MEASUREMENT OF STRESS AND TEMPERATURE IN CURING PROCESS OF EPOXY ADHESIVES

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ABSTRACT

This study is concerned with stress and temperature in the curing process of epoxy adhesives measured using a system that can simultaneously measure the stress and temperature in structures in time series. The stress and temperature were measured by a digital photoelastic technique in the form of phase stepping and a thermographic technique, respectively. The specimens consisted of two epoxy resin plates (3 mm thick, 26 mm wide and 76 mm long) with a layer of adhesive 0.1, 0.2 or 0.3 mm thick between the plates. Immediately after the application of adhesive between the plates, the photoelastic fringe and thermal images were captured. Results showed that the curing of the area where the adhesive was applied was not uniform and the residual stress in the adhesive after curing was low.

Introduction

Recently, adhesive has been often used for jointing members in structures and pipes in pipe arrangements, instead of using bolts and rivets, which are conventional fasteners. This is mainly because of the resulting reduction of the weight of structures and the improvement of workability for jointing structure members. The quality of a glued connection is easily affected by variations in temperature and humidity and depends on the worker’s skill to a greater extent than that in joints using bolts and rivets. Therefore, the glued connection possesses lower reliability of strength than bolted and riveted joints.

A way of improving the reliability of the strength of the glued connection is to understand the relationships among the curing process of the adhesive, the stress generated in the adhesive during its curing and the residual stress after its curing under various conditions. The curing process of the adhesive can be indirectly observed via the temperature distribution in the adhesive, which is generated by the chemical reaction in the adhesive. Therefore, to understand the relationships, it is desirable to simultaneously measure stress and temperature in the whole field during curing in time series.

In general, the thermographic technique is used to measure the temperature on the surface of a specimen. In the case of a glued connection, the technique cannot be directly used to measure the temperature on the surface of the adhesive because the adhesive is between adherends. However, by determining the difference between the temperatures of the adhesive with and without an adherend by preliminary measurements using the thermographic technique, the temperature of the adhesive can be obtained.

For the whole-field measurement of stress, the photoelastic technique is employed. For a glued connection, the adhesive cannot be seen because it is between the adherends. However, when transparent adherends are used, stress in the adhesive can be indirectly measured via the adherends. Therefore, the combination of both photoelastic and thermographic techniques enables the simultaneous measurement of stress and temperature in the whole field [1-4]. On the basis of the concept of the combination, a device that can simultaneously measure stress and temperature by the combination of both techniques is thus required, since photoelasticity and thermography use visible light and infrared light, respectively [3-5].

In recent years, a system for simultaneously measuring stress and temperature in structures in time series has been developed by the authors [6]. The system uses a digital photoelastic technique in the form of phase stepping for measuring stress and a thermographic technique for temperature. The system has been successfully applied to the measurement of stress and temperature in the curing process of UV curing resin illuminated with UV rays [7].

In this study, stress and temperature in the curing process of epoxy adhesives were measured using the system that can simultaneously measure stress and temperature in time series.

System for Simultaneous Measurement of Stress and Temperature

Figure 1 systematically shows the main part of the measurement system for simultaneously measuring stress and temperature in the whole field of a tested specimen. The system consists of photoelastic apparatus for measuring the stress (GFP2100,
Stress Photonics Inc.), thermographic apparatus (Radiance HS, Raytheon Co.) and a beam splitter, which can reflect infrared light and transmit visible light.

The photoelastic apparatus consists of an achromatic circular polarizing projector, which can generate achromatic circular polarized light at an accuracy of 1% over the range of 450 to 700 nm, a charge-coupled device (CCD) camera, which can simultaneously capture four photoelastic fringe images, and a data processor. The apparatus can record a sequence of four photoelastic images at a maximum sampling rate of 15 fps. The differences in the principal stresses and their directions under dynamic phenomena are analyzed from the photoelastic fringe images obtained using the apparatus. In addition, the shear stresses called shear 45 deg, which denote the shear stresses acting on planes at an angle of 45 degrees to the horizontal plane, can be obtained. The accuracy of the measurement is in the order of 1/100 of the fringe order.

For measuring the temperature, the thermographic apparatus consists of an infrared camera and a data processor. The apparatus can record thermal images at a maximum sampling rate of 6400 fps. The accuracy of the measurement is in the order of 0.025 K.

![Figure 1. Main part of the system for simultaneous measurement of stress and temperature](image)

**Experimental Procedure**

The system shown in Figure 1 was used to measure both stress and temperature generated in epoxy adhesives during and after their curing. Figure 2 shows the details of the specimens, which consist of two transparent epoxy plates (3 mm thick, 26 mm wide and 76 mm long) as adherends and epoxy adhesive. The adhesive was prepared by mixing epoxy as the base resin with polythiol as the hardener, then applying it onto an area of 26 mm × 26 mm at one end of an epoxy resin plate. Thereafter, one end of the other epoxy resin plate was placed over the 26 mm × 26 mm area in the opposite direction to the first epoxy resin plate. Three different thicknesses of adhesive, 0.1, 0.2 and 0.3 mm, were used.
Immediately after the specimens, both ends of which were fixed between steel blocks, were placed on the stage, photoelastic fringe and thermal images were captured at intervals of 10 s for 120 min at room temperature (about 24 °C). The time from the beginning of the mixing of the base resin with the hardener to capturing the first photoelastic fringe and thermal images was 100 s.

**Experimental Results**

Figures 3-5 show respectively the isochromatic fringes, principal stress ($\sigma_1$) directions and shear 45 deg images at $t=5$, 10, 15, 20 and 120 min for thicknesses of the adhesive layer, $h$, of 0.1, 0.2 and 0.3 mm. Figure 6 shows the thermal images at $t=0$, 10, 15, 20 and 120 min. Figure 7 shows the variation of temperature with time at point 1 located at the center of the bonded area, as shown in Figure 6. In Figures 3-6, the bonded areas in the specimens are shown by broken lines. The temperature on the surface of the epoxy resin plate was measured by the thermographic technique described in the preceding section.

![Isochromatics](image-url)
The fringe order of the isochromatics generated in the curing adhesive increased with time up to about $t=10$ min for $h=0.1$ mm and up to about $t=20$ min for $h=0.2$ and 0.3 mm, as shown in Figure 3. In particular, the fringe orders along the upper and lower edges of the bonded area were higher than those at other parts. The maximum fringe orders of isochromatics for $h=0.1$, 0.2 and 0.3 mm were about 0.1, 0.12 and 0.15, respectively. The residual stress hardly existed for all thicknesses of the adhesive layer at $t=120$ min.

The directions of the principal stresses, $\sigma_1$ and $\sigma_2$, for $h=0.1$ mm were substantially different from those for $h=0.2$ and 0.3 mm, as shown in Figure 4. For $h=0.1$ mm, the direction of $\sigma_1$ was toward the center of the bonded area and the direction of $\sigma_2$ was concentric around the center of the bonded area. For $h=0.2$ and 0.3 mm, the direction of $\sigma_1$ was concentric around the center and that of $\sigma_2$ was toward the center. These characteristics of the directions of the principal stresses were significant when isochromatics with the maximum fringe order were generated at $t=10$ min for $h=0.1$ mm and at $t=15$ min for $h=0.2$ and 0.3 mm; they then almost disappeared with time.

![Figure 4. Principal stress ($\sigma_\parallel$) directions](image)
From shear 45 deg images shown in Figure 5, for $h=0.1$ mm the compressive stress in the horizontal direction acted along the upper and lower parts of the bonded area, and the tensile stress acted at the middle part. It is inferred that this is because the upper and lower parts of the adhesive moved toward the left and right sides of the adhesive, and the middle part shrank from the left and right sides toward the center of the adhesive. In contrast, for $h=0.2$ and 0.3 mm the tensile stress in the horizontal direction acted along the upper and lower parts of the bonded area, and the compressive stress acted at the middle part. Therefore, the adhesive for $h=0.2$ and 0.3 mm moved oppositely to that for $h=0.1$ mm. The reason for these movements of the adhesive is unclear.

Figure 5. Shear 45 deg
The increase in temperature in the bonded area implies an increase in the curing reaction rate of the adhesive, which is due to a chemical reaction. Therefore, the thermal images shown in Figure 6 reveal that the curing of the adhesive started from the lower part near the center of the bonded area, and proceeded circumferentially with time. The maximum temperature occurred locally at the lower part near the center of the bonded area, where the curing started. The maximum temperatures for $h=0.1$, 0.2 and 0.3 mm were 26.3 °C at $t=14$ min, 27.0 °C at $t=16$ min and 27.7 °C at $t=18$ min, respectively, as shown in Figure 7. After that the temperature gradually decreased. These results showed that the maximum temperature during the curing of the adhesive increased with increasing thickness of the adhesive layer, and the maximum stress during the curing was generated 2-4 min after the maximum temperature occurred in the adhesive.

Figure 6. Thermal images
Conclusions

Stress and temperature in the curing process of epoxy adhesives were measured using a system that can simultaneously measure the stress and temperature in structures in time series. Results showed that the curing of the area where the adhesive was applied was not uniform and the residual stress in the adhesive after curing was low. Furthermore, the maximum temperature during the curing of the adhesive increased with increasing thickness of the adhesive layer, and the maximum stress during the curing was generated 2-4 min after the maximum temperature occurred in the adhesive.

References