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Effect of long-term neutral salt spray exposure on durability of adhesive-bonded Zr–Ti coated aluminum joint

Yongrong Wu [a], Jianping Lin [a,*], Pei-Chung Wang [b], Rui Zheng [a], Qianqian Wu [a]

[a] *School of Mechanical Engineering, Tongji University, 4800, Cao'an Highway, Shanghai 201804, China*
[b] *Global Research & Development Center, General Motors Corporation, Warren, MI 48090-9055, USA*

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A B S T R A C T

In this study, the effect of long-term neutral salt spray (NSS) exposure (i.e., 50 g/L concentration of salt solution at 25 °C) on the retained strengths of Zr–Ti coated and bare lap-shear aluminum joints bonded with hem flange adhesive Henkel Terokal 8021 NB was investigated. A one-part toughened epoxide adhesive and Zr–Ti coated aluminum substrates (i.e., AA6014-T4 and AA6016-T4) were selected. Adhesive-bonded coated aluminum joints (ACJ) and bare aluminum joints (ABJ) were fabricated and exposed in NSS environment for various times. Quasi-static tests were conducted immediately following removal of the joints from the salt chamber at the ambient condition. It was found that while NSS exposure for 720 h degraded little the strength of ACJ, it decreased the strength of ABJ. The Zr–Ti coating protected aluminum substrates from electrochemical reaction and consequently minimized the strength degradation. As the exposure time was prolonged to 1400 h, the strength of ACJ was reduced drastically while the strength of ABJ was only degraded slightly. Fractography, differential scanning calorimetry, electrochemical potentiostatic polarization measurements, and X-ray photoelectron spectroscopy analyses revealed the strength degradation of ACJ was caused primarily by the corrosion of Zr–Ti coating. The passive film on bare aluminum provided a long time protection against NSS exposure, and consequently minimized the strength degradation for ABJ.

1. Introduction

The use of adhesive bonding of aluminum alloys for automotive application has several advantages in comparison with other joining techniques [1–3]. Despite these advantages, the use of adhesive-bonded aluminum in vehicle structural applications has been limited, partially because of concerns regarding the crashworthiness and environmental durability. Of particular concern is the effect of neutral salt spray (NSS). Recently, Zr–Ti based coatings have been developed to improve the corrosion resistance for automotive applications [4–8]. In order to utilize the full potential of these Zr–Ti based coatings, the effect on long-term salt exposure on the mechanical strength of adhesive-bonded Zr–Ti coated aluminum alloys must be understood.

A few studies have focused on the effect of Zr–Ti based coatings on the performance of the adhesive-bonded aluminum [9–11]. Critchlow et al. [9] compared the performances among several pre-treatments for the adhesive-bonded aluminum and the results showed that the conversion coatings provided better durability performance than the mechanical treatments. Lunder et al. [10] investigated the effect of long-term hot–humid exposure (i.e., 96% RH and 40 °C for 8 weeks) on the crack length growth of the adhesive-bonded Zr–Ti coated aluminum joints and found that Zr–Ti based coating improved the durability significantly. Furthermore, Rechner et al. [11] reported the results on the effect of NSS (i.e., concentration of 50 g/L at 25 °C) on the retained strength of Zr–Ti coated aluminum joints bonded with structural adhesive Dow Betamate 1496. They found that pre-exposure the joints for 1000 h degraded significantly the strength and failure mode of the joints. While most of the studies focused on the durability of Zr–Ti coated aluminum joints bonded with structural adhesives, little information is available concerning the influence of long-term salt spray exposure (> 1000 h) on the retained strength of the coated aluminum joints bonded with hem flange adhesive.

In this study, the effect of long-term neutral salt spray exposure on the retained strength of Zr–Ti coated aluminum joints bonded with hem flange adhesive (i.e., Henkel Terokal 8021 NB) was investigated. There are three main parts in this study; the first presents the experimental procedure including material, sample fabrication, neutral salt spray exposure testing, mechanical testing, fractography, DSC, corrosion resistance assessment and surface characterization. This is followed by the test results including the

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* Corresponding author. Tel.: +86 139 0171 9457; fax: +86 21 69589485.
E-mail address: jplin58@tongji.edu.cn (J. Lin).

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Table 1
Chemical compositions of uncoated AA6014-T4 and AA6016-T4 aluminum alloys (wt%).

Aluminum	Al (%)	Mg (%)	Si (%)	S (%)	Ti (%)
AA6014-T4	Balance	0.5	0.58	0.01	0.03
AA6016-T4	Balance	0.3	1.2	0.02	0.04

effects of NSS exposure on the retained strength and failure modes of ACJ and ABJ, the adhesive properties, the corrosion characteristics of aluminum substrates. Finally, X-ray photoelectron spectroscopy (XPS) analysis was performed to confirm the degradation mechanisms for adhesive-bonded Zr–Ti coated and bare aluminum alloys in neutral salt spray exposure.

2. Experimental procedures

2.1. Materials

Wrought Zr–Ti coated 1.0 mm thick Novelis AC170PX (AA6014-T4) and Zr–Ti coated 0.9 mm thick Novelis Ecodal-605 (AA6016-T4) aluminum sheet were selected in this study. The chemical compositions of the uncoated bulk aluminum alloys are listed in Table 1 and the mechanical properties are listed in Table 2. The adhesive used in this study is Henkel Terokal 8021 NB adhesive (hereafter refer to HT adhesive), a one-part toughened epoxide used for hem flange bonding. Per manufacture's data sheet, Table 3 lists the mechanical properties of the adhesive.

2.2. Sample fabrication

To study the effect of Zr–Ti coating on the retained strength of NSS exposed adhesive-bonded aluminum joints, adhesive-bonded bare aluminum joints were also included in this study. To make sure the same aluminum substrates were used for sample fabrication, the Zr–Ti coating on AA6014-T4 and AA6016-T4 aluminum substrates was carefully removed with 5000 grit SiC sandpaper.

All substrates were machined into coupons of 25 × 100 mm^2, and cleaned with Trichloroethane 1-1-1. The single lap shear joints were fabricated from 25 × 100 × 1.0 mm^3 and 25 × 100 × 0.9 mm^2 aluminum coupons (i.e., AA6014 bonded with AA6016). The detailed configuration and dimensions of a lap-shear joint are shown in Fig. 1. The adhesive-bonded specimens were fabricated as follows: (1) applying the adhesive through a hand-held injection gun on one of the two substrates; (2) positioning the substrates with and without dispensed adhesive using a fixture; (3) bringing the substrates together by a fixture under ambient laboratory conditions; (4) pressure was applied via the fixture so that a bondline thickness (set up by the metal shim) of 0.25 mm can be maintained; (5) curing the specimens in the oven per supplier's recommended curing procedure (i.e., 20 min at 170 °C). To consistently fabricate the joints, the adhesive was properly dispensed and small cambered spew fillet was formed at the edge of the overlap.

2.3. Neutral salt spray (NSS) exposure

To simulate the extended exposure in a neutral salt spray environment, specimens were exposed in a standard salt spray chamber (i.e., JW-60-SS) shown in Fig. 2. The adhesive-bonded joints were exposed in a neutral salt spray environmental condition (i.e., 50 g/L at 25 °C [12]) for various times (i.e., 240, 480, 720, 960, 1200, and 1400 h). Quasi-static tests were performed immediately after removal of the joints from the salt chamber at the ambient condition.

Table 2
Mechanical properties of AA6014-T4 and AA6016-T4 aluminum alloys.

Aluminum	Yield strength (MPa)	Ultimate tensile strength (MPa)	Total elongation (%)
AA6014-T4	100.2	207.3	23.7
AA6016-T4	114.2	229.3	21.9

Table 3
Mechanical properties of Henkel Terokal 8021NB adhesive.

Adhesive	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)
Henkel Terokal 8021 NB	2.8	4.7	8.0

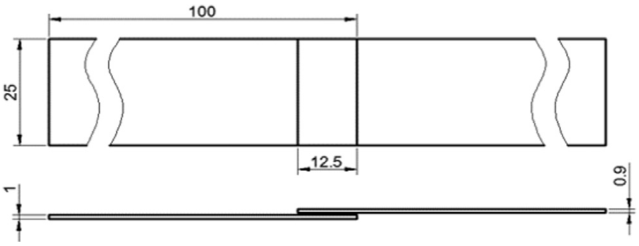

Fig. 1. Joint configuration (dimension in mm).

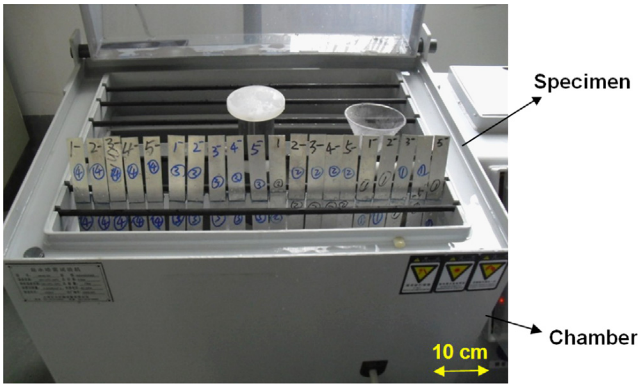

Fig. 2. Specimens exposed in a JW-60-SS salt spray chamber.

2.4. Quasi-static testing

The cured specimens were kept 24 h at room temperature, quasi-static lap-shear tests were performed by loading each specimen to failure in a Zwick/Roell—Z050 tensile tester according to the standard ASTM D1002—2001 [13]. To minimize bending stresses inherent in the testing of single-lap shear specimens, filler plates were attached to both ends of the specimen using masking tape to accommodate the sample offset. Load vs. displacement curves were obtained as the specimens were loaded at a crosshead speed of 10 mm/min. The joint strength is evaluated by the peak load. Three replicates were performed, and the average peak loads reported.

2.5. Fractography

Specimens were machined from the broken overlap sections for failure analysis. Post-failure fractographic analysis was performed