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A design of experiments assessment of moisture content in uncured adhesive on static strength of adhesive-bonded galvanized SAE1006 steel

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ABSTRACT

As part of a cooperative research program to develop and implement crash-resistant toughened adhesives targeted for future vehicles, this paper summarizes a study of the influence of pre-exposure of uncured adhesive and steel sheets in a humid and elevated temperature environment on quasi-static strength of bonded hot dipped galvanized SAE1006 steel joints.

In this study, we use a DOE (design-of-experiment) program called DEXPERT to design the experiment and to analyze the effects of exposure temperature, exposure time, curing temperature and curing time on joint strength of adhesive-bonded galvanized SAE1006 steel. Prior to adhesive curing, the adhesive and galvanized steel coupons were pre-exposed to various relative humidity levels and temperatures. The experimental results were then analyzed by DEXPERT and the relative contributions of each factor on variance in joint strength were calculated. It was found that curing temperature is the most influential factor affecting the strength of adhesive-bonded galvanized SAE1006 steel joints. The curing of a joint at 180 °C can increase the robustness of the process and provides the greatest strength regardless of the variation of other factors. The joint strength curing at 150 °C shows a strong sensitivity to the curing time, while the adhesive cannot cure at 130 °C at all under all conditions. It has also been found that the pre-exposure of adhesive and steel for an hour can slightly decrease the joint strength at high temperature and humidity. Therefore, the effect of long time exposure of the uncured adhesive and steel still needs to be further investigated.

1. Introduction

Epoxy based adhesive-bonded steel has a wide range of applications in light-weight vehicle structures for good stiffness and strength [1,2]. To obtain a good bonding strength, the bonded steel components in the vehicles must be held at a certain temperature for a certain length of time to reach full cure [3–6]. This curing process is often performed in the paint shop without a dedicated curing oven for cost consideration. In the paint shop, the ambient curing temperature and the curing time are two main variables, which can significantly influence the adhesive cure and the bonding strength [7,8]. Before the curing of the bonded steels, the adhesive and steels could be exposed to the ambient hot humid environment due to the variable weather conditions, since the bonded components cannot immediately come into paint shop after adhesive was dispensed on the steels. It is known that the humid environment can lead to the strength degradation of adhesive bonded steels [9–11]. In the past, most studies have been undertaken on the effects of environmental condition on the mechanical properties of cured adhesive-bonded steels [12–14]. These researches provide valuable information on the humid environmental aging of adhesive bonded steel. However, little information is available concerning the effects of exposure of uncured crash-resistant toughened adhesive in hot humid environment.

In this study, the combined effects of pre-exposure of uncured adhesive and steel in hot humid environment and the curing conditions on the quasi-static strength of cured crash-resistant toughened adhesive-bonded steels are investigated. To understand the combined effects of pre-exposure and the curing, we use a DOE (design-of-experiment) program called DEXPERT [15] to design the experiment and to analyze the effects of exposure temperature, exposure time, curing temperature, curing time and humidity levels on joint strength of adhesive-bonded galvanized SAE1006 steel. Adhesive is first dispensed on a steel sheet and both dispensed adhesive and steel adherends are exposed in 65% or 95% relative humidity at 20 or 40 °C for a period of 0, 10 or 60 min. After curing, mechanical tests were conducted in ambient environment. In this study we used lap-shear specimens made from 0.75 mm thick galvanized SAE1006 steel and adhesive. The details of the experimental design, sample fabrication and data analysis are presented in the following section of this study.

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This study is a part of a larger program to develop a more quantitative and predictable description of environmental durability of crash-resistant toughened adhesive-bonded steels.

2. Design of experiment

For high volume automotive applications, simpler, faster and more environmental friendly processes are required. Normally, the sheet metal is covered with oil during transportation from steel mill to stamping plant. After delivery to the plant the steel is sheared and stamped. The next stage of the process is the application of the adhesives for weld-bonding. This is achieved by using equipment of varying complexity, from hand-operated pumps to sophisticated robotic dispensing systems. The robot is programmed to apply a bead of adhesive. After the adhesive has been applied, the panels are married together to form the parts. The panels are then put through paint oven with temperature ranging from 180 to 200 °C. The typical time in the oven is from 20 to 30 min. Since there are many processing parameters (e.g., temperature and humidity in the plant, curing temperature and curing time in paint oven) to control in this production environment, it is conceivable that bonded components may experience a range of humidity level, curing temperature and time. Therefore, it is essential that an understanding of the effects of these variables on the strengths of bonded joints be obtained. The present study was undertaken to experimentally evaluate the effects of these process variables.

Table 1 shows the input factors and responses for this experiment. The design consists of five input factors—exposure time, exposure temperature, curing time, curing temperature and humidity level. There is only one response variable, namely joint strength. Shown in Table 1 are the details of the input factors as defined in DEXPERT. Curing temperature, curing time and exposure temperature each have 3 levels. Exposure time and humidity have only two levels. This DOE is a full factorial design with 3 replicates, which requires a total of 324 specimens.

3. Experimental procedure

3.1. Material

Low carbon hot-dipped galvanized (HDG60) steel SAE1006 was used in this study. Chemical composition and mechanical properties of the steels are shown in Table 2 and Fig. 1(a), respectively. The adhesive used in this study was Henkel one-part adhesive, a proprietary crash-resistant toughened epoxy. Bulk adhesive specimens were fabricated (based on manufacturer's recommended curing procedure (i.e., 180 °C and 30 min)) and tested. Fig. 1(b) shows the mechanical properties of the adhesive.

Table 1
Details of input factors in DEXPERT.

Factor	Level
Curing time (min)	10, 15, 20
Curing temperature (°C)	135, 150, 180
Exposure time (min)	0, 10, 60
Exposure temperature (°C)	20, 40
Humidity (%)	65, 95

3.2. The environmental chamber

To simulate the extended exposure in the humid environment, the adhesive was dispensed on a steel adherend and then exposed in a laboratory environmental chamber (0.6 m × 0.6 m × 1.5 m) as shown in Fig. 2. Dispensed adhesive and steel adherends were suspended in the humidity chamber. The water vapor was supplied through the humidifier. The vapor is added in the form of steam by boiling faucet water in a stainless steel tank maintained at a constant temperature. The vapor is pumped into the chamber and is circulated inside the chamber by means of a circulating fan (placed inside the chamber) to maintain a uniform environment throughout the chamber. The relative humidity (R.H.) inside the chamber is controlled by means of an optical dew point hygrometer. Selected workpieces were periodically removed from the humidity chamber for joint fabrication.

3.3. Specimen fabrication

The lap-shear specimen configuration, shown in Fig. 3, was fabricated from 38 mm × 127 mm hot-dipped galvanized steel sheets (SAE1006). To simulate the production environments, the steel sheets used in this study were not specifically cleaned (i.e., as-received condition). Shims are bonded on the adherends to keep the load plane of the specimen coincident with central plane of the tensile tester. The adhesive-bonded specimens were prepared as follows: (1) applying the adhesive through a hand-held injection gun on one of the two adherends, which were stored in an ambient laboratory environment (20 °C and 50% R.H.); (2) positioning the adherends with and without dispensed adhesive with a fixture in the humidity chamber; (3) after removing the steel adherends from the humidity chamber the adherends were brought together by a fixture under ambient laboratory conditions; (4) applying the pressure through the fixture so that a adhesive thickness (set up by a 0.25 mm thick metal shim) of 0.25 mm can be maintained; (5) curing the specimens in the oven for period of a time and temperature as prescribed by to DOE schedule. All finished specimens are examined and the spew fillets around the edge of the overlap were remained to simulate the real production conditions.

3.4. Static testing

Static tests were performed by loading each specimen to failure in a tensile tester. To minimize bending stresses inherent in the testing of lap shear specimens, filler plates were attached to both ends of the sample using masking tape to accommodate the sample offset. Load vs. displacement curves were obtained as the specimens were loaded at a stroke rate of 2 mm/min. Three replicates were tested, and the average peak loads were reported.

Table 2
Chemistry (wt%), coating and sheet gage for SAE1006 steel.

Steel	C	Mn	P	Si	Ni	S	Al	Cr	Ca	Ti	Gage (mm)
SAE1006 (HDG60)	0.006 max.	0.2 max.	0.025 max.	–		0.02 max.	0.015 min.	–	–	0.03/0.08	0.75