Preparation of Bulk Adhesive Test Specimens

Summary

The design of adhesively-bonded structures often involves the use of Finite Element Analysis (FEA) to predict component performance. Accurate predictions require accurate material properties data. These data are often best obtained from bulk test specimens [1]. The use of bulk test specimens brings advantages in the measurement of strain (through larger gauge sections) and in the interpretation of the data (since the absence of adherends leads to simpler stress distributions). However, the bulk specimens may differ from the adhesive in the joint by incorporating voids (that act as stress concentrators to promote premature failure) or by experiencing different thermal histories (thus reaching a different state of cure). Thus, specimen preparation is an extremely important aspect of the test programme.

Techniques for preparing bulk test specimens were addressed in the DTI funded Performance of Adhesive Joints research programme. Standard preparation methods for structural adhesives that cure without liberating gases, such as epoxies, acrylics and polyurethanes, were proposed to the International Standards Organisation (ISO TC 61) [2, 3]. These describe methods by which one-part and two-part adhesives should be dispensed into a mould and subsequently cured. Test specimens can be cut from the manufactured plaques [4]. This document illustrates some aspects of these sample preparation methods.

The cure state of the bulk test specimens used to obtain the mechanical properties data should be similar to that of the adhesive in the bonded structure. Cure schedules should be selected to ensure that the thermal histories of the materials are similar in each case. Dynamic Mechanical Thermal Analysis (DMTA) measurements can be made to compare the final state of cure of the materials. Real time monitoring of the development of the mechanical properties of the adhesive can be performed using either ultrasonic or rheological techniques [5]. Conditions experienced by the specimens after cure can also influence their properties.

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Dispensing into a mould

All specimen preparation methods should take into account the hazardous nature of the adhesives. COSHH procedures should be followed to minimise operator exposure.

The mould for preparing bulk specimens should be made mostly from metal to ensure good thermal conductivity. The surfaces in contact with the adhesive should be flat, dry and free from defects. They should be covered with a release coating (e.g. thin PTFE sheet) to facilitate release of the specimen. Frames or spacers are required to control the specimen thickness - 2-3 mm thickness is suitable for many tests [1]. Clamps or weights should be used to hold the mould closed whilst the specimen cures.

Low viscosity adhesives may be dispensed directly into shaped moulds for the production of test specimens. However, many adhesives will not flow readily and such an operation is likely to be ineffective. These adhesives are best produced as flat plaques from which test specimens can be taken after the adhesive is cured [4].

The dispensing operation needs to be done such that additional air is not trapped in the final specimen. Procedures such as those shown in Figure 1 can be used, when dispensing through a nozzle, to produce specimens with minimal incorporation of additional air [2, 3].

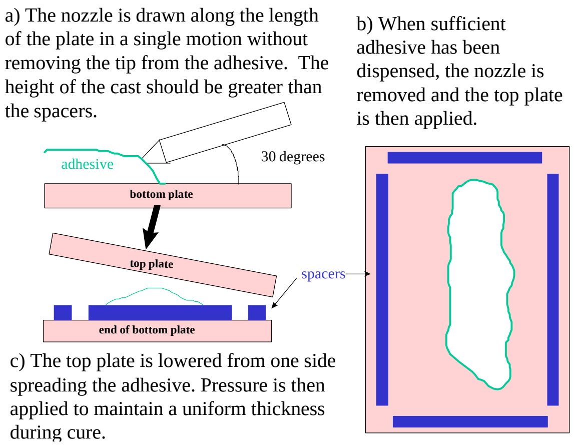

Figure 1: Dispensing a viscous adhesive to manufacture a plaque of bulk adhesive having a uniform thickness..

Where possible, two-part adhesives should be mixed by dispensing through static mixing nozzles (either from cartridges or using dispensing equipment). This ensures a uniform mix and a short dispensing time. Hand mixing tends to mix air into the adhesive.

As-supplied adhesive contains varying proportions of entrapped air. Techniques such are vacuum stirring or centrifuging have been suggested as methods for removing air from the adhesive. These techniques have varying degrees of success depending on the viscosity of the adhesive but add considerably to the cost of specimen preparation. Plaques can be inspected after manufacture (either visually or by ultrasonic C-scan) to identify regions of low void content from which test specimens can be cut.

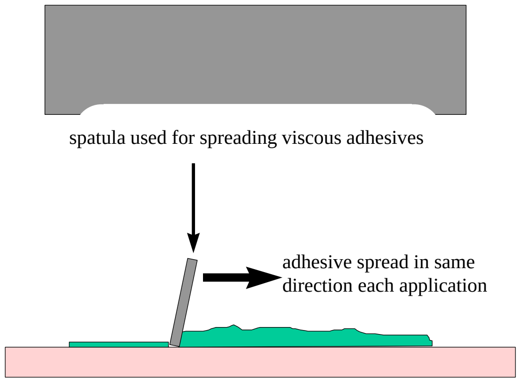

Figure 2: Bulk specimen manufacture technique for a highly viscous adhesive.

High viscosity adhesives can be difficult to dispense into a single 'cast' as described above. An alternative method for dispensing these adhesives into the mould is to build up layers of the adhesive by spreading using a specially shaped spatula (Figure 2). A small amount of adhesive is applied at first. This is spread as a thin layer. More adhesive is added gradually and smoothed out along the original layer. The action of spreading the adhesive acts to displace air that may become entrapped in the adhesive. This is a much slower operation for preparing specimens than dispensing directly from a cartridge. Consequently, it is unsuitable for adhesives with short working lives.

Curing the adhesive

Bulk adhesive specimens should be cured under conditions that approximate those that occur in the adhesive when the bonded component is cured. There is a danger that the low thermal conductivity of the adhesive may not allow the heat generated by exothermic cure reactions to dissipate. This could lead to excessive temperature rises in the bulk adhesive in comparison to those in the thin layers in joints. Metal moulds should act as heat sinks and prevent excessive temperature rises. However, temperatures in the adhesive during cure should be monitored using a thermocouple embedded in the adhesive.

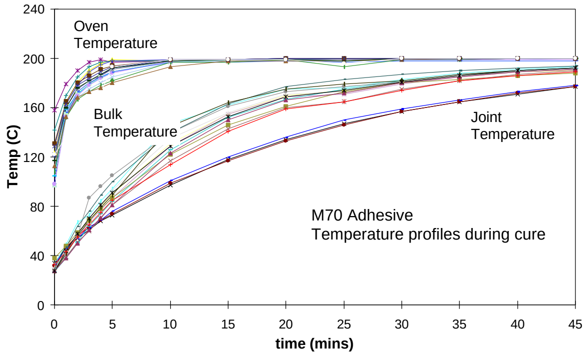

Figure 3: Thermal histories of bulk and joint specimens cured under identical conditions.

For adhesives cured at elevated temperatures, differences in the effective thermal mass of the bulk specimen mould from the bonded joint may lead to differences in the thermal histories of the adhesives. Simply placing both fixtures into an oven at the same temperature for the same duration does not guarantee equivalent states of cure.

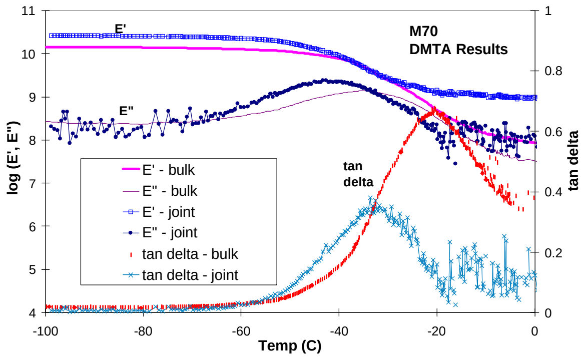

Figure 4: DMTA results showing the properties of adhesive cured in the bulk specimen and joint specimen preparation fixtures.

Figure 3 shows temperature measurements made whilst curing bulk adhesive specimens and adhesively bonded lap joint specimens. The temperature of the specimens lags that of the oven. The joint specimens are slower to heat than the bulk test specimens. The final temperature of the adhesive in the joint at the end of the cure period is significantly lower than in the bulk adhesive.

The Dynamic Mechanical Thermal Analysis (DMTA) data in Figure 4 show that these differences between thermal histories may lead to differences between the mechanical properties. Since the objective of testing the bulk specimens is to determine material properties for use in designing joints, differences between the material properties will lead to inaccuracies in the design predictions.

Curing behaviour of the adhesive can be studied directly from the development of the material modulus (Figure 5). This can be measured by either oscillatory rheometry or ultrasonic methods [5]. Such studies enable the establishment of optimised cure schedules. However, equally important is information on how rapidly the bonding fixture heats. With this information the cure schedules for the bulk specimens can be adjusted to give comparable thermal histories and, therefore, representative test specimens can be produced. In the example above, it was found that increasing the oven temperature by 15 °C when curing joint specimens gave material with mechanical properties comparable to the bulk specimens.

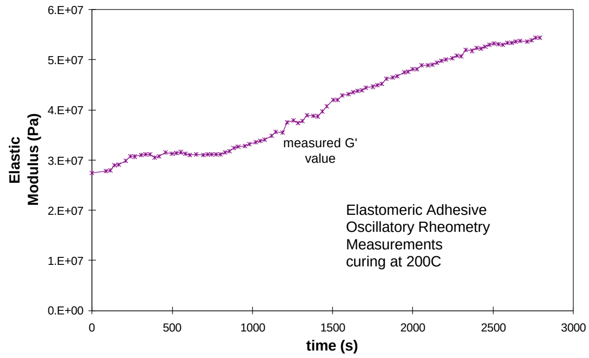

Figure 5: Development of modulus during cure

Whilst specimen preparation is key to producing representative test specimens, the storage of the sample between manufacture and testing can have significant influence on the material properties. For example, adhesives that cure at room temperature often continue to cure for a significant period of time after cure is nominally 'complete'. These specimens should either be post-cured or stored at low temperatures to reduce the variability in results due to specimen age. The moisture content of the adhesive will affect its mechanical properties. Therefore, post-manufacture moisture adsorption by the specimens is another factor to consider.

Concluding remarks

Tests on bulk test specimens are the most accurate method for obtaining design data for adhesives. However, to obtain data that is truly representative of the materials in joints requires careful specimen preparation. Standard methods for preparing such specimens have been developed. However, there are a number of key points that should be considered when producing test specimens.

- Specimens should be prepared using methods that minimise the inclusion of air in the test specimens.
- Cure schedules need to be developed that reproduce the thermal history experienced by the adhesive layer in the manufactured joint.
- The mechanical properties of the test specimens will be affected by the conditions that they experience after they have been 'cured'. These also need to be specified and controlled.

Acknowledgements

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