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Round-Robin Validation Exercise for Plate Twist Test Method

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ABSTRACT

A round-robin validation exercise has been undertaken to obtain precision data for **"the plate twist test method for determination of in-plane shear modulus"**. Six fibre-reinforced, polymer matrix composite materials representing a range of composite materials and shear moduli (1-6 GPa) were tested. The tests were undertaken by eight laboratories, seven of which had not previously used the method, according to the draft test procedure developed by NPL. The exercise was organised and the results were analyzed according to ISO 5275 using 95% confidence limits. The detailed results are given in Appendix I.

Repeatability values, when expressed as a percentage of the mean, were between 2.80% and 5.38%. The reproducibility, when similarly expressed, was generally less than 7%, with a higher value obtained for the lowest shear modulus material.

The precision data have been used to support a UK proposal, prepared by NPL, for a New Work Item (NWI) within the ISO work programme. The test method has been studied and the improved analysis validated as part of the NPL programme on test methods for composites, funded by the DTI.

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1 INTRODUCTION

A round-robin validation exercise has been undertaken to obtain precision data for **"the plate twist test method for determination of in-plane shear modulus"**. Full details of the plate-twist method are given in [1]. Six fibre-reinforced polymer matrix composite materials representing a range of composite materials and shear moduli (1-6 GPa) were tested. The tests were undertaken according to the attached test procedure (Appendix II). The tests were undertaken by eight laboratories, seven of which had not previously used the method. The exercise was organised and the results were analyzed according to ISO 5275 [2] using 95% confidence limits.

The programme was undertaken to provide precision data to support a UK proposal, prepared by NPL, for a New Work Item (NWI) on this test method within the ISO work programme. The particular attractions of this test method are the absence of special specimen preparation, application to a wide range of composites, simple loading fixtures and test machine requirements, which result in a cheap test method. Shear moduli are important for composites as they are frequently low relative to the tensile modulus as compared to isotropic materials. Suitable orientaton of the fibres can improve the shear modulus.

2 ROUND-ROBIN PROGRAMME

2.1 PARTICIPANTS

Centre for Composite Materials, Imperial College
Short Brothers PLC
Leicester University
Shell Research BV
Queen Mary and Westfield College
RAPRA Technology
DRA (Farnborough)
National Physical Laboratory

2.2 MATERIALS

The following materials, designated by codings, were tested:-

Material 1	RR1-5	Unidirectional glass-fibre/epoxy
Material 2	RR6-10	SMC (glass-fibre/filler/polyester)
Material 3	RR11-15	Woven glass-fibre/epoxy
Material 4	RR16-20	Random glass-fibre/polypropylene
Material 5	RR21-25	Injection moulded glass-fibre/nylon
Material 6	RR30-34	Unidirectional carbon-fibre/epoxy

For each material 5 plates, 150 mm x 150 mm, were prepared by cutting with a water-cooled diamond-faced cutting blade.

2.3 ROUND-ROBIN PROCEDURE

The test method applies only a small elastic strain to the specimen, so a single batch of 5 plates for each material could be circulated to each participant in turn. This enabled the same plates to be tested by each participant, thus reducing the variability of the results compared with destructive round-robin exercises. After testing, the plates were either dispatched directly to the next participant or returned to NPL for dispatch. The results were sent to NPL for collation and analysis. The analysis of the results for reproducibility and repeatability was undertaken using software written at NPL to the method described in ISO 5725 [2] using 95% confidence limits.

3 EVALUATION OF RESULTS

3.1 REPEATABILITY AND REPRODUCIBILITY

The results given below are calculated using the method of ISO 5725 [2], after removal of outliers as described in that method. The detailed individual results are given in Appendix I. The data are also shown in Figures 3-8. The outliers were removed and the reasons for their removal are given in Section 3.3.

Table 1. Repeatability, Reproducibility and Mean Shear Moduli

Material	Repeatability Conditions		Reproducibility Conditions		Mean Result (GPa)
	S_r	r	S_R	R	
1	0.164	0.459	0.302	0.846	5.852
2	0.137	0.385	0.184	0.516	4.296
3	0.106	0.296	0.307	0.859	4.386
4	0.096	0.269	0.098	0.274	1.784
5	0.061	0.171	0.165	0.461	1.156
6	0.200	0.559	0.309	0.865	5.171

Table 2. Repeatability and Reproducibility Values as Percentage of Mean Values

Material	Value as Percentage of Mean			
	Repeatability Conditions		Reproducibility Conditions	
	S_r	r	S_R	R
1	2.80	7.84	5.41	14.4
2	3.19	8.96	4.29	12.0
3	2.42	6.75	7.00	19.6
4	5.38	15.1	5.50	15.4
5	5.28	14.8	14.27	39.8
6	3.87	10.8	5.98	16.7

Note: Definitions taken directly from ref [2]

Repeatability value, (r): The value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95%.

Reproducibility value, (R): The value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95%.

Repeatability standard deviation, (Sr): The standard deviation of test results obtained under repeatability conditions.

Reproducibility standard deviation, (SR): The standard deviation of test results obtained under reproducibility conditions.

3.2 CRITICAL DIFFERENCE VALUES

These results reflect how well the measured data coincide with the statistical description of the sample based on the normal distribution. A perfect fit is suggested when the critical difference value is 5%. The larger the dataset analyzed the more likely that a value of 5% will be achieved.

Table 3. Critical Differences

Material	Critical Difference Values (%)	
	Repeatability	Reproducibility
1	2.9	4.8
2	5.3	7.1
3	0.0	9.5
4	3.8	10.7
5	2.5	3.6
6	7.5	3.6

Note: Definitions taken from ref [2]

Repeatability critical difference CrD: the value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95%.

Reproducibility critical difference CrD: the value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95%.

3.3 DATA REMOVED FROM THE ANALYSIS

The following datasets were removed after the initial analysis for the reason stated:

Material 3 data from site 3 failed the Cochran test for outliers and the Dixon test for stragglers.

Material 1 data from site 3 failed the Cochran cell variance test, the Dixon cell averages test, and the maximum and minimum values for the Material 1 dataset were in this cell.

The data were then re-analyzed with these datasets removed.

4 DISCUSSION

The participants were able to use the supplied test method without further support. No difficulties in building the text fixture were reported. Participants were generally surprised at the low load levels reached and suggested that a note to this effect be made in the test method. It was also suggested that a linear regression technique for determination of the slope should be included in the method as an alternative to the chord modulus approach.

The removal of Material 1 data measured at site 3 is fully justified. The spread of values between specimens from this site is high as shown by the coefficient of variance 11.6% compared to that of the other sites of between 3.3% and 1.9%. This effect is clearly shown in Figure 3.

The removal of Material 3 data measured at site 3 is also fully justified. The spread of values between specimens from this site is high as shown by the coefficient of variance 6.3% compared to that of the other sites of between 3.2% and 1.7%. This effect is clearly shown in Figure 5.

The critical difference values are close to 5% and therefore indicate that the data collected can be approximated to a normal distribution with mean and variance values as shown. The practicalities of the round-robin exercise do not permit an increased number of replications which would increase the closeness of this approximation. Treatment of the data by the method chosen, which is based on the normal distribution, is therefore justified. The repeatability of test results shows an imperfect linear relationship with measured shear modulus (Figure 1). For reproducibility values the pattern is less clear and other factors, such as the reproducibility of the thickness measurement, may influence the result (Figure 2).

The higher scatter for the lowest modulus material may possibility be associated with loading point indentation.

5 CONCLUSIONS

- The draft test method for measurement of in-plane shear modulus by the plate twist method has been validated.
- The precision data have been used to support a UK proposal, for a New Work Item (NWI) within the ISO work programme.
- The participants were able to use the draft test method without further assistance.
- The critical difference values support the approximation of the data to the normal distribution and validate the statistical tests used.
- Repeatability values were between 2.80% and 5.38% of the mean.
- Reproducibility values were generally less than 7% of the mean, although a higher value was obtained for the material with the lowest shear modulus.

ACKNOWLEDGEMENTS

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The authors wish to thank the participants, without whose efforts the round-robin would not have been possible, and Anne Woolf of the Division of Materials Metrology at NPL who wrote the software.

REFERENCES

(1) Sims G D , Nimmo W, Johnson A F and Ferriss D H "Analysis of Plate-Twist Test for In-Plane Shear Modulus of Composite Materials". NPL Report DMM(A)54*.

(2) ISO 5725 - 1986: Precision of Test Methods. Part 1 "Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory test".

FIGURES

Figure 1 Repeatability vs Shear Modulus

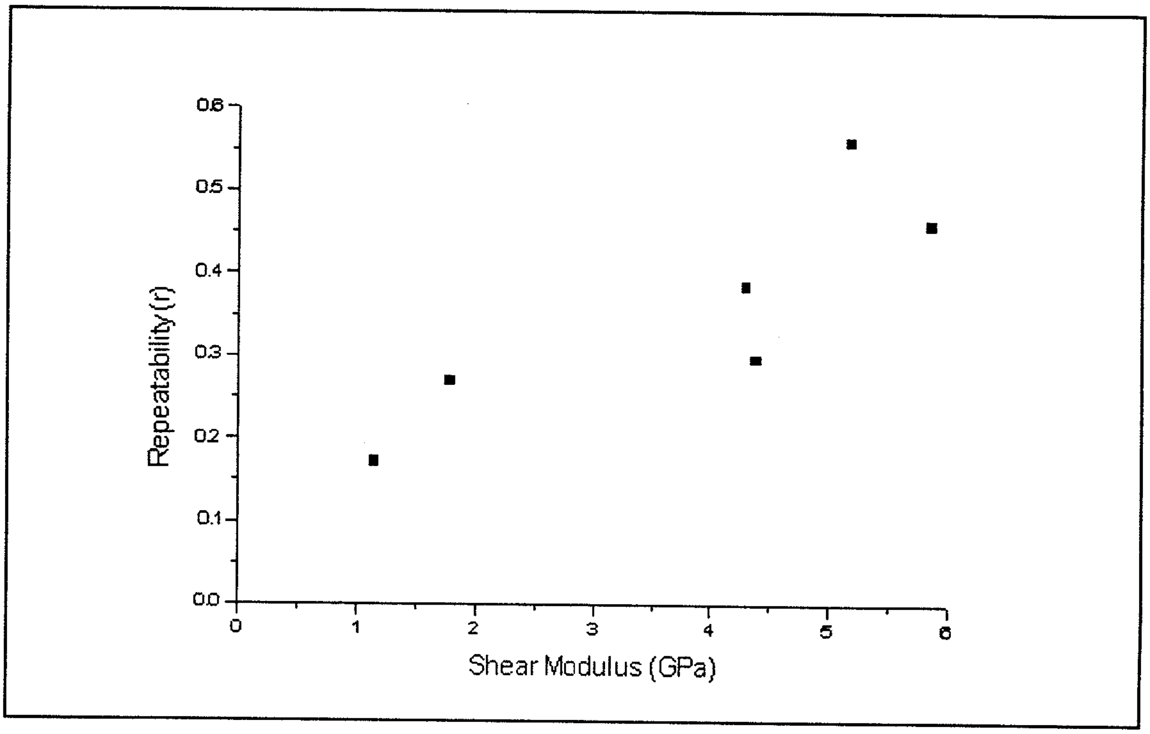


Figure 2 Reproducibility vs Shear Modulus

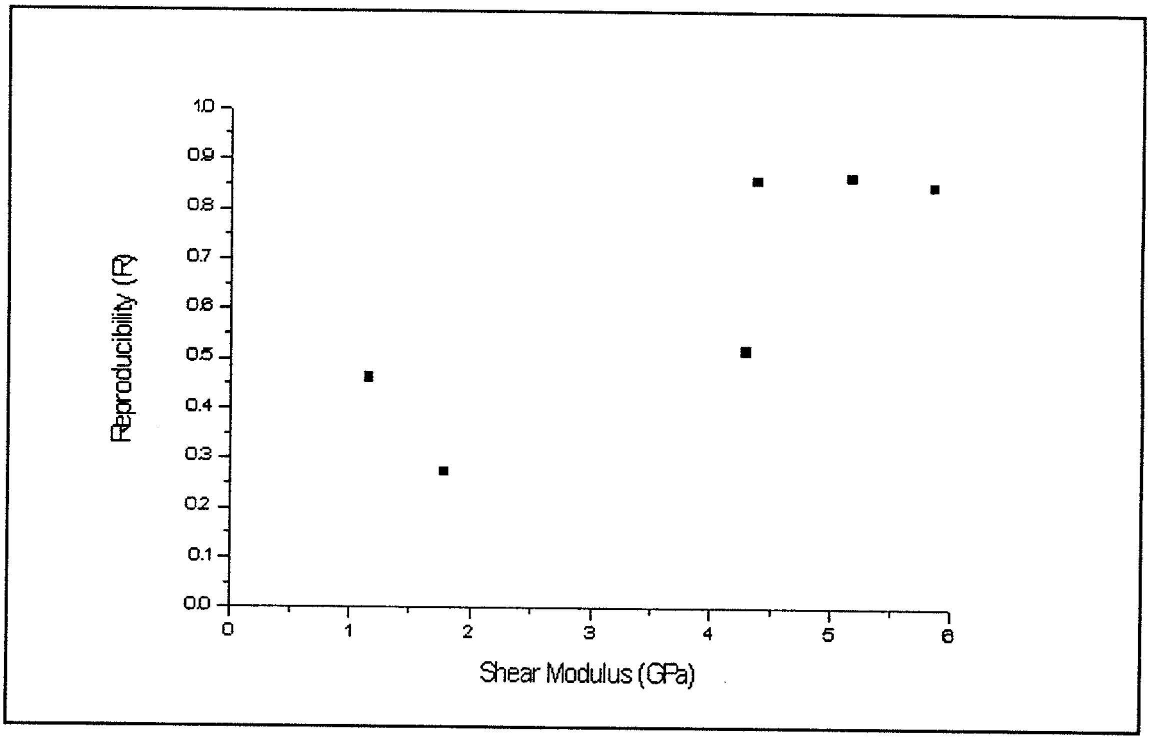


Figure 3 Material 1 Data from all sites

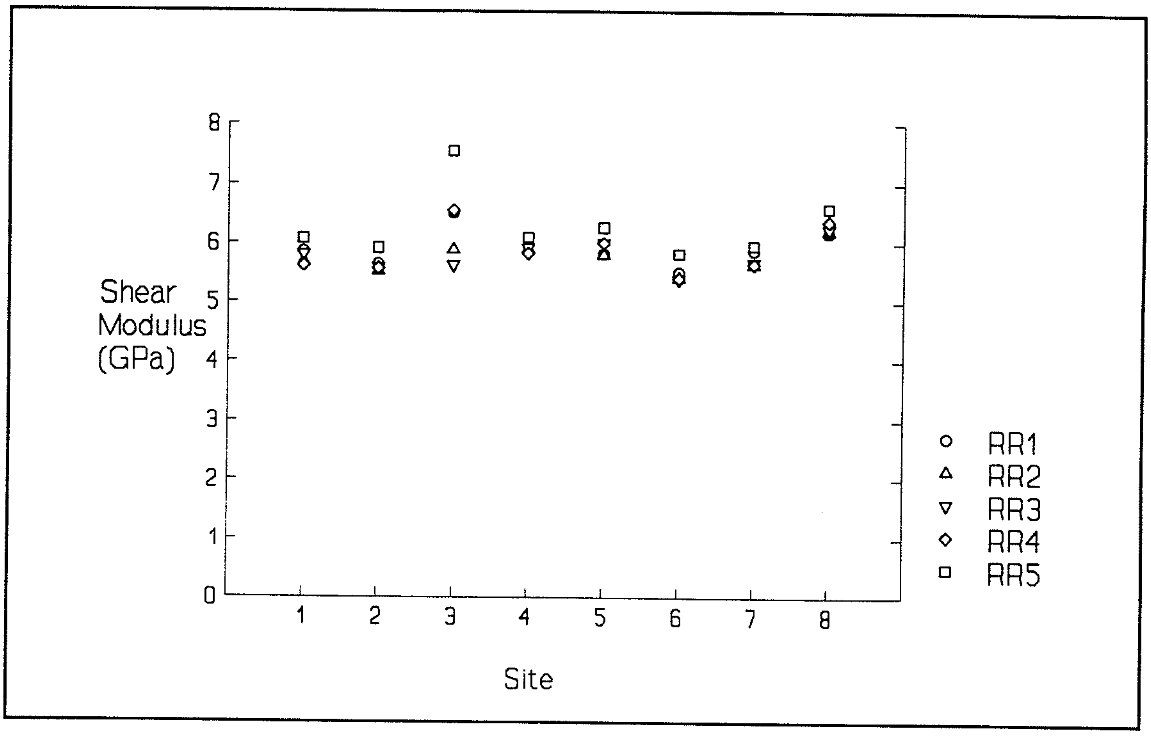


Figure 4 Material 2 Data from all sites

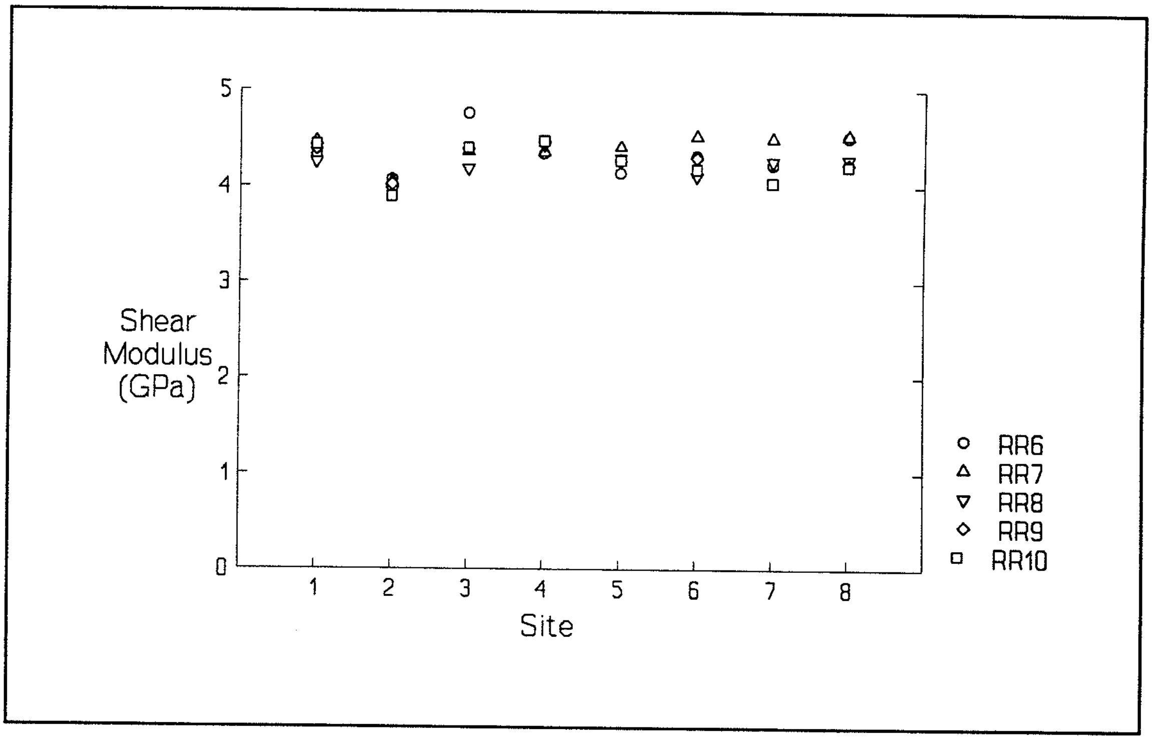


Figure 5 Material 3 Data from all sites

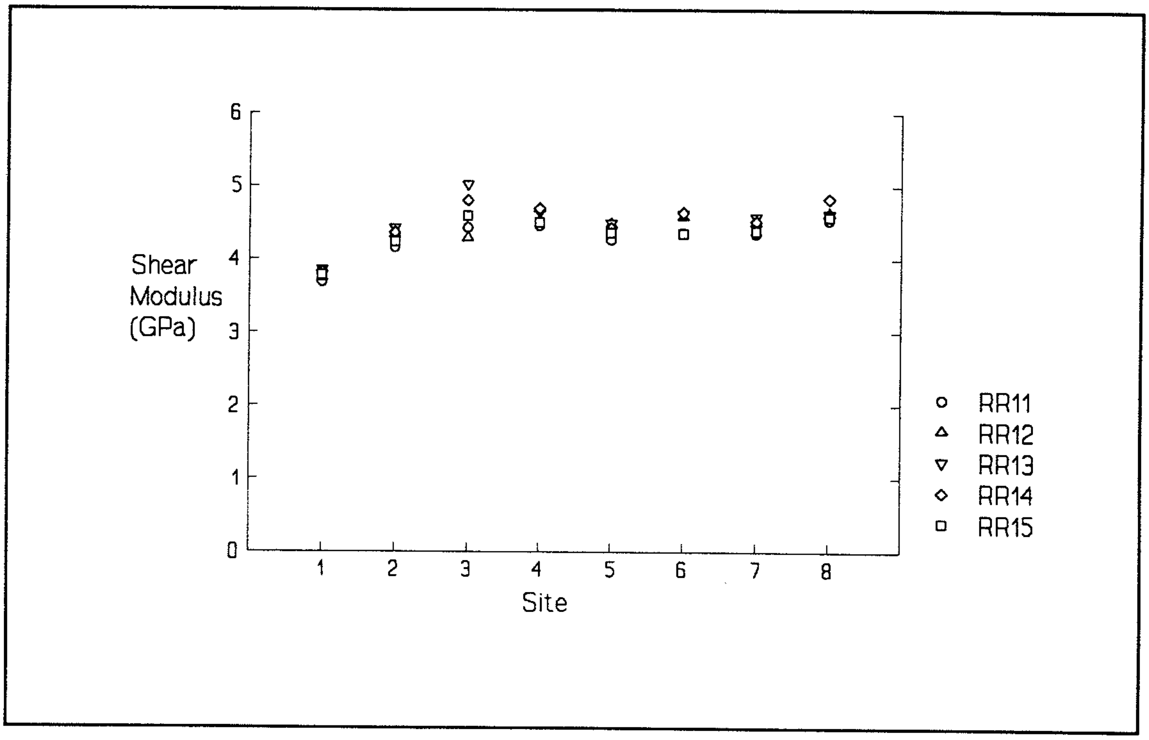


Figure 6 Material 4 Data from all sites

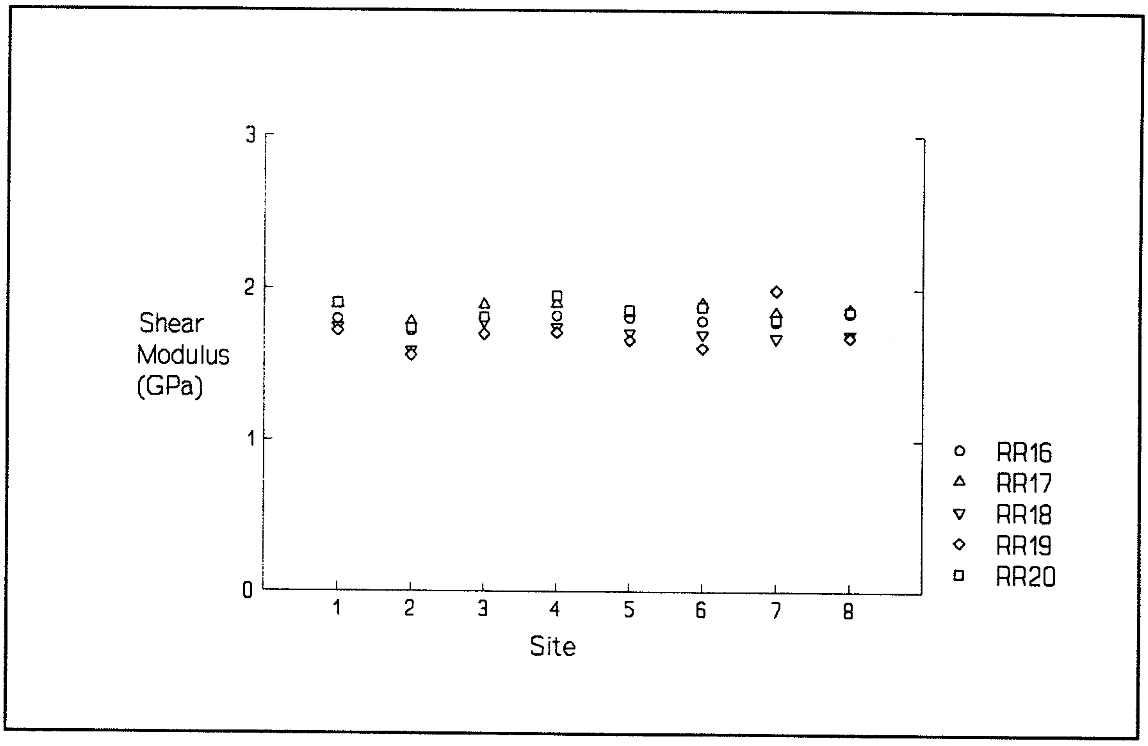


Figure 7 Material 5 Data from all sites

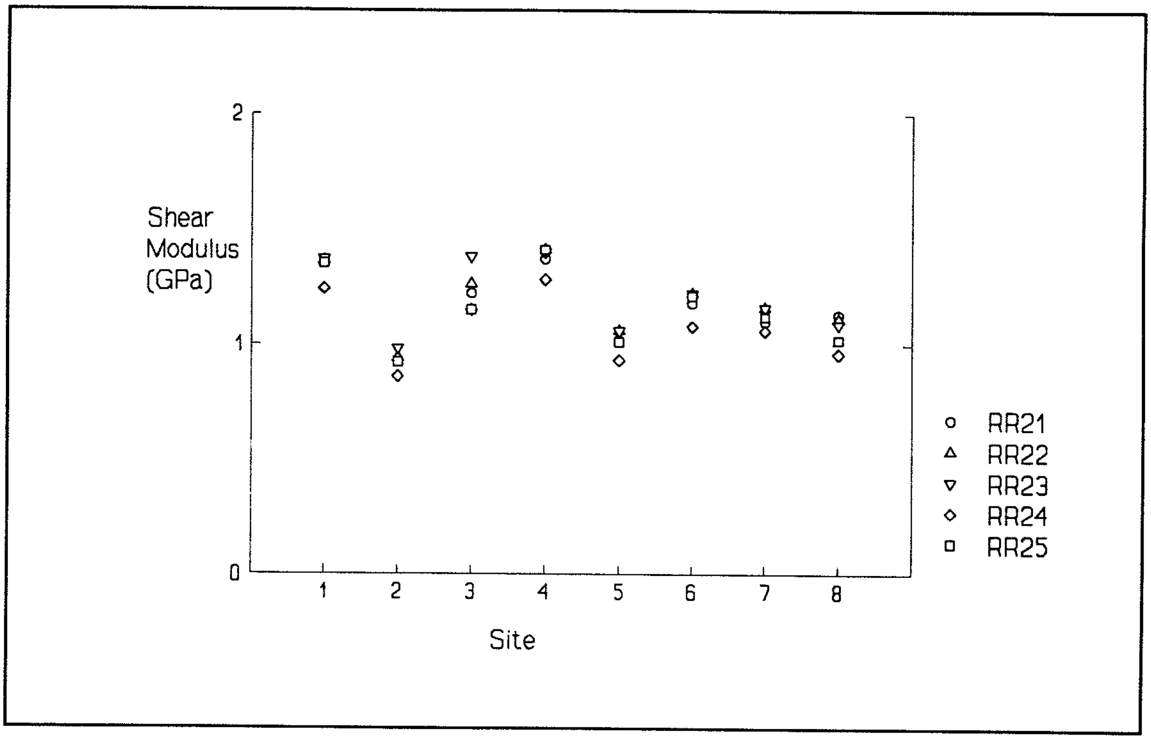
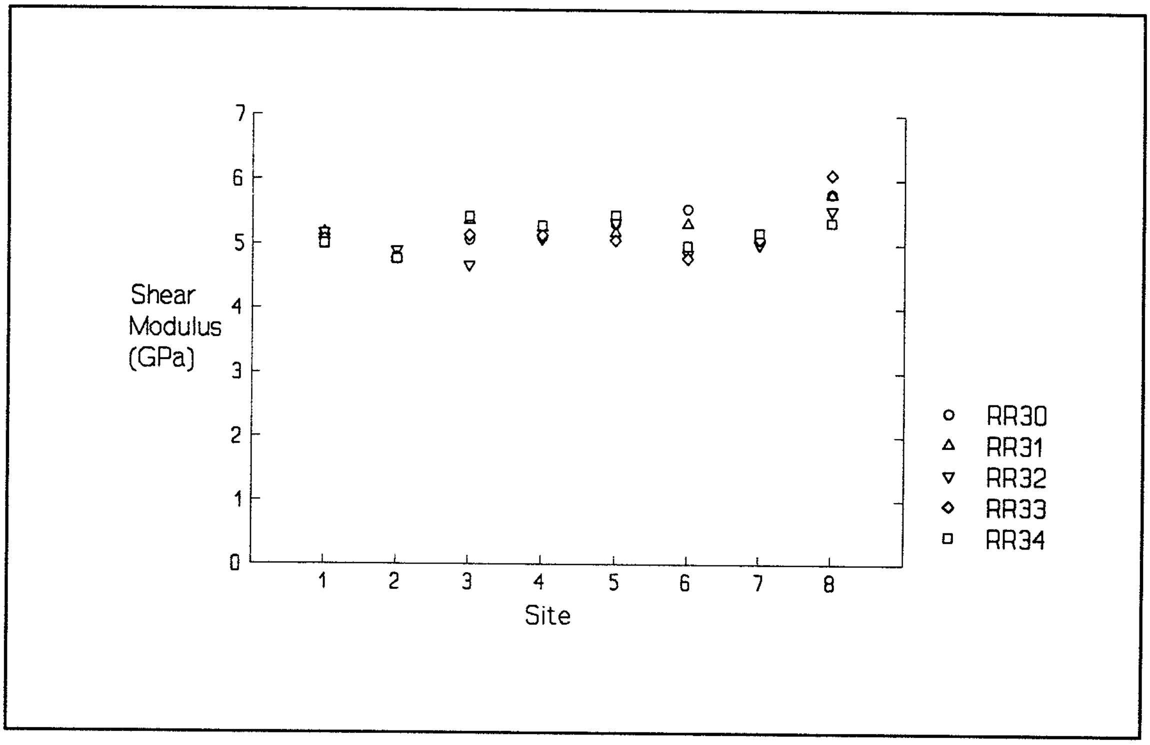


Figure 8 Material 6 Data from all sites



Appendix 1

Table A.1. Results from all sites on all specimens of material 1.

Specimen Identity	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8
RR1	5.86	5.64	6.51	6.07	5.82	5.51	5.88	6.18
RR2	5.67	5.51	5.88	5.89	5.80	5.41	5.64	6.21
RR3	5.80	5.58	5.62	5.91	6.00	5.39	5.67	6.25
RR4	5.61	5.57	6.55	5.82	6.00	5.41	5.65	6.37
RR5	6.07	5.91	7.54	6.09	6.26	5.82	5.96	6.59

Table A.2. Results from all sites on all specimens of material 2.

Specimen Identity	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8
RR6	4.32	4.07	4.76	4.34	4.14	4.32	4.23	4.52
RR7	4.46	4.04	4.36	4.35	1.41	4.52	4.50	4.53
RR8	4.25	3.95	4.18	4.43	4.29	4.11	4.26	4.28
RR9	4.38	4.02	4.39	4.46	4.27	4.30	-	4.23
RR10	4.43	3.89	4.40	4.47	4.27	4.18	4.04	4.21

Table A.3. Results from all sites on all specimens of material 3.

Specimen Identity	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8
RR11	3.69	4.16	4.43	4.46	4.26	4.36	4.35	4.54
RR12	3.74	4.34	4.29	4.69	4.48	4.58	4.50	4.63
RR13	3.85	4.42	5.02	4.65	4.49	4.62	4.58	4.62
RR14	3.82	4.36	4.80	4.69	4.43	4.64	4.52	4.83
RR15	3.78	4.24	4.59	4.50	4.36	4.35	4.39	4.58

Table A.4. Results from all sites on all specimens of material 4.

Specimen Identity	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8
RR16	1.80	1.72	1.81	1.82	1.81	1.79	1.78	1.84
RR17	1.89	1.78	1.89	1.90	1.84	1.90	1.84	1.86
RR18	1.75	1.59	1.78	1.75	1.71	1.70	1.68	1.70
RR19	1.72	1.56	1.70	1.71	1.66	1.61	1.99	1.68
RR20	1.90	1.74	1.81	1.95	1.86	1.88	1.80	1.85

Table A.5. Results from all sites on all specimens of material 5.

Specimen Identity	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8
RR21	1.36	0.93	1.22	1.37	1.02	1.18	1.10	1.13
RR22	1.36	0.94	1.26	1.41	1.06	1.22	1.16	1.11
RR23	1.37	0.98	1.38	1.40	1.06	1.22	1.16	1.09
RR24	1.24	0.86	1.15	1.28	0.932	1.08	1.06	0.96
RR25	1.35	0.92	1.15	1.41	1.01	1.21	1.12	1.02

Table A.6. Results from all sites on all specimens of material 6.

Specimen Identity	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Site 7	Site 8
RR30	5.12	4.77	5.07	5.10	5.32	5.55	5.18	5.78
RR31	5.17	4.77	5.36	5.25	5.17	5.31	5.09	5.77
RR32	5.17	4.90	4.67	5.08	5.33	4.85	5.00	5.53
RR33	5.07	4.78	5.13	5.13	5.06	4.78	5.08	6.08
RR34	5.00	4.77	5.42	5.28	5.45	4.97	5.18	5.34

Appendix II

Reinforced Plastics - Determination of in-plane shear modulus by the plate twist method.

1 Scope

1.1 This draft specifies a method for determining the in-plane shear modulus (G_{12}) of reinforced-plastics/composites using a standard plate specimen. When applied to isotropic materials the shear modulus measured is independant of direction.

1.2 The method is used to investigate the shear modulus of the test specimens but not for determining the shear strength. It applies to a plate supported on one diagonal and loaded, on the other diagonal by movement of the crosshead.

1.3 The method is suitable for use with the following range of materials:

filled and reinforced compounds; including laminates;

fibre-reinforced thermoset and thermoplastics composites incorporating unidirectional or non-unidirectional reinforcements such as mat, woven fabrics, woven rovings, chopped strands, combination reinforcement and hybrid rovings.

NOTE 1: This method can also be applied to unreinforced polymers and other materials (e.g. metals and ceramics, metal or ceramic matrix composites).

1.4 The method is performed using specimens which may be either moulded to the chosen dimensions, machined from flat areas of products or machined from semi-finished products such as mouldings, laminates and extruded or cast sheet.

1.5 The method specifies preferred dimensions for the specimen. Tests which are carried out on specimens of other dimensions, or on specimens which are prepared under different conditions may produce results which are not comparable. Other factors, such as the speed of testing and the conditioning of the specimens can influence the results. Consequently when comparative data are required, these factors must be carefully controlled and recorded.

NOTE 2: The stress strain response in shear is very non-linear. This test method measures the modulus within a low strain region and is not applicable to higher strains.

2 Normative references.

The following standards contain provisions which, through references in this test, constitute provisions of this draft. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to use the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291: 1977, *Plastics - Standard atmospheres for conditioning and testing.*

ISO 293: 1986, *Plastics - Compression moulding test specimens of thermoplastic materials.*

4 Definitions

For the purposes of this draft the following definitions apply.

4.1 Plate deflection w. The distance over which the loading points move relative to the support points.

4.2 Modulus of elasticity in shear; in-plane shear modulus, G_{12}. It is expressed in Gigapascals GPa and for isotropic materials it equals the shear moduli in other directions. It is measured as the ratio of shear stress to shear strain between plate deflections of 0.1h and 0.3h (where h = plate thickness).

4.3 Speed of testing. The rate of movement of the loading points and support points expressed in mm/min.

4.4 Span, S. The distance between the two support points, S1, which is equal to the distance between the two loading points, S2 (see figure 2).

4.5 Diagonal, D. The distance between the diametrically opposed corners of the plate.

4.6 Widths, a, b. The lengths of the sides of the specimen.

4.7 Thickness, h. The mean thickness of the specimen.

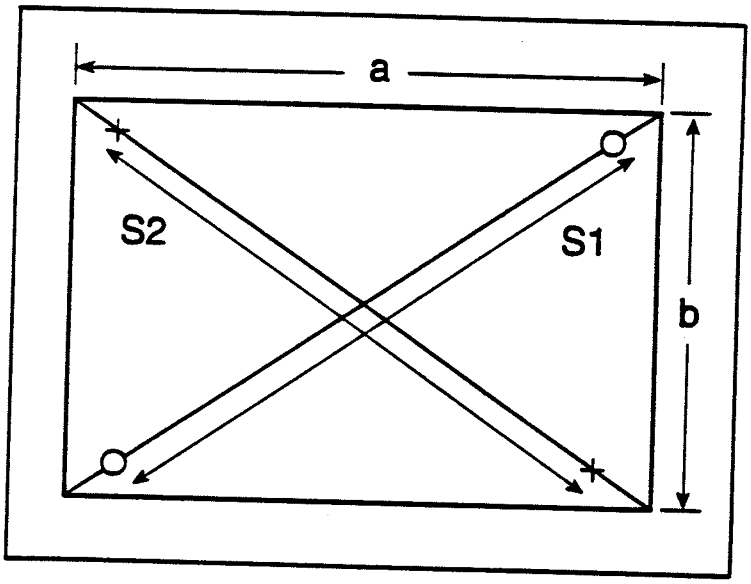


Figure 2. Showing position of loading and support points.
+ supporting point o loading point

5.2 Micrometers and gauges

5.2.1 Micrometer or equivalent, reading to less than or equal to 0.01 mm, for measuring the thickness h of the test specimen.

5.2.2 A vernier caliper or equivalent, accurate to within 0.1 mm, for determining the span and specimen widths and diagonals.

6 Test Specimens

6.1 Shape and dimensions

The test specimens shall be a square and flat plate of the material under test.

6.1.1 Standard plate specimen

Material	Specimen Width (a,b) (mm)	Span (S) (mm)	Thickness (h) (mm)
Reinforced-plastics (particle and discontinuous fibre)	150+/-1.5	201	3+/-0.5
Unidirectional and multi-directional composites	150+/-1.5	201	2+/-0.5

NOTE 5-For this standard in any one test the specimen thickness shall nowhere deviate by more than 5% from its mean value. The corresponding maximum deviation for width is 1%. The cross section shall be rectangular and without rounded edges.

6.1.2 Other test specimens

When the thickness of the plate is not within the standard range given in 6.1.1 the following limits shall apply;

The widths of the test specimen shall be as given below;

$$a = b \geq 35h$$

NOTE 6- Specimen thicknesses may only be reduced by machining from thicker materials when the material's structure is homogeneous throughout the thickness of the material.

8.2 Measure the widths a and b of the test specimens to the nearest 0.5mm at three positions along the specimen edge and calculate the mean value.

8.3 Measure the thickness of the specimen to the nearest 0.02 mm at the mid point of each edge, 25mm from the edge and calculate the mean value. Discard any specimen(s) with a thickness exceeding the tolerance of +/- 5 % of the mean value. Replace the specimen by another one, sampled by chance.

8.4 Adjust the span S so that the loading point and support spans are equal and;

$$S1 = S2 = 0.95\ D$$

Measure the spans S_1 and S_2 to the nearest 0.5mm, and calculate the mean S.

8.5 Measure the diagonals D1 and D2 of the plate to the nearest 0.5mm and calculate the mean value D.

8.6 Set the speed of testing according to clause 5.1.2.

8.7 Place the test specimen symmetrically on the two supports.

8.8 Deflect the loading points to a maximum of 0.5h.

8.9 Record the deflection and corresponding force during the test using, if practicable, an automatic recording system that yields a complete load /deflection curve for this operation.

9 Calculation and expression of results

9.1 Shear Modulus

For the calculation of the shear modulus measure the deflections w1 and w2 and the corresponding loads P1 and P2.(See Figure 4).

9.2 Statistical parameters

Calculate the arithmetic mean of the test results and, if required, the standard deviation and the 95 % confidence interval of the mean value using the procedure given in ISO2602.

9.3 Significant figures

Calculate the modulus to three significant figures.

10 Precision.

The materials tested and precision data obtained in the validation of this test method are listed in Appendix 1.

11 Test report

The test report shall include the following information:

a) a reference to this draft;

b) complete identification of the material tested, including type, source, manufacturer's code number, form and previous history, where these are known;

c) the date of measurement;

d) the shape and dimensions of the test specimens;

e) the method of preparing the specimens; the test conditions and conditioning procedures, if applicable;

f) the number of specimens tested;

g) the length of span used;

h) the speed of testing;

i) the accuracy grading of the test machine (see ISO 5893);

j) the individual test results, if required;

k) the mean values of the individual results;

l) the standard deviations and the 95 % confidence intervals of the mean values, if required.