MATC(MN)46

Round-robin validation exercise for the thick adherend shear test in compression

Summary

This Measurement Note details a round-robin exercise undertaken in order to provide precision data to support the proposal of the thick adherend shear test (TAST) in compression as a proposed new work item for ISO test methods.

The exercise was organised to principally assess the specimen manufacture stage of the tests, as past experience had shown this to be the most important part of the process. The exercise involved the manufacture of specimens by five participating organisations, using two different adhesives. The testing of all specimens was performed at NPL. Statistical analysis was performed on the test results according to ISO 5725-2[1] using 95% confidence limits to determine their repeatability and reproducibility.

The work was carried out as part of the "Measurements for Materials Systems" (MMS) programme funded by the Department of Trade and Industry.

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INTRODUCTION

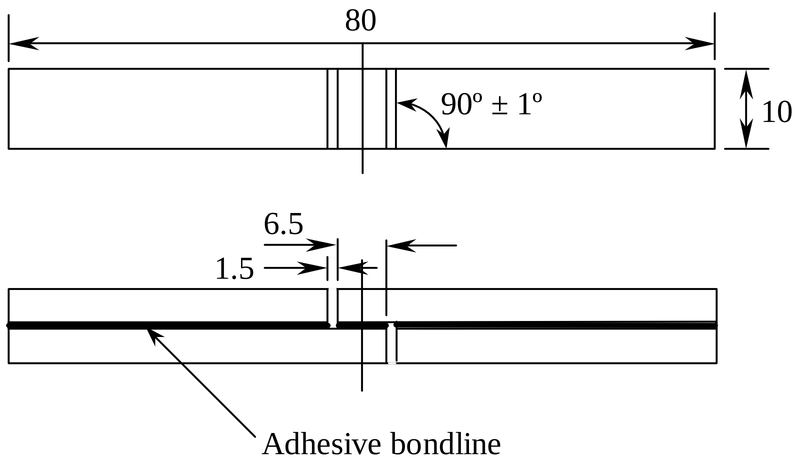
Currently the thick adherend shear test method is standardised only in the tension mode. To provide precision data to support the progress of the compression version of the test method a round-robin validation exercise was organised. Five laboratories took part in the exercise, each laboratory manufacturing the specimens which were then sent back to NPL for testing.

The specimen is placed in an anti-buckling fixture, based on the fixture in ASTM D 695[2], and the ends loaded in compression until shear failure of the adhesive bond in the centre of the specimen occurs. The cross-head displacement and load are recorded throughout the test.

Previous round-robins conducted by NPL have shown that manufacturing differences cause the most variability in results[3]. hence the round-robin concentrated on this aspect of the test and all specimens were therefore tested at NPL by the same operator.

MANUFACTURE AND TESTING

All the materials used in the round-robin were supplied by NPL. The specimens were manufactured from 6082T6 aluminium alloy strips and two adhesives: Adhesive A, a two-part room temperature-curing epoxy, and Adhesive B, a single-part high temperature-curing epoxy. Four pre-cut aluminium sections, 4 mm thick, were supplied for each specimen and assembled as shown in Figure 1.



(all dimensions in mm – not to scale)

Figure 1 Thick adherend shear test compression specimen dimensions

Five organisations participated in the round-robin:

- MERL (Materials Engineering Research Laboratory) Ltd
- MIRA (Motor Industry Research Association) Ltd
- Permabond UK
- Stanger Environmental Science
- National Physical Laboratory

Each organisation was sent the materials needed to assemble and manufacture five specimens using each adhesive, along with a draft bonding and test procedure, and report sheets to fill in detailing the various stages in the specimen manufacture. Details of the manufacturing differences between the test laboratories are given in Table 1.

Manufacturing stage	Manufacturing processes used
Surface preparation	60-80 alumina grit blast; 180 grade SiC paper; 240 grade SiC paper
Degreasing solvent	Acetone; isopropyl alcohol; methylated spirits
Method of specimen alignment	Clamped in PTFE angle section; lined up against V-shaped frame; by eye
Control of bondline thickness	Ballotini glass beads supplied by NPL; none
Control of fillet	PTFE strip
Method of clamping pressure applied	Bulldog clips; weight on specimens; G-clamped between metal plates; bound with tapes
Clamping pressure	30-40 N
Cure temperature	As specified

Table 1 Variations in specimen manufacture between participating laboratories

The manufactured specimens were then sent back to NPL and tested to failure by a single operator. Each specimen was placed in an anti-buckling fixture, which is a modified version of the fixture described in ASTM D 695[2] as shown in Figure 2 and Figure 3. A compressive force is applied to the top end face of the specimen at a constant displacement rate of 2 mm/min, resulting in a state of pure shear in the adhesive layer between the two notches. The force and displacement of the cross-head until failure occurred were recorded.

The decision to use a single operator at NPL to carry out all the testing was undertaken for two reasons for this: firstly, previous experience at NPL have shown that the main cause of variability within the results is caused by differing specimen manufacture processes and that differences due to different operators are less important; and secondly, it was thought that the D 695 anti-buckling fixture would not be widely available to many of the participants and that sending NPL's own fixture to each in turn for testing to be carried out would prove to be time-consuming.

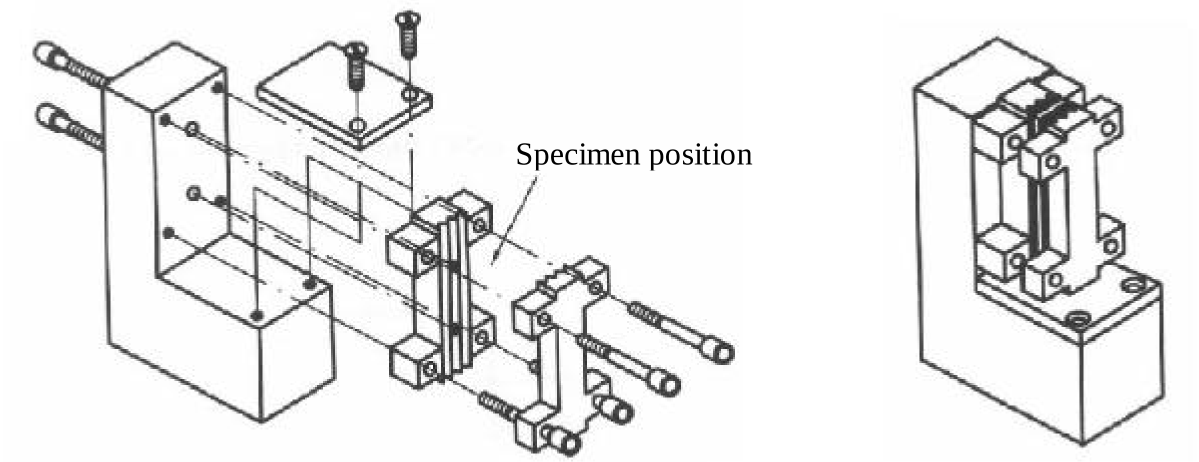


Figure 2 Schematic of anti-buckling fixture

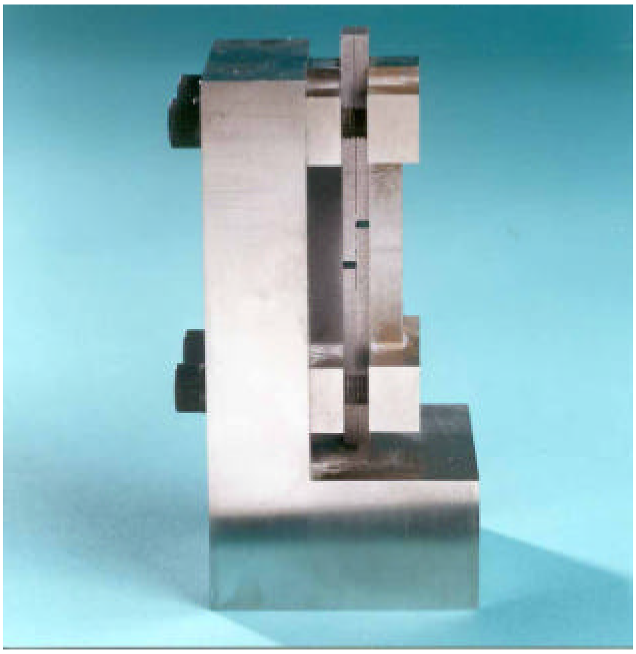


Figure 3 Specimen in anti-buckling fixture

Participant	Individual results (MPa)	Average failure stress (MPa)	Standard deviation
1	23.99, 22.11, 29.25, 21.37, -	24.18	3.56
2	25.83, 22.58, 26.72, 25.22, 27.39	25.55	1.66
3	12.90, 18.16, 13.67, 21.48, 15.15	16.27	3.17
4	12.30, 25.13, 17.48, 12.77, 20.66	17.67	4.85
5	33.27, 28.88, 32.64, 35.69, 32.42	32.58	2.18

Table 2 Results of specimens manufactured with Adhesive A

Participant	Individual results (MPa)	Average failure stress (MPa)	Standard deviation
1	37.82, 37.61, 34.12, 37.61, 33.85	36.20	1.81
2	41.20, 40.90, 43.35, 40.07, 44.81	42.07	1.75
3	47.38, 37.72, 45.52, 40.28, 39.99	42.18	3.65
4	53.13, 53.86, 45.61, 47.46, 55.94	51.20	3.96
5	41.31, 43.15, 38.14, 44.30, 41.03	41.59	2.10

Table 3 Results of specimens manufactured with Adhesive B

EVALUATION OF RESULTS

The results of the tests are detailed in Table 2 and Table 3 (the order of the participants has been randomised from the list on page 2). The results were then analysed using the statistical methods detailed in ISO 5725-2[1] for repeatability and reproducibility within 95% confidence limits. Repeatability refers to tests performed under conditions that are as constant as possible, with the tests performed during a short interval of time in one laboratory by one operator using the same equipment. Reproducibility refers to tests performed in widely varying conditions, in different laboratories with different operators and different equipment. Thus the value of repeatability, r, and the value of reproducibility, R, are the values below which the absolute difference between two single test results may be expected to lie within a probability of 95%, under repeatability and reproducibility conditions respectively. S_r and S_R are the standard deviations of repeatability and reproducibility respectively.

When the data were analysed no outliers were found. The repeatability and reproducibility of the specimens, their standard deviations, and their respective coefficients of variation at the 95% confidence limits are shown in Table 4.

Adhesive	Mean stress (MPa)	r	S_r	C.o.V.	R	S_R	C.o.V.
Adhesive A	23.21	10.02	3.58	15.4 %	20.81	7.43	32.0 %
Adhesive B	42.65	8.83	3.15	7.4 %	17.04	6.09	14.3 %

Table 4 Repeatability and reproducibility values for test results

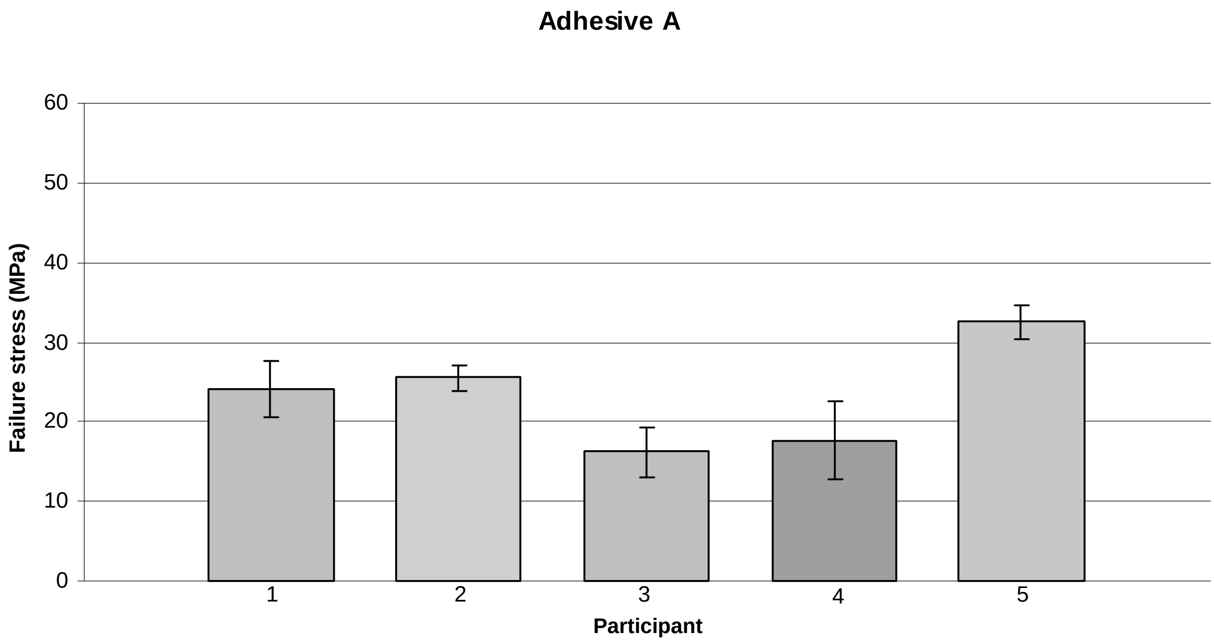


Figure 4 Comparison of average failure stresses (with error bars) of specimens manufactured with Adhesive A from each participant

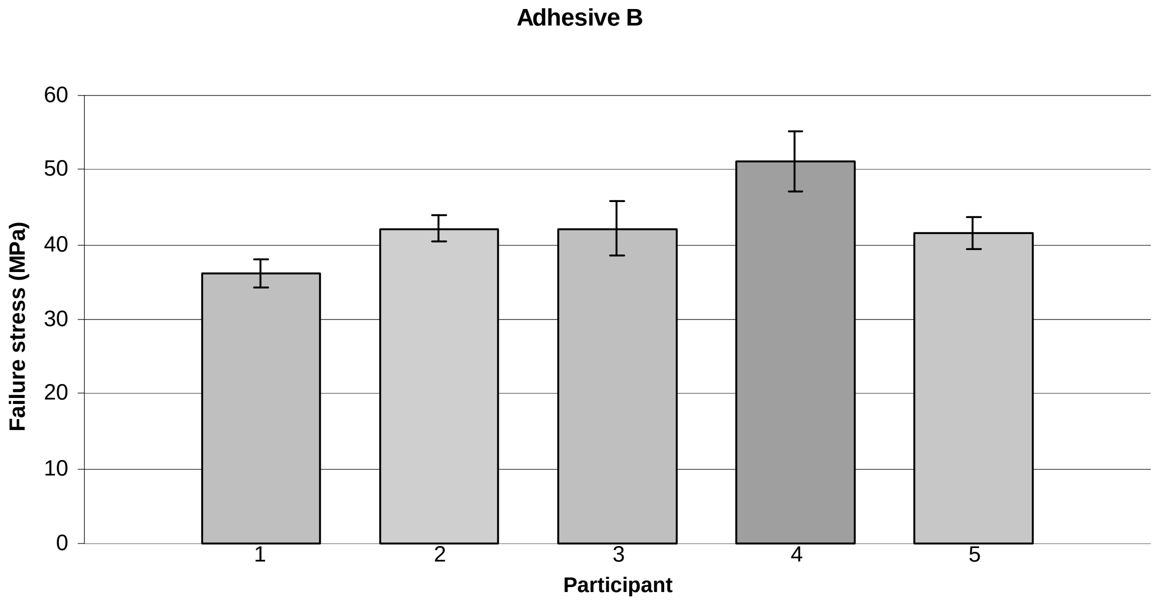


Figure 5 Comparison of average failure stresses (with error bars) of specimens manufactured with Adhesive B from each participant

DISCUSSION OF RESULTS

There is a marked variation in average results between the five test laboratories for both adhesives. The results from participants 3 and 4 both displayed considerably lower values for Adhesive A than the other laboratories. The failure mode of this adhesive is predominantly interfacial, so the surface preparation of the adherends is more critical. Inspection of the failure surfaces of specimens from these two batches showed that the abrasion of the surface was generally poor: these were the two participants that abraded the surfaces manually using silicon carbide paper rather than grit blasting the surfaces.

For Adhesive B the lowest failure stresses were from the specimens manufactured by participant 1. This participant was the only one to use a large steel bonding fixture to align the specimens during assembly and high temperature cure, rather than using bulldog clips: it is thought that the fixture, and therefore the specimens in it, would have heated up more slowly and as a result these specimens may be under-cured compared to those from the other participants. The highest results were achieved from participant 4: this laboratory used only tape to assemble the materials during curing and no metal parts were used.

FURTHER TESTS

Additional tests were undertaken at NPL only on other systems as background research. Four different adherend materials were tested in addition to aluminium: mild steel, titanium, a unidirectional carbon fibre-reinforced plastic, and a glass fibre-reinforced plastic pultrusion. The thickness to be used for each adherend was chosen to provide a flexural stiffness value, EI, at least as high as the value for the 4 mm thick aluminium reference material. Tests were also carried out on thinner steel adherends and thicker aluminium adherends to determine whether the change in thickness affected the overall strength properties. The results are shown in Table 5.

Material	Thickness (mm)	Average strength (MPa) ± standard deviation	
		Adhesive A	Adhesive B
Mild steel	2.5	28.71 ± 1.59	48.63 ± 0.66
CFRP	3.0	29.37 ± 4.76	37.90 ± 1.26
Titanium	3.0	22.45 ± 5.74	43.61 ± 3.76
Al alloy	4.0	24.18 ± 3.56	36.20 ± 2.03
GFRP pultrusion	5.2	24.65 ± 1.19	21.69 ± 1.81
Mild steel	1.5	Not tested	47.52 ± 0.55
Al alloy	6.0	Not tested	35.05 ± 0.95

Table 5 Results of additional tests on other material systems

For Adhesive A it appears that the failure strength is largely independent of the adherend type and its thickness. This may be due to the adhesive failing in adhesion which would mean the surface preparation would be the critical factor in determining the failure strength. For Adhesive B it can be seen that the shear strength is not wholly dependent on the flexural stiffness and that there is an effect of adherend material, as the strength is highest for the stiffest adherend (steel) and lowest for the least stiff adherend (pultrusion).

Changing the thickness of the adherend appears to have had little effect on the failure strength of the specimens, suggesting that the maximum strength as obtained from the bulk adhesive has already been reached and that for a sufficiently stiff adherend there is no increase in the strength value.

CONCLUSIONS

The round-robin supported findings from previous round-robin exercises that variability in the preparation of the specimens can have a marked effect on the failure load and the repeatability and reproducibility of the results. Results for repeatability and reproducibility of the specimens made using the two-part Adhesive A are comparable with those obtained in a previous round-robin for single-lap joints[3] – the results for Adhesive B are significantly better.

REFERENCES

[1] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[2] ASTM D 695:2002, *Standard test method for compressive properties of rigid plastics*

[3] Broughton, B and Gower, M, *Preparation and Testing of Adhesive Joints*, NPL Measurement Good Practice Guide No. 47, 2001

ACKNOWLEDGEMENTS

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