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Characterizing the constitutive properties and developing a stress model for adhesive bond-line readout

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ABSTRACT

Visible deformation on the exterior surface of adhesively-bonded automotive body panels in the vicinity of the adhesive application line is referred to as bond-line readout (BLRO). Differential shrinkage of the adhesive and substrate as an assembly cools from bonding temperature to room temperature is the primary factor responsible for BLRO. The gradual relief of bondline readout over time suggests that relaxation of residual stresses within the adhesive layer occurs. This work addresses determination of viscoelastic and thermal expansion characteristics of representative epoxy and polyurethane adhesives for input into numerical models to predict bondline readout. Besides the constitutive properties, usefulness of the bimaterial curvature technique was also investigated for measuring residual stresses in a coating layer, as well as the stress-free temperature. Knowing the constitutive properties and stress-free temperature, the residual stresses and deformations in a steelepoxy bimaterial specimen were modeled using a simple recursive approach.

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1. Introduction

Adhesive bonding in automotive exterior panels offers a number of advantages such as improved stiffness and durability, reduced noise and vibration, and improved sealing against environmental challenges. This assembly method becomes a primary joining option in automotive closure panels if the substrates are made of dissimilar metals or reinforced composites. Typically, a bead of adhesive is applied to the surface of one component. The two panels are positioned and pressed together. The assembly is often heated to accelerate the cure of the adhesive bond. Additional curing of the adhesive may take place at room temperature or during subsequent thermal excursions to which the bonded structure is exposed. In spite of numerous advantages associated with the use of adhesives for automobile manufacturing, a continuing difficulty arises in that a surface deformation along the adhesive bond line is sometimes visible on exposed surfaces of the exterior panels. This bond-line readout (BLRO) effect is usually viewed as a defect in the surface of the door or panel assembly when it is visible to the user of the vehicle (defect on a 'class A' surface). The human eye is remarkably perceptive at detecting slight waviness (\sim 10 μ m) in reflected images from

surfaces [\[1\]](#page--1-0), and such visible distortions in the surface are considered to be unacceptable by customers. Blunk defines three major categories of BLRO—ridging, optical and mechanical [\[2\].](#page--1-0) Mechanical BLRO describes the deformation in the panel and is attributed to the coefficient of thermal expansion difference and moduli difference between the adhesive and the substrate. This type of BLRO is the focus of the paper.

Thermal shrinkage of adhesives and the resulting residual stress buildup due to thermal mismatch of adhesive and adherends is the primary factor contributing to BLRO. The gradual relief of bondline readout suggests that relaxation of residual stresses within the adhesive layer may occur over time [\[3\]](#page--1-0). Epoxy and polyurethane adhesives are widely used for bonding metallic and sheet molding compound (SMC) panels or other reinforced polymer panels. These adhesives provide good bond strength in joining complementary panels or in attaching panels to metal frame members. But these adhesives generally have higher coefficients of thermal expansion (CTE) than that of the composite or metal panels or frame members. Due to the difference in the CTE between the adhesive and the adherends, each experiences different expansion or shrinkage during thermal cycling for adhesive curing, paint baking, or other processing conditions. Recent studies, however, have shown that it is the first thermal cycle only that develops BLRO, and the subsequent thermal cycles do not contribute [\[4–6](#page--1-0)]. The thermal shrinkage and not the chemical shrinkage due to crosslinking in adhesive is by far the more dominant phenomenon leading to residual stresses [\[7–9\]](#page--1-0). Additional deformation modes and residual stresses also arise

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when the two adherends are dissimilar, due to differences in the CTE [\[10\]](#page--1-0).

Fernholz et al. discussed the recent efforts to understand the relationship between BLRO severity and various materials and process variables through completion of a series of experiments [\[7,11](#page--1-0)]. The goal of the experiments was to determine ways to minimize the distortion without the cost and weight penalty associated with increasing the thickness of the outer panel. They examined the severity of the distortions under a variety of conditions, including experiments in which the geometry of the inner panel was modified so that relatively large bond standoffs, very small bond standoffs and/or adhesive 'dams' were molded into the inner panel of the assembly. In one of these experiments, some assemblies were bonded with the standoffs and dams on the inner panel in contact with the outer panel, a so-called 'hard-hit' condition. Their work concluded that the location and volume of adhesive, and thus the adhesive cross-sectional geometry, are the two most critical factors that must be controlled to minimize the occurrence and severity of the BLRO-distortions when the type of adhesive, outer panel thickness, and bond fixture temperature are fixed [\[7,11](#page--1-0)]. In conjunction with the studies by Fernholz et al., Fuchs et al. used analytical tools to predict the surface distortion observed after adhesively bonded sheet molding compound (SMC) composite assemblies at elevated temperatures [\[12\]](#page--1-0). Initial studies using a finite element analysis (FEA) based approach showed good agreement with experimental observations and highlighted the importance of accounting for viscoelastic adhesive material properties. FEA model predictions based on viscoelastic material properties for the adhesive and linear elastic material properties for the substrate resulted in substantially better correlation between predicted and measured distortions [\[13\].](#page--1-0) Hahn and Jendrny investigated the influence of thermal expansion and curing shrinkage, as well as relative movements of the adherends on the resulting deformations [\[14\]](#page--1-0). They used viscoelastic properties of adhesive to simulate the BLRO deformation.

Usefulness of the bimaterial curvature technique to measure residual stresses in a coating, as well as the stress-free temperature (T_{sf}) has been reported in references [\[15–19](#page--1-0)]. Because of the similarity of the stress state in this configuration to that experienced by the adhesive in a typical automotive bond-line, the bimaterial curvature technique offers a means to characterize residual stresses, the T_{sf} , and an alternate but indirect method to measure the difference in coefficient of thermal expansion between the coating and the substrate [\[20\]](#page--1-0). These simple, selfloaded specimens can also provide insights into how stresses evolve as a function of aging time. Thermal (heat–cool) profiles relevant to automotive processing conditions can be run in a commercial dynamic mechanical analyzer (DMA), as the end deflection of a cantilevered bimaterial strip is measured. Because adhesives typically have larger coefficients of thermal expansion than metallic and SMC substrates, the polymer will typically experience tensile stresses as the bimaterial strip is cooled. If the substrate is initially flat, any bending of the bilayer strip indicates that residual stresses are present in both the polymer and the substrate. By measuring the curvature, and knowing the geometric configuration and elastic properties of the substrate, one can nominally determine the CTE and residual stresses in the adhesive [\[15\].](#page--1-0) Furthermore, by noting the temperature at which the curvature begins to build substantially, one may readily establish the T_{sf} , which is often within \pm 10–20 °C of the glass transition temperature of the polymer, but is affected by the cooling kinetics and viscoelastic relaxation associated with the thermal history and aging conditions. Knowing the CTE and T_{sf} , a simple linear relationship may be used to estimate the residual stresses, as these are responsible in developing the BLRO.

In this paper we report: (1) characterization of the viscoelastic and thermal properties of representative adhesives for input into predictive numerical and analytical models for BLRO, (2) use of the bimaterial curvature technique for measuring residual stresses in the coating, and the T_{sf} and (3) an existing analytical recursive formulation to model the bending curvature by modifying time-independent Stoney equation to incorporate viscoelasticity of the adhesive.

2. Experimental procedure

2.1. Materials

The three adhesives characterized in this study were epoxy 'A', epoxy 'B', and polyurethane.

Due to the extremely high room temperature viscosity of two of the adhesives, they had to be heated before dispensing into the mold. In order to do so, the adhesive supplier provided industrial dispensing units for their epoxy A and polyurethane adhesives. Epoxy B had a relatively low room temperature viscosity and could be dispensed using handheld cartridges. Bake-hardenable 210 steel substrates with the dimensions of $40 \text{ mm} \times$ 4 mm \times 0.85 mm (L \times W \times T) were used to conduct the bimaterial curvature measurements.

2.2. Specimen preparation

Neat adhesive specimens with nominal dimensions of 55 mm \times 15 mm \times 1.7 mm ($L \times W \times T$) were cast in a silicone rubber mold (Smooth-Sil™ 940-Reynolds Advanced Materials[®]) that was designed to make five specimens at a time. Excess adhesive was dispensed into the mold ensuring that adhesive filled the corners of the mold and no air was entrapped in the specimens. The excess adhesive was then scraped from the top of the mold to create specimens as close to the specified dimensions as possible. The adhesives were cured at $90^{\circ}C$ (per manufacturer's specification) for 6 h. After cure, the specimens were easily removed from the silicone mold without damage. Typically at least three specimens from each adhesive were tested to ensure repeatability. No significant variation in the properties was observed from specimen to specimen, giving confidence in the casting as well as the testing procedure.

The bimaterial specimens were made by coating a $75-100 \mu m$ layer of epoxy A on 0.85 mm thick steel strips to ensure that the adherend to adhesive thickness ratio remains about 10 to maintain a more uniform residual stress state in the adhesive and simplify the analysis. More on this will follow in [Section 3.](#page--1-0) The adhesive coat did not entirely cover the length of the steel substrate, and left the length equal to the length of the grip, uncoated. This was done in order to ensure that there was no adhesive underneath the clamp. In order to ensure uniform thickness of the adhesive, a drawdown bar (Precision Gage and Tools, Dayton, OH) was used. The bimaterial strips were cured following the same procedure as mentioned before. Five bimaterial specimens were tested to ensure the repeatability in the data.

2.3. Linear viscoelastic characterization

The anticipated strain levels in the adhesives due to cooling from 250 \degree C to room temperature would be on the order of 0.4–0.5% based on the CTE data. This temperature range is also important from a practical standpoint as it represents most likely thermal excursions the panels are subjected to. To remain within the linear viscoelastic region, mechanical loading of the three adhesives was limited to a strain level of 0.15%. At this strain

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