FAILURE CRITERIA FOR TRANSPARENT ACRYLIC ADHESIVE JOINTS UNDER STATIC, FATIGUE AND CREEP LOADING

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Abstract

Modern acrylic adhesives make possible the bonding of glass and polycarbonate in large sheets of laminated glass using thin adhesive layers. Glass polycarbonate laminates with thin adhesive layers should have good mechanical properties and thus allow for lighter transparent constructions. Asymmetric double butt strap specimens of glass and polycarbonate were manufactured using photobond 53517 adhesive. These were tested at room temperature in laboratory air in zwick Z10 universal testing machines. Specimens were tensile tested at different strain rates and subjected to creep and low cycle fatigue tests with different hold times. The results show that several mechanisms are active in a fatigue cycle. Damage is mainly a function of the hold time at maximum stress rather than a cycle by cycle approach. No single stress/time or strain time parameter can explain the results.

Introduction

The general problems in using adhesives in structural applications have been looked at many times. The 1997 overview by Kinlock (1) summarises the results of several decades of research. Particular problems are the creep and fatigue behaviour of adhesively bonded joints. For most common adhesive systems adequate reference data, such as the paper by Ashcroft and Shaw (2) on epoxies, exists. In new adhesive systems or problems the research has to be done anew. One particular problem where inadequate data is available is that of transparent adhesive systems. Glass laminates are usually made by bonding glass sheets using a PolyVinylButyral rubber interlayer. This type of laminate is autoclaved and bonds without adhesives. For glass polycarbonate laminate polyurethane adhesive are used. These are usually in thicknesses of > 100 µm and suffer considerable creep and fatigue degradation leading to delamination. More modern adhesives such as the DELO photocatalytic acrylic adhesives of the photobond series in theory can be applied in much thinner layers, typically 10 to 50 µm. This should lead to improved mechanical behaviour because thin adhesive layers are less affected by creep. Research by Veer et al. (3,4,5) on the photobond 4455 adhesive resulted in the conclusion that the fatigue properties of this adhesive were strongly dependent on the method of adhesive application. The main problem is the introduction of invisible micro defects during the adhesive application before curing compared to the conventional problem of macro defects being created during curing such as described for steel/epoxy combinations by Melander et al. (6).

Considering the problems of the photobond 4455 adhesive research has continued with the photobond 53517 adhesive which does not have the problems associated with applying the adhesive found in the 4455 adhesive.

Experimental method

To study the problem asymmetric double buttstrap specimens were made of glass and polycarbonate. The dimensions and shape are given in figure 1. All components were cleaned with propanol and dried using compressed air before bonding. Delo photobond 53517 acrylic adhesive was used to manufacture the specimens, jigs were used to ensure proper positioning of the specimen components. This adhesive is designed to have good wetting properties on low surface energy polymers such as polycarbonate and is photo-catalytic. On exposure to blue light of sufficient intensity the adhesive cures in 30 seconds. The advantage of this is that the adhesive is free to flow until exposed to blue light and after exposure the voids and defects are rapidly "frozen" in the cured adhesive. This adhesive is thus very suitable for theoretical studies and also has significant technical usage. The asymmetric double buttstrap specimens were fatigued in a zwick z10 universal testing machine using test expert version 8.1 software. Specimens were loaded from zero stress to a specified maximum shear stress, τ_{max} , then held at this stress for a fixed hold time, then loaded to a lower stress level for the same hold time as kept at before. Thus block loading was used rather than sinusoidal loading. In some tests different combinations of hold time at maximum or minimum stress were used. The standard displacement rate was 100 mm/minute. All data was recorded at every 1 µm of displacement. Thus complete stress/strain data are available. All tests were conducted in laboratory air at a temperature of 20°C.



Figure 1: Asymmetric double buttstrap specimens used

Results of tensile tests

Tensile tests were conducted at three speeds. The results of these tests are given in table 1. The results show that the shear strength of the adhesive is strongly dependent on the strain rate. At high strain rate the adhesive is stronger than at low strain rates.

| displacement rate (mm/min) | Average τ_{max} (MPa) | Standard deviation |
|----------------------------|----------------------------|--------------------|
| 1 | 2.98 | 0.38 |
| 10 | 4.40 | 0.06 |
| 100 | 4.73 | 0.21 |

Table 1: Failure stress in tension at different test speeds, average of three tests

Results of fatigue tests at R=0.1

The numbers of fatigue cycles to failure , n_f , at R=0.1 are summarised in table 2. The individual test systematically show the pattern illustrated in figure 2. Three stages can be identified. A first stage at the beginning of the test where the displacement increases rapidly, a second stage where the displacement increases slowly, and the final stage where the displacement increases to final failure. This should not be considered in terms of fatigue crack propagation curves as threshold, Paris regime and final failure. In this type of adhesive joint the first stage is one of a viscoelastic adjustment of the adhesive to the loading while cracks start to initiate. In the second stage the cracks propagate slowly cycle by until the propagation rate accelerates to final failure.



Figure 2: change in displacement with increasing number of cycles, τ_{max} =1.2 MPa R=0.1



Figure 3: displacement plotted against time in second stage regime of test at τ_{max} =1.6 MPa R=0.1

Figure 3 shows a detail of a test in the second stage area. It can be seen that the displacement starts to increase at the beginning of the loading cycle and that the rate of increase of displacement slows down as the load is kept constant. In the unloading part of the cycle there is a recovery of sorts. This recovery is however not constant as the next cycle starts at a higher displacement that the last cycle. This is assumed to be caused by a combination of crack growth which causes the increase in displacement per cycle and viscoelastic behaviour which causes most of the increased displacement in the loading cycle and the recovery during the unloading cycle.

| τ_{max} (MPa) | Hold time (s) | n _f | n _f second test | $t_{hold} \times n_f(s)$ |
|--------------------|---------------|----------------|----------------------------|--------------------------|
| 1.2 | 2 | 25000 | 22456 | 48000 |
| 1.2 | 10 | 3914 | | 39000 |
| 1.2 | 50 | 1131 | 1209 | 60000 |
| 1.6 | 1 | 21305 | | 21000 |
| 1.6 | 5 | 9226 | | 46100 |
| 1.6 | 10 | 1557 | 1691 | 16000 |
| 1.6 | 50 | 362 | 341 | 17500 |
| 1.6 | 250 | 74 | 83 | 20000 |
| 2.4 | 1 | 23873 | | 24000 |
| 2.4 | 5 | 727 | | 3600 |
| 2.4 | 10 | 408 | 434 | 4200 |
| 2.4 | 50 | 47 | 42 | 500 |

Table 2: Results of fatigue tests at R=0.1 at different holdtimes

Results of fatigue tests at R=0.5

The results of R=0.5 are similar to those at R=0.1. The results are summarized in table 3. Figure 4 compares the fatigue life times at R=0.1 and R=0.5 for the tests at identical holdtimes and at the same τ_{max} of 1.6 MPa. The effect on holdtime fatigue life is in both cases similar. The reduced stress range at R=0.5 increases fatigue life but not as significantly as would be expected on a pure fatigue loading damage mechanism. Figure 5 shows the displacement/time data in the stage 2 regime for a test with a holdtime of 50 seconds. This shows the same initially quick

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increase in displacement at the onset of the loading cycle followed by slow deformation during the load cycle. At unloading there is a rapid elastic decrease in displacement followed by a slow viscoelastic decrease. In the subsequent loading cycle the initial displacement is slightly higher than in the previous cycle.

| τ _{max} (MPa) | Hold time (s) | n _f | n _f second test | $t_{hold} \times n_f$ |
|------------------------|---------------|----------------|----------------------------|-----------------------|
| 1.6 | 1 | 15794 | | 16000 |
| 1.6 | 5 | 7432 | | 37000 |
| 1.6 | 10 | 4894 | 4924 | 49000 |
| 1.6 | 50 | 618 | | 32000 |
| 1.6 | 250 | 333 | 362 | 80000 |

Table 3: Results of fatigue tests at R=0.5 at different holdtimes





Figure 4 : Comparison of cycles to failure At τ_{max} of 1.6 MPa at R=0.1 and R=0.5

Figure 5: Displacement against time in second stage regime, holdtime=50 seconds

Results of fatigue tests with irregular holdtimes

In most test the holdtime at minimum and maximum load were identical. A number of tests were conducted with different holdtimes. The results of these tests are summarized in table 4. Figures 6 and 67 show the local displacement/time data for these tests. The combination of long hold time at maximum load and short holdtime at minimum load leads to a situation where little deformation can be seen at maximum load and a lot of viscoelastic recovery at minimum load. The tests with a short hold time at maximum load show some viscoelastic behaviour there but almost complete recovery at minimum load. In both cases there is no significant difference

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between the tests with irregular holdtimes compared to the tests with regular holdtimes if we take the holdtime at maximum load as reference. The number of cycles to failure seems to only depend on the maximum holdtime. This would seem to show that the viscoelastic behaviour at maximum and minimum load does not play a significant role in the damage process.

| τ_{max} (MPa) | R | T _{hold} max (s) | T _{hold} min (s) | n _f | n _f second test |
|--------------------|-----|---------------------------|---------------------------|----------------|-------------------------------|
| 1.6 | 0.5 | 50 | 5 | 735 | 779 |
| 1.6 | 0.5 | 50 | 50 | 618 | |
| 1.6 | 0.5 | 5 | 50 | 7305 | 7558 |
| 1.6 | 0.5 | 5 | 5 | 7432 | |

Table 4 : results of fatigue tests with irregular hold times





Figure 6: Plot of time/displacement for specimen Figure 7: Plot of time/displacement for specimen with short holdtime at maximum

Discussion

The duplicate tests show that the life times are quite reproducible between tests. The data is thus valid for analysis. However the results are not easy to interpret. The results of the tests with irregular holdtimes show the time to final failure is only dependent on hold time at maximum load. Longer holdtimes at minimum load allow for relaxation of the viscoelastic flow that occurred during the holdtime at maximum load. This relaxation does not seem to remove the damage that occurred during the cycle. If we multiply the holdtime with the number of cycles to failure no unique value results that is valid for a single stress level. This implies that the test cannot be regarded as a creep test with interruptions.

In a fatigue cycle thus two reasons for increases of the displacement exist. :

- fatigue damage which is dependent on the length of the loading cycle and the maximum stress
- some form of delayed elasticity which is dependent on the stress level, hold time and probably temperature

As the two forms of displacement cannot be distinguished during the load cycle the crack growth cannot be detected. Various post-test processing of the load-displacement –time data have been used to see if the data can be correlated to a stress/time or strain/time based damage parameter. Sofar this has been markedly unsuccessful.

The fatigue life times have been plotted against the hold time for the tests at R=0.1 in figure 8 and R=0.5 in figure 9. A rough power law between the two can be deduced from the log-log linearity but this fails at very low holdtimes. The exponent of the power law would anyway be stress dependent as the exponent seems to increase with stress. The data can be curve fitted on a single mathematical function with stress and hold time as variables but as this would give no physically meaningful result the equation has been omitted.

An important practical result is the extreme dependence of the fatigue life time on the hold-time. At a shear stress level of 2.4 MPa the life times varies from 24000 at a holdtime of 1 second to 45 at a hold time of 50 seconds. This extreme frequency effect implies that this particular adhesive cannot be used for any application where it is subjected to load for a long time. An example would be glass-polycarbonate laminates subjected to a summer-winter heating cycle. Although the adhesive is quite strong in static tests, it would delaminate during the summer due to the strains imposed by the expanding polycarbonate.



Figure 8: Relation between the number of cycles of failure and the hold time at three stress levels at load ratio R=0.1



Figure 9: Relation between the number of cycles of failure and the hold time at τ_{max} = 1.6 MPa and a load ratio R=0.5

Conclusions

From the results it is concluded that for the photobond 53517 adhesive:

- crack growth and reversible viscoelastic deformation take place in a single fatigue load cycle
- only the crack growth contributes to final failure
- the holdtime at maximum load is the most important factor in the fatigue life time
- the crack grows during the whole period at maximum load, but not a constant speed
- the hold time at minimum load has no effect on the fatigue life
- the fatigue process cannot be considered as an interrupted creep test
- no single stress/time or strain/time based parameter can describe the data
- it is not suitable for applications where the adhesive has to carry loads for a long duration

References

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