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Effects of processing conditions on bondline void formation in vacuum bag only adhesive bonding: Modelling, validation and guidelines

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A B S T R A C T

Analytical modelling and adhesive characterizations were used to predict void nucleation and growth in an adhesive film under ideal, and then realistic conditions of pre-bond moisture, consolidation pressure, and curing temperature. Void growth evolutions were measured *in-situ* by means of an instrumented transparent tool plate. Void development observations were found to be in close agreement with the analytical estimations obtained by a water diffusion-based model. The results identified the source of void formation: pre-bond moisture, while adhesive solvent was not found to cause void nucleation for the studied adhesive film. The findings also highlight the sensitivity of void formation to process variations. For example, the constructed processing maps identified that for very low content of pre-bond moisture (as received ~0.1 wt%), moisture-induced voids can nucleate and grow in the adhesive if the temperature exceeds the recommended cure temperature or if the applied pressure is reduced, for example, in repair shops at altitude. Processing widows are proposed to provide guidelines to adhesive bonding practitioners.

1. Introduction

1.1. Background

The growing share of composite materials in primary structures of commercial aircraft pushes the need for cost-effective and reliable repair methods in the manufacturing and maintenance industry. While adhesive bonded repair methods are attractive, they lack robustness for airworthy certification other than for cosmetic repairs [1]. Co-bonded scarf repairs quality and subsequent performance are highly process-dependent. Co-bonded repairs are typically carried out using an epoxy adhesive film and repair plies that are consolidated out of autoclave, with a single vacuum bag, under one atmospheric pressure only (~ 1000 mbar). Additionally, in a repair environment, adhesive films are sensitive to moisture uptake at room temperature, and pre-bond moisture may be present within the structure to be repaired [2]. Under conditions of limited available compaction pressure, the presence of pre-bond moisture and/or volatiles within the uncured repair materials may generate voids in the adhesive by off-gassing dissolved species at elevated temperatures.

1.2. Importance and source of porosity in bonded applications

The presence of voids, or empty cavities within the adhesive microstructure, is typically large in bonded repairs. Porosity levels of 5–10% of porosity are common in wet-lay-up [3] and prepreg repairs [4]. This is a major concern for repairing load-bearing structures since bondline porosity significantly reduces repair strength. A strength reduction of 5% per bondline areal void content percent was reported for scarf bonded repairs [5].

During processing, presence of bubbles in the adhesive is caused by three distinct gas-induced mechanisms: air entrapment, volatile and moisture release [6]. First, entrapped air may be evacuated during the initial vacuum application stage (debulk) if the adhesive film allow transverse gas transport at room temperature by means of perforations and embossing of adhesive films, or partial impregnation strategies for prepregs [7]. A second source of gas-induced porosity may arise during the polymerization reaction by the release of volatiles contained in the resin. To mitigate this source of void, thermal treatment, such as B-staging of adhesive film is found to lower the initial amount of volatile in un-cured materials measured by Fourier Transform Infrared Spectroscopy (FTIR) [6,8,9]. Third, un-cured resins are also readily susceptible to moisture absorption during storage and at room temperature [10]. At higher temperatures, the subsequent release of water vapour may occur and cause void to nucleate and grow [11].

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Nomenclature

C_{bulk}	Water concentration in adhesive bulk [kg/m^3]
C_{vs}	Water concentration at the void surface [kg/m^3]
d	Bubble diameter [mm]
D	Moisture diffusion coefficient [mm^2/min]
E_a	Activation energy [J/mol]
k	Water saturation constant [-]
M	Molecular weight [-]
p_{water}	Partial pressure of water [bar]
r	Bubble radius [mm]
S_m	Moisture saturation level [%]
β	Void growth driving force [-]
ΔH	Water enthalpy [J/(K mol)]
λ	Pressure loss constant [%]
$P_{adh.}$	Adhesive pressure [bar]
ρ_a	Adhesive density [kg/m^3]
ρ_g	Bubble gas density [kg/m^3]
φ	Relative humidity [%]

1.3. Void formation conditions and modelling during processing

Bubble size depends on the equilibrium between the gas pressure in the bubble P_{void} and the surrounding hydrostatic adhesive pressure P_r. If $P_{void} > P_{adh}$ then a bubble grows, and on the contrary, a bubble shrinks when $P_{void} < P_{adh}$. Adhesive pressure is a function of the applied pressure, and gas pressure in the bubble depends on the nature and content of the gas components, as well as the imposed temperature [6,12].

In vacuum bag only processing, because the applied consolidation pressure is limited, all three types of gas-induced void mechanisms can lead to unacceptable levels of porosity. Several researchers have developed models to predict void formation and growth during composite processing. Kardos's popular model [12] is constructed upon a simplified approach of Scriven's detailed early work [13]. Both models framework rely on water transfer though the resin/bubble interface between an infinite medium of resin saturated with water and a bubble of gas. Epstein [14], and Wood and Bader [15] also studied diffusion-controlled void growth. These authors included surface tension effects and initial nuclei size in their framework, but concluded that these factors were only important at the very early stage of void nucleation. Recently, in an attempt to refine void diameter predictions, Ledru, Bernhart [16] added the thermo-mechanical effects induced by resin viscosity changes during the process to previous diffusion-based void growth models.

These various models approaches were used to predict void evolution for autoclave cure of various thermoset composites [12,17], and were also later applied to forecast void growth in partially impregnated prepreg materials in vacuum bag only processing [11]. Nonetheless, while the aforementioned studies show good agreements for void nucleation predictions, each model tends to over-predict by several orders of magnitude the final void sizes compared to measurements by optical microscopy.

2. Objective, approach and materials

The present paper proposes to verify the robustness of a diffusion-based void growth model in the context of adhesive bonding under vacuum bag pressure only. The objective of this study is to predict the onset of void growth and final content in a common adhesive film under various processing conditions of humidity, temperature, and pressure. First, a diffusion-based model is presented along with the characterization of model input parameters in a common adhesive film. Then, after the model is compared to void growth measurements by means of an instrumented glass-tool plate, model assumptions and limitations are discussed. Finally, the validated model is used to produce repair process maps. These process windows predict final adhesive porosity under a wide range of realistic and deficient processing conditions and provide guidelines to repair practitioners on how to prevent the formation of moisture-induced porosity in adhesive bonding.

2.1. Materials

The adhesive considered for this work was a toughened epoxy B-staged adhesive film: Cytec FM® 300-2 M. This typical repair adhesive film has an areal weight of 293 g/m^2 and a nominal thickness of 0.25 mm. Cure kinetics and rheological analytical models have been proposed in [9] and were used to predict the gelation of the adhesive used in this study. Moreover in [9], Fourier Transform Infrared (FTIR) gas Spectrometry was used to characterize the volatiles released during cure. Metyl Ethyl Ketone (MEK) was identified near its vaporization temperature under ambient pressure at 79.6 °C, and water traces were detected near its boiling point until adhesive gelation.

3. Void growth modelling

3.1. Model framework – governing transport equations

The void evolution model employed here is based on water transfer from the surrounding uncured epoxy into the void at a bubble/adhesive interface, as shown in the schematic in Fig. 1. Considering a bubble of radius *r(t)* at the centre of an infinite pseudo-homogenous epoxy medium, which has a known initial concentration of water C_{bulk}, the water concentration varies with time and the radial coordinate r from C_{vs} at the void surface $r = R$, to C_{bulk} at $r = \infty$. Assuming a constant moisture diffusion coefficient D, second Fick's law can be applied for this spherical geometry:

$$\frac{1}{D}\frac{\delta C}{\delta t} = \frac{1}{r}\frac{\delta^2}{\delta r^2}(rC) \tag{1}$$

With the solving details presented in [18], at the void surface $r = R$, the water concentration gradient becomes:

$$\left.\frac{\delta C}{\delta r}\right|_{r=R} = \frac{C_{bulk} - C_{vs}}{R}\left(1 + \frac{R}{\sqrt{\pi D t}}\right) \tag{2}$$

The rate of water mass m, transferred from the bulk adhesive into the void, is modelled as an inward flux J at the void surface expressed

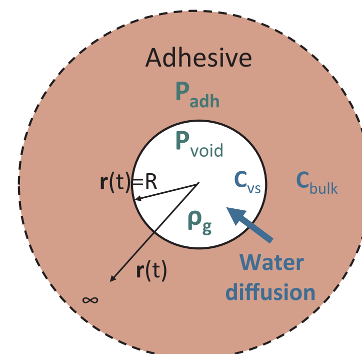

Fig. 1. Schematic of the void growth cellular model showing a spherical void of internal pressure under adhesive pressure, and of known adhesive water concentration and void surface water concentration.

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