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Material Properties

Thermal analysis and degradation of properties in carbon fiber/epoxy laminate riveting at high temperatures

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A R T I C L E I N F O A B S T R A C T *Keywords:*Joints/joining Thermal analysis Mechanical testing Finite element analysis (FEA) A riveting process was developed to fasten carbon fiber/epoxy composite laminates. This process heats a thermoplastic composite rivet blank placed into the joint hole, above melt temperature, to form a rivet fastening the joined components. This process poses a threat to the integrity of the structure's epoxy matrix. To verify the state of matrix degradation, a pin-loaded mechanical test was applied on composite test pieces heat treated at various temperatures. The heat treatment was specifically designed to recreate the riveting process of the test piece during the heat treatment. For use in the finite element model, emissivity was characterized through infrared image analysis, and thermal conductivity was characterized with an inverse method. The results show

laminates. However, the process at 450 °C does affect the bearing properties.

1. Introduction

In recent years, advanced composite materials have seen an increase in use. Their high stiffness to weight and strength to weight ratios make them an interesting alternative to traditional metallic materials in structural applications [1]. The aerospace industry adopted composite materials in an effort to further decrease the weight of aircraft for environmental and economic reasons [2]. Initially used in aircraft secondary structures, composite materials are now widely used across the various structures of modern aircraft. Due to the nature of advanced composite materials and their relative novelty, design of composite structures is more complex than for their metallic counterparts. Among the challenges of composite structure design, joining poses a problem since the traditional joining methods developed for metallic materials give rise to several issues when applied to composite structures. For instance, galvanic corrosion, mainly between carbon fiber composites and aluminum alloys, occurs when the materials are in contact. Solutions to impair corrosion such as sealants or using titanium fasteners usually imply added weight and diminish the advantage of composite materials [3], [4]. The use of metallic fasteners also creates a safety hazard in the case of a lightning strike on the aircraft. Metallic fasteners form an ignition site which could be catastrophic around a fuel tank. Integral protection of composite structures has been studied by Pridham et al. [5] as a way to reduce the damage to the structure and the risk of sparks at fastened joints. Sealing the fastener is another alternative to

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https://doi.org/10.1016/j.polymertesting.2018.02.002 Received 21 November 2017; Accepted 2 February 2018 Available online 03 March 2018 0142-9418/ © 2018 Published by Elsevier Ltd. avoid sparks in case of a lightning strike [6].

that a riveting process at nominal temperature of 350 °C will not affect the bearing properties of the composite

The issues listed above show the need for a joining method adapted to advanced composite structures. Non-metallic fasteners have seen some development in recent years [7]. The use of similar materials in the fastener and structure eliminates the incompatibility issues. A new riveting process for aerospace composite structures is in development [8]. The process involves a thermoplastic matrix composite rivet blank heated in-situ. Schematic representation of the process before and after the forming of rivet heads can be seen in Fig. 1. The rivet blank is heated by resistive heating. An electrical current and a riveting force are applied simultaneously on the rivet blank. Once the matrix has reached its process temperature, the rivet heads are formed. This process may be detrimental to the composite structures to be joined since high performance thermoplastics have melting points well above the glass transition temperature (T_g) of aerospace epoxy at around 180 °C.

At high temperature, thermoset degradation depends on both the time and temperature exposure. More than one degradation mechanism comes into play [9–11]. Polymeric chains can be decomposed by chainend scission, random scission at weaker bonds in the chain or scission of side groups called chain stripping. In epoxy, random scission is the dominant decomposition mechanism and can be activated by heat (thermolysis) or by the presence of oxygen (thermo-oxidation) [10]. McManus and Chamis [12] and Zhang et al. [9] have studied the thermo-oxidation of composites and neat resin. Thermo-oxidation is usually superficial and requires long exposure to affect the material's







Fig. 1. Thermoplastic composite riveting process: a) Before forming of rivet heads; b) After forming of rivet heads.

properties. Chatterjee [13] and Anderson [14] have studied the degradation of epoxy by thermolysis. Thermolysis occurs at higher temperature and affects various properties of thermosets such as T_{g} , dielectric properties and mechanical properties. Fatigue and static mechanical properties of composites has been studied by Gonzalez et al. [15] and Flore et al. [16]. Flore et al. found that mass loss was the most defining factor in predicting the residual mechanical properties of thermally aged composites. Considering these heat degradation mechanisms, understanding and modeling the thermal history experienced by the joint members during this riveting process is essential for its adoption by the industry.

Heat propagation modeling through composite materials requires accurate prediction of their thermal conductivity. Several factors influence the properties of a laminate. Fiber orientation, fiber volume fraction and interfacial thermal resistance are among the factors impacting the thermal conductivity of a composite laminate. On one hand, analytical models were shown to be overly simplistic in the evaluation of thermal conductivity of composites [17] and experimental methods are costly and require large specialized equipment. On the other hand, inverse methods offer a way to evaluate parameters that are difficult to measure experimentally. Inverse methods have been used with success in evaluating the thermal conductivity of advanced composite materials [18-21]. At moderate temperatures, heat loss by radiation can be significant when compared to convection losses. The emissivity of materials must consequently be characterized for radiation to be taken into account. Emissivity depends on a material's surface condition, wavelength and direction [22]. Usually, a reflectometer is used to measure hemispherical and directional emissivity for several wavelengths. Madding [23] used thermal imaging with a reference material with a known emissivity as a way of calculating the emissivity of other materials.

In riveted joints, the bearing properties relate to the mechanical behavior of the joined material in the hole where the fastener is installed. This property is critical for the design of structural joints. In composite materials, the failure modes of fastened joints is more complex than in isotropic and ductile metallic materials [24], [25]. The bearing response involves an intricate progression of localized compressive failures [26]. Environmental conditions can affect the bearing properties of polymer matrix composites because of their interactions with the matrix [27]. Johnson et al. [28] have shown that aging at elevated temperature has an effect on the bearing capacity of bolted joints. Hygrothermal aging was studied by Parida et al. [29] and was found to have an impact on the bearing strength of carbon fiber/epoxy composites. It is clear that the thermal history of composite laminates affects the performance of fastened joints.

The purpose of this study is to model the temperature evolution during the riveting thermal cycle and characterize its effect on the bearing properties of composite structures. To do so, two experiments and corresponding finite element (FE) models were used jointly in an inverse method to characterize the thermal conductivity of the composite laminate. Emissivity and thermal conductivity were characterized and validated for the FE model. Mechanical tests were conducted on composite test pieces heat treated with a thermal cycle similar to the riveting process. A FE model was elaborated to further explore the temperature exposure history in the composite.



Fig. 2. Infrared image analysis for characterization of emissivity of composite laminate at 75 $^\circ\text{C}.$

2. Materials and methods

2.1. Characterization of emissivity

Emissivity was characterized using infrared image analysis software (VarioAnalyze, JenOptik). Two pieces of aerospace-grade pressure sensitive tape (PSA) with a known emissivity of 0.96 were applied on a composite sample. The sample was heated to set temperatures of 25, 50, 75 and 100 °C and let stabilize. An infrared image (VarioCam, JenOptik) was then captured for each set temperature. Fig. 2 shows an example of the infrared image analysis. It illustrates the composite sample on the heating apparatus. Two regions identified as "Tape" and "Sample" can also be seen. By comparison to the known emissivity of the PSA, the emissivity of the "Sample" region was then manually adjusted until the average temperature of the two regions matched. The process was repeated for the infrared image of each set temperature.

2.2. Characterization of thermal conductivity

For the thermal conductivity characterization, an inverse method was used. It consisted of two steady state experiments and corresponding finite elements models. Temperatures were measured experimentally once using each set-up. The two experiments and FE models are designated in-plane and out-of-plane for the direction of the temperature gradient. In both experiments, the same test piece equipped with embedded thermocouples was used. The test piece was manufactured from a prepreg twill weave with a $[0F/45F]_{6S}$ stacking. The prepreg resin was Solvay 977-2 and was cured in an autoclave at 177 °C according to the manufacturer's recommendations. The sample was 76.2 mm by 76.2 mm with a thickness of 7.8 mm. The thermocouples were disposed on the greater diagonal of the sample, at every four plies.

For the out-of-plane experiment, the same heating apparatus and sample was used as for the emissivity characterization. As seen in Fig. 3 a), it consisted of a metallic block with cartridge heaters. The sample was placed on top of the block, heating it from below. A thermocouple placed near the heating surface controlled the temperature via a PID. Insulation was placed around the block to decrease heat loss. The experiment was carried out in air, as all other experiments of this study. Temperatures in the sample were recorded for 5 min on a GraphTec GL-220 data logger once thermal equilibrium was reached. Temperature recordings were done in a steady state to simplify the heat transfer

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