

National Physical Laboratory

Results of the UK Interlaboratory Strength Test Exercise

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SUMMARY

The UK interlaboratory strength test exercise on ceramic materials is described, and the results from the participating laboratories are discussed. Large differences in uniaxial flexural test results between laboratories can be ascribed to differences in test jig geometry and mode of operation, in particular to an inability to align the specimen, to distribute the applied load evenly and to minimise friction effects. Determination of the static fatigue parameter " n " by undertaking tests at a variety of loading rates gave a wide spread of results. A simple three-ball support disc test was also evaluated, and gave fairly reproducible results although analysis of the data proved to be complicated by the existence of a bimodal distribution of strengths of the alumina ceramic used. Some guidelines for improved practice are established.

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Foreword

This report was originally compiled in December 1990 following a closed UK round robin under the auspices of BSI Committee RPM/13, but was prepared with restricted distribution to participants and the Committee only. In the light of the non-publication of the results of the 1987 VAMAS round robin on subcritical crack growth it has been decided, with no objections from the original participants, to place the results into the public domain without alteration. In doing so, the authors recognise that there have been similar round robins since, including those organised by ESIS (European Structural Integrity Society) and in Japan. However, the authors feel that the results remain valid and provide an illustration of some of the issues involved in obtaining consistent results when testing ceramic materials.

The main text of the report is unaltered from that prepared in 1990, but some additional footnotes have been included to explain some of the developments since, based in part on this work.

1. INTRODUCTION

The UK Strength Test Round-Robin was instigated in 1987 to evaluate the UK's ability to undertake flexural strength testing of ceramic materials in parallel with the concurrent VAMAS international exercise organized by the National Institute of Standards and Technology (NIST, formerly National Bureau of Standards). The total number of test-pieces available for the international project was too small to permit all interested UK laboratories to take part, so the UK project was organised by NPL.

The NIST exercise proposed the evaluation of the reproducibility of determining the static fatigue parameter “ n ” from so-called “dynamic fatigue” tests on flexure test bars with and without flaws produced by Vickers indentations, using three different specimen loading rates. It can be shown mathematically that, assuming a subcritical crack growth law of the type:

$$v = A K_c^n$$

where v is the crack velocity, K_c is a critical stress intensity factor, and A and n are constants, there is a relationship between the mean strengths, σ , under different loading rates, $\dot{\sigma}$, of the form:

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{\dot{\sigma}_1}{\dot{\sigma}_2} \right)^{1/(n+1)}$$

Thus, plotting $\log \sigma$ vs. $\log \dot{\sigma}$ should yield a straight line of slope $1/(n + 1)$, allowing easy evaluation of n . The proposed use of Vickers indentations in the NIST exercise was intended to reduce the scatter of strengths such that a more reliable value of “ n ” might be produced.

Flexural testing of ceramic materials is an essential part of many commercial activities, from materials development, through specifications, to quality control. At that time (1987) there were no standards in use in Western Europe that were suitable for use on advanced technical ceramics (¹). In the UK, different companies involved in testing ceramics used different procedures. The UK Round-Robin therefore set out to examine several factors:

- (1) The effect of test jig type on the strength results produced on a single batch of test-pieces by the participating laboratories.
- (2) The ability to determine the static fatigue parameter “ n ” on the test material by testing at high and low loading rates.
- (3) The ability to undertake simple biaxial strength tests using a method similar to that in ASTM F394 for testing electronic substrate materials.

This report summarises the results of the round-robin and draws some conclusions concerning the state of the art of testing.

¹ Since this time, there have been concerted efforts in the US, UK, Japan, France and Germany to produce standards suitable for such materials. European standards supersede the first British, French and German standards. A programme is also underway in ISO and new standards are appearing.

2. TEST MATERIAL

The test material was “Hilox 961”, a fine-grained brown-coloured 96% alumina from Morgan Matroc Ltd, Stourport-on-Severn, Worcs., UK (Figure 1), and was prepared by dry pressing in the form of 5 x 3 x 50 mm test bars and discs 18.6 mm diameter and 3 mm thick according to drawing nos. 81764 and 81763 respectively (Figure 2). The test surfaces were lapped flat and parallel as stated on the drawings, the final lapping stage being 6 µm grit size. The maximum level of chipping along the edges of the test bars was 0.2 mm, but no edge chamfering of the bars was undertaken ⁽²⁾. Bars and discs were individually wrapped for transportation.

Since the disc specimens could not be made any thinner than 3 mm by the automated machinery at Morgan Matroc, but at this thickness were considered to be unacceptably thick for the disc test, they were subjected to a second grinding procedure at NPL, using a vertical spindle grinding machine and a cup grinding wheel. One face only was ground, and was marked as such. The same procedure was used for each set machined.

NPL undertook randomisation of the batches of test-pieces, and then supplied them to the participants together with instructions.

3. TEST INSTRUCTIONS

Participants were supplied with 50 bars and 30 discs. With the bars, they were asked to perform 30 tests with their currently used flexure testing jig to obtain a reliable mean strength. Then, two batches of 10 bars were to be tested, one at a much faster rate and the other at a much slower rate, for determination of the “n”-value. Either three or four-point bending could be used over a 36-40 mm span, and some participants received sufficient specimens to undertake tests in both geometries.

The test discs were to be broken all at the same rate, with the marked face in compression, and the original Morgan Matroc face in tension. The test jig could either be one loaned by NPL or be manufactured by the participant. Specimen dimensions were to be measured, and the strengths calculated.

For each set of conditions a results sheet was to be filled in and returned to NPL for correlation of results. Since the test equipment in the bar tests was not standard, participants were asked to supply some details of their jig’s design and function. Specifically, they were asked if the loading and support rods were able to rotate freely or were fixed, whether the two support rods were in fixed positions parallel to each other, whether the loading rod (or rods in four-point bending) was able to align itself relative to the support rods, and whether there was any device for centralising the specimens on the loading axis.

The test jig supplied by NPL for the biaxial tests was of the three-ball support type (Figure 3) as described in ASTM F394 (1), but unlike this standard, the loading was via a ball with a prescribed flat area, rather than via a guided flat-ended punch. A similar design of jig, but without the prescribed flat area has been described by Godfrey (2).

⁴ In the light of the developments since, which have shown the importance of chamfering, this operation should have been made in order to reduce risks of fracture from variable edge damage flaws.

4. BASELINE NPL TESTS

The test series was first undertaken by NPL using a precision-made test jig in which both the loading and support rollers were housed in bearings allowing rotation. The two rollers in each pair were in fixed relative positions, but the pair of loading rollers (or single roller in three-point bending) could self-align on the test specimen. This situation is similar to that specified for accurately machined size B test specimens in MIL-STD-1942 (3). The roller diameters were 10 mm, this relatively large size being necessary to minimise bending of the roller, which was supported only at its ends.

The test series on the bar specimens was performed over as wide a range of loading rates as could be accommodated on various test machines. The main strength distribution based on 30 tests was determined using a cross-head speed of 0.5 mm/min, to obtain failure in about 20–30 s. Two faster and two slower rates were used on 10 tests each to obtain baseline static fatigue data. The tests at the faster two rates were performed using a machine-independent quartz load cell, because previous experience had shown that the Instron machine used had a slow-response load cell which would be inaccurate for the loading rate applied. The results are shown in Table 1 for both three-point and four-point bending over a 40 mm span (20 mm loading span in 4-point bending). In both types of loading, the strength distributions at the medium loading rate were very similar (Figures 4 and 5), and the mean strengths were separated by a small amount in accordance with usual volume/area effects on strength of brittle materials. The relationship between the three-point and four-point strengths can be shown to be (4):

$$\frac{\sigma_3}{\sigma_4} = \left\{ \frac{V_4 \Sigma(V_4)}{V_3 \Sigma(V_3)} \right\}^{1/m}$$

where V_3 and V_4 are the volumes of the test-pieces in three and four-point bending (equal in this case), $\Sigma(V)$ is the stress-volume integral (4), and m is the Weibull modulus which characterises the width of the distribution of strengths. A similar equation can be written in terms of the area of the specimen, A . For four-point bending with 40 mm support span and 20 mm loading span, $V_3 = V_4$, $\Sigma(V_3) = \frac{1}{2}(m+1)^2$ and $\Sigma(V_4) = (m+2)/4(m+1)^2$, so that the above equation reduces to:

$$\frac{\sigma_3}{\sigma_4} = \{(m+2)/2\}^{1/m}$$

For the strength data in Table 1, this can be solved numerically to give $m = 18.7$. Independent Weibull plots of the two test series give $m_3 = 15.7$ and $m_4 = 11.4$ after least squares fitting the mean-ranked data points to straight lines. However, if the values of the mean strengths are considered to be in error (either high or low) from the true value by one standard error (standard deviation divided by square root of the number of tests), then the above equation would predict upper and lower limits to m of 28 and 13.3 respectively. In view of this, one can argue that there is broad agreement, but the result is an interesting demonstration of the risks associated with size extrapolation, even with populations of 30 specimens. (Note: for case of area, or indeed edge length, the formulation leads to the same answer as for the volume case.)

Inspection of the Weibull distributions shows that they have a single mode, and that there are no signs of occasional very weak specimens which might result from the infrequent presence of large defects of a different type.

Inspection of the positions of failure within the central span of the four-point loaded specimens suggested some small degree of eccentricity of loading (Figure 6). After readjusting the position of the central loading roller pair, a small improvement was achieved, and the mean strength was increased slightly. The recalculated value of m using this four-point strength figure (Jig 1A in Table 1) is 13.6. After completion of the round-robin, a second type of jig, Jig 2, was made with minimal eccentricity and incorporating individually alignable 4.5 mm diameter rollers capable of free rolling on flat surfaces. A third batch of specimens was tested with this jig under the same conditions and yielded very similar mean strength results and sensibly no positional bias of fracture. The value of m was found to be 14.0. In neither of these two cases does the modified value of m significantly affect the three-point to four-point bend strength ratios.

The effect of loading rate shown in Table 1 was determined using Jig 1, and is plotted in Figure 7. This plot covers five orders of magnitude in loading rate, and thus readily satisfies one of the criteria found in the VAMAS Round-Robin that at least three orders of magnitude must be employed. The behaviours in the two loading geometries are almost identical. However, the results present something of an anomaly, because those at the very slowest rate appear to be similar to those at the next fastest, suggesting that there is the possibility of a fatigue limit involved with this material, or that the results at the slowest rate have been affected by the machine used, which was of different type to that employed for the remainder of the tests. Otherwise, the mean fracture stress increases monotonically with increasing loading rate. Least-squares fitting to all the data yields a line, the slope of which gives a static fatigue parameter " n " of about 25. However, the lines drawn in Figure 7 exclude the slowest results, and give values of " n " of about 20.

The disc tests were performed at a single cross-head speed of 0.25 mm/min, giving failure in 10 – 20 s. Plotting the results as a Weibull distribution (Figure 8) shows a very marked bimodal plot and a wide spread. Fitting the data with a single line seems highly inappropriate. After the bulk of the results had been returned from participants with reports of variable numbers of weak test-pieces in the lower parts of the respective distributions, a second batch of discs was tested by NPL after first UV dye testing them. This time, rather than 8 results in the clear low-strength "tail", there were only two, but nevertheless, the shape was similar. This subject is discussed further later in the report.

Based on the bar test results, the material was assumed fit for the purpose of a round-robin, and was distributed to the participants.

5 PARTICIPANTS' RESULTS

The participating laboratories are listed in Table 2. In order to preserve anonymity, the listed order in this Table is different to the numerical order given for describing the results. Table 3 lists details of the test procedures and jigs reported by the participants. Table 4 provides a summary of all the participants' results on the bar test-pieces, and Table 5 summarises the results of the disc tests.

5.1 Discussion of bar tests results

Considering first the intermediate loading rate tests, there is clearly a significant range of mean values, between 281 and 321 MN m⁻² for three-point bending and between 248 and 306 MN m⁻² for four-point bending (Figure 9). The NPL results were the lowest in both cases. It should be noted that the results for Laboratory 4 in four-point bending were obtained at a cross-head speed a factor of 6 faster than that used by the remaining laboratories, and the test jig had a 16.7 mm inner span compared with 20 mm for the others. Their results should therefore be corrected downwards on both counts by a total of perhaps 6 – 10 MN m⁻² to compensate for the different conditions. The humidity levels reported for the test environments were all closely grouped between 35 and 50%. For the purposes of the following discussion, these small differences have been ignored.

Inspection of the Weibull plots of each set of results shows that the distributions obtained could be fitted by simple two-parameter Weibull relationships in all cases, with “m” values in the range 10 to 16. Unlike the disc tests (see below), it is therefore likely that the differences seen between participants are due to differences in the test jig arrangement.

In particular, it is known that overestimates of strength can arise from friction between the specimen and the support or loading rollers (in four-point bending), to a level of 10% or more if the rollers are not free to roll or rotate to accommodate changes in specimen chord length with bending. Different degrees of freedom for the support points to roll or slide could account for the range of results seen. Three laboratories reported that their rollers could rotate *when not loaded*, but were constrained in position in such a way that free rotation under specimen load was very doubtful. Laboratories 4 and 6 reported high four-point bend results, while Laboratory 3 reported a rather higher three-point bend result than the NPL result. In contrast, the NPL jigs had rollers that were unrestricted in their ability to rotate, and this correlates with apparent strength levels substantially below those of the other Laboratories.

A second factor that applies particularly to four-point bending is non-symmetrical loading. If the loading rollers are not symmetrically positioned relative to the support rollers, or if the loading rollers are constrained to move such that they cannot naturally equalise their loads, there is a tendency for the maximum surface stress in the test-piece under one loading roller to be higher than under the other, and to be greater than that anticipated by centralised loading. Baratta *et al.* (5) show that for quarter-point bending over a 40 mm span, an eccentricity of as little as 0.4 mm (1% of span) gives a stress error of 2% provided that the load remains distributed equally to the two loading rollers. If the system is restrained rigidly such that loading is unequal, the error is increased four-fold. In practice both of these factors would lead to lower strengths being recorded. From this conclusion is derived the requirement in MIL-STD-1942 to try to keep the system below a maximum 0.2 mm eccentricity. The tendency of the participants' jigs to produce eccentric loading can be assessed from the distribution of failure positions. If there is a predominance for failure to one side, this could result from eccentric loading. Figure 10 shows the mean failure position plotted against mean fracture stress. Increasing eccentricity of fracture location from the central mean position, corresponding to random position of failure over 50 test-pieces, appears to correlate with decreasing strength for the group of results employing fixed rollers. The preferred failure positions are more clearly seen in the histograms plotted in Figure 11. Laboratories 1 and 7 have distinct preferred loading to one side, and Laboratory 7 admitted the deliberate removal of shims normally used to improve equalisation of load on the two loading rollers. Thus in Figure 9, the relatively low strength levels recorded in four-point

bending by these two participants can be explained by an eccentricity error. The NPL results (Figure 6) have relatively low eccentricity, and are similar for the two types of jig used. In contrast, for three-point bending, ref (5) shows that 5% eccentricity of loading results in only 1% error in stress, so this type of misalignment cannot be used to explain the scatter of the three-point bend test results.

Other errors potentially involved and discussed by (5) are small for the test geometry used, of the order 1%, for geometry in question. The remaining potential error not discussed by (5) is that of laterally off-centre loading such that an element of specimen twisting is involved, resulting in non-uniform stress across the width of the specimen. No attempt has been made to estimate the magnitude of this potential error, but it is easy to see qualitatively that introduction of a torsional element of loading will have a similar effect to eccentric loading in increasing the stress preferentially on one side of the test-piece, and reducing it on the other. The net effect in testing ceramics would therefore be a reduction in the apparent mean strength, and a tendency to fail consistently on one side. The magnitude of this error is difficult to estimate on the individual participants' jigs without careful assessment with strain-gauged specimens. Some of the participants reported a distinct preference for edge failures, either to one side or the other. Inspection of the returned test-pieces did not always provide the same answers, due to the difficulties of interpreting the fractographic evidence. Free alignment of the loading rollers by rotation in a plane perpendicular to the specimen length should minimise the risk of misalignment of load in this plane. On the NPL jigs, this alignment could occur readily, and resulted in only about 25% edge failures, despite not applying an edge chamfer to the test-pieces. Figure 12 shows some examples of fracture surfaces of test bars.

The effect of loading rate for three and four-point bending is shown in Figures 13 and 14 respectively. In these figures, no attempt has been made to obtain the actual stressing rate of the test-pieces. The abscissa is plotted in terms of nominal cross-head speed, which, for the purposes of determining the value of " n " from a logarithmic plot is perfectly adequate (³). The values of " n " obtained are shown as the final column in Table 4, and show a considerable scatter.

Some obvious points arise. Laboratory 5 recorded low results in both three and four-point bending at their fastest speed, which is symptomatic of the load-recording device not responding sufficiently fast to allow the true load at fracture to be determined with accuracy. In four-point bending, Laboratories 7 and 10 showed a similar effect. (Note that at a given cross-head displacement rate, four-point bend specimens load at twice the rate of three-point bend specimens tested over the same span, making it more likely for the effect to be seen in four-point bending.) Discounting these results, individually calculated values of " n " varied between 15 and 114, with the majority between 15 and 30. These values are based on the mean strength results, and do not take into account the possible unreliability due to the small numbers of test-pieces. Compared with the international VAMAS round-robin, the results are quite promising, and are in a generally much narrower band. The essential difference is that the in the present tests, they are based on batches of 10 specimens, whereas in the VAMAS project, they were based on only five, which increases the uncertainty considerably. However,

³ Although it would be more correct to use actual measured stressing rate, the ratio of such rates in any given machine would be the same as the ratio of cross-head speeds to within a negligible error. Use of a nominal cross-head speed is thus adequate for the present purpose.

the levels of uncertainty remain significant, as shown for example by the large difference between three and four-point bend results from Laboratory 7, even discounting the fast four-point bend result.

Several factors could influence the results of a test of this type. Firstly, the machine response in terms of the rate of stressing of a test-piece may not be exactly linear with the chosen gearing, especially at low machine loads where the response may be distinctly non-linear. To check on this difficulty, it would be necessary to measure the actual stressing rate from load/time curves, a procedure not requested by the round-robin instructions. Secondly, there may be changes in the humidity level between batches. Ideally, humidity levels should be constant. Thirdly, probabilities of significant errors arise in selecting small numbers of test-pieces. In the present work, the estimated standard errors of the mean strengths are about $\pm 10 \text{ MN m}^{-2}$ for a batch of ten specimens, and this is significant in terms of the resulting “*n*” value when compared with the expected change in fracture strength that changes in test rate should cause. Next, there are instrumental recording errors, especially at high rates. In order to make this error insignificant, it is necessary to limit the cross-head displacement rate to a value such that the machine response time is perhaps less than 1% of the time to fracture. Finally, the range of rates of loading must be large to maximise the changes in mean strength relative to the possible errors in the mean values. Most laboratories used three rates separated by factors of 10, which typically covers the range limits of sensible operation of test machines. This may not be adequate, yet not all laboratories have access to equipment that will even cover this range, especially low cross-head speeds on the one hand, and fast and accurate data capture on the other.

Regarding any recommendations from this work that might be made for the generation of new standards for testing, a compromise will have to be reached in terms of cost of sophisticated equipment and number of tests made on the one hand, and the demand for accuracy of result on the other. The present material appears to have an “*n*”-value of around 20 in air, which is quite low. An accuracy of perhaps ± 5 , or 25%, is probably adequate to rank materials for susceptibility to subcritical crack growth, in which case, the conditions employed in this round-robin, i.e. 10 specimens over three orders of magnitude of loading rate, should be adequate for this purpose. For higher values of “*n*”, the situation becomes more critical, because the differences in mean strength become smaller, and the proportionate potential error resulting from small numbers of tests increases. Thus when $n = 100$, a 25% error in n would require at least 30 test-pieces at each rate for a range of three orders of magnitude of rate. If $n \gg 100$, then the errors become such that for typical values of m , n can never be measured with any sensible accuracy. For all intents on purposes, the test is useless, but on the other hand, such a material would not suffer badly from delayed failure effects. Overall, it is clear that some care will be required in the wording of the scope and meaning of an standardised test procedure (⁴).

5.2 Discussion of disc test results

The disc test results are summarised in Table 5. For this test, all the test jigs were of the same geometry, so variations between laboratories should have been an absolute minimum, limited only by load calibration and dimension measurement errors. Despite this, the mean strength

⁴ A European provisional standard, ENV 843-3, has since been published, detailing the test procedure and its limitations, and a similar method and a similar document has been published as ASTM C1368.

varies between 257 and 316 MN m⁻² (Figure 15), and the standard deviations were much larger than in the bar tests, between 51 and 83 MN m⁻² on batches of 30 specimens. The origin of this becomes clear on examination of Weibull distributions which show that different participants found different percentages of particularly weak test-pieces in their batch. Under such circumstances, even the “mean” strength, let alone the standard deviation or Weibull modulus, is not particularly meaningful. It is necessary strictly to consider statistical fitting using a bimodal Weibull function, but in view of the wide range of percentages of clearly weak test-pieces between laboratories, this was felt not worthwhile. The randomising of specimens clearly did not result in identical populations. Instead, all the results were combined into a single Weibull plot (Figure 16) which clearly shows the existence of two separate characteristic populations.

Using the assumption that it was valid to separate the weak specimens from the strong ones, the mean strengths and slopes of the top parts of the distributions were calculated with the principal objective of comparing the relative results of the individual participants. The results are also shown in Table 5. The mean values are now much more closely grouped, and the standard deviations are much smaller, as might be expected. The Weibull modulus still has considerable scatter, varying from 6.3 for Laboratory 1 (assuming that the calculations have been appropriately corrected from the values originally supplied) to 17.2 for NPL2, but with most in the range 7.5-11.5. The low result from Laboratory 5 may have resulted from a load cell problem, highlighted in the bar test, whereby at fast loading rates the response of the system is too slow to give an accurate recording of fracture load. However, the spread of mean results is quite small and could be accounted for since they are mostly within one standard deviation of the overall mean figure.

To examine this further, some batches were subjected to fractographic analysis. Because of the small size of the test-pieces, this proved difficult and mostly inconclusive. It was clear that the basic material was not particularly homogeneous on the scale of the test-piece, and the fractures did not show clear origins or mirror regions. Examination optically at up to 64x magnification using a “macroscope” revealed the presence of voids, multiple cracking, and red spots (origin unknown and not present in the bar test-pieces). Some specimens were examined by scanning electron microscopy, and this technique showed little evidence to explain why some of the test-pieces were substantially weaker than others. The principal type of defect observed was pores of such geometry that suggested that they originated from incomplete packing of spray-dried granules in pressing the discs. No large grains or regions of obvious inclusion or contamination were found. Some examples are shown in Figure 17. The presence of pre-existing cracks from the machine-lapping process also cannot be ruled out. The second batch tested by NPL, NPL2, was UV dye tested before fracturing in the test jig. Only one large radial crack in one specimen was observed. No other pre-existing defects of significant size could be found, but as it transpired, there were only two weak specimens in this batch. Nonetheless, the second weak one could not be pre-determined. Thus one can only conclude that the material was basically unsuitable for fractography, and was certainly different in disc form from that in bar form, despite assurances from the manufacturer that there should have been little difference between the batches of this material, which is made in tonnage quantities.

This illustrates the problem of placing reliance on limited sampling of batches of nominally identical components. When two flaw populations exist, one less frequent than the other, the appearance of the strength distribution, and the statistical parameters calculated from it, can

be positively misleading. It appears that little reliance can be placed on them unless the numbers of test-pieces showing the less-frequent distribution are much larger than those discovered in this exercise.

One of the objectives was to gain experience with using this simple biaxial test. With the exception of those participants who possessed horizontal mechanical testing machines, no problems with using the test were reported. With the horizontal machines, ensuring that the loading ball flat area remained on contact with the test-piece during setting up proved the biggest problem. The re-working of the data to study the reproducibility of the stronger specimens shows that the participants all obtained closely spaced results, demonstrating that the disc test can be used reproducibly by any laboratory.

Analysis of the fracture pattern from the fragments shows that the 3-fold symmetry of the support balls plays a major role in determining the path of the growing crack. The defined flat area on the loading ball should provide uniform biaxial stress within the loaded area, but it appeared that the majority of fractures began within but close to the edge of the loaded area, with only a small proportion outside this area (Figure 18). The origins appeared to be in random initial positions relative to the support balls, as might be expected, but the subsequent fracture paths clearly tended to run in curves to avoid the support balls, and to exit at the edge of the test-piece near the mid-point between support points. This is clearly a result of the fact that along the line between a support ball and the loaded area the stress on the tensile surface is a maximum parallel to this line, while between the balls it tends to be maximum perpendicular to the radial direction. Most specimens broke into 2 or 3 major fragments only, but locally there were often multiple crack paths giving much loose debris on fracture surfaces. Occasionally, cracks would halt in one direction, and secondary cracks would appear running perpendicular to the primary crack. Often, fracture faces were very tortuous, suggesting several false starts in progressing to final fragmentation of the test-piece, and this contributed to the difficulties of identifying failure origins. It is suggested that the presence of porous regions in the material, corresponding to incomplete compaction of spray-dried particles, allows this behaviour by providing a network on the scale of 50-150 μm along which cracks can readily propagate.

6 CONCLUSIONS

A UK round-robin to evaluate strength test procedures in uniaxial and biaxial flexure has been carried out and the results evaluated. It is concluded that:

Existing test practice among major UK users as of 1987 varied widely, and generally did not conform to the standards currently under development. The reasons are principally associated with the design of test jigs, which did not have adequate degrees of centralisation and alignment, nor eliminated friction by allowing support and loading rollers to rotate under load.

2. Participants generally experienced no problems with undertaking a simple biaxial disc strength test using three-ball support based on ASTM F394, but loading with a ball with a defined flat area.

- 3 The test material, chosen on the basis of availability and cost, posed some problems of fractographic analysis because of the distribution of porosity. In the form of discs, it possessed a bimodal strength distribution, which made analysis of the results less certain than in the uniaxial flexure tests.
4. The typical errors involved in determining the delayed failure parameter “n” are large, especially when the range of loading rates is small. Some test machines produce a substantial error when the time to failure is less than 10 s, due to long response times of load cells and recording equipment. At least three, preferably more, orders of magnitude range in loading rates are required in order to obtain reliable figures. If a standard procedure is to be devised for this test, a compromise between accuracy of result and cost of equipment and testing will have to be made.

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Table 1 – NPL results summary

Test	Test rate, mm/min	No of tests	Mean strength, MN m ⁻²	Standard deviation, MN m ⁻²	Weibull modulus, <i>m</i>	Value of “ <i>n</i> ”
3-point: Jig 1	0.005	10	260	14.6	(14.6)	25 (20)
	0.05	10	256	20.1	(11.5)	
	0.5	30	281	19.2	15.7	
	5.0	10	330	33.6	(9.0)	
	50	10	351	11.1	(29.3)	
4-point: Jig 1	0.005	10	230	31.0	(6.7)	25 (20)
	0.05	10	231	20.3	(10.4)	
	0.5	30	248	22.7	11.4	
	5.0	10	294	30.4	(8.1)	
	50	10	314	36.5	(7.1)	
Jig 1A	0.5	30	262	20.7	13.6	
Jig 2	0.5	30	257	20.0	14.0	
Biaxial:						
NPL1	0.25	30	285	77.1	3.0	
NPL2	0.25	30	308	64.8	1.3	

Jig Jig with rollers aligned parallel in pairs and fixed in needle roller bearings. Loading rollers self-aligning relative to support rollers.

Jig 1A As Jig 1, but with the centralising of the loading rollers improved.

Jig 2 New design of jig with 5 mm rollers, independent movement.

Note: Weibull moduli figures in brackets refer to such small specimen numbers as to be subject to considerable error. Values of *n* in brackets refer to value calculated ignoring the slowest-speed test.

Table 2 – Participants

Company	Contact
British Aerospace, Sowerby Research	H Evans
Coors Ceramics Wales Ltd, Tonyrefail*	G Richards
DFVLR, Köln, Germany	G D Quinn**
Fairey Tecramics, Stone, Staffs	D Taylor
UKAEA Harwell Laboratory	R Piller
Lodge Ceramics Ltd, Rugby	B R Franklin
Morgan Materials Technology Ltd, Stourport	R Stannard
National Engineering Laboratory, East Kilbride	R Dower
Pilkington R and D, Lathom	B Proctor
Geo. Wade Ltd, Burslem, Stoke-on-Trent	C G Martin

* Now Vesuvius Zyalons Ltd, part of Cookson Group

** On leave from US Army Materials Testing Laboratory, Watertown, Mass., USA

Table 3 – Details of participants' flexure jigs

Lab. No	Jig span, mm		Rotation of support rods perp. spec	Rotation of support rods para. spec.	Loading rods constrained rel. to support	Centralising method for specimen
	major	minor				
1	40	20	No	No	Uncertain	No
2	38	–	No	No	Yes	No
3	39.5	–	Some	No	Yes	No
4	37.8	16.7	Yes	Yes	Yes	No
5	40.0	19.9	No	No	Yes	Using card
6	40	20	Some	Yes	No	Yes
7	38.5	18.5	No	No	Yes (3-pt) No* (4-pt)	Yes (3-pt) No (4-pt)
8	35.3	–	No	No	No	No
10	40	20	No	No	Yes	No
NPL1	40	20	Yes	No	No	Yes
NPL2	40	20	Yes	Yes	No	Yes

* Normally used with packing to improve alignment; removed for this study.

Table 4 – Participants’ results on bar tests

Lab	3 or 4 point loading	Results at:									Value of “n”
		Fast rate			Medium rate			Slow rate			
		Rate	σ	SD	Rate	σ	SD	Rate	σ	SD	
1	4	ns	278	20.2	0.05	264	22.8	ns	246	14.9	–
2	3	0.3	313	19.6	0.05	285	21.9	0.015	263	14.9	15
3	3	0.5	321	15.9	0.05	304	19.5	0.005	276	25.9	29
4	4	0.635	320	17.2	0.318	298	24.8	0.159	291	24.0	14
5	3	0.5	274	26.6	0.05	314	26.4	0.005	290	20.5	28*
	4	0.5	238	19.9	0.05	281	33.8	0.005	272	22.0	42*
6	4	0.5	342	30.2	0.05	306	24.6	0.01	303	23.3	32
7	3	0.2	334	25.9	0.05	313	17.6	0.005	276	19.3	18
	4	0.2	253	48.5	0.05	261	34.7	0.005	256	29.5	113*
8	3	0.5	335	26.0	0.05	321	30.7	0.5	335	14.3	90
10	3	0.5	312	24.5	0.05	291	22.9	0.005	274	20.3	34
	4	0.5	247	30.4	0.05	287	23.0	0.005	250	26.2	16*

Key: Rate = testing rate in terms of crosshead speed in mm/min
 σ = mean strength in MN m⁻²
SD = standard deviation about mean strength in MN m⁻²
ns = not specified
* = ignoring fastest rate

Table 5 – Summary of disc test results

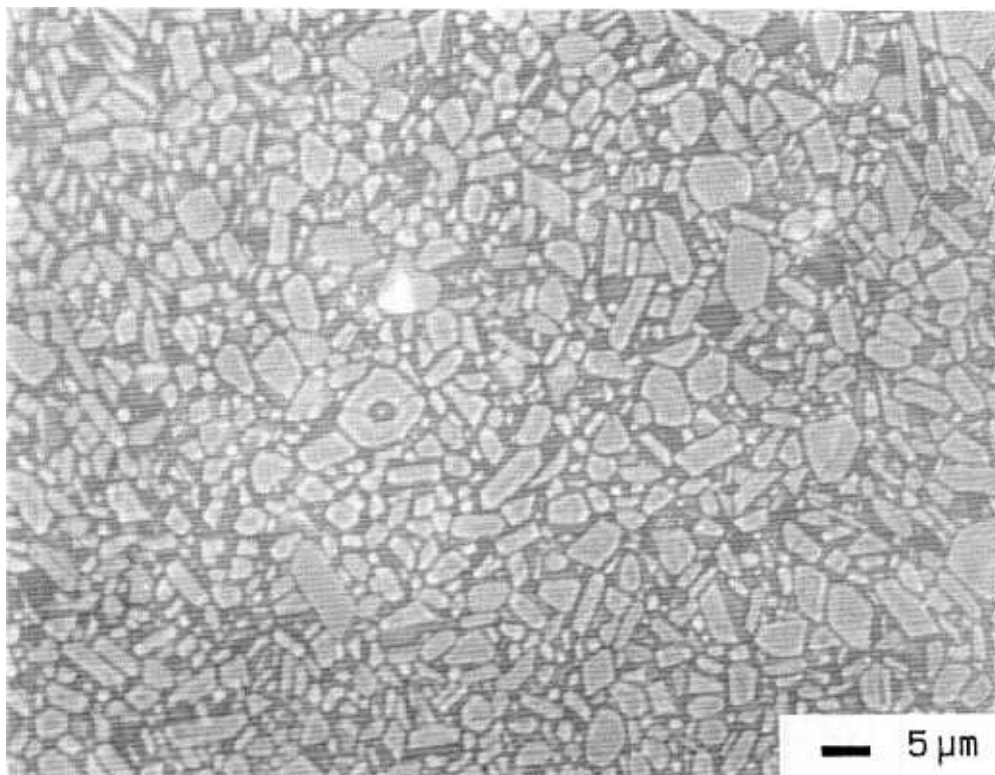
Lab. no.	Crosshead speed, mm/min	No. of tests	Strength, mean MN m ⁻²	Strength, st.dev., MN m ⁻²	Weibull modulus "m"	No. in tail*	Removing tail**		Modified Weibull modulus
							Mean MN m ⁻²	st.dev MN m ⁻²	
1	0.25	30	155***	48	2.3				
	0.25	29	(270)	(83.7)	(2.3)	6	309	32	6.3
2	0.5	30	257	65	3.6	8	292	25	7.2
3	0.5	30	316	51	4.9	4	330	26	10.3
4	ns	30	300	51	5.2	0	330	25	8.0
5	0.25	30	273	50	4.3	4	290	22	10.2
6	0.5	30	316	52	5.3	3	333	22	11.5
7	0.5	30	310	77	3.2	6	345	30	8.0
8	0.25	30	308	56	3.8	1	314	40	7.5
9	0.25	30	292	80	2.6	7	333	22	9.8
NPL1	0.25	30	285	77	3.0	10	329	21	8.1
NPL2	0.25	30	308	65	1.3	2****	326	16	17.2
	0.25	29	318	37	5.8	1			

Key: * Number of specimens obviously in a weak tail of the distribution.

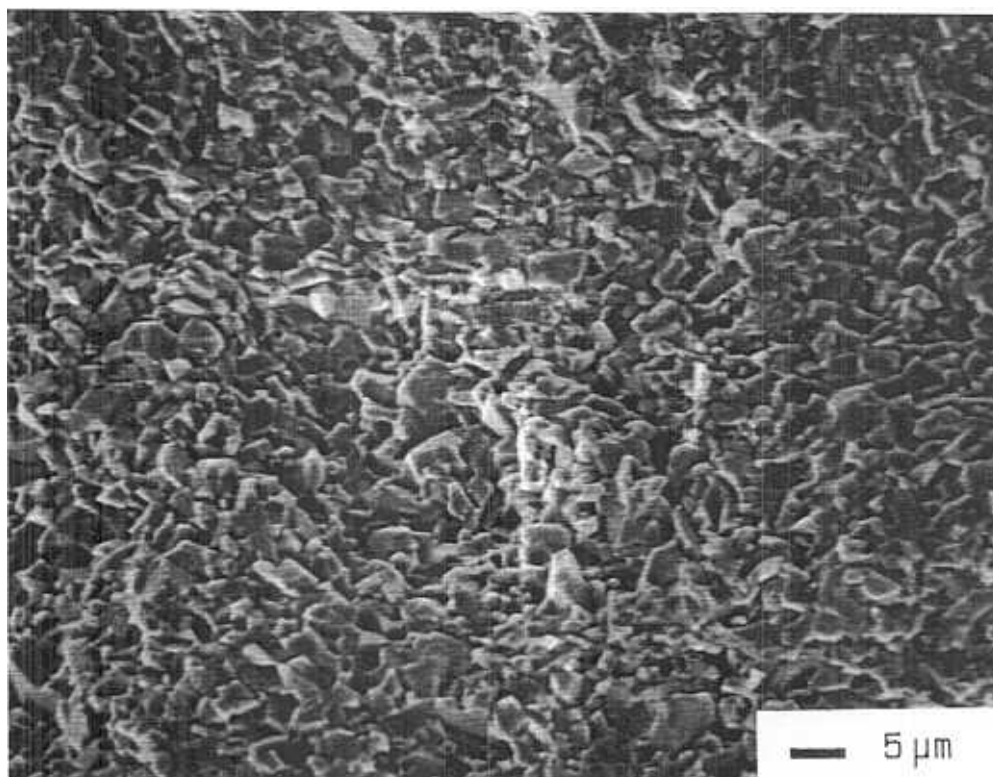
** Results calculated for the stronger part of the distribution only.

*** Participant asked to check calibration of testing machine since results approximately a factor of two low. The calibration was confirmed, but the error was found to be in the calculation of results, the corrected figures being shown in brackets in the following line.

**** One exceptionally weak specimen (18 MN m⁻² only) biasing the overall results. The results recalculated ignoring this specimen are shown on the following line.

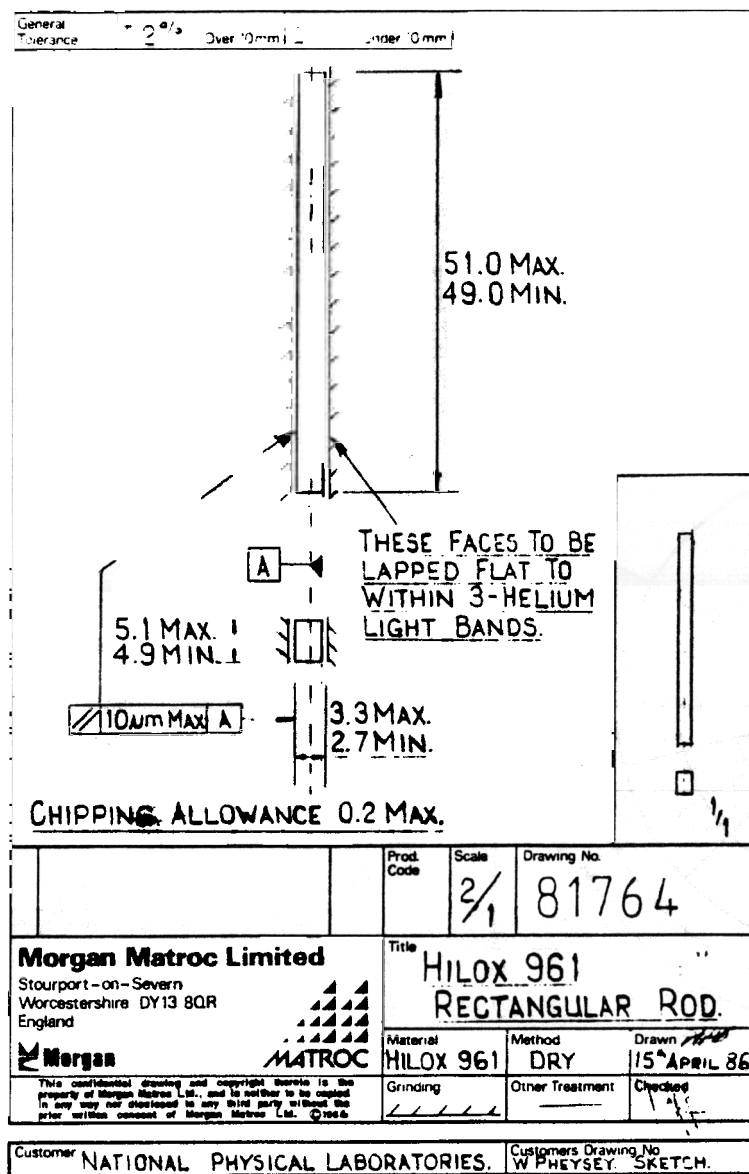


(a)

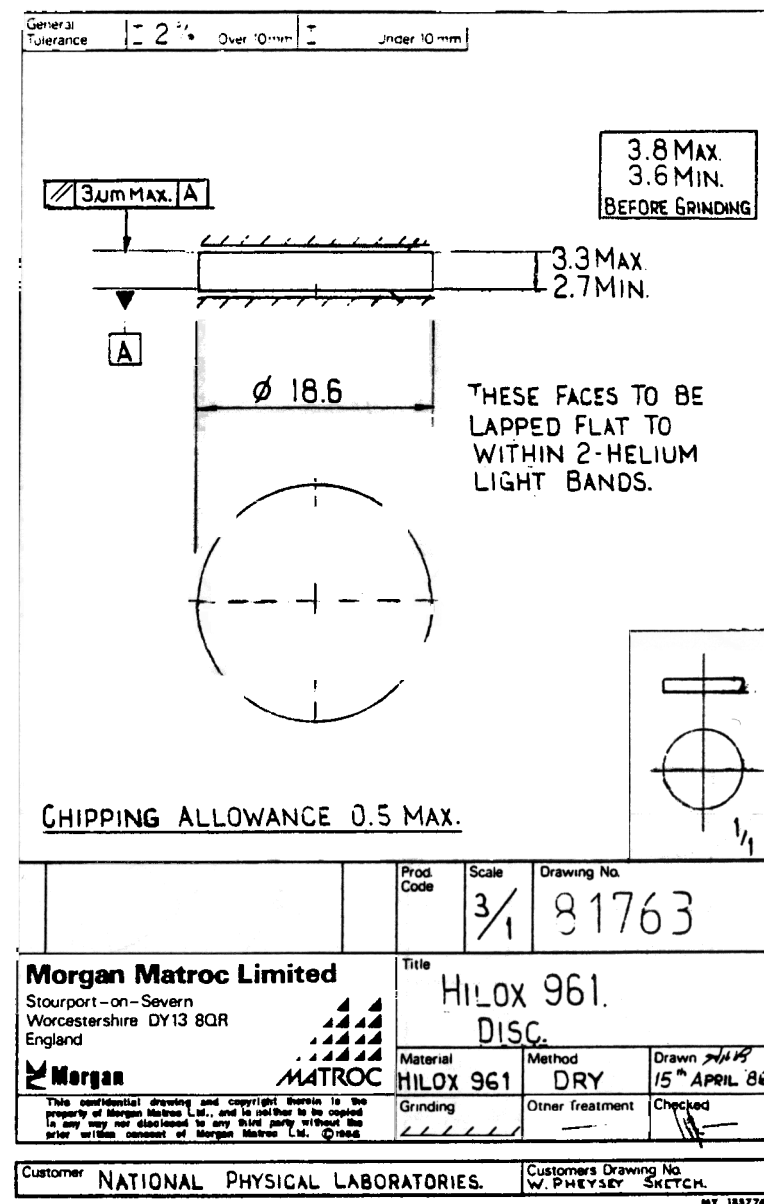


(b)

Figure 1 Microstructure of "Hilox 961" alumina ceramic (a) optical image of polished and thermally etched surface, (b) scanning electron micrograph of similar specimen.



(a)



(b)

Figure 2. Drawings of the supplied test-pieces, (a) bar test-piece, (b) disc test-piece.

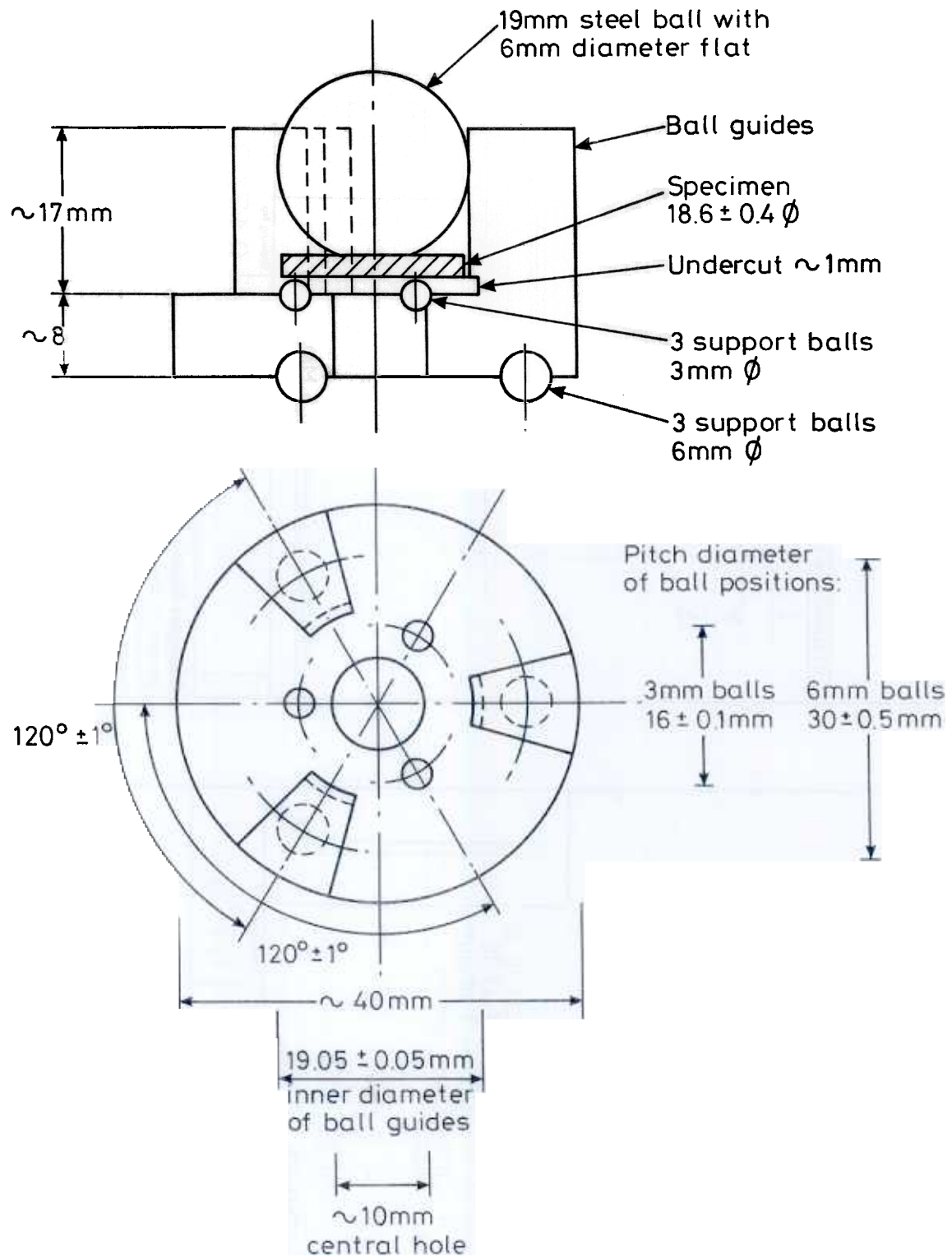


Figure 3. Schematic diagram of the three-ball support biaxial test jig.

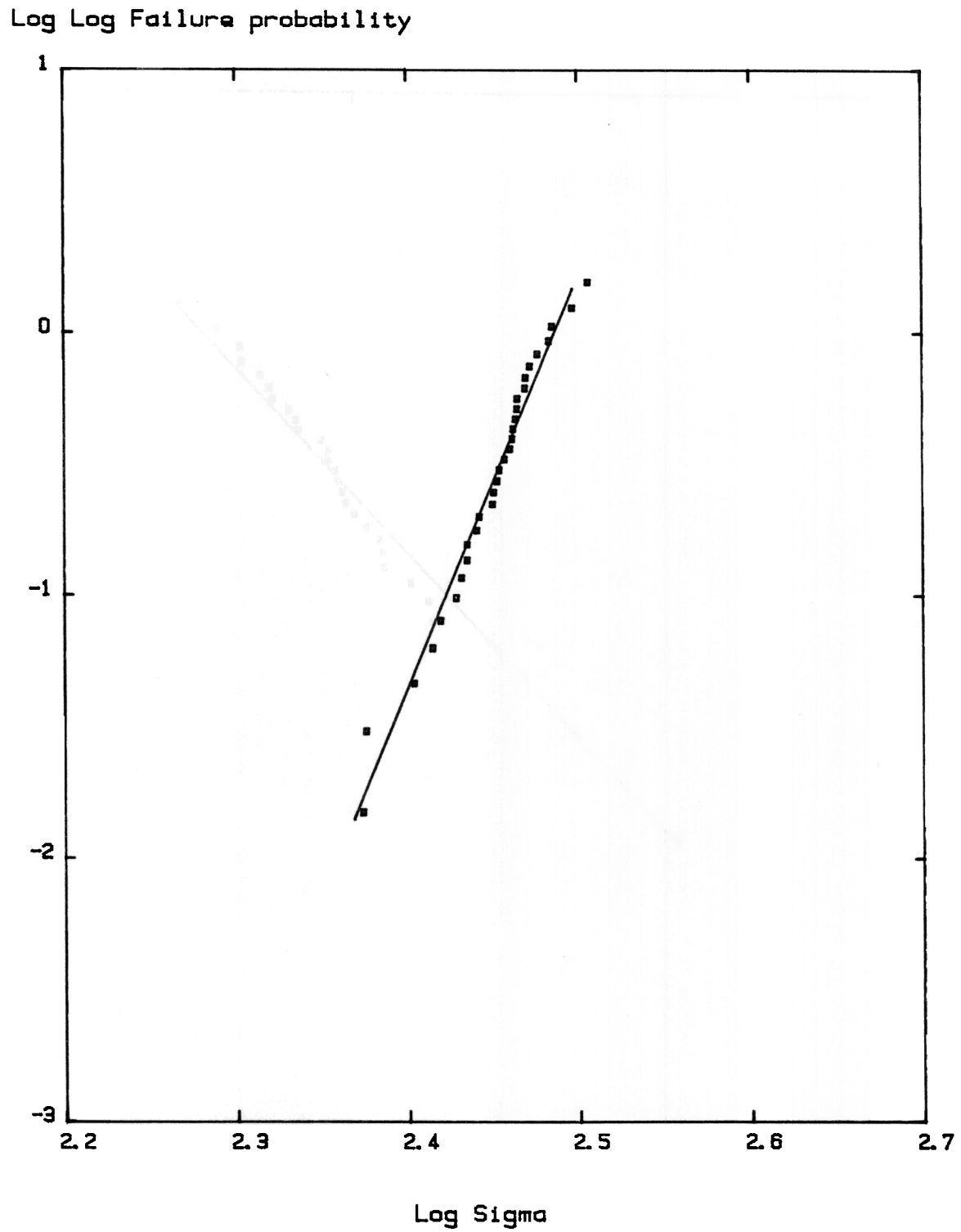


Figure 4. Weibull distribution of the NPL three-point bend test results.

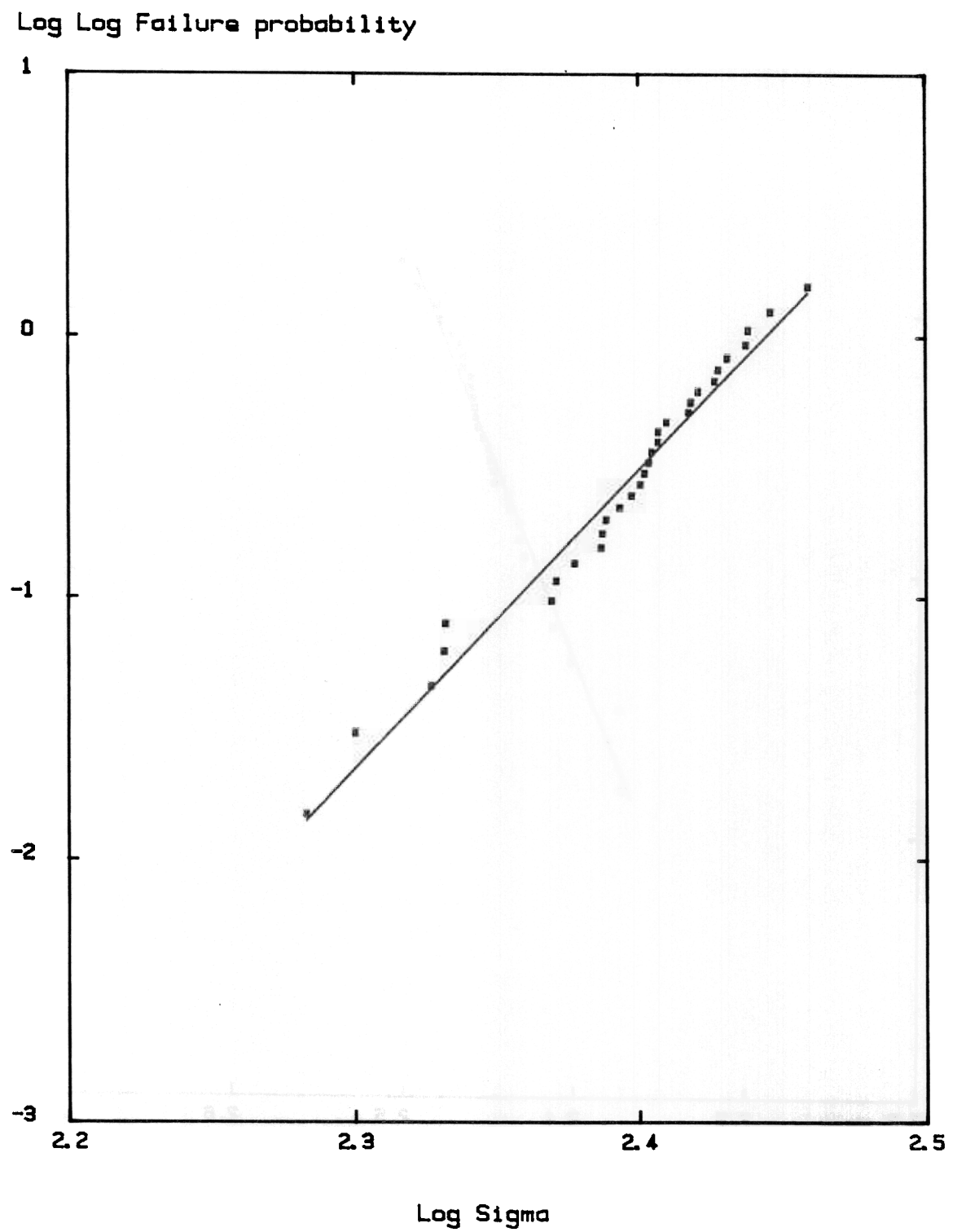


Figure 5. Weibull distribution of the NPL four-point bend test results.

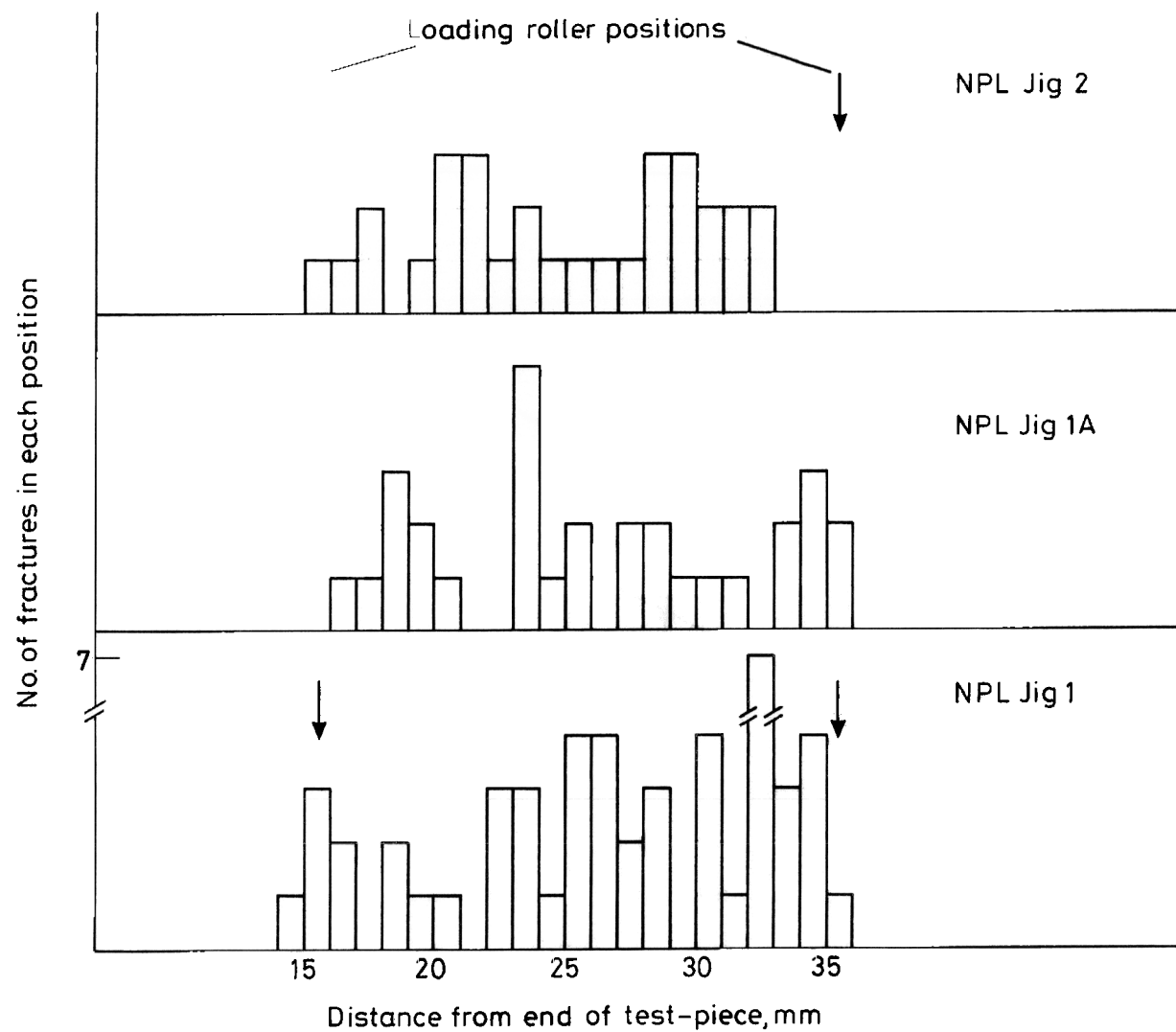


Figure 6. Position of failure in NPL four-point bend tests, (top) Jig 2, (centre) Jig 1A (adjusted for alignment), and (bottom) Jig 1.

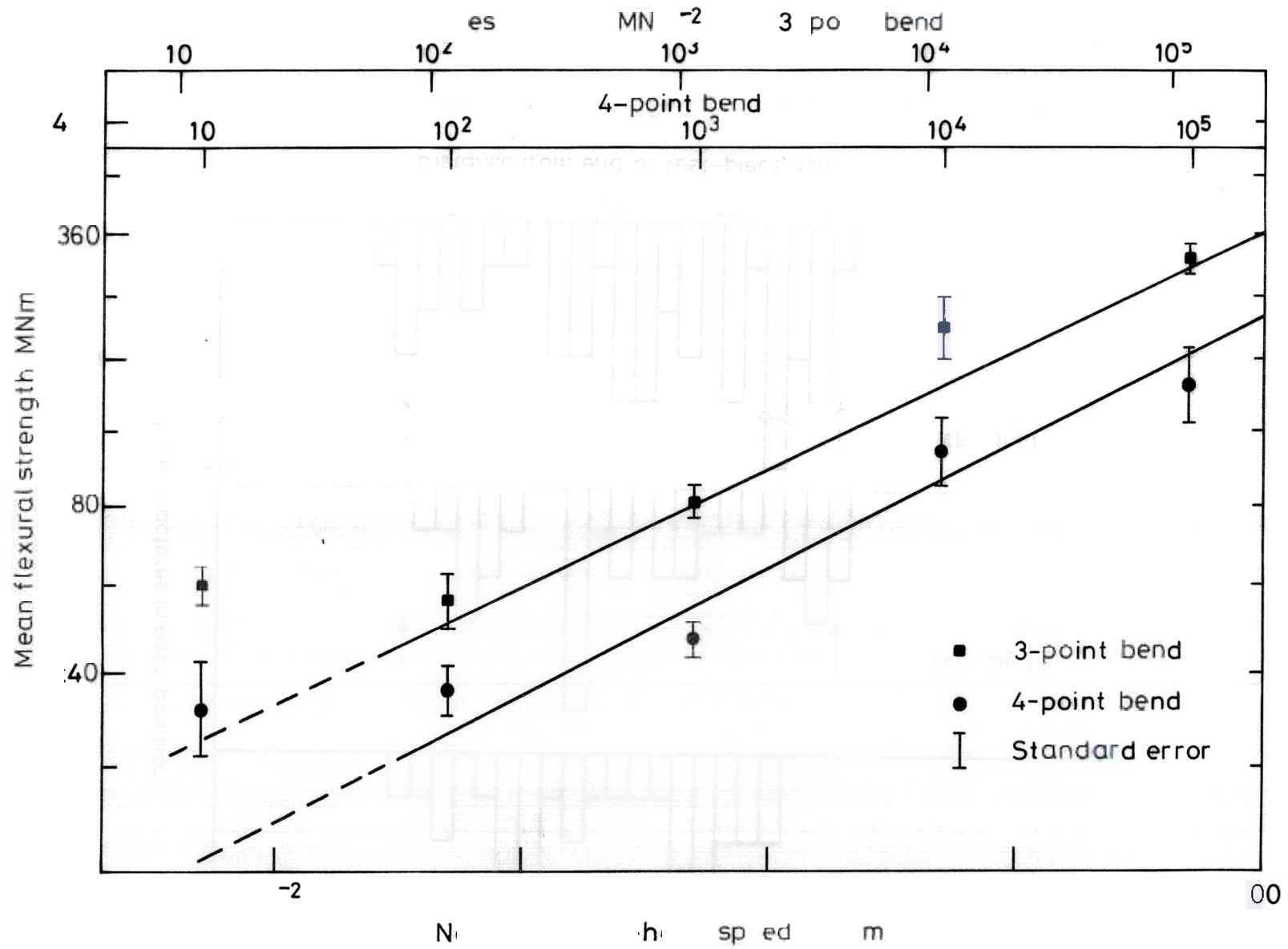


Figure Mean fracture str fo different testing tes lotted nominal ss-head speeds

Log Log Failure probability

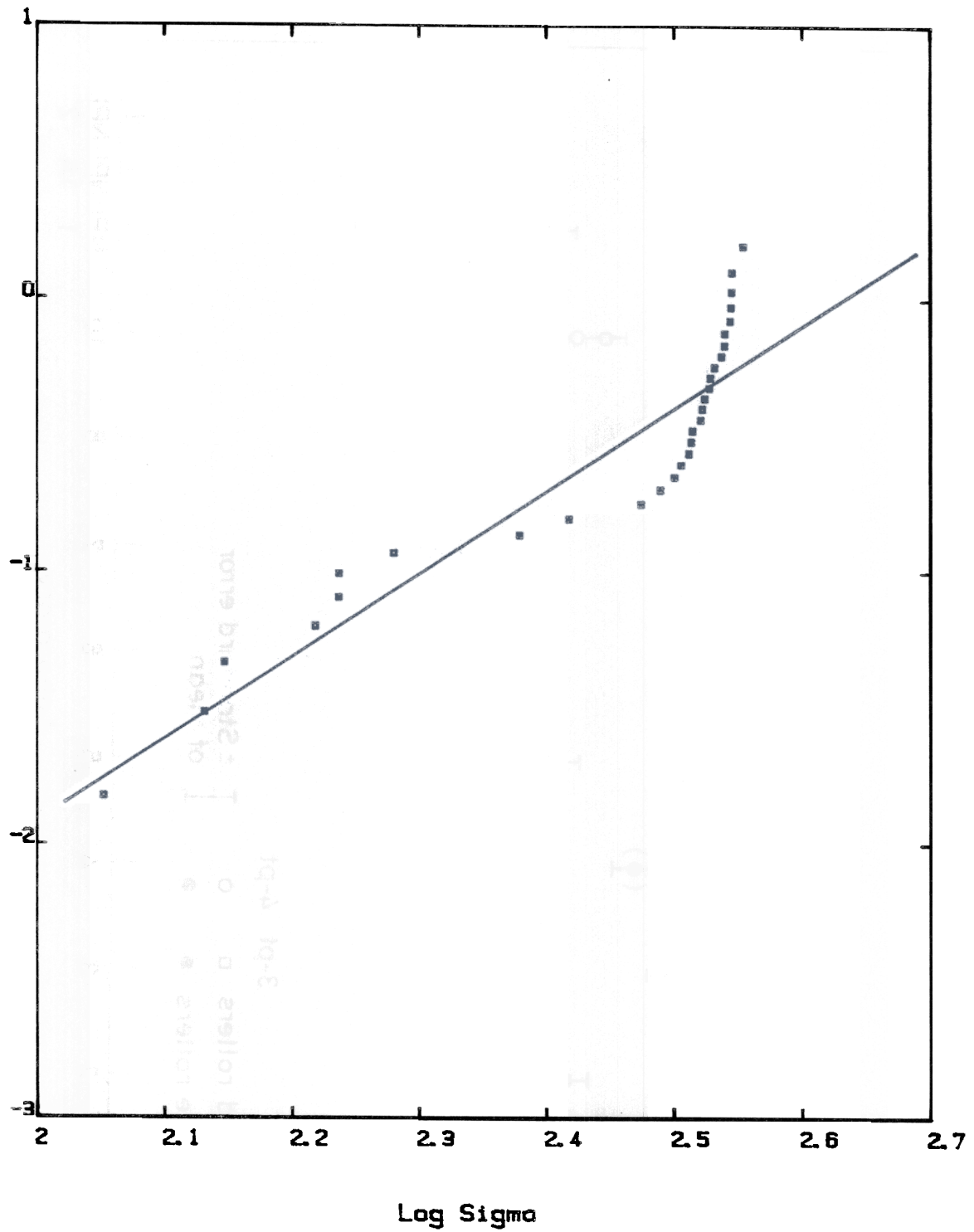


Figure 8. First batch of NPL results on the disc test, showing bimodal distribution of strengths.

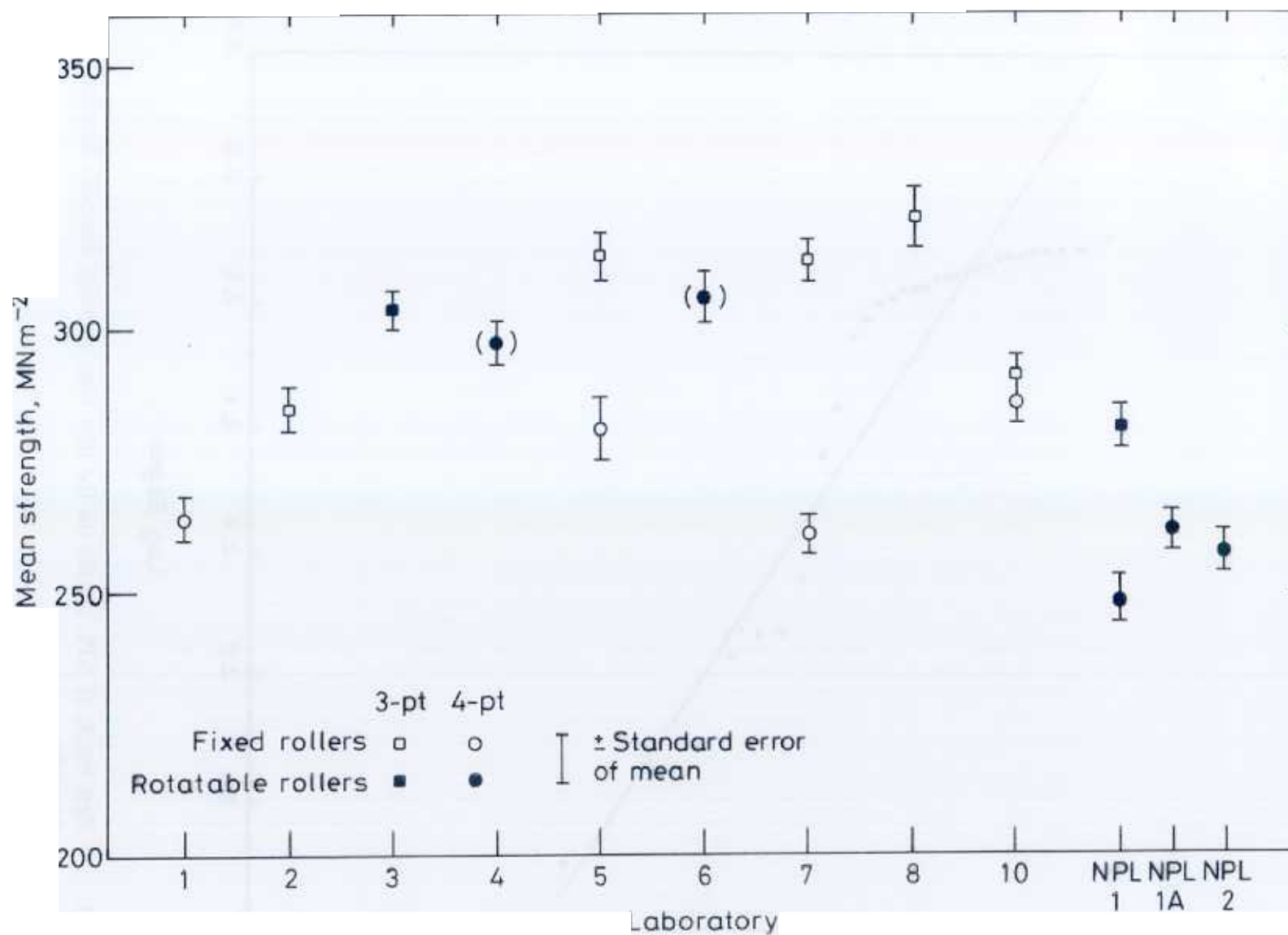


Figure 9. Mean strengths in three-point and/or four-point bending tests for each of the participants.

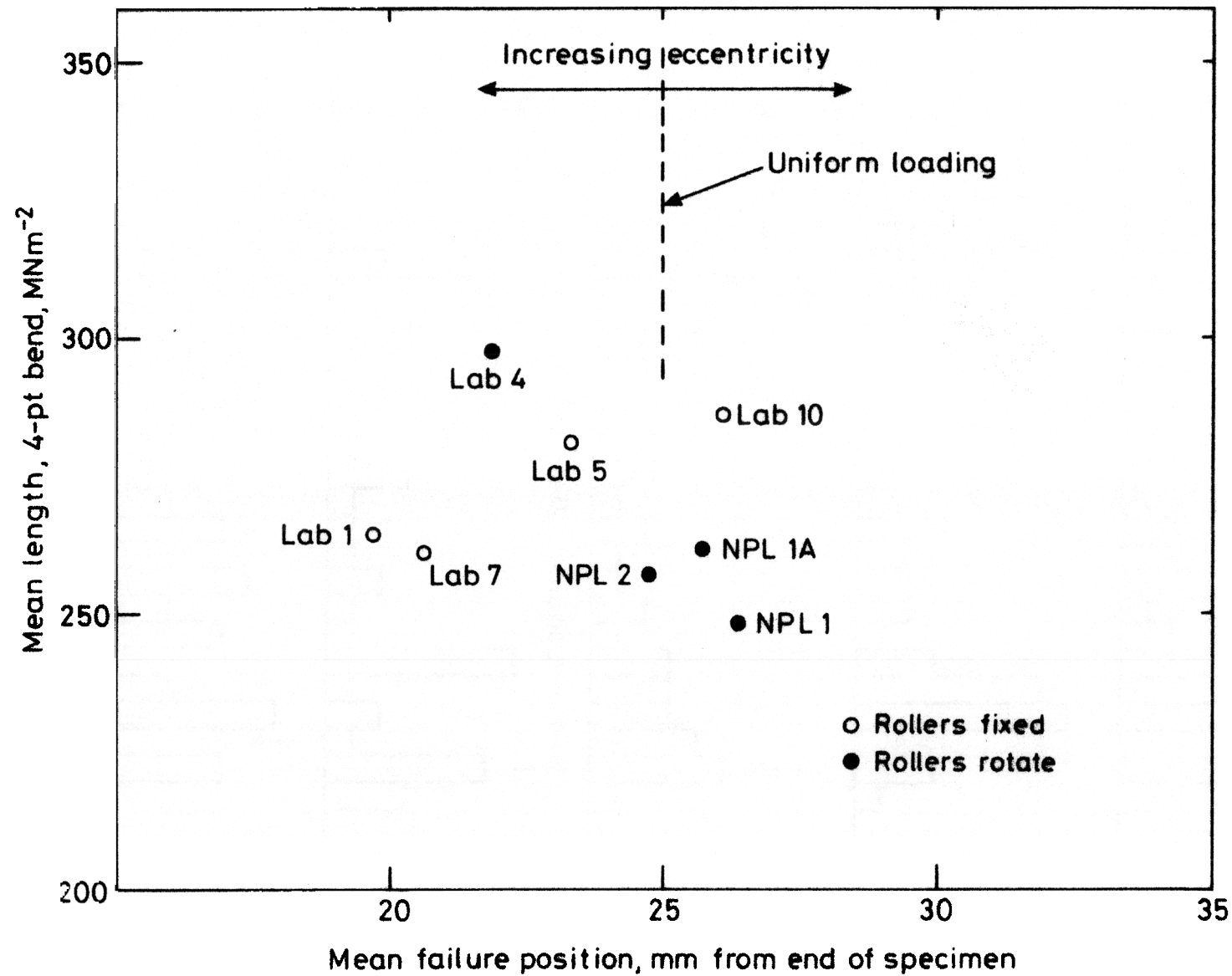


Figure 10. Mean failure position in four-point bending for each of the participants.

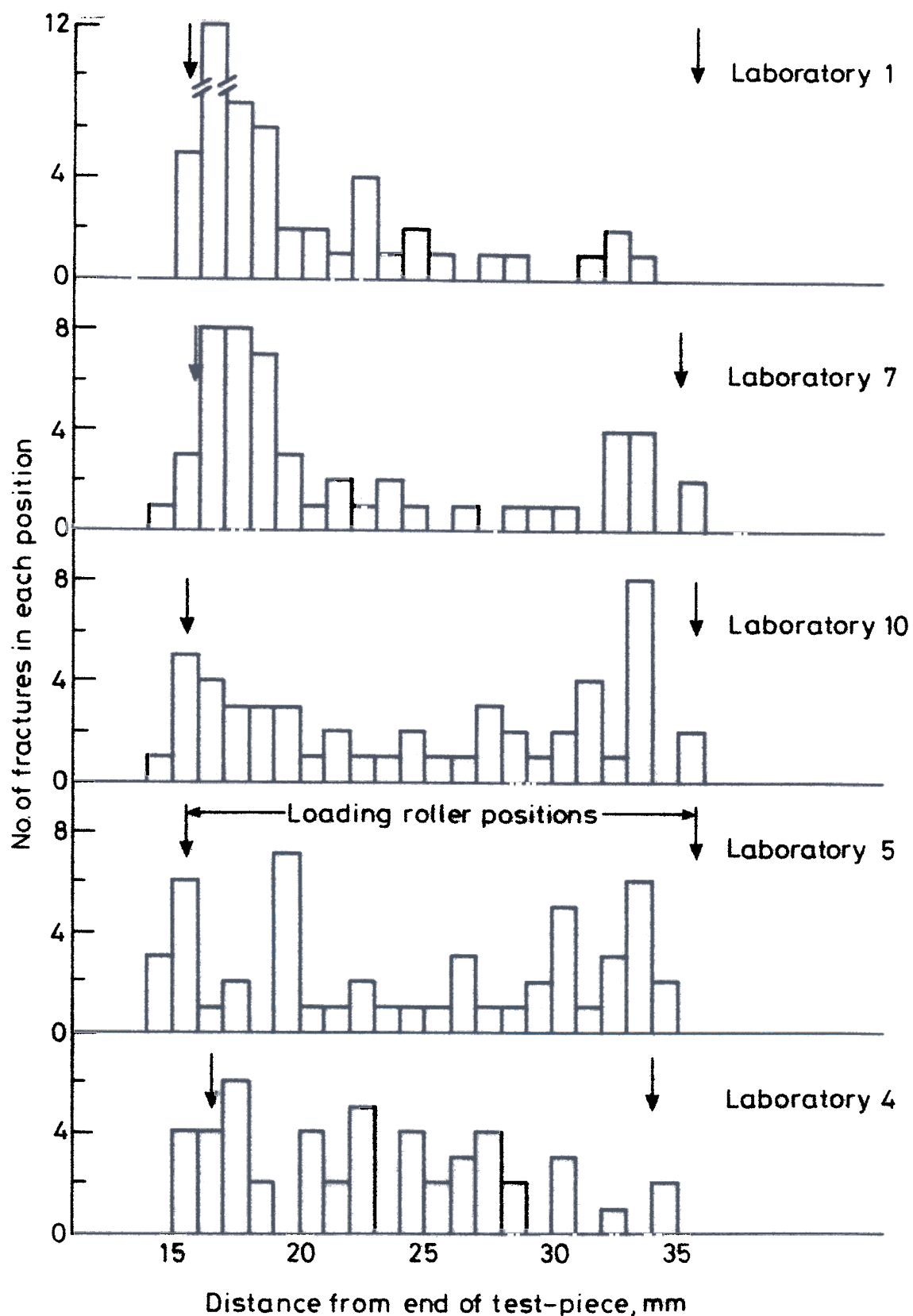


Figure 11 Distributions of failure positions for the four-point bending tests performed by the participants.

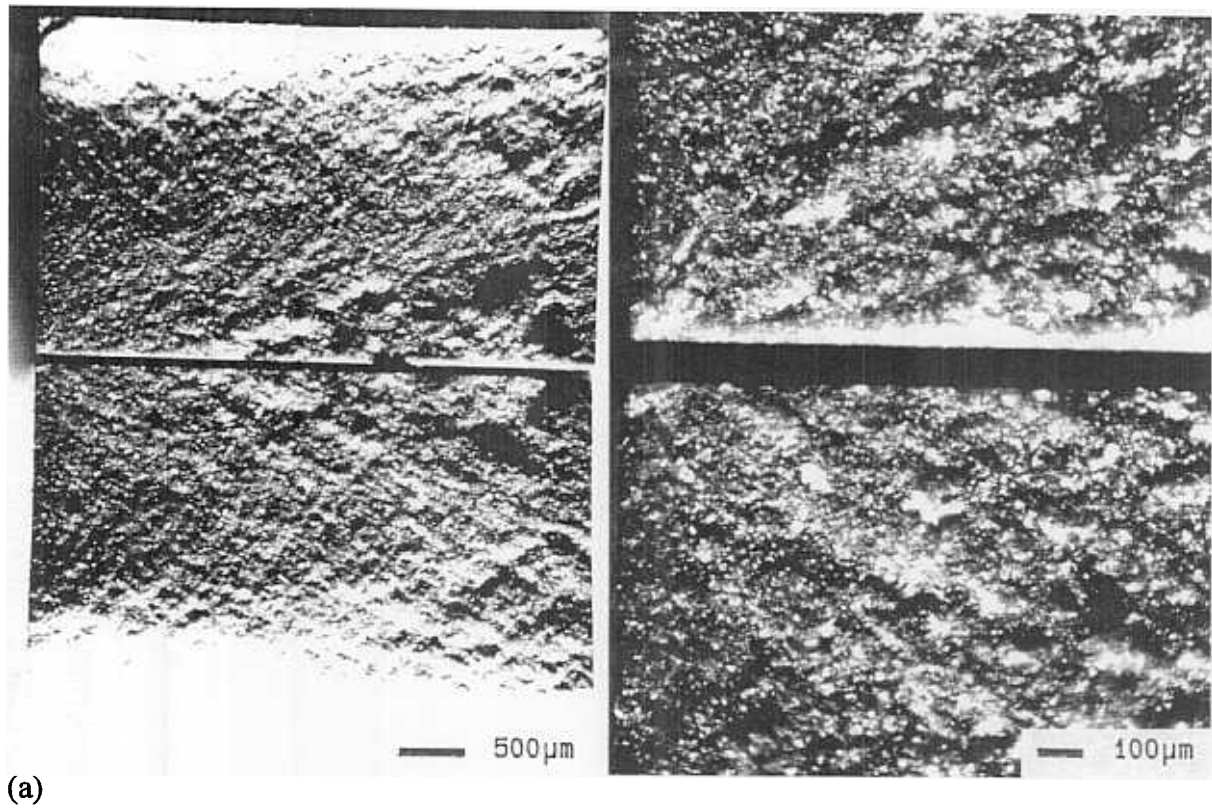
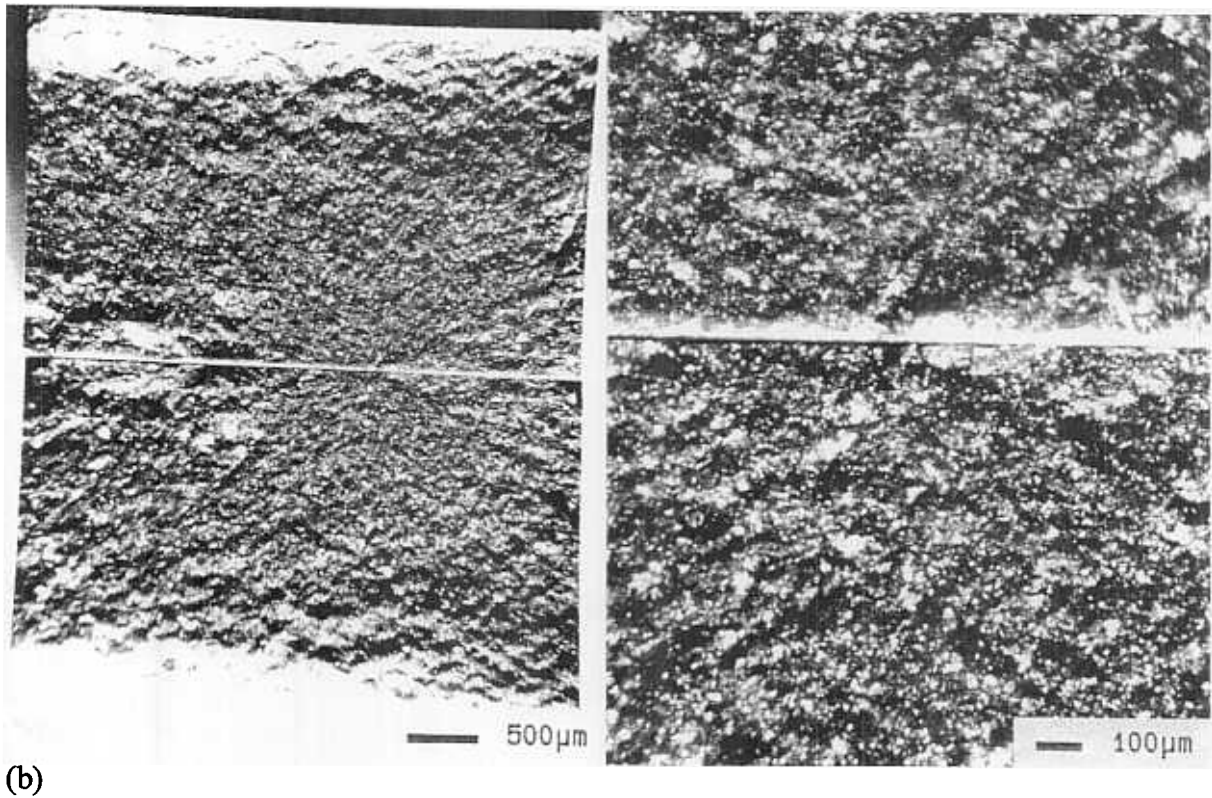
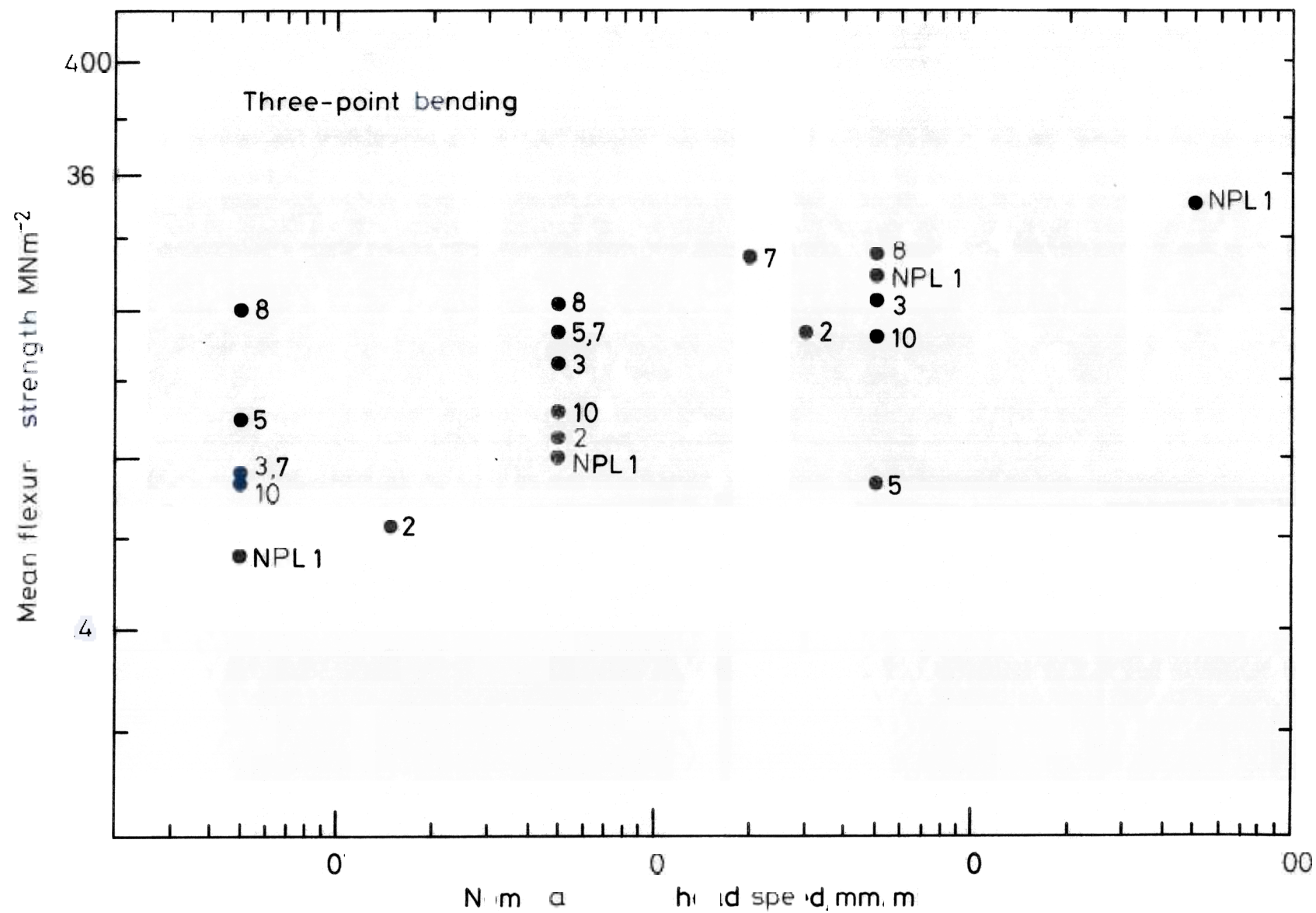


Figure 12. Matched halves of fractured test bars showing (top) typical bar edge failure, and (bottom) typical face centre failure. Note lack of clear mirror regions around fracture origins.



Mean fracture strength as function of nominal head speed for three-point bend test of all participants

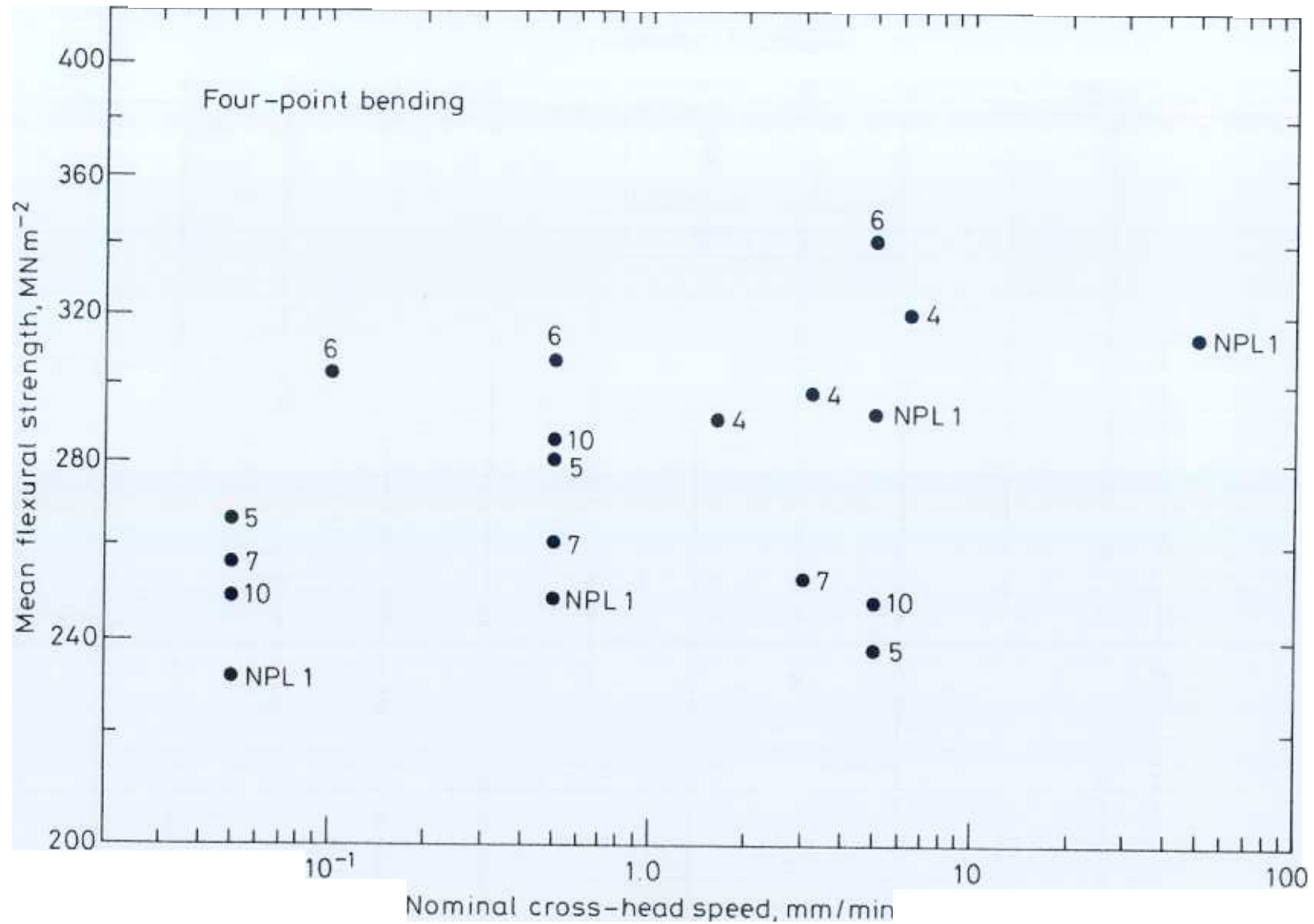


Figure 14. Mean fracture stress as a function of nominal cross-head speed for four-point bend tests of all participants.

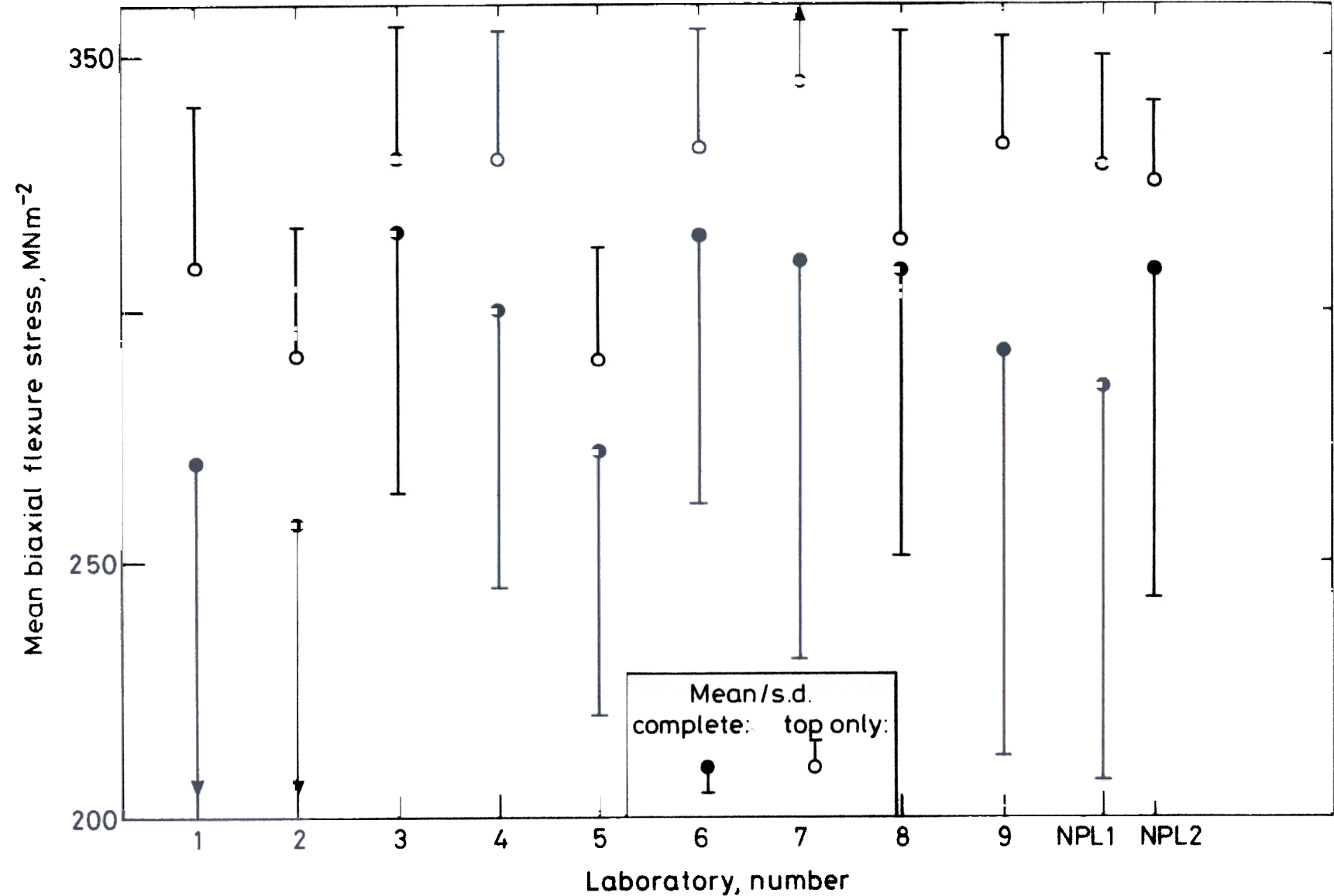


Figure 15. Mean strength results for all specimens, and for batches excluding clearly weak specimens, for each participant.

Log Log Failure probability

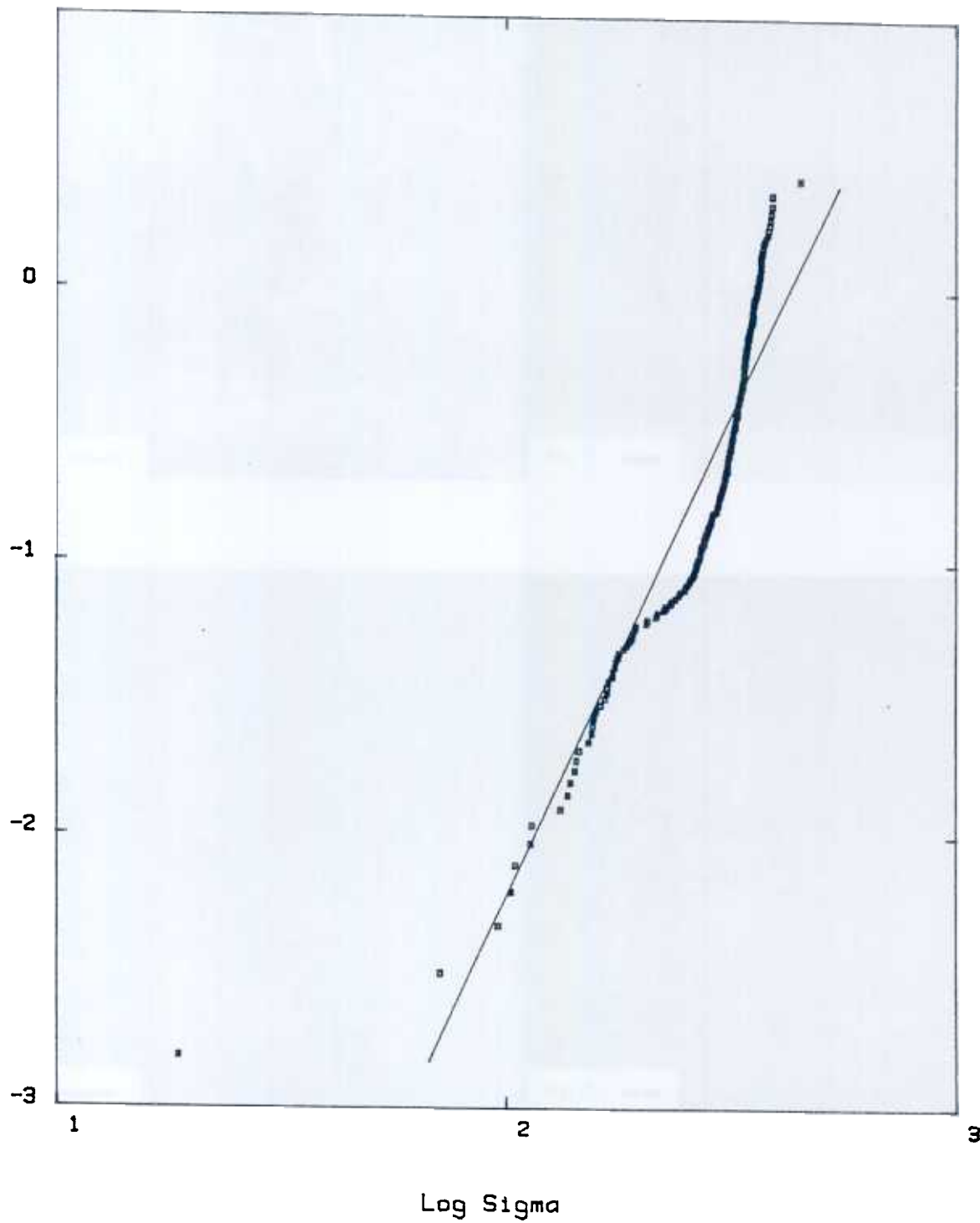


Figure 16. Combined distribution of strength results for all participants from the disc test, showing a bimodal distribution.

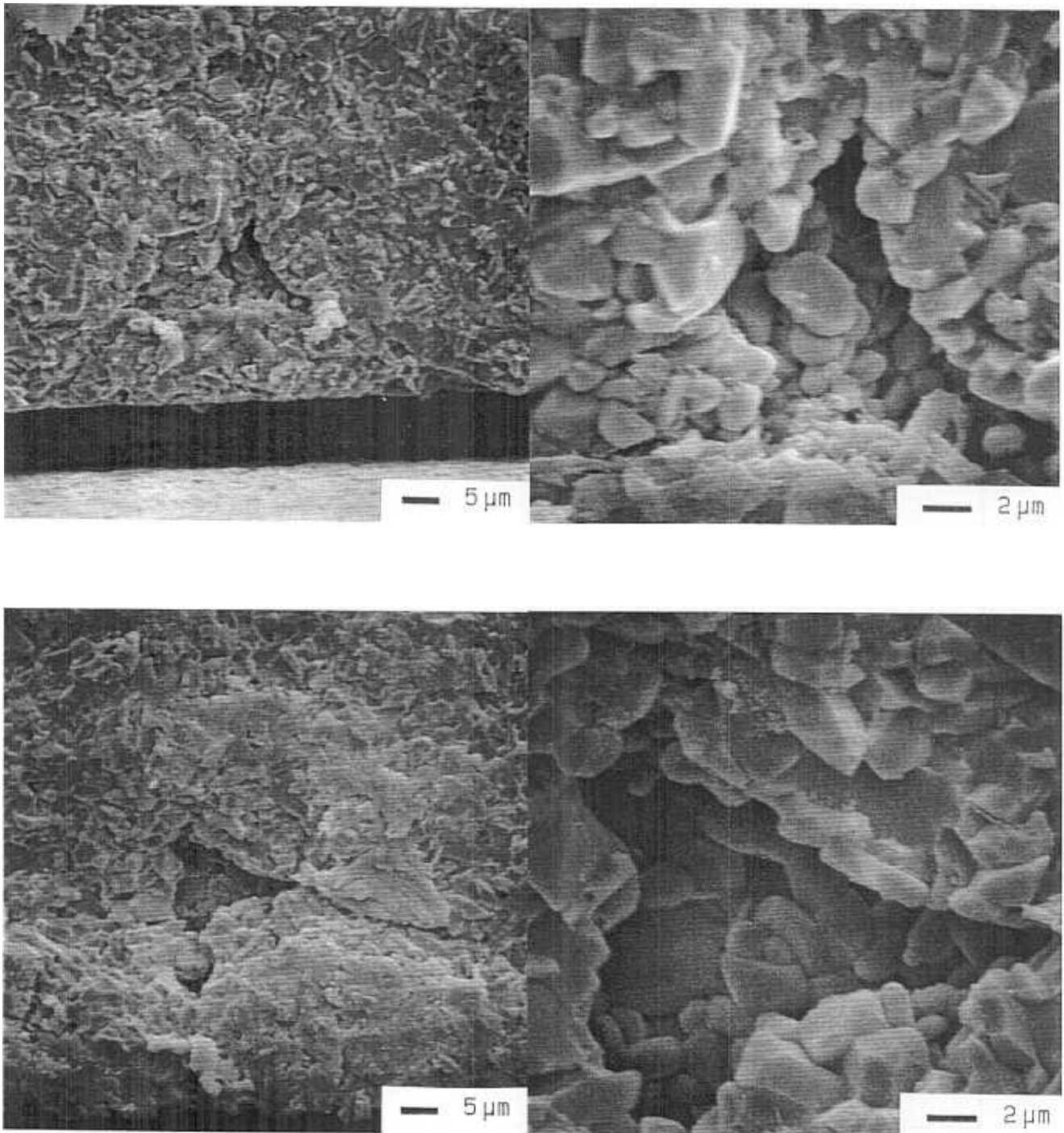


Figure 17. Scanning electron micrographs of large pores near fracture surfaces in disc test specimens, illustrating lack of complete compaction of spray-dried granules in pressing.

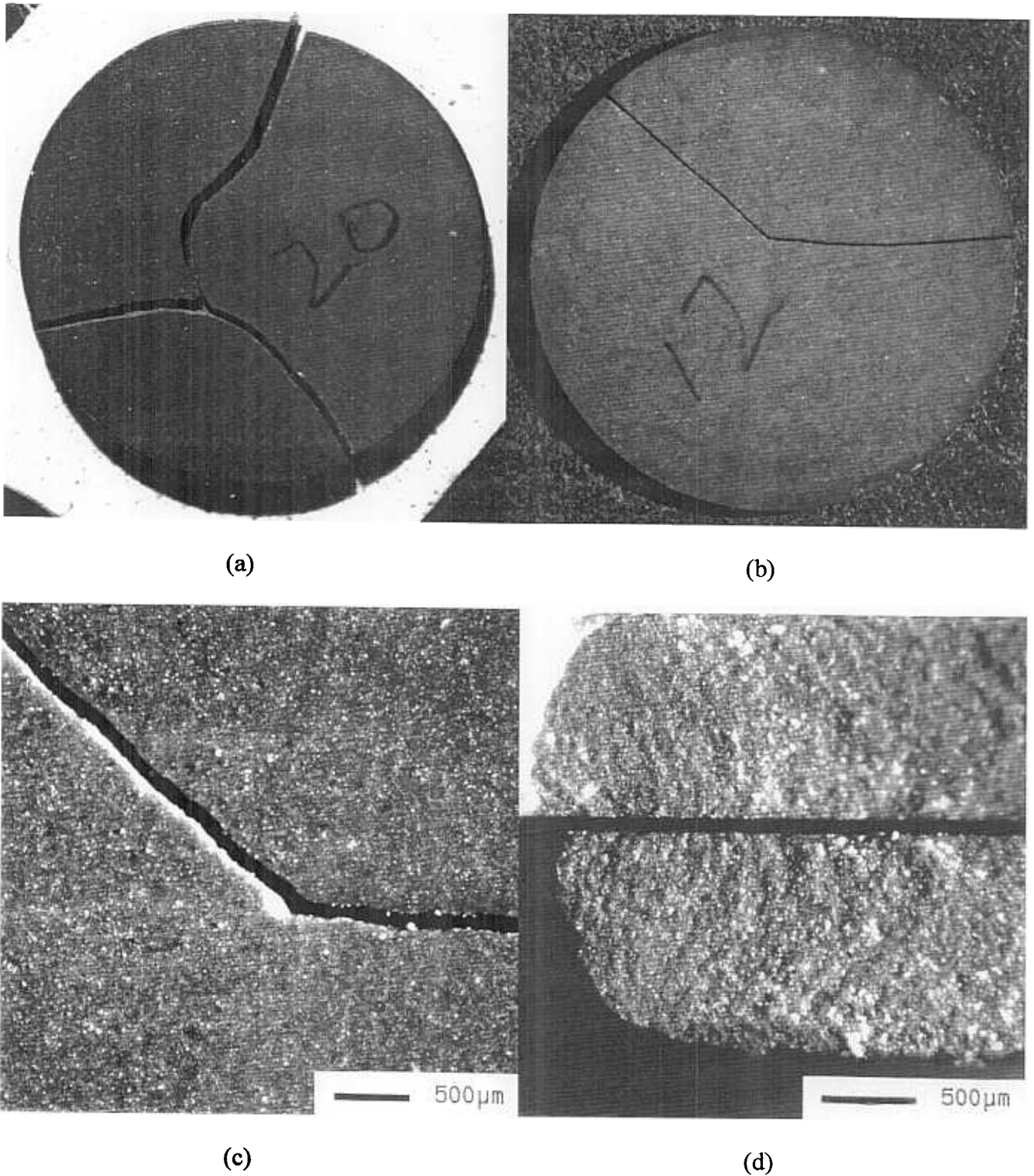


Figure 18. Fracture patterns in disc specimens, (a) typical high-strength failure, (b) typical low-strength failure, (c) as (b), but showing trace of crack halted during failure, (d) as (c) but matching fractured halves close to crack bifurcation showing possible failure initiation site.