SMART CURE CYCLE TO IMPROVE TENSILE LOAD STRENGTH OF THE ADHESIVELY BONDED JOINT

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Abstract

Generally, the thermal residual stress which is generated during the curing process of the adhesive decreases much tensile load capability of adhesively bonded joints. In this work, the smart cure cycle with abrubt cooling and post curing at room temperature was devised to eliminate the thermal residual stress and obtain sufficient interfacial wetting simultaneously. For monitoring and controlling of the curing reaction, the dielectrometry was used, where the dissipation factor of adhesive joint was measured. From these results, it was found that the mechanical properties of adhesively bonded joint fabricated by the smart cure cycle, was greatly enhanced because the thermal residual stress was reduced and the sufficient interfacial wetting between adhesive and adherend was achieved simultaneously.

1. Introduce

Generally, the structure designed without joints is desirable because joints can be the weakest point of the structure or cause the excessive increase in weight. However, the joint configuration is essential because there are many factors (size of the processed products, inspection, repair, delivery and assemble) to be considered in large structures. Therefore, the design of joints for the assembly of separated structural parts has been an important research topic because the efficiency of structures with joints is established not by its basic structure but by its joints with very few exceptions [1,2].

There are two main types of joints : mechanical and adhesively bonded joints. The adhesively bonded joint can distribute load over a larger area than the mechanical joint, requires no holes, adds very little weight to the structure and has excellent fatigue resistance [3,4]. For these reasons, adhesively bonded joints have been widely used in various structure.

However, the mechanical performance of adhesively bonded joints are weakened due to the thermal residual stress which is generated during the curing process of the adhesive due to the difference of coefficient of thermal expansion (CTE) between adhesive and adherend. It has been well known that the thermal residual stress decreases much tensile load capability of adhesively bonded joints [5]. These effects appear more worse when the joints have a thick adhesive thickness and cause lower interfacial fracture than the bulk adhesive failure [6]. Also, the joints cured at room temperature to avoid the residual thermal stress, has lower adhesive strength because of the poor interfacial wetting between adhesive and adherend.

In this study, the smart cure cycle with cooling followed by the postcuring at the room temperature, was devised to eliminate the thermal residual stress. For cure monitoring and controlling of the cure cycle, the dielectrometry method was used, where the dissipation factor of adhesive was monitored during the curing operation of the joints.

In this work, tubular single lab joint was used. The steel adherends of the joints were used as the electrode to measure the dissipation factor of the epoxy adhesive during the cure cycle.

2. Experimental

2.1 Preparation of tubular single lap joint

In this work, the epoxy adhesive (Araldite, AW106 / HV953U, Huntsman) was used for the adhesive joint. The steel tubular single lap joints are designed as shown in Fig.1. The adhesively bonded joint for tensile test specimen was fabricated by adhering the tube-shaped outer adherend and cylinder-shaped inner adherend with epoxy adhesive. The inner diameter of the outer adherend was varied to adjust the adhesive thickness as 0.1mm, 0.4mm, 0.7mm and 1.0mm, respectively.

The roughness of the steel adherend was controlled in average 2um by using 80# sand paper as the average 2um roughness of steel surface has optimal adhesive fatigue strength [7]. In order to remove remaining impurities, the steel surface was cleaned by acetone. And the adhesive length was adjusted to be 15 mm by using the teflon block, inside the joint (see Fig.1) as the failure load was saturated is 15mm of the bonding length [6].

In order to adjust eccentricity, the V-block was used during the cure cycle (Fig. 1). The adhesively bonded joint was cured in various curing conditions such as manufacturer recommended cycle as shown in Fig. 2 and the room temperature (20°C) for 48 hours, respectively. After curing operation, excess adhesives of all of the joints were removed by using lazor blade.

2.2 Dielectrometry for cure mornitoring and Smart cure cycle

The dielectrometry method has been widely used to monitor the curing process of the polymeric material. In this work, epoxy adhesive was used as the dielectric material and the steel inner adherend and outer adherend body was used as the counter electrodes. Therefore, the cylindrical steel adhesively bonded joint can be considered as the parallel capacitor which is filled with dielectric material between 2 electrodes (Fig. 3). The charge accumulated in the capacitor depends on the mobility of dipoles and ions present in the adhesive to follow the alternating electric field and varies with the state of cure. The degree of cure is related to the movement of dipoles and ions, which have high mobility when the epoxy adhesive is uncured [9]. The movement is restricted abruptly when the epoxy adhesive becomes gel state or solidifies. The degree of movement can be expressed by the dissipation factor *D*, which represents the ratio of the energy loss by movements of dipoles and ions to the supplied energy. The dissipation factor *D* for the equivalent circuit can be obtained as equation (1).

$$D = \left|\frac{I_R \cdot V_m}{I_C \cdot V_m}\right| = \left|\frac{I_R}{I_C}\right| = \left|\frac{Z_C}{Z_R}\right| = \frac{1}{\omega \cdot R_m \cdot C_m} \qquad (1)$$

where, I and Z are electric current and quivalent impedance, respectively, V_m is alternating voltage with angular frequency ω applied to the equivalent circuit and subscripts R and C represent resistance and capacitance of the equivalent circuit model, respectively. In this study, the dissipation factor *D* of the epoxy adhesive was measured using a LCR meter (U1732B, Agilent Technologies).

2.3 SEM image of adhesive cross section

In order to observe the degree of interfacial wetting between the epoxy adhesives and the steel adherend, the cross section of the interface between the adhesive and adherend was observed by using a scanning electron mivroscope (SEM, JSM-6300)

2.4 Tensile test of adhesively bonded joints

To evaluate the mechanical load capability and fracture mode of the joints, static tensile test was performed. The tension test was performed with MTS universal tensile tester, with 0.7mm/min of loading speed. For each test conditions, 5 specimens were tested.

After the tensile test, the fracture mode of each joint was observed and compared the failure mode.

The tensile strength τ of the adhesively bonded joint can be obtained as equation (2).

$$\tau = \frac{P}{d \cdot l} \qquad (2)$$

where, P is tensile fracture load, d and l represent adhesive diameter and adhesive length, respectively.

3. Result and discussion

Fig. 4 shows the dissipation factor *D* of the epoxy adhesive. It was known that from the result of Kwon et al [10], the dissipation factor has maximum value when the epoxy adhesive has lowest viscosity, at which the dD/dt crossed zero point as the dipoles and ions has largest movements compared to the supplied energy. When the gelation point of the epoxy resin is passed by, the dissipation factor decreases quickly at which dD/dt became the minimum value [10]. Based on the cure mornitoring, the smart cure cycle was performed to reduce the thermal residual stress of the adhesively bonded joint, where the joints specimen was taked out from the oven and applied room temperature curing immediately when the epoxy starts curing process.

Table.1 shows the adhesive strength of the adhesively bonded joint fabricated by conventional cure cycle and room temterature cure with respect to the adhesive thickness. In the case of conventional cure cycle, the adhesive strength was decreased as the adhesive thickness was increased. It was because the thermal residual stress generated in the adhesive was increased as the adhesive thickness is increased [6]. Fig. 5 shows the failure modes of the adhesive joint cured with conventional cure cycle. It was found that as the thickness of adhesive is thicker, the failure mode is changed from a bulk failure to the interfacial failure which is similar to the result of Lee et al [6]. The failure mode change is attributed to the thermal stress.

In the case of room temperature curing, the adhesive strength was lower than the case of the conventional cure cycle regardless of the adhesive thickness (Table.1). The failure mode of the room temperature curing case was observed as shown in Fig.6. It was found that the interfacial failure occurred in all the specimens regardless of the adhesive thickness. The interfacial failure mode might come from the fact that the poor interfacial wetting was achieved due to insufficient liquefying of the epoxy adhesive at the room temperature curing.

Fig.7 shows the tensile strength of the adhesively bonded joint favricated by smart cure cycle with respect to the adhesive thickness as 0.4mm and 1.0mm. Improvement of the adhesive strength of 38% and 55% could be obtained respectively, using the smart cure cycle compared to those of specimens fabricated using the conventional manufacturer's recommended cure cycle, which was due to the reduction of the thermal residual stress of the epoxy adhesive. For the same reason, the strength improvement rate of the 1.0mm adhesive thickness specimens was higher than that of the specimen 0.4mm adhesive thickness, because the thermal residual stress was increased as the adhesive thickness is increased.

Fig. 8 shows the failure modes of the adhesive joint cured with smart cure cycle. It was found that the failure mode is more slowly changed from bulk failure to the interfacial failure compared to those of failure mode of adhesive joint fabricated under the the conventional cure cyle and room cure temperature.

Fig. 9 shows SEM images of interface between the adhesive and adherend cured under the various cure cycle. In the case of conventionally cured specimen having more close wetting condition compared to specimens cured under the room temperature. The specimen applied smart cure cycle, shows sufficient interfacial closeness between adhesive and adherend compare to the speciemen cured under the room temperature. This result shows that smart curing process of adhesively bonded joints can reduce the thermal residual stress and obtain the sufficient interfacial wetting between adhesive and adherend simultaneously.

4. Conclusions

In this work, a cure system using dielectric method was devised to on-line monitor and control the cure cycle of the adhesively bonded joints to decrease the thermal residual and to obtain optimal interfacial wetting between adhesive and adherend. From the experimental results, the following conclusions were made.

(1) Both the thermal residual stress reduction and sufficient interfacial wetting between adhesive and adherend was obtained by the smart cure cycle using a dielectric method, in which adhesively bonded joint were applied room temperature curing at the point the maximum *D*.

(2) The developed cure method could obtain the sufficient interfacial wetting as high temperature curing between the adhesive and adherend and improved 38% and 55% of the adhesive strength at 0.4mm and 1.0mm adhesive thickness of joints respectively, itself compared to the conventional manufacturer's recommended cure cycle.

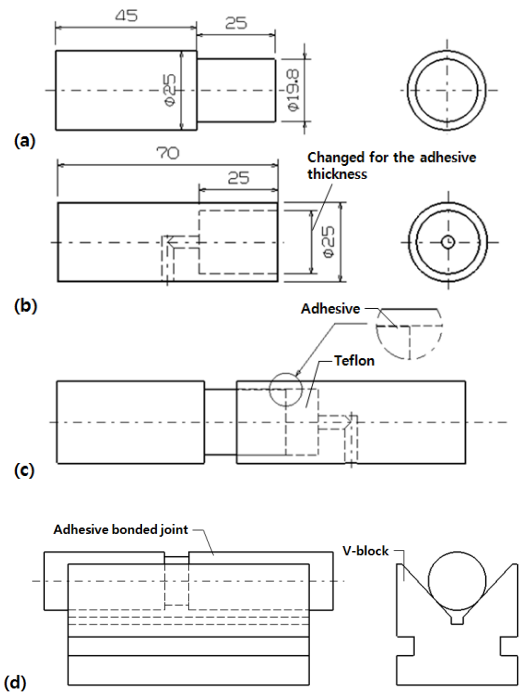

Fig.1. Specimen of the tubular single lap joint: (a) Inner adherend; (b) Outer adherend; (c) Shape of the joint; (d) V-block and adhesively bonded joint.

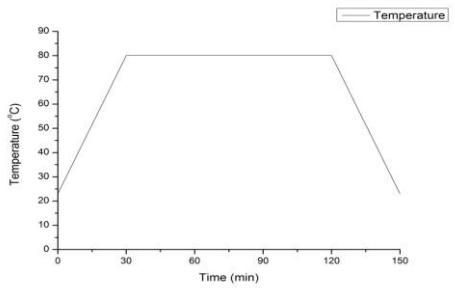

Fig.2. Conventional curing cycle for the epoxy adhesive.

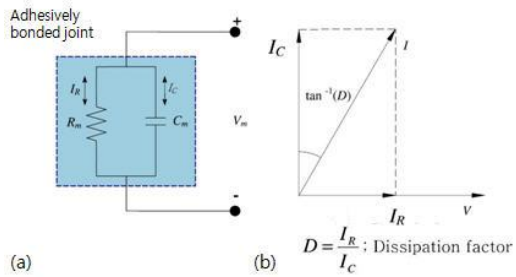

Fig.3. Principle of the measuring dissipation factor: (a) equivalent electrical circuit model for adhesively bonded joint; (b) electrical current through the dielectric material and definition of the dissipation factor.

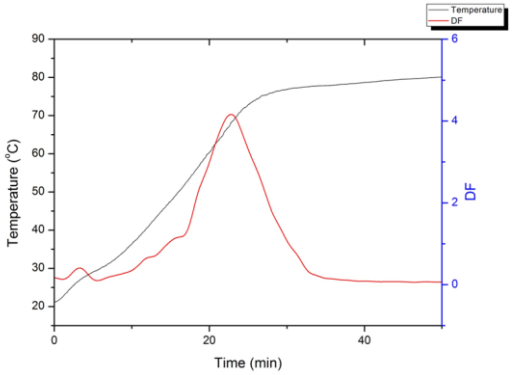

Fig.4. Dissipation factor D of the adhesively bonded joint at conventional cure cycle

Adhesive thickness	Conventional cure	Room cure
0.1 mm	22.03932	13.01893
0.4 mm	18.4984	12.61318
0.7 mm	14.09226	14.23954
1.0 mm	13.05113	13.52545

(Tensile strength unit : MPa)

Table.1. Tensile test results of the adhesively bonded joint with conventional cure cycle and room temperature cure.

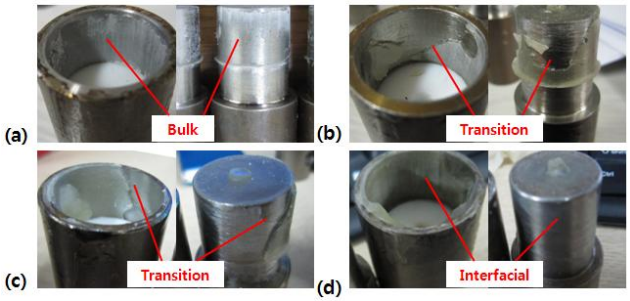

Fig.5. Failure modes of steel adhesively bonded joint on the conventional cure cycle: (a) 0.1mm; (b) 0.4mm; (c) 0.7mm; (d) 1.0mm.

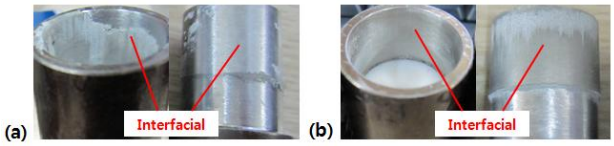

Fig.6. Failure modes of steel adhesively bonded joint on the room temperature cure: (a) 0.1mm; (b) 0.4mm.

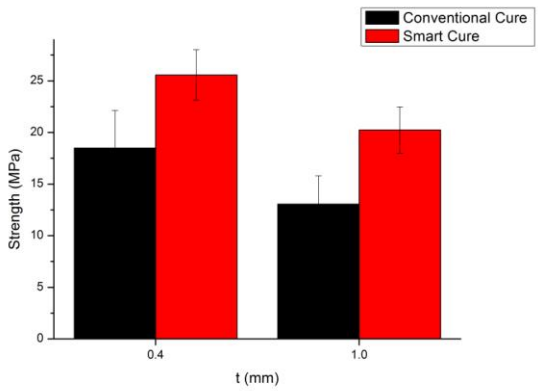

Fig.7. Tensile test results of the adhesively bonded joint fabricated by smart cure cycle.

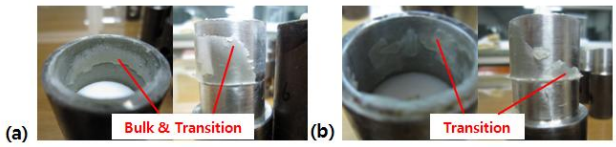

Fig.8. Failure modes of steel adhesively bonded joint on the smart cure cycle: (a) 0.4mm; (b) 0.7mm.

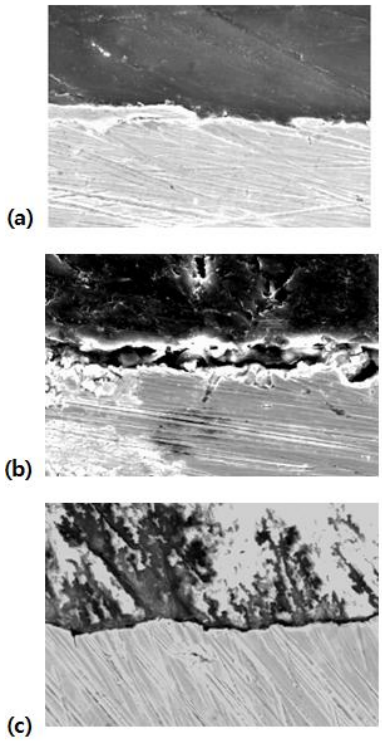

Fig.9. SEM image of Steel/Adhesive interface: (a) conventional cure; (b) room temperature cure; (c) smart cure.

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