linked to the CTE. Understanding the thermal expansion of PyroSic will help to determine the thermal fatigue resistance of the composite and structures constructed from it. Given the relatively low CTE values over the temperature range, the material is likely to perform well in thermal fatigue, which is another indication to the potential of polysialate composites in high temperature structures. The data attained is especially useful in the accurate modelling of hot structures, as changes in CTE can impart significant stresses within materials.

3.4. Tensile properties

Ultimate tensile strength, strain and Young's modulus were recorded from room temperature to 760 °C. Higher temperature testing was not conducted due to limitations in the test equipment, which included the available proximity of infrared heating emitters to the test coupon. Failure remained in the gauge section of coupons and all were of a relatively linear

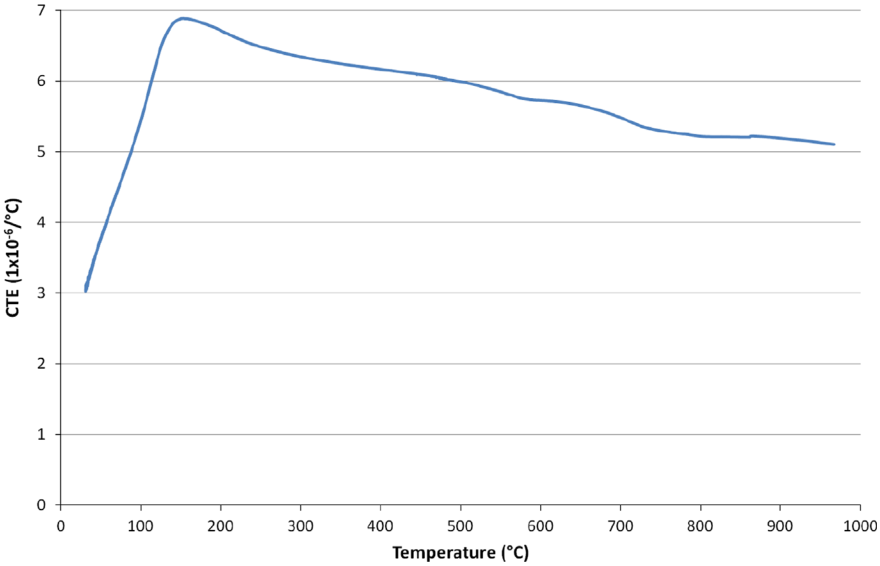

Fig. 4. Polysialate composite in-plane CTE.

stress strain nature, as shown in Fig. 5, with brittle failure observed throughout.

Significant changes were exhibited in the tensile properties of the polysialate composite coupons over the test temperature range. Strain increases across the temperature range, whereas a decrease in strength and Young's modulus was observed. Ultimate strain increases slightly from approximately 1.0% at room temperature to 1.1% at 300 °C. After this, ultimate strain remained almost constant, creating a kink between 300 °C and 500 °C, before again increasing linearly to approximately 1.3% at 760 °C, as shown in Fig. 6. A similar, albeit reversed, trend was observed in the ultimate strength results shown in Fig. 7. Ultimate strength decreased slightly from room temperature to 300 °C, from 288 MPa to 281 MPa, after which the decrease was much more severe. There is a clear kink between 300 °C and 500 °C in the strength performance of the polysialate composite, where the largest gradient is observed. The ultimate strength continued to reduce between 500 °C and 760 °C but at a much reduced gradient, concluding in a ultimate tensile strength of 213 MPa at 760 °C. Ultimately these values result in a negative effect on the Young's modulus, reducing it steadily from 32.0 GPa at room temperature to just 17.3 GPa at 760 °C. The behaviour of the Young's modulus is illustrated in Fig. 8, where stiffness reduces at a much shallower gradient from 300 °C to 500 °C, owing to the trend seen in the strain results.

Overall, ultimate strain increased by 30%, whereas the Young's modulus reduced significantly to less than 54% of the room temperature value over the 760 °C temperature range, indicating an improvement in material toughness. The reduction in ultimate strength is not as severe, with a loss of only 25%, representing useful strength retention at 760 °C. Softening of the matrix and degradation of the fibre bond are possible causes for the reduction in mechanical properties. However, an interesting observation is the kink seen in the

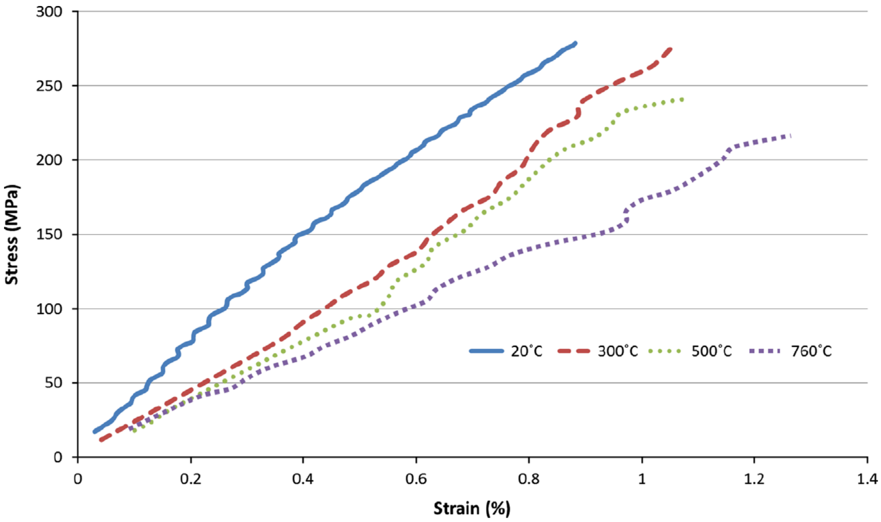

Fig. 5. Polysialate composite stress strain curves over the tested temperature range.

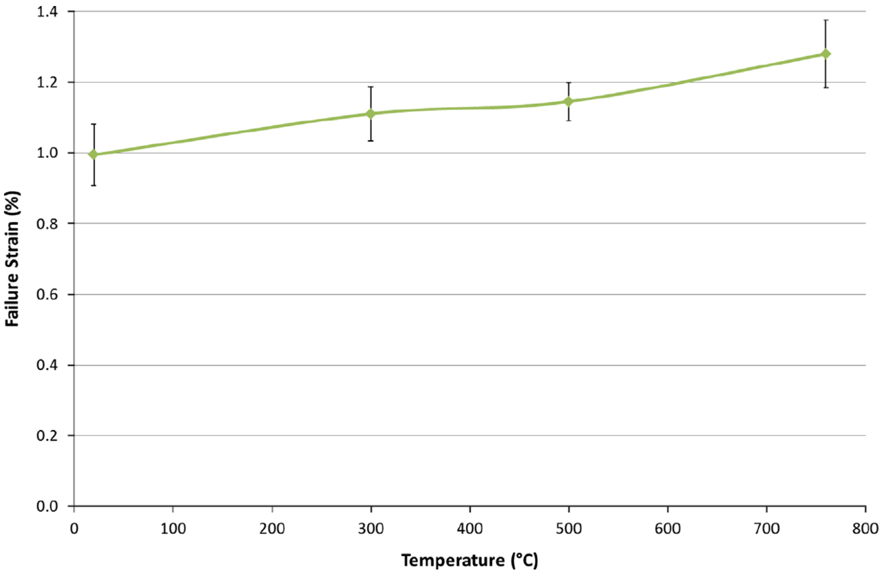

Fig. 6. Polysialate composite ultimate strain performance up to a temperature of 760 °C, error bars represent ± one standard deviation.

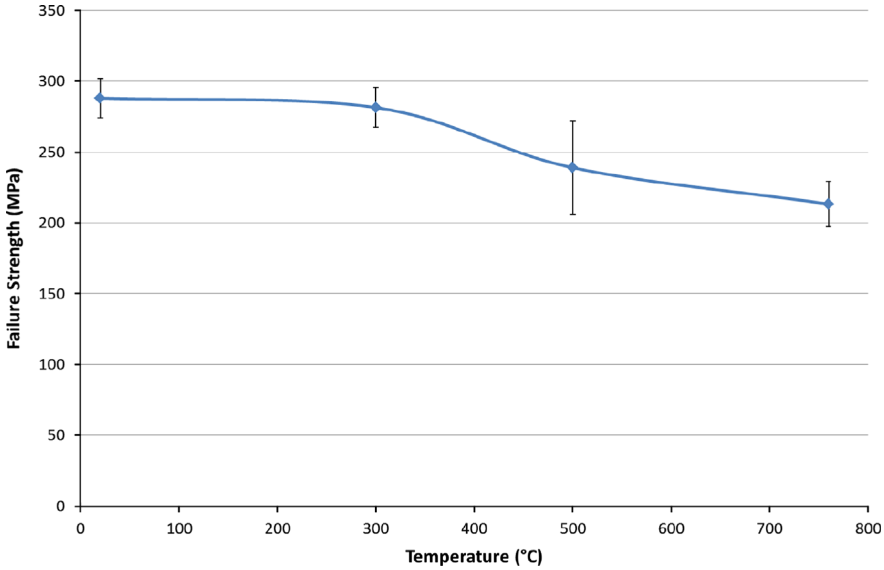

Fig. 7. Polysialate ultimate strength performance up to a temperature of 760 °C, error bars represent ± one standard deviation.

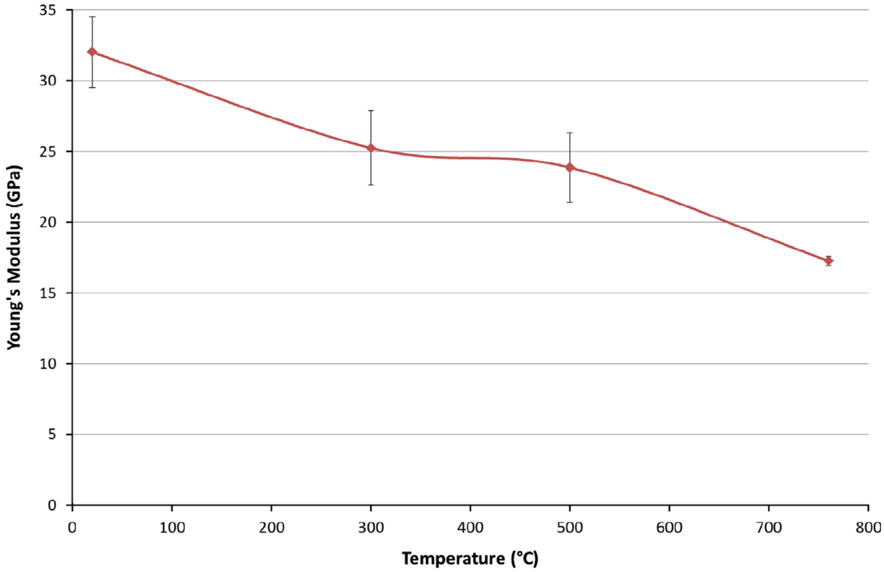

Fig. 8. Polysialate composite Young's modulus properties up to a temperature of 760 °C, error bars represent ± one standard deviation.

ultimate strength results, and to a lesser extent the ultimate strain and Young's modulus results. Here the gradient at which values change differs considerably to that at the temperatures either side of it, a pattern which appears to mimic the mass loss behaviour given in Fig. 2. Although the exact mechanisms are not known, it would appear that there is a correlation between the increased loss of mass between 300 °C and 500 °C, and the increased reduction in ultimate strength over the same temperature range. This would imply that some thermal degradation has occurred, which would also manifest itself at room temperature following exposure to these temperatures.

Overall error is low, with the vast majority of results achieving a coefficient of variation of less than 10%. The error experienced in the higher temperature results is comparable to that in the room temperature tests. This gives confidence in the data set, and would suggest that the error present is more likely due to bulk material inconsistencies, such as voids and fibre irregularities, rather than the test procedure.

4. Conclusion

The thermal properties and tensile properties of 0/90° polysialate composites reinforced with silicon carbide fibres have been examined up to temperatures required for practical consideration as materials for use in high temperature structures.

Minimal mass loss up to 1000 °C highlighted the material's high temperature resistance, with scope for future work regarding understanding of the mechanisms involved in this mass loss and subsequent loss of thermal history. Thermal conductivity and specific heat capacity values were in line with expectations for ceramic materials and the constituent elements that make up the composite. Thermal conductivity measurements in both the through thickness and in-plane orientations provide useful and often neglected values for the design and modelling of heat transfer, which is of great consideration when using materials at high temperatures. The thermal conductivity in all directions is low enough to confirm its use as a heat shield material, but also its future potential in high temperature structures.

In terms of CTE, the results achieved provide temperature dependent in-plane data up to 1000 °C. Low CTE response is observed over the whole temperature range, which suggests good thermal fatigue performance. Data is verified through a level of agreement with the independent CTE data for the matrix and reinforcing fibres used in the composite.

Tensile mechanical properties show an overall increase in ultimate strain, but decrease in both ultimate strength and Young's modulus from room temperature to 760 °C. However, there is still sufficient retention of ultimate tensile strength over the temperature range to suggest potential for use in high temperature structures. The decrease in stiffness also suggests a degree of toughness improvement in the material. Broadening the range of mechanical data, such as investigation into shear and compression properties at elevated temperatures, offers the potential for future work. In addition to this, interesting correlations with mass loss and mechanical property reduction indicate that further investigation into this relationship could prove useful.

The results and data set achieved highlight the potential for polysialate composites for use in high temperature structures. In addition to this, the data presented is required in the accurate modelling of heat transfer and stress conditions, relating to the thermal and mechanical loads that high temperature structures are subjected to. Orthotropic properties also make thermal modelling of polysialate composite laminate behaviour much more accurate and representative of real-world problems.

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