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N A T I O N A L P H Y S I C A L L A B O R A T O R Y

DIVISION OF MATERIALS APPLICATIONS

THE VAMAS HARDNESS TESTS ROUND-ROBIN ON CERAMIC MATERIALS

by

D M Butterfield, D J Clinton and R Morrell

SUMMARY

This report analyses the results of a VAMAS round-robin to examine the utility of conventional hardness test methods to hard ceramic materials. Two high-alumina ceramics were characterised by HR45N, HV1.0, HV0.2 and HK0.2 tests at NPL, and then by 22 participants in the UK, US, Japan, France, Germany and JRC Petten. Participants remeasured NPL's Vickers and Knoop indentations, and then used their own equipment to repeat the test series. Analysis of the results has revealed the typical scatter involved in measurement, the bias of individuals and the bias of instruments, leading to estimations of the reliability with which hardness data can be produced. It is shown that potential errors of measurement as a consequence of these factors may be as high as 10-15% in hardness number on the HV and HK scales and ± 1 (an equivalent inaccuracy) on the HRN scale. These limitations need to be recognised if hardness test methods for ceramics are to be standardised.

CONTENTS

LIST OF PARTICIPANTS:

UK	BP Research, Sunbury-on-Thames British Ceramic Research Ltd, Stoke-on-Trent British Aerospace, Sowerby Research Centre, Bristol Imperial College, London Lodge Ceramics, Rugby Morgan Materials Technology Ltd, Stourport National Engineering Laboratory, East Kilbride Pilkington Brothers, R & D Laboratories, Lathom University of Oxford
US	National Institute of Science and Technology, Gaithersburg, MD (two sets, one with three observers)
FRANCE	ENSCI, Limoges
F R GERMANY	BAM, Berlin
EEC	JRC, Petten, Netherlands
JAPAN	Mechanical Engineering Laboratory Japan Fine Ceramics Centre, Nagoya Asahi Glass Company, Tokyo National Institute for Research in Inorganic Materials, Ibaraki-ken GIRI, Nagoya National Research Laboratory of Metrology

DISTRIBUTION:

Participants (see list above)	(2 each) 44 total
VAMAS organisation (B Steiner, NIST)	2
NPL	10
Overall total	56

1. INTRODUCTION

Hardness measurements are widely employed in the metallurgical industry as a quality-control tool and as a means of specifying the resistance of a material to localised deformation, scratching or erosion. The tests are based on indenting the test-piece with a hard indenter which may be spherical, conical or pyramidal. Such tests have been subject to considerable research and standardisation, particularly with regard to the establishment of indentation procedures, geometry of the indenters, and the provision of certified hardness test blocks.

Microhardness tests are widely employed in the glass industry for determining the scratch resistance of optical glass, and in the paints and coatings industry for the same purpose.

The existence of convenient methods of determining hardness has resulted in their application to ceramic materials without the development of any firm basis for their validity. Notwithstanding the extensive studies of indentation fracture processes [1,2,3] in brittle materials, there remains a lack of experimental evidence to support the use of hardness tests as a means of quality-control or specification. In particular, the high levels of hardness (> 1000 HV5), the small size of indentations, and the combination of plastic flow, fragmentation and gross cracking, lead to observational difficulties, and hence to considerable scatter between indentations and to differences between observers [4].

As engineering ceramics become more widely employed in conditions in which "hardness" is an important parameter, such as in shaft seals, slurry pumps, guides, rollers and other applications where erosion is a problem with metallic materials, it becomes necessary to understand the implications of using hardness tests. Important questions include:

(1) what types of tests are meaningful?

(2) what are the limitations imposed by material factors such as grain size and porosity?

(3) what are the limitations imposed by cracking and fragmentation?

(4) what are the limitations imposed by observational difficulties,

including poor contrast and small indentation size?

Previous work at NPL [4,5] has been concerned with the utility of tests as applied to a wide range of high-alumina ceramics. It has been shown that cracking tends to be unacceptably severe above HV1.0 in materials with grain sizes above about 8 µm, and above HV5 in finer-granined ones. When indentation sizes fall below about 25 µm for HV and 50 µm (long diagonal) for HK, the scatter of indentation sizes increases markedly due to a combination of increased observational error and the smaller volume of microstructure tested by the indenter. It was concluded that the most reliable type of measurement was Rockwell superficial (N-scale) which although could produce extensive damage, the results suffered least scatter, and the test could distinguish between differing levels of porosity and grain size.

In order to verify these conclusions the VAMAS Ceramics Project was used as a vehicle for conducting a round-robin test of hardness measurement to provide a body of statistical evidence.

2. ROUND-ROBIN DESIGN

The round-robin was designed to examine the ability of participants to make measurements of hardness using three common test techniques, Rockwell Superficial (HR45N), Vickers (HV) and Knoop (HK). Each participant was supplied with two test discs, both of alumina, but different types:

A : A white, high porosity (99.9%), fine-grained (~ 1.5 µm), low porosity (< 0.5%) product: "Vitox", manufactured by Morgan Matroc Ltd (Anderman Division), East Molesey, Surrey, UK.

B : A pink, general purpose alumina, 95% nominal Al_2O_3 content, medium grain size (~ 5-8 µm), moderate porosity (3-5%) product, with about 8-10% by volume of a second, glassy phase: "Sintox FA", manufactured by Lodge Ceramics Ltd, Rugby, Warwickshire, UK.

Material A, by virtue of its nearly fully dense, fine-grained microstructure, was employed in order to present participants with as near a problem-free high-hardness specimen as possible in terms of ability to observe and measure HV and HK indentations. Variations from point to point should be minimised because of uniform grain size and high density. The material is used for replacement hip joints, for which ISO 6474 contains a hardness specification. Material B was chosen in order to present more problems to the participant in terms of microstructural inhomengeneity, porosity, cracking and spalling around indentations, requiring decisions from the participant as to the acceptability and measurability of indentations.

Each test disc was divided into four zones using baked-on ink lines in order to define indentation positions, as shown in Figure 1. NPL placed HR45N indentations in one top zone, and a line of HV1.0, HV0.2 and HK0.2 indentations in the adjacent central zone. Two experienced observers with similar judgement criteria were employed for the optical measurements of the HV and HK indentations in order to provide a systematic set of data covering all test discs. This allowed statistical monitoring of the quality of each disc whilst reducing scatter due operator bias.

Participants were asked to remeasure the NPL HV and HK indentations with their own available system. A comparison of these measurements with those of NPL would then show any reading biases of the participants relative to that of the NPL observer. An analysis of the differences can be used to determine whether they are random or systematic, and whether they are of sufficient magnitude compared with the scatter of individual results to demonstrate unacceptability of the test for quality-control purposes. [Note: The HR45N data cannot be remeasured because the hardness scale is linked empirically to the depth of penetration of the indenter after application of a major load but while still under a minor load.]

Participants were then asked to use their own equipment to make equivalent sets of indentations in the remaining two zones and to measure these. The differences between the two sets of participants' measurements would then indicate the differences between their machines and/or indenters and those at NPL.

Finally, one of the NPL observers would recheck some of the participants' indentations, especially in cases of wide deviation from the expected results.

The tests were therefore designed to examine:

1. machine differences

2. systematic biases of measurement

3. scatter of data

4. criteria for acceptance/rejection of indentations due to excessive cracking or other problems.

5. suitability of the test method as a quality-control tool in terms of repeatability and discrimination.

The test instructions and the results form for return by the participants are included as Appendices I and II.

3. PARTICIPANTS

Test discs and instructions were distributed to participants either directly or through the Japan Fine Ceramics Center (coordinating Japanese involvement). Twenty-eight sets were sent, and twenty have been returned and analysed in this report.

The results have been communicated in confidence to all participants, from whom agreement has been received to publish the results in full.

4. TEST DISC PREPARATION

As-fired discs of the materials described in Section 2 were purchased from the respective suppliers in an as-fired form. They were mounted, six of one type at a time, in a Struers Abramatic automatic polishing machine, and after initial polishing trials, were all given the same polishing sequence to give a flat metallographic-quality finish with a minimum of scratching and grain pluck-out.

The final sequence was as shown in Table 1.

5. TEST DISC CHARACTERISATION AT NPL

5.1 HR45N

Test discs in pairs of material A and material B were subjected to five Rockwell HR45N indentations on an Eseway Type S superficial tester with a 15s dwell time. Before each test disc was indented, the machine was checked using a hardened steel block of hardness 74 HR45N supplied with the machine. No significant trend of result over the test series was found (Table 3, Figs. 2, 3).

5.2 HV1.0 and HV0.2

The tests were carried out using a Leitz Miniload II microharness tester at both load levels. The diamond indenter used was supplied by the manufacturers to the National Bureau of Standards (now National Institute of Science and Technology) recommendations as embodied in ASTM Standard E92.

Before undertaking measurements on the ceramic discs, indentations were made in a 576 HV0.1 steel test block supplied by the manufacturer and certificated by the Staatliches Materialprüfungsamt Nordrhein-Westfalen (F.R. Germany). The results of these measurements should in principle be a guide to the observer as to his criterion for placing the cross-wires at the ends of the diagonals and for checking the instrument performance. However, the value of the test data are questionable when the test pieces have such different optical contrast that there can be no equivalence of observation between the test block and the unknowns. It becomes unclear as to how the observer should adjust his criterion in order to match the test block result. The maximum deviations observed were about 0.5 µm in an indentation of diagonal approximately 18 µm, with the observer's measurements being greater than expected from the test block. [It has to be borne in mind that the calibration of the test block by one observer is similarly subject to both systematic errors of observation and potential errors of diamond geometry.] It was concluded that the deviations observed were within expected observational criteria differences and thus the machine performance was judged to be adequate. No deliberate adjustment of observer's criterion was made except insofar as the tests were used to allow the observer's eyes to become accustomed to the microscope system.

A series of indentations was then placed in each test disc, first using HV1.0 and HV0.2. At least five HV1.0 indentations were used, more if some were judged as being unacceptably damaged such as to preclude measurement. At least ten HV0.2 indentations were made using the same criterion. The indentations were placed in a line starting 3 mm in from the edge of discs, the larger HV1.0 indentations acting as a guide to locating the smaller HV0.2 indentations. Each indentation was numbered and entered into the participants' results form so that identification of unacceptable ones could be made by the participant.

5.3 HK0.2

After a batch of test discs had been indented with a Vickers diamond, the indenter was changed for a Knoop diamond supplied by Leitz and certified as being of correct geometry by the Staatliches Materialprüfungsamt Nordrhein-Westfalen and equivalent to the National Bureau of Standards recommendations embodied in ASTM Standard E384.

No test block was available for calibration purposes. A series of at least ten indentations was placed in the same line as the Vickers indentations and the long diagonals were measured.

6. PARTICIPANTS' RESULTS

Participants were asked to make the series of remeasurements of the NPL indentations, and to make their own series of measurements with their own machines after calibration checks and according to the suppliers instructions (Appendix 1). The completed results form and the test discs were then returned to NPL for checking and analysis. Participants' age and eyesight data, potentially relevant to measurements requiring microscope viewing, are given in Table 2.

6.1 HR45N

Only one third of the participants had equipment to measure HR45N; two used HRA, and one, HRC. Only three participants had HR45N test blocks with which to check the machine; the others used HR30N, HRC or HRA test blocks as noted in Table 3. No particular testing difficulties were noted.

6.2 HV1.0

All participants were able to measure the NPL-placed indentations, although some indentations in material B proved difficult. It is suggested that the damage introduced at indentation corners resulted in loss of material by spalling in the period between initial testing production at NPL and their examination by the participants. Material A caused no problems.

Laboratory 16 used three observers to make separate assessments.

All but two laboratories made their own HV1.0 indentations, again with no problems in material A, but some difficulties with material B. Laboratory 9 used a universal mechanical testing machine equipped with an indenter, rather than a purpose-built hardness tester.

The measurement of indentation radial crack lengths emanating from the corners of the indentations posed considerable problems for the participants. Although overall lengths were of the order of 70 µm in material A, a number of participants suggested they were not sufficiently long or well defined to measure. No specific illumination instructions were given, but it is likely that participants attempted to use the same conventional measurement system as for measuring indentations, which would not illuminate narrow cracks particularly well. In consequence, there was a large scatter of results. Material B did not produce adequately well-defined single cracks, and multiple cracking rendered measurement uncertain. Nevertheless, some participants recorded measurements without comment.

6.3 HV0.2

There were few reported problems in identifying the NPL microhardness indentations despite the fact the some were ignored as not measurable. Two participants were not certain as to the identification of indentations in material B, and one commented that the damage around some indentations rendered them unsuitable for measurement. Some additional damage may have occurred after the NPL characterisation. Only one participant commented that he would not consider the result on material B reliable because of the uncertainties of indentation geometry.

No problems were reported by participants in making their own indentations, and only one laboratory could not do the test. The majority of participants needed to make only ten indentations in material A to obtain ten acceptable readings, but it needed an average of 13.2 (and in one case 20) in material B to obtain ten acceptable ones. None reported any chipping damage to the indenters used, but some reported that they were unable or reluctant to remove the indenters from the instruments for examination because of problems of realignment.

6.4 HK0.2

Thirteen participants possessed appropriate equipment for this test. There were no reported problems in identifying and measuring the NPL-made Knoop indentations, nor in placing their own indentations, in either of the materials. Most laboratories required only 10 indentations in material A to obtain 10 satisfactory results, and the average requirement was 11 (with a maximum of 14) for material B. No damage to indenters was recorded.

7. ANALYSIS OF RESULTS

7.1 HR45N

Figures 2 - 5 and Table 3 summarise all the test data. The NPL data show that the discs are reasonably consistent in properties, but a few showed wider variations among the five indentations made than the majority (Figures 2,3). There was no correlation between the small variations in individual indentations in the 74 HR45N test block and the recorded data on the ceramics, suggesting that the repeatability of testing with the instrument used was limited to about ± 0.5 about the mean result 74.3. Standard deviations in the ceramic in each block varied from 0.1 to 0.8 for material A and 0.2 to 3.2 for material B, with overall standard deviations of the mean results for all test discs of 0.4 for material A and 0.3 for material B. In other words, the consistency of mean results from the ceramics was of the same order as the consistency of indenting a reference block over a period of time.

Figures 4 and 5 show, in histogram form, a comparison of the NPL data with those from participants indenting using the HR45N method. For individual discs, mean results are within 1 scale division for all but two participants for material A, and within 0.5 scale division for all but two participants for material B. Laboratories 26 and 28 appear to read consistently higher than the others for both materials. Close inspection of these figures reveals that all laboratories measure consistently either higher or lower than others. This suggests that there are systematic differences between machines, probably in the individual diamonds used. Because it was not possible to circulate a reference test block to obtain a baseline to confirm such variations, one is forced to conclude that in the absence of the use of a high-hardness HR45N test block, considerable variations in mean results can arise. In addition, the calibration of a machine using a test block on a different scale (HR30N, HRA, HRC, etc), while being a useful check that the machine is functioning in the correct manner, does not necessarily guarantee that the correct results are obtained on a different scale or different part of the same scale. This is particularly the case with Laboratory 28, where despite acceptable calibration with a 62.1 HR45N test block, large deviations from the NPL results, and from all the other participants, have occurred at above 75 HR45N. Examination of the indentations in question revealed that they were not spherical pits, but had a large angular area protruding on one side. Such a geometry could not have arisen as a result of slight chipping of the diamond, as this would tend to produce indentations with inward deviations to their periphery, not outward ones. It is suspected that there was major damage on the scale of a 120-140 μm effective indentation diameter, but limited to within the scale of 200-230 μm diameter of the test block indentations at 62.3 HR45N such that the test block read correctly after the tests on the ceramics. In this respect the results of Laboratory 28 should be ignored in the overall assessment, underlining the need to inspect the form of small Rockwell indentations in very hard materials.

7.2 HV1.0

Table 4 summarises the optical readings of both NPL and participants' indentations in material A in terms of the average diagonal lengths of at least five indentations, and Table 5 is the equivalent for material B. The averages and standard deviations are calculated in the normal arithmetic manner.

NPL readings of the initial set of indentations on material A are in quite a narrow size band. Average results for the different test specimens cover the range 30.4 to 31.3 µm, about 3% variation. Standard deviations on the set of five indentations are typically 0.5 µm, varying between 0.2 and 0.8 µm. Participants' measurements of these indentations showed a considerably wider spread of mean values: 29.6 to 32.5 µm, with standard deviations up to 1.0 µm. Mean results were both higher and lower than the corresponding NPL figures by up to 1.8 µm in the extreme case, as demonstrated by Figures 6 and 7.

In the case of material B (Table 5) the acceptability of indentations for measurement was, as expected, much reduced. The coarse-grained inhomogenous microstructure caused fragmentation at corners of indentations with a risk of loss or displacement of indentation corners. Some participants needed to make in excess of 10 indentations before obtaining 5 acceptable ones from the measurement point of view. NPL's initial indentations had mean values in each test specimen ranging from 38.1 to 40.4 µm, about 5% variation, with standard deviations ranging from 0.6 to 2.9 µm, about three times that of material A. Participants' mean results on remeasuring the NPL indentations ranged from 36.9 to 41.3 µm (Figure 8), somewhat wider than the NPL range, and standard deviations ranged from 0.3 to 3.3 µm (Figure 9). Again mean results were both higher and lower than those made by NPL by up to 2.9 µm at greatest. A small proportion of the acceptable NPL indentations were deemed unmeasurable by the participants, either as a result of different acceptability criteria or due to further losses of fragments around indentations. The averages quoted may therefore be for less than five indentations.

Unlike the case with the HR45N, a comparison of the mean HV1.0 results for the two materials (Figures 6 and 8) shows no obvious systematic relationship between the two sets of results for the participants. Those obtaining results high compared with NPL measurements on material A did not necessarily also obtain high results on material B, e.g. participants 21 and 26.

The mean and standard deviation data have been converted into hardness numbers in Tables 6 and 7 to put the spread of data into the terms normally used to express data. The average of the two indentation diagonals was used in the calculation of each hardness number, and this has had the effect of significantly reducing the spread of results. For material A, NPL mean results of 1891 to 2004 HV1.0, and standard deviations up to 100 scale units compare with a range of 1759 to 2116 HV1.0 for participants' measurements, with standard deviations up to 129 scale units. For material B, NPL's range of 1141 to 1283 HV1.0 with standard deviations up to 159 compare with the participants' range of 1091 to 1385 with standard deviations up to 238.

Whereas the Rockwell HR45N data are essentially independent of the machine operator, HV1.0 data depend on two human factors:

(1) personal bias in placing a measurement cross-wire at the corner of the indentation;

(2) ability of the eye to resolve the position of the indentation corner within the optical limitations of the system and the contrast produced.

These are in addition to machine factors:

(3) load applied

(4) calibration of the magnification of the system

(5) accuracy of the indentation resulting from accuracy of geometry of the indenter and its alignment in the machine.

Participants were asked to check magnifications and calibrations with a graticule or other suitable device, so factor (4) should result in a consistent bias in all participants' measurements. Factors (3) and (5) are not involved in participants' measurements of NPL's indentations, and so the comparison of NPL's measurements and participants' remeasurements of the same indentations is principally a comparison of the human factors.

It is desirable to distinguish between the elements of randomness of measurement and randomness of small genuine size variations of indentations. To do this requires comparison of individual indentation diagonal lengths measured by NPL and each participant. However, since there was no instruction given as to which of the two diagonals should be recorded first, this test can only be carried out on the mean results of pairs of diagonals. It was felt that the best statistical test to use was to determine the correlation coefficient:

$$r = \frac{S_{xy}}{S_x S_y}$$

where $S_{xy} = \frac{\Sigma(y-\bar{y})(x-\bar{x})}{n-1}$ is the covariance

$S_x^2 = \frac{\Sigma(x-\bar{x})^2}{n-1}$ is the variance of x

$S_y^2 = \frac{\Sigma(y-\bar{y})^2}{n-1}$ is the variance of y

It was felt that a value of $r = 1$, implying full correlation of measurement groups x and y, means in the present case that the variations in real indentation size are truly followed by the both sets of measurements. A value of $r = 0$ implies complete randomness of the sets, while a value of $r = -1$ implies that the two sets are completely anticorrelated, which in the present case has to be interpreted as a freak case of randomness (or a most peculiar eye condition!)

Correlation coefficients for the NPL and participants' sets of measurements are given in Tables 4 and 5, and are illustrated more clearly in Figure 10. There is a wide spread of correlation coefficients from - 0.86 to 0.99, the range being wider for material B than for material A. Interpretation of these results depends upon the assumptions one makes about the consistency of the NPL observations. If the NPL observations by only two persons truly reflect the real variations in indentation sizes, then the participants' measurements correlate quite well in material B for more than 50% of the participants, but in material A for only 20% of the participants. However it is likely that there is an element of randomness in the NPL results to the level of perhaps 0.3 µm, which is about the same as the standard deviation of measurements treated as averages of the diagonal pairs. In this event, it would explain why correlation was poorer for material A than for B. Material A has a smaller real variation in indentation size than B as a consequence of its low porosity level and homogeneous fine grain size. The correlation sought between NPL's and participants' measurements is thus more likely to be obscured by random errors in material A than in material B. Figure 11 shows a plot of NPL and participant standard deviations (plotted as joined pairs of points) against computed correlation coefficient. There appears to be no distinct correlation between these factors for material A, but a marked one for material B in cases where the standard deviations exceed about 1.5 µm. From this one can draw the conclusion that recorded data from participants can be said to follow the NPL-observed variations if the standard deviations exceed 1.5 µm. In view of the small population of indentations used (5), there are questions as to the significance of the computed correlation coefficients, but it seems likely that observational uncertainties rule the results on material A, and also on material B in cases where apparent standard deviations are less than 1 µm.

Also shown in Tables 4 and 5 are the mean results and standard deviations of tests made by the participants with their own machines. Eighteen participants were able to perform the tests. NPL checked three participants' data on material A, and two on material B.

The mean results were typically within 0.5 µm of those recorded by the same participant on NPL indentations for material A, and only two deviated by more than 1 µm. The NPL measurements on Laboratory 14's indentations showed significantly lower results, more in line with the general trend. On material B, deviations were larger, up to 2 µm or more. The two NPL check measurements made confirmed participants' results. Standard deviations on material A were similar to those recorded on NPL's indentations with two exceptions (Laboratories 9 and 24). NPL confirmed the figures for Laboratory 9. The mean result for Laboratory 24 was a consequence of bias from one large indentation out of the 10 made. The remaining nine gave a result very much in line with the rest of the data. On material B, standard deviations tended to be larger than those recorded on NPL's indentations, probably a consequence of variations of individuals' assessment of measurability in contrast to the preselection of those NPL indentations they measured. Figures 12 and 13 illustrate the variation in mean results for materials A and B respectively.

The radial crack lengths measured from tip to tip are reported as averages in Table 8 and Figure 14. These data were obtained from participants' own indentations. Mostly, cracks in material A were well-formed and posed no measurement difficulties although some participants declined to take part in the exercise because of difficulties of adequate resolution of crack ends with the optical system available. Cracks in material B tended to be poorly formed, irregular, and frequently bifurcated. A number of participants refused to measure them as having insufficiently well-defined geometry. Those who did undertake the measurements gave a wide spread of results with high standard deviations. One laboratory commented that in their opinion, it required HV10 indentations to produce adequately well defined cracks for the purpose of measurement of fracture toughness. The results of the present exercise show that there is a need to exercise care over illumination and resolution conditions, and that variations in perception of length are as high as ±20 μm, a considerable error.

7.3 HV0.2

Results are presented in Tables 9 and 10 for materials A and B respectively. The mean results on all test discs of material A as measured by NPL are closely grouped in the range 13.1 to 13.9 μm, with standard deviations up to 0.5 μm (Figures 15 and 16) indicating a fair degree of consistency of material and measurement criterion combined. Participants' mean measurements are more broadly spread, 12.1 to 14.2 μm with standard deviations up to 0.7 μm. Differences between NPL and the participants are typically up to 1 μm in mean values, with one result at 1.5 μm. Correlation coefficients obtained as described for HV1.0 indentations are poor, with no results above 0.82, suggesting that either the NPL measurements or the participants measurements (or both) are subject to random errors of around 0.5 μm or greater which obscure true size variations. This fact is not surprising considering all the optical resolution problems at this level of measurement. Material B showed a range in NPL-measured mean values of 14.7 to 16.7 µm, a considerably broader spread than for material A, with correspondingly larger standard deviations up to 1.7 µm (Figures 17 and 18), clearly reflecting the inhomogeneous nature of the material. Participants readings were 15.3 to 17.7 µm with standard deviations up to 2.1 µm. Differences in mean values between NPL and participants are equally positive or negative up to 1 µm. Correlation coefficients were remarkably high for many sets of data, as Figure 19 shows, but, as demonstrated by Figure 20 which shows a correlation between correlation coefficient and standard deviation, this is principally a result of the high standard deviations. Thus, as in the analysis of the HV1.0 indentations, one concludes that only when real size variations exceed 1 µm is it possible to argue that random measurement errors are not the principle contribution to scatter.

In terms of the hardness numbers as conventionally determined, Tables 11 and 12 show a very wide spread of results with differences in mean value between NPL and participants up to 280 hardness numbers (and in one case, 537) for material A, and up to 260 hardness numbers (and in one case, 417) for material B. These differences in material A are larger than the scatter in mean NPL results for the series of specimens, but those in material B are similar to the overall spread of the NPL results. In percentage terms they readily exceed 10% for both materials.

Data on indentations made by nineteen participants are given in Tables 9 and 10. Results on material A (Figure 21) are very similar to those recorded by the participants on NPL indentations. Differences do not exceed 0.8 µm and are typically less than 0.3 µm, and standard deviations are of the same order. Five sets of indentations were remeasured by NPL as a check. Only those indentations from participant 6 were measured to be significantly different from the participant's values, confirming that the low results obtained by the participant are principally due to individual bias larger than that of any other participant.

Results on material B (Figure 22) showed a higher degree of scatter, but similar to that measured by the participants on the NPL indentations. Remeasurements by NPL of the somewhat low values recorded by participant 14 were confirmed, and since the equivalent values on material A were slightly higher than NPL's measurements, it has to be concluded that if the result on B is genuine, it is due to freak choice of indentation locations. Considerable difficulties arose with some of the remeasurements owing to inconclusive identification of the indentations.

7.4 HK0.2

Results of HK0.2 measurements are summarised in Tables 13 and 14. The NPL results on material A are moderately consistent, mean values varying from 35.9 to 41.6 µm with standard deviations in the range 0.3 to 1.6 µm, but with one result at 4.0 µm (Figures 23 and 24). Participants' measurements were mostly within 1 µm in mean result with similar levels of standard deviations. Despite the relatively large standard deviations, correlation coefficients were widely spread with a number being negative, indicating the considerably larger element of randomness in determining the positions of the ends of the indentations than in the HV1.0 or HV0.2 indentations.

NPL's results on material B also show a wide spread (Figures 25 and 26) from 41.4 to 48.3 µm in mean value, with standard deviations of 1.2 to 3.0 µm. The participant's results covered a similar range, 41.2 to 49.4 µm in mean value and 1.2 to 3.0 µm standard deviation. Correlation coefficients were much higher than with material A, with only five results being less than 0.5 (Figure 27). The correlation between correlation coefficient and standard deviation shown in Figure 28 appears to confirm that random errors of measurement predominate when standard deviations are less than about 1.5 µm, but even up to 3 µm there are poor levels of correlation with some participants.

Thirteen participants were able to make their own HK0.2 indentations, and these were spread at least as widely as those of NPL's indentations: 36.0 to 40.4 µm in A, with standard deviations of 0.2 to 1.6 µm, and 42.3 to 47.1 µm in B, with standard deviations of 1.3 to 6.7 µm (see Figures 29 and 30). Check remeasurements were made by NPL in a few cases, notably where there appeared to be large differences between the NPL indentations and those of the participants. In all cases, the check data were within one standard deviation of the participant's values, implying that the indentation sizes were genuinely different.

Because of the relatively large long-axis dimension of the HK0.2 indentations compared with the square-based HV0.2 indentations, the large standard deviations recorded for the HK0.2 measurements are ameliorated to some degree in the calculation of Knoop hardness number (Tables 15 and 16). Nevertheless, the ranges of mean hardness numbers, 1650 to 2110 HK0.2 for material A and 1216 to 1670 HK0.2 for material B, based on NPL measurements, and 1545 to 2488 HK0.2 for A, and 1178 to 1681 for B based on participants' measurements, are considerably wider than those found for the HV0.2 tests.

8. DISCUSSION

8.1 Materials Problems

The two materials were chosen deliberately to show different characteristics. Material A, with is homogeneous fine-grained structure, would always have many grains under the indenter area, and thus should behave in a uniform, relative consistent manner.

Material B, with much coarser grain size, two-phase structure and significant residual porosity, was chosen as an example of an inexpensive, general-purpose ceramic which would not respond ideally in hardness tests. This reasoning turned out to be true in the round-robins. Material A gave reproducible results in the initial NPL tests, and also when these same indentations were remeasured by participants. It appeared straightforward to identify NPL's indentations, and they were still in good condition. As far as the spread of data shows, the set of test specimens were very similar in nature.

Figure 31 shows a correlation for material A between HV1.0 data and those obtained on other hardness scales by the two NPL participants. There is no correlation with the HR45N, indicating that at least one of the test series was subject principally to random errors. With HV0.2, there was a much more convincing correlation with most of the pairs of data lying in a band more or less parallel to the HV1.0 = HV0.2 line plotted in the Figure. With HK0.2 indentations, there was a very wide spread of points and no distinct correlation can be seen. Thus small variations in mean indentation size between the test specimens could only be resolved by Vickers-type tests, and using such tests, hardness numbers were consistent to within ± 50 HV1.0 or ± 80 HV0.2. It has to be borne in mind that the specimens could have been significantly inhomogeneous on the test surface. Rockwell indentations were in a different zone to the line of HV1.0 indentations. The microhardness indentations were towards the disc centres where densities could have been slightly different. It is not feasible to test for such inhomogeneity in all the test blocks, so it has to be assumed that the blocks are homogeneous.

Material B proved to be much more inhomogeneous than material A. As Figure 32 shows, there was no correlation between the HV1.0 results and those of any other test type. The HR45N tests had a spread about as broad as that for material A, suggesting that it could be considered as equally mechanically homogeneous on the scale of 150 µm. On the scale of 30 µm, the HV1.0 indentations gave a spread in mean hardness numbers of ± 70 HV1.0, while on the scale of a microhardness indentations, covering only 230-250 μm^2, the spread was ± 200 HV0.2 or HK0.2. It was perfectly feasible for a small indentation to be greatly affected by invisible subsurface pores or, equally well, closely packed groups of alumina grains with no porosity or little secondary phase. Although this material proved to be variable and it was difficult to obtain useful data from analysis of the results, it has shown up dangers in relying on numerical hardness data for such materials.

On a further note, variations of ± 0.5 HR45N represent ± 0.5 µm variation in mean penetration depth. With material A of hardness 88 HR45N, this represents ± 2.8 µm in the 136 µm diameter indentation produced, or ± 2.1%, equivalent to ± 4% in hardness number determined from the $(\text{dimensions})^2$ of an optically measured indentation. This figure is similar to the spread of mean HV1.0 results determined conventionally of ± 5%. A variation of ± 0.5 HR45N is thus a perfectly reasonable spread of results to expect. In material B, the same spread in HR45N would lead to only a ± 2% variations in an optically measured value compared with ± 6% variation in the HV1.0 results. Again this indicates the level of inhomogeneity below the scale of 150 µm.

8.2 Participants' Difficulties

Aside from the limited availability of HR45N and HK0.2 tests within the set of participants, the principal difficulties encountered were said to be identification of the NPL-made micro-indentations in material B, and the number of indentations this material required in order to obtain 5 (HV1.0) or 10 (HV0.2, HK0.2) acceptable ones from the measurement point of view. None of the participants stated that they had difficulties with the optical contrast, nor in judging where the measurement cross-wires should be placed, but clearly there were wide differences between NPL and participant measurements in a number of cases indicating the adoption of radically different criteria.

Overall, the worst problems appeared to be with the radial crack length measurement in material B owing to bifurcation.

8.3 Optical Measurement Criteria

It should be noted that no optical criteria were given to participants as to how to place the measuring cross-wires at the corners of indentations. Their own judgement was required, and this can result in a significant bias to the results of any individual. It is interesting to note that the NPL researchers are almost in the middle of the population as determined by the range of differences in results between NPL and the individual participants. However, it does not appear possible to calculate individual biases from this exercise because of lack of consistency of real indentation size and the random error involved, but they could be as large as ± 1 μm in Vickers indentations and ± 3 μm in Knoop indentations.

The participants' stated eyesight conditions covered the range from 20/20 vision to astigmatic, and ages varied from 19 to 54. NPL operators were aged 19 (20/20 vision) and 58 (short-sighted). Attempts were made to correlate results with eyesight and age, but no clear pattern emerged to explain the spread of data. The results are thus most likely to be controlled by a combination of random variations in the real indentation sizes, random errors in measurement, and individual bias.

8.4 Limitations of Hardness Tests for Hard Ceramics

Table 17 summarises the estimated reliability of hardness measurements on the two materials in each of the tests. Five aspects are listed and are described in the table footnotes. The variability of the materials themselves, based on the spread of mean NPL results, is of the same order as the spread of results on each specimen determined blind by each participant. Comparing NPL and participant measurements of the NPL indentations reveals that the bias of the participants relative to NPL was usually less than the two factors above. Comparing the participants' measurements on the NPL indentations and their own set reveals the possible machine bias due to variations in loading, diamond geometry and calibration of magnification.

The overall ability of any two observers operating any two machines to agree that a material is or is not harder than a set level, e.g. in conformance with a specification, is dependent on all the factors above. The manner is which the possible errors and biases combine is uncertain, and the end result will depend on how this is done. The final assessment in Table 17 is based on simple root sum of squares of factors (2) to (4). These figures are confirmed by the correlation coefficient data in Figures 11, 20 and 28 which indicate the typical differences in indentation sizes that are required in order that two observers correlate to a level with $r > 0.9$. Material variability has <u>not</u> been added as it may be the factor that needs to be investigated as part of testing to a specification.

The final assessment shows that the possible resolvability in Rockwell testing is about ± 1 HR45N for both materials, which is proportionately a much larger difference for high hardness material A than low hardness material B in terms of percentage variation in penetration depth.

The Vickers and Knoop tests all have similar levels of uncertainty in hardness number for both materials (150 to 230 units) despite differences in percentage variations of indentation dimensions, and Knoop measurements are no less variable than Vickers indentations at the microhardness level. Possible larger errors in percentage terms with material B are offset by the larger indentation size and the small hardness number resulting.

It should be noted that the possible errors can be significantly reduced by reducing participant bias, or the combination of participant and machine biases. For this, wider availability of high-hardness test blocks would be required in order that observers can redefine their measurement criteria. The use of low-hardness test blocks may not be a sufficiently sensitive means of identifying changes the observer should make to his criteria, and, as we have seen with HR45N data from participant 28, may not reveal calibration problems due to damaged indenters.

8.5 Recommendations for Standardised Tests

1. Hardness tests may used for engineering ceramic materials provided that it is recognised that errors and biases lead to high levels of uncertainty which increase with increasing hardness level.

2. Microstructural features must be much smaller than the size of the indentation used.

3. An adequate number of indentations must be used, preferably 10 or more of good geometry. (Part of the possible scatter in mean HV1.0 test results may be due to the use of only 5 indentations.)

4. Badly damaged indentations must be ignored. Cracking from corners has to be accepted, but the impression of the corners must be undamaged.

5. The machine/observer combination must have a means of calibration, preferably a high-hardness test block, especially for HR45N.

6. The geometry of the diamond indenter must be checked at intervals especially in the high-load HR45N tests, by inspecting either the diamond or the quality and shape of the indentation.

7. It should be recognised that possible systematic errors in hardness numbers are in the range 10-15%, or even greater on high hardness materials (> 2000 HV or HK), when the numbers are to be compared with a specified level.

8. Rockwell hardness numbers above about 85 HR45N risk large errors due to poor discrimination between different hardness levels.

9. CONCLUSIONS

1. The VAMAS international hardness round-robin has been analysed and assessed to enable some recommendations to be made and limitations to be established with regard to the hardness testing of hard ceramics.

2. Observer biases and random errors are the major sources of possible error in hardness measurements which contribute towards errors of 10-15% in hardness numbers produced by Vickers and Knoop tests. Machine biases are ± 0.7-0.9 in HR45N tests.

3. There is a clear need for the use of a high-hardness test block for the calibration of HR45N machines for operation above 75 HR45N to reduce machine bias. Test blocks are also required to ensure observers have correct measurement criteria for HV and HK tests.

4. In using hardness tests to compare a material with a specification or other independent source of data, the errors listed at 2, above must be recognised as a limitation. These errors will be less in instances where a single observer ranks materials.

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Table 1 - Polishing Procedure

Grit size, µm	Lap type	Time, s	Load,* N
20	Petrodisc M[1]	60	100
6	Petrodisc M	300	200
6	DP Plan[2]	600	200
3	DP Plan[2]	600	200
1	DP Plan[2]	600	200

* Total load applied to 6 specimens
1 Petrodisc M is a rigid filled polymer lap with embedded diamond grit
1 DP Plan is a synthetic woven cloth

Table 2 - Eyesight and Age

Participant	Age	Eyesight code*	Glasses	Comment
4	52	3	N	Mildly
5	39	5	Y	
6	25	2	N	Very mild
9	23	2	N	
11	23	1	N	
12	49	4	Y	
13	24	1	N	
14	41	5	Y	
15	28	1	N	
16A	33	3	Y	Contact lens
16B	19	2	N	
16C	33	2	Y	
19	28	2	N	
20	21	1	N	
21	25	1	N	
22	24	2	Y	
23	27	4	N	
24	25	4	N	
25	39	4	N	
26	36	4	N	
27	25	2	Y	
28	54	4	N	
NPL 1	19	1	N	
NPL 2	58	2	Y	

* The eyesight code used was that shown in Appendix II, i.e. 1 = 20/20 vision, 2 = short-sighted, 3 = long-sighted, 4 = middle-sighted, 5 = significantly astigmatic.

Table 3 - Rockwell superficial, HR45N, results

Disc No	NPL Tests						Laboratory Tests								
	Reference block		Material A		Material B		Reference block ***					Material A		Material B	
	Nom.	Results *	Mean	s.d.	Mean	s.d.	Nom.**	Mean 1	s.d.	Mean 2	s.d.	Mean	s.d.	Mean	s.d.
4+	74.0	74.0, 74.1	87.9	0.3	77.5	3.2	89.5A	89.6	0.5A	89.6	0.2A	91.0	0.3A	80.2	0.7A
5		74.1, 74.3	88.3	0.2	78.4	0.3	76.5N	76.9	0.2	77.0	0.2	89.4	0.4	78.7	0.7
6		74.0, 74.1	88.5	0.3	78.3	0.3	60.7C	59.6	0.6C	58.9	0.4C	80.7	0.4C	67.1	1.5C
9		74.7, 74.4	88.6	0.2	78.4	0.3	-								
11		73.8, 74.7	88.5	0.2	78.3	0.2	-								
12		74.5, 74.0	88.4	0.3	78.1	0.6	-								
13		74.2, 74.0	88.1	0.8	78.1	0.5	-								
14		74.4, 74.0	88.2	0.3	78.6	0.5	-								
15		74.7, 74.2	88.2	0.4	78.6	0.6	-								
16		74.5, 73.3	87.4	0.8	78.4	0.4	-								
19		74.3. 74.5	88.4	0.4	78.3	0.4	-								
20		74.4, 74.1	88.2	0.1	78.4	0.9	-								
21		74.0, 74.0	88.2	0.2	78.4	0.3	-								
22		74.1, 74.4	88.3	0.1	78.2	0.3	22.2C	21.8	0.05C	21.9	0.04C	89.2	0.4	78.5	0.3
23		74.0, 74.5	88.3	0.2	78.5	0.9	-	79.9	0.1	79.8	0.1	87.7	0.4	77.7	0.4
								(HR30N)		(HR30N)					
24		74.7, 75.0	87.5	0.5	78.7	0.3	31.4C	76.2	0.2A	81.6	0.3A	97.3	3.1A	97.8	0.5A
25		73.9, 74.0	88.4	0.3	78.2	0.3	22.1C	21.6	0.1C	21.7	0.1C	89.1	0.9	78.1	0.3
26		74.1, 74.5	88.3	0.5	78.5	0.7	78.4N	74.8	0.1	74.7	0.1	89.9	0.1	78.9	0.5
27		74.8, 74.1	87.3	1.1	78.3	0.2	66.5	65.6	0.2	65.9	0.3	88.7	0.4	77.8	0.5
							(HR30N)	(HR30N)		(HR30N)					
28		74.5, 73.9	88.1	1.3	79.1	0.2	62.1	62.3	0.3	62.7	0.3	92.1	0.2	81.2	0.4

* Single tests before testing Materials A and B respectively

** Nominal value of test block, A = HRA, C = HRC, N = HR45N - note some test blocks were HR30N

*** Mean 1 = before tests, Mean 2 = after tests on ceramics

\+ Participant reports the use of an indenter specially purchased for hard materials, such as hardmetals.

Table 4 - Material A, HV1.0, dimensions

Disc No.	NPL indentations, µm							Lab. indentations, µm					
	NPL readings Mean	NPL readings s.d.	Lab.readings Mean	Lab.readings s.d.	Difference Mean	Difference s.d.	Corr. coeff.	Lab.readings Mean	Lab.readings s.d.	NPL readings Mean	NPL readings s.d.	Difference Mean	Difference s.d.
4	31.2	0.4	32.2	0.5	-1.02	0.46	0.34	31.6	0.5	-			
5	31.3	0.6	32.1	0.7	-0.73	0.26	0.77	-		-			
6	31.2	0.3	31.0	0.5	0.19	0.37	0.00	-					
9	31.0	0.6	31.3	0.7	-0.33	0.59	-0.83	31.8	2.4	31.8	2.1	0.06	1.31
11	31.3	0.2	31.1	1.0	0.24	0.84	-0.08	31.5	0.4	30.9	0.9	-0.55	1.05
12	30.9	0.5	31.1	0.5	-0.50*	0.19	0.87	30.9	0.7	-			
13	30.8	0.5	31.5	0.8	-0.74	0.69	-0.44	32.7	0.7	-			
14	31.0	0.5	32.6	0.3	-1.61	0.32	0.53	34.1	0.6	32.6	0.5	-1.51	0.60
15	30.6	0.5	30.2	0.6	0.31	0.51	0.15	30.7	0.3	-			
16A	30.8	0.6	31.3	0.7	-0.47	0.57	0.41	31.0	0.4	-			
B			30.5	0.5	0.24	0.44	0.08	-		-			
C			29.9	0.9	0.92	1.03	-0.25	-		-			
20	30.4	0.8	29.6	0.8	0.82	0.80	0.17	29.8	0.7	-			
21	31.3	0.5	32.5	1.0	-1.18	0.80	0.64	32.1	0.4	-			
22	30.5	0.4	31.4	0.4	0.94	0.36	0.15	32.5	0.9	-			
23	30.4	0.6	32.2	0.8	1.82	0.62	-0.08	31.5	0.4	-			
24	31.7	0.3	32.5	0.2	-0.78	0.17	0.05	33.1**	1.8	-			
25	31.2	0.4	32.4	0.4	-1.13	0.29	0.48	31.7	0.6	-			
26	31.2	0.6	32.3	0.6	-1.04	0.57	0.33	32.2	0.6	-			
27	30.7	0.8	31.5	0.4	-0.70	0.27	-0.61	31.1	0.6	-			
28	31.0	0.4	30.4	0.2	0.64	0.40	-0.84	30.1	0.1	-			

* Three indentations only.
** 32.6 ± 0.8 ignoring one of ten indentations

Table 5 - Material B, HV1.0, dimensions

Disc No.	NPL indentations, µm									Lab. indentations, µm					
	NPL readings		Lab.readings		Difference		Corr. coeff.		Lab.readings		NPL readings		Difference	
	Mean	s.d.	Mean	s.d.	Mean	s.d.			Mean	s.d.	Mean	s.d.	Mean	s.d.
4	38.3	1.1	41.3	1.6	-2.92	1.49	-0.09		39.6	1.6	-			
5	39.0	1.2	38.8	1.0	0.23	1.33	-0.36		-		-			
6	39.0	1.0	39.5	0.5	-0.49	1.29	-0.55		-		-			
9	39.2	0.7	40.0	1.5	0.80	0.79	0.17		39.4	2.7	-			
11	39.1	1.1	36.9	3.3	2.18	2.49	0.76		39.3	2.1	-			
12	39.0	1.1	40.7	1.0	-1.39	0.45	0.74		40.5	1.1	-			
13	38.8	1.1	38.3	1.7	0.41	0.81	0.81		40.4	2.1	-			
14	38.3	1.1	40.7	1.3	-2.35	1.47	-0.63		41.5	3.1	41.2	2.0	0.31	1.48
15	38.1	1.3	37.9	1.0	0.28	1.49	0.70		38.8	2.0	-			
16A	39.8	2.9	40.7	2.7	-0.89	1.10	0.93		42.8	5.5	-			
B			-						-		-			
C			39.8	2.2	-0.05	1.08	0.99		-		-			
20	38.4	1.6	38.5	1.8	-0.06	1.45	0.57		38.4*	2.7	38.0*	1.0	0.43	2.12
21	38.1	0.8	38.5**	-	-	-	-		39.1	1.0	-			
22	39.2	1.5	40.8	1.4	-1.63	0.78	0.94		41.6	1.2	-			
23	39.1	2.0	39.7	1.5	-0.54	0.94	0.89		37.9	2.5	-			
24	39.5	0.8	39.7	0.8	-0.20	0.79	-0.86		38.5	1.2	-			
25	38.8	0.6	39.4	1.0	-0.57	0.73	0.52		40.5	1.0	-			
26	38.3	0.7	38.5	1.2	-0.14***	1.38	0.27		38.1	2.2	-			
27	40.4	1.9	40.6	2.2	-0.15	0.88	0.92		39.8	1.5	-			
28	38.8	1.0	37.9	0.3	0.88	0.81	0.18		38.7	0.5	-			

* Excluding one result
** One result only considered measurable.
*** Correlation uncertain

Table 6 - Material A, HV1.0, hardness numbers

Disc No	NPL indentations, HV1.0					Lab. indentations, HV1.0				
	NPL readings Means	s.d.	Lab.readings Means	s.d.	Difference in means	Lab.readings Means	s.d.	NPL readings Means	s.d.	Difference in means
4	1911	27	1793	44	+118	1857	27	-		
5	1891	54	1806	53	+ 85	-		-		
6	1905	32	1928	19	- 23	-				
9	1931	60	1890	41	+ 41	1849	259	1853	236	+ 4
11	1894	12	1927	106	- 43	1876	46	1946	108	+ 70
12	1946	54	1902*	26	+ 44	1947	66	-		
13	1953	46	1870	64	+ 83	1735	46	-		
14	1925	55	1740	30	+185	1600	42	1752	26	+152
15	1988	55	2029	47	- 41	1963	36	-		
16A	1958	36	1901	78	+ 56	1926	22	-		
B			1989	57	- 31	-		-		
C			2086	129	-131	-				
20	2004	101	2116	86	-112	2088	88	-		
21	1896	39	1758	100	+138	1798	46	-		
22	1999	42	1881	27	+118	1755	37	-		
23	2004	29	1786	66	+218	1871	24	-		
24	1849	7	1759	20	+ 90	1708	147	-		
25	1902	27	1771	34	+131	1860	66	-		
26	1901	62	1781	52	+120	1792	51	-		
27	1984	66	1871	21	+113	1915	42	-		
28	1927	24	2009	30	- 82	2040	19	-		

* Only three remeasured

Table 7 - Material B, HV1.0, hardness numbers

Disc No	NPL indentations, HV1.0					Lab. indentations, HV1.0				
	NPL readings Means	NPL readings s.d.	Lab.readings Means	Lab.readings s.d.	Difference in means	Lab.readings Means	Lab.readings s.d.	NPL readings Means	NPL readings s.d.	Difference in means
4	1265	64	1091	45	+174	1188	92	-		
5	1222	75	1234	22	- 12	-	-	-		
6	1221	67	1189	21	+ 33	-	-	-		
9	1207	41	1161	68	+ 46	1208	163	-		
11	1217	58	1389	238	-172	1209	110	-		
12	1220	63	1119	42	+101	1133	59	-		
13	1238	69	1266	93	- 28	1142	109	-		
14	1265	46	1124	50	+141	1125	177	1100	116	- 25
15	1283	80	1301	54	- 18	1240	130	-		
16A	1186	159	1133	142	+ 53	1054	246	-		
B			-			-		-		
C			1174	106	+ 12	-				
20	1262	94	1259	107	+ 3	1269*	171	1286	71	+ 17
21	1277	48	1251**	-	-	1213	58	-		
22	1210	93	1112	48	+ 98	1074	51	-		
23	1216	110	1181	76	+ 35	1302	165	-		
24	1188	31	1176	22	+ 12	1253	67	-		
25	1232	41	1197	52	+ 35	1130	42	-		
26	1258	38	1257	80	+ 1	1284	134	-		
27	1141	107	1134	118	+ 7	1173	85	-		
28	1232	52	1288	10	- 56	1239	24	-		

* Ignoring both indentation much smaller than the rest

** Only one measured

Table 8 - Crack length data, HV1.0

Disc No.	Material A, µm Mean	s.d.	No.	Material B, µm Mean	s.d.	No.
4	87.1	5.2	14	78.7	12.9	10
5	-			-		
6	-			-		
9	58.0	10.2	17	64.3	12.4	15
11	64.2	6.4	19	65.4	8.0	4
12	84.5	1.6	10	n.m.		
13	85.6	6.6	10	n.m.		
14	-			-		
15	-			-		
16A	77.4	7.6	10	67.9	12.0	8
19	-			-		
20	-			-		
21	70.8	4.7	10	n.m.		
22	n.m.			n.m.		
23	82.1	7.7	12	38.0	2.5	12
24	68.5	8.7	20	72.7	10.7	7
25	67.9	6.2	10	68.9	-	1
26	67.3	7.5	10	61.2	10.9	10
27	84.8	5.3	10	57.1	4.0	3
28	70.6	4.8	10	75.1	13.4	10
Overall mean and s.d. of means	74.5	9.5		64.9	11.5	

s.d. = standard deviation, n.m. = not measured

Table 9 - Material A, HV0.2, dimensions

Disc No.	NPL indentations, µm							Lab. indentations, µm					
	NPL readings Mean	NPL readings s.d.	Lab.readings Mean	Lab.readings s.d.	Difference Mean	Difference s.d.	Corr. coeff.	Lab.readings Mean	Lab.readings s.d.	NPL readings Mean	NPL readings s.d.	Difference Mean	Difference s.d.
4	13.8	0.4	14.2	0.4	-0.41	0.41	0.18	13.7	0.3	-			
5	13.6	0.4	13.6	0.3	0.01	0.50	-0.62	13.7	0.3	-			
6	13.7	0.4	12.1	0.6	1.52	0.44	0.55	11.6	0.5	13.4	0.3	1.82	0.39
9	13.6	0.3	13.0	0.6	0.58	0.37	0.67	13.4	0.5	-			
11	13.6	0.3	13.8	0.4	-0.22	0.36	0.38	14.2	0.6	-			
12	13.5	0.3	14.0	0.5	-0.54	0.39	-0.45	13.2	0.3	13.6	0.3	0.45	0.29
13	13.5	0.3	13.7	0.4	-0.17	0.23	0.55	12.9	0.4	-			
14	13.3	0.3	14.0	0.3	0.60	0.32	-0.22	13.9	0.8	13.2*	0.9	*	
15	13.2	0.3	13.1	0.3	0.03	0.23	0.47	13.5	0.5	13.5	0.6	-0.01	0.42
16A	13.1	0.2	13.3	0.3	-0.13	0.21	0.47	13.3	0.3	-			
B			12.9	0.3	0.21	0.30	0.16	-		-			
C			12.7	0.4	0.47	0.29	0.25	-		-			
19	13.2	0.2	13.6	0.3	-0.43	0.27	0.13	13.7	0.2	13.4	0.5	**	
20	13.2	0.2	12.5	0.5	0.77	0.34	0.60	-		-			
21	13.3	0.3	14.2	0.6	-0.92	0.40	0.34	13.9	0.3	-			
22	13.3	0.4	14.1	0.7	-0.92	0.43	0.17	13.8	0.3	-			
23	13.1	0.2	14.0	0.5	-0.96	0.22	0.50	14.0	0.4	-			
24	13.7	0.4	13.1	0.3	0.71	0.32	0.40	14.3	0.7	-			
25	13.9	0.5	13.7	0.7	0.15	0.33	0.82	13.7	0.3	-			
26	13.5	0.3	14.0	0.3	-0.45	0.26	0.40	14.2	0.4	-			
27	13.2	0.3	13.6	0.4	-0.42	0.17	0.75	13.4	0.3	-			
28	13.5	0.2	13.0	0.3	0.47	0.27	0.16	13.1	0.2	-			

* Correlation uncertain
** Correlation uncertain, only 7 remeasured by NPL

Table 10 - Material B, HV0.2, dimensions

Disc No.	NPL indentations, µm							Lab. indentations, µm					
	NPL readings Mean	NPL readings s.d.	Lab.readings Mean	Lab.readings s.d.	Difference Mean	Difference s.d.	Corr. coeff.	Lab.readings Mean	Lab.readings s.d.	NPL readings Mean	NPL readings s.d.	Difference Mean	Difference s.d.
4	16.3	0.8	16.6	1.1	-0.20	1.30	0.22	16.7	0.8	-			
5	15.9	1.2	15.5	1.6	0.42	0.72	0.94	15.0	0.8	-			
6	16.2	0.8	15.6	2.1	0.58	2.19	0.11	14.4	0.8	15.4	1.0	*	
9	16.6	1.0	15.8	1.2	0.82	0.37	0.96	15.9	0.8	-			
11	16.0	0.7	16.4	0.6	-0.41	0.81	0.32	16.5	0.6	-			
12	16.3	0.7	16.5	0.9	-0.40	0.39	0.89	15.5	0.8	16.3	1.6	*	
13	16.4	0.8	16.2	0.8	0.20	0.39	0.87	15.6	1.1	-			
14	15.2	0.9	15.9	1.2	-0.96*	-	-0.28*	11.8	3.8	12.5	3.1	*	
15	15.1	0.9	15.3	1.1	-0.16	0.69	0.75	16.2	1.0	16.2	0.8	-0.03	0.91
16A	15.7	1.3	16.2	1.5	-0.54	0.34	0.98	16.1**	1.5	-			
16B				-	-	-	-	-		-			
16C			16.2	1.3	-0.51	0.51	0.94	-		-			
19	15.6	0.8	15.9	0.7	-0.32	0.38	0.47	16.3	1.5				
20	14.7	0.7	15.0	0.4	-0.28	0.51	0.54	-		-			
21	15.1	0.8	17.7	1.8	-2.6	-	-0.03*	17.2	0.5	-			
22	15.4	1.0	16.9	1.3	-1.47	0.51	0.92	16.4	1.2	-			
23	15.8	0.9	16.2	1.2	-0.37	0.33	0.97	16.6	1.8	-			
24	15.7	0.8	15.5	0.9	0.21	0.54	0.65	17.9	1.7	-			
25	16.7	1.4	16.9	1.5	-0.19	0.40	0.97	15.7	1.0	-			
26	15.9	1.2	16.1	1.2	-0.20	0.75	0.78	16.0	1.3	-			
27	15.8	0.6	15.7	0.9	0.12	0.55	0.77	16.1	0.9	-			
28	16.5	1.7	16.0	1.7	0.44	0.15	0.996	15.6	1.3	-			

* Uncertain correlation
** Ignoring one data point

Table 11 - Material A, HV0.2, hardness numbers

Disc No	NPL indentations, HV0.2							Lab. indentations, HV0.2				
	NPL readings Means	s.d.	Lab.readings Means	s.d.	Difference in means		Lab.readings Means	s.d.	NPL readings Means	s.d.	Difference in means
4	1939	83	1829	86	+ 90		1970	60	-		
5	2001	96	2003	67	- 2		1995	69	-		
6	1993	124	2530	203	-537		2759	142	2060	71	-699
9	2011	72	2201	165	-190		2080	144	-		
11	1999	86	1938	98	+ 61		1848	140	-		
12	2051	57	1899*	97	+152		2138	86	1999	54	-139
13	2043	67	1993	80	+ 50		2251	123	-		
14	2097	74	1894	64	+203		1932	207	2151*	327	+219
15	2145	70	2155	74	- 10		2035	141	2030	130	- 5
16A	2157	51	2116	75	+ 41		2095	69	-		
B			2230	104	- 73		-		-		
C			2321	103	-164		-		-		
19	2145	70	2011	59	+134		1969	52	2079	121	+110
20	2126	38	2399**	154	-273		-		-		
21	2091	86	1833	110	+258		1928	72	-		
22	2115	105	1864	114	+251		1945	29	-		
23	2167	57	1883	73	+284		1899	68	-		
24	1970	91	2190	87	-220		1819	153	-		
25	1936	126	1981	163	- 45		1984	60	-		
26	2025	81	1896	56	+129		1853	71	-		
27	2125	71	2000	71	+125		2075	67	-		
28	2040	45	2190	84	- 50		2146	61	-		

* Only 7 remeasured
** Only 9 remeasured

Table 12 - Material B, HVO.2, hardness numbers

Disc No	NPL indentations, HVO.2					Lab. indentations, HVO.2				
	NPL readings Means	NPL readings s.d.	Lab.readings Means	Lab.readings s.d.	Difference in means	Lab.readings Means	Lab.readings s.d.	NPL readings Means	NPL readings s.d.	Difference in means
4	1401	147	1356	187	+ 45	1333	124	-		
5	1494	207	1593	287	- 99	1652	150	-		
6	1429	131	1596	355	-167	1788	142	1590	208	-198
9	1359	146	1512	190	-153	1484	142	-		
11	1450	111	1377	96	+ 73	1358	85	-		
12	1398	125	1364	135	+ 32	1552	152	1443	249	-109
13	1387	133	1420	128	- 33	1546	241	-		
14	1619	186	1481	181	+138	2812	1242	2945	1859	+130
15	1644	194	1618	236	+ 22	1424	168	1416	143	- 8
16A	1529	209	1431	210	+ 98	1460	247	-		
B			-			-		-		
C			1440	23	+ 89	-		-		
19	1542	153	1477	120	+ 65	1434	246	-		
20	1659	85	1727	147	- 68	-		-		
21	1632	167	1215	234	+417	1256	72	-		
22	1585	199	1324	185	+261	1403	207	-		
23	1497	166	1436	200	+ 61	1389	268	-		
24	1508	135	1545	117	- 37	1210	264	-		
25	1355	196	1327	198	+ 28	1527	172	-		
26	1485	209	1446	194	+ 39	1448	219	-		
27	1494	110	1522	156	- 28	1437	165	-		
28	1407	250	1485	266	- 77	1542	232	-		

Table 13 - Material A, HKO.2, dimensions

Disc No.	NPL indentations, µm							Lab. indentations, µm					
	NPL readings Mean	NPL readings s.d.	Lab.readings Mean	Lab.readings s.d.	Difference Mean	Difference s.d.	Corr. coeff.	Lab.readings Mean	Lab.readings s.d.	NPL readings Mean	NPL readings s.d.	Difference Mean	Difference s.d.
4	40.7	1.2	41.1	1.2	-0.35	0.51	0.91	39.4	0.3	-			
5	40.2	0.8	39.3	0.8	-0.91	0.68	0.63	-	-	-			
6	40.9	1.3	40 *	-	-	-	-	-		-			
9	39.1	1.6	40.2	0.7	-1.09	1.74	0.07	37.9	1.0	-			
11	40.3	1.5	41.5	0.7	-1.15	1.58	0.13	38.2	0.5	38.6	0.5	0.42	0.25
12	41.6	1.1	42.9	1.1	-1.38	1.02	0.58	37.1	0.7	36.9	0.8	-0.28***	
13	41.2	0.8	41.9	1.4	-0.79	0.73	0.91	-		-			
14	37.1	0.7	35.2**	1.0	1.90	1.20	0.08	-		-			
15	38.3	0.7	35.2	0.8	3.09	1.09	-0.10	37.1	1.6	38.9	1.7	1.79	0.80
16A	38.1	0.4	38.7	0.6	-0.66	0.52	0.47	-		-			
16B			38.1	0.4	-0.32	0.59	-0.28	-		-			
16C			38.0	0.3	0.07	0.61	-0.57	-		-			
20	38.3	0.6	33.8	1.0	4.44	1.15	-0.11	-		-			
21	38.2	0.4	39.2	0.6	-1.00	0.85	-0.33	39.3	0.4	-			
22	38.3	0.3	39.5	1.0	-1.14	1.00	0.11	37.6	0.4	-			
23	38.2	1.0	38.8	1.2	-0.61	0.87	0.72	38.9	0.6	-			
24	36.9	4.0	37.1	1.7	-0.22	0.73	0.93	37.9	0.7	-			
25	37.9	1.2	38.6	0.9	-0.73	0.47	0.91	38.2	0.8	-			
26	37.7	0.6	37.5	1.0	0.13	1.36	-0.43	36.0	0.5	-			
27	37.0	0.6	39.9	0.6	-2.92	0.10	0.38	40.4	0.3	-			
28	37.3	1.5	37.9	0.5	0.61	1.17	0.75	39.1	0.2	-			

* Measured to 1 µm only
** Assuming factor 2 error in measurement by Lab.
*** Uncertain correlation

Table 14 - Material B, HKO.2, dimensions

Disc No.	NPL indentations, µm							Lab. indentations, µm					
	NPL readings Mean	NPL readings s.d.	Lab.readings Mean	Lab.readings s.d.	Difference Mean	Difference s.d.	Corr. coeff.	Lab.readings Mean	Lab.readings s.d.	NPL readings Mean	NPL readings s.d.	Difference Mean	Difference s.d.
4	43.2	1.6	45.4	1.6	-2.18	1.49	0.58	44.2	1.6	-			
5	45.3	2.3	44.2	2.1	1.09	1.91	0.61	-	-	-			
6	45.8	2.2	44.1	2.4	1.71	2.97	0.19	-		-			
9	48.3	3.0	48.2	3.0	0.14	0.52	0.98	42.3	2.1	43.4	1.9	1.12*	
11	45.9	1.9	45.6	2.3	0.32	0.97	0.94	45.3	5.3	-			
12	47.6	2.6	49.4	2.2	-1.76	0.66	0.96	44.7	2.3	42.8	2.0	-0.86**	1.29
13	44.7	1.2	45.4	1.8	-0.70	0.98	0.85	-		-			
14	41.7	1.6	45.4	2.8	3.72	2.04	0.25	-		-			
15	42.9	2.7	40.0	2.3	2.84	1.29	0.88	42.7	3.0				
16A	42.3	2.4	43.9	2.9	-1.55	1.96	0.37	-		-			
B			-	-			-	-		-			
C			44.1	2.9	-1.78	2.06	0.08	-		-			
20	42.9	2.4	42.6	2.3	0.38	1.18	0.87	-		-			
21	43.2	1.6	44.1	2.4	-0.97	1.08	0.93	44.1	1.3	-			
22	42.9	2.0	44.3	2.4	-1.35	0.90	0.93	44.4	2.5	-			
23	42.4	2.3	41.8	2.3	-0.56	1.38	0.82	47.1	5.1	-			
24	41.7	2.5	44.0	2.4	-2.24	1.42	0.83	46.2	6.7	-			
25	41.6	1.5	42.7	1.2	-1.01	1.13	0.51	46.1	2.3	-			
26	41.4	2.3	41.2	1.7	0.21	2.19	0.44	42.7	4.1	-			
27	41.7	1.8	44.3	2.1	-2.57	1.02	0.88	46.1	4.0	-			
28	42.4	1.5	42.6	1.7	-0.20	1.36	0.65	42.7	2.2	-			

* poor correlation
** only 8 remeasured

Table 15 - Material A, HKO.2, hardness numbers

Disc No	NPL indentations, HKO.2					Lab. indentations, HKO.2				
	NPL readings Mean	NPL readings s.d.	Lab.readings Mean	Lab.readings s.d.	Difference in means	Lab.readings Mean	Lab.readings s.d.	NPL readings Mean	NPL readings s.d.	Difference in means
4	1716	98	1691	98	+ 25	1835	30	-		
5	1763	69	1847	71	- 84	-		-		
6	1708	106	1778	0	- 70	-		-		
9	1872	152	1764	56	+108	1983	98	-		
11	1751	126	1657	60	+ 94	1952	56	1906	47	- 46
12	1650	92	1545	78	+105	2065	75	2096	98	+ 31
13	1681	64	1621	103	+ 60	-		-		
14	2067	75	2301	134	-234	-		-		
15	1945	71	2303	100	-358	2072	168	1878	154	-194
16A	1966	41	1899	55	+ 67	-		-		
B			1932	50	+ 34	-		-		
C			1973	30	- 7	-		-		
20	1943	55	2488	137	-545			-		
21	1849	54	1947	45	- 98	1843	32	-		
22	1939	34	1829	89	+110	2013	41	-		
23	1951	101	1890	115	+ 61	1882	58	-		
24	2110	211	2082	172	+ 28	1983	77	-		
25	1990	118	1914	89	+ 76	1955	84	-		
26	2008	67	2024	102	- 16	2202	56	-		
27	2079	72	1786	57	+293	1742	23	-		
28	2051	166	1978	52	+ 73	1865	22	-		

Table 16 - Material B, HK0.2, hardness numbers

Disc No	NPL indentations, HK0.2					Lab. indentations, HK0.2				
	NPL readings Mean	NPL readings s.d.	Lab.readings Mean	Lab.readings s.d.	Difference in means	Lab.readings Mean	Lab.readings s.d.	NPL readings Mean	NPL readings s.d.	Difference in means
4	1524	110	1385	98	+139	1457	98	-		
5	1389	128	1458	128	- 69	-		-		
6	1359	123	1463	147	-104	-		-		
9	1232	147	1227	138	+ 5	1590	145	1509	126	- 81
11	1348	116	1376	143	- 28	1429	272	-		
12	1272	162	1173	104	+ 99	1521	145	1559	142	+ 38
13	1424	75	1381	104	+ 43	-		-		
14	1638	118	1394*	165	+244	-		-		
15	1562	149	1789	191	-227	1582	214	-		
16A	1589	164	1475	167	+114	-		-		
B			-	-		-		-		
C			1463	174	+126	-		-		
20	1554	161	1570	159	- 26			-		
21	1533	114	1461	147	+ 72	1462	82	-		
22	1543	133	1446	142	+ 97	1443	149	-		
23	1216	110	1178	84	+ 38	1284	150	-		
24	1649	181	1484	152	+165	1416	372	-		
25	1648	127	1568	88	+ 80	1349	128	-		
26	1670	179	1681	139	- 11	1596	273	-		
27	1642	138	1459	135	+183	1418	199	-		
28	1585	111	1572	119	+ 13	1572	147	-		

* Assuming factor 2 error in measurements

Table 17 - Resolvability of Differences in Hardness

Factor	Material A				Material B			
	HR45N*	HV1.0	HV0.2	HK0.2	HR45N*	HV1.0	HV0.2	HK0.2
Material variability µm (1) No.of test blocks	± 0.4 20	± 0.5 20	± 0.4 20	± 1.5 20	± 0.3 20	± 1.0 20	± 0.7 20	± 2.0 20
Single participant µm variability (2) No.indents.	± 0.4 5	± 1.0 5	± 0.4 10	± 1.0 10	± 0.5 5	± 1.5 5	± 1.0 10	± 2.0 10
Single participant µm bias (typical) (3) No.labs	± 0.8 6	± 1.0 20	± 0.5 22	± 0.8 21	± 0.5 6	± 1.5 20	± 0.5 22	± 1.5 21
Machine bias (4) µm	± 0.8 (same as (3))	± 0.5	± 0.3	±1.0**	± 0.5 (same as (3))	± 0.5	± 0.3	± 2.0**
Difference required µm before resolvable No.indents (5) % Hardness no.	± 0.9 5 7.5 ± 0.9	± 1.5 5 4.6 ± 180	± 0.7 10 5.4 ± 220	± 1.6 10 4.3 ± 180	± 0.7 5 3.2 ± 0.7	± 2.2 5 5.7 ± 150	± 1.2 10 7.5 ± 230	± 3.2 10 7.1 ± 180

* In units of HR45N scale, and excluding laboratory 28

** Some very large deviations, especially on material B, may be due to choice of indentation area.

(1) Defined as standard deviation of the averages of the mean of determininations on each test disc as determined by NPL, but clearly includes random errors of measurement of the NPL operator.

(2) Defined as standard deviation typically found by participants over number of indentations.

(3) Difference between mean values of participants' and NPL's results, with number of laboratories involved.

(4) Suspected bias introduced by machine (same as (3) for HR45N) by comparing participants' results on their own and NPL's indentations.

(5) Difference between materials needed before there is a good probability that the difference will be resolved reliably by any observer on any machine using the indicated number of indentations.

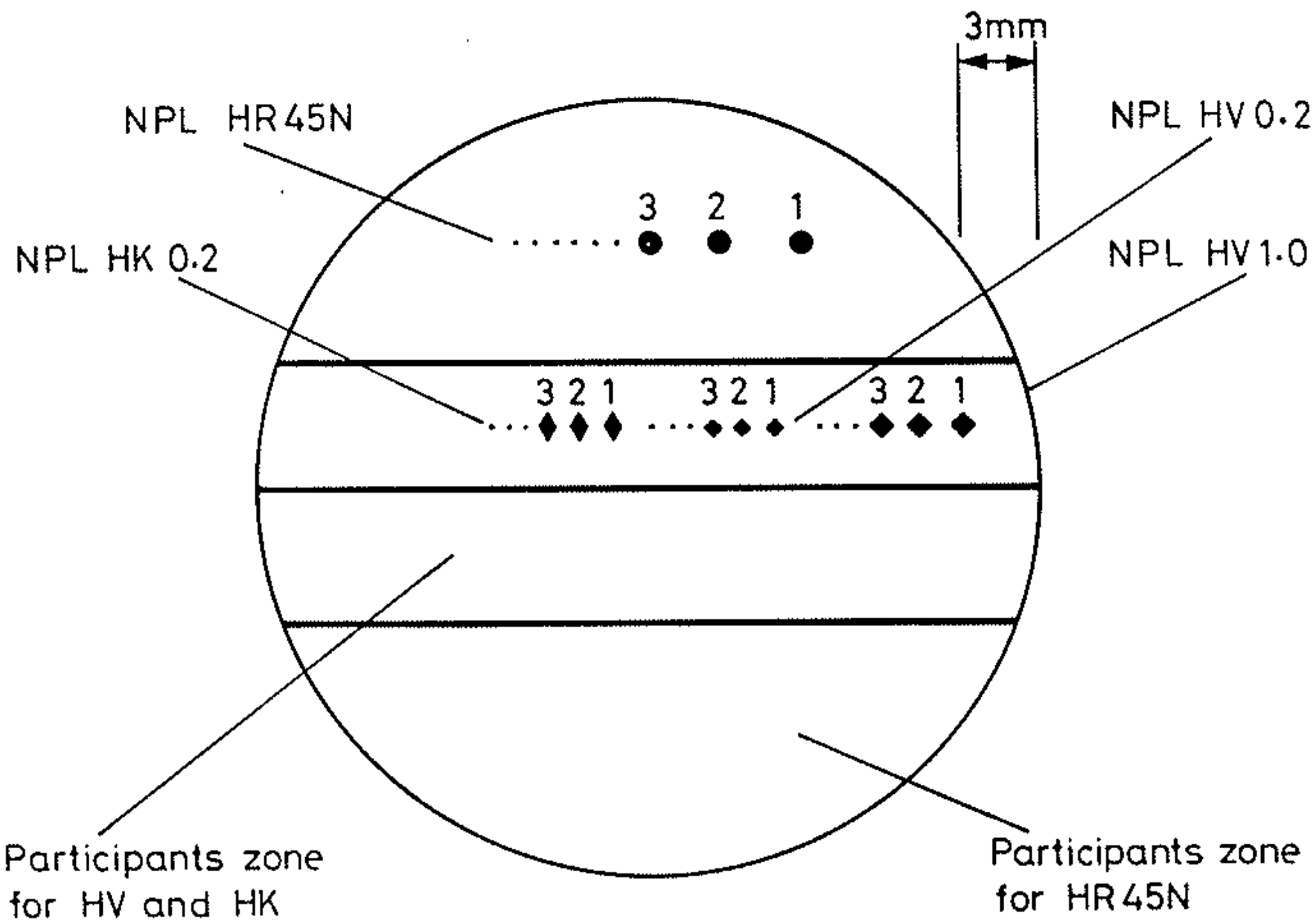

Figure 1 Scheme of layout of indentations in test discs

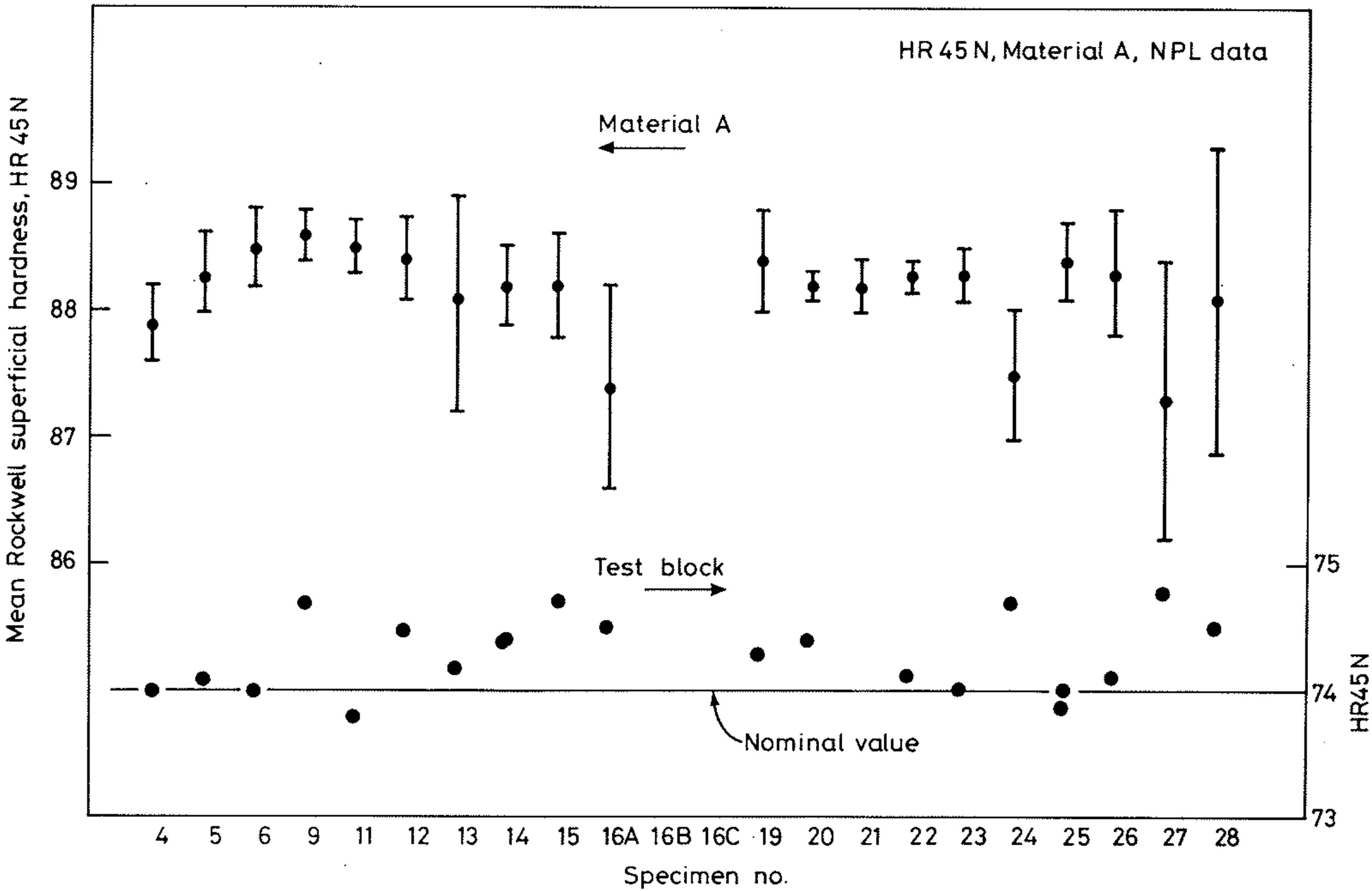

Figure 2 Distribution of mean HR45N results on material A produced by NPL, with single intervening 74 HR45N standard test block indentations.

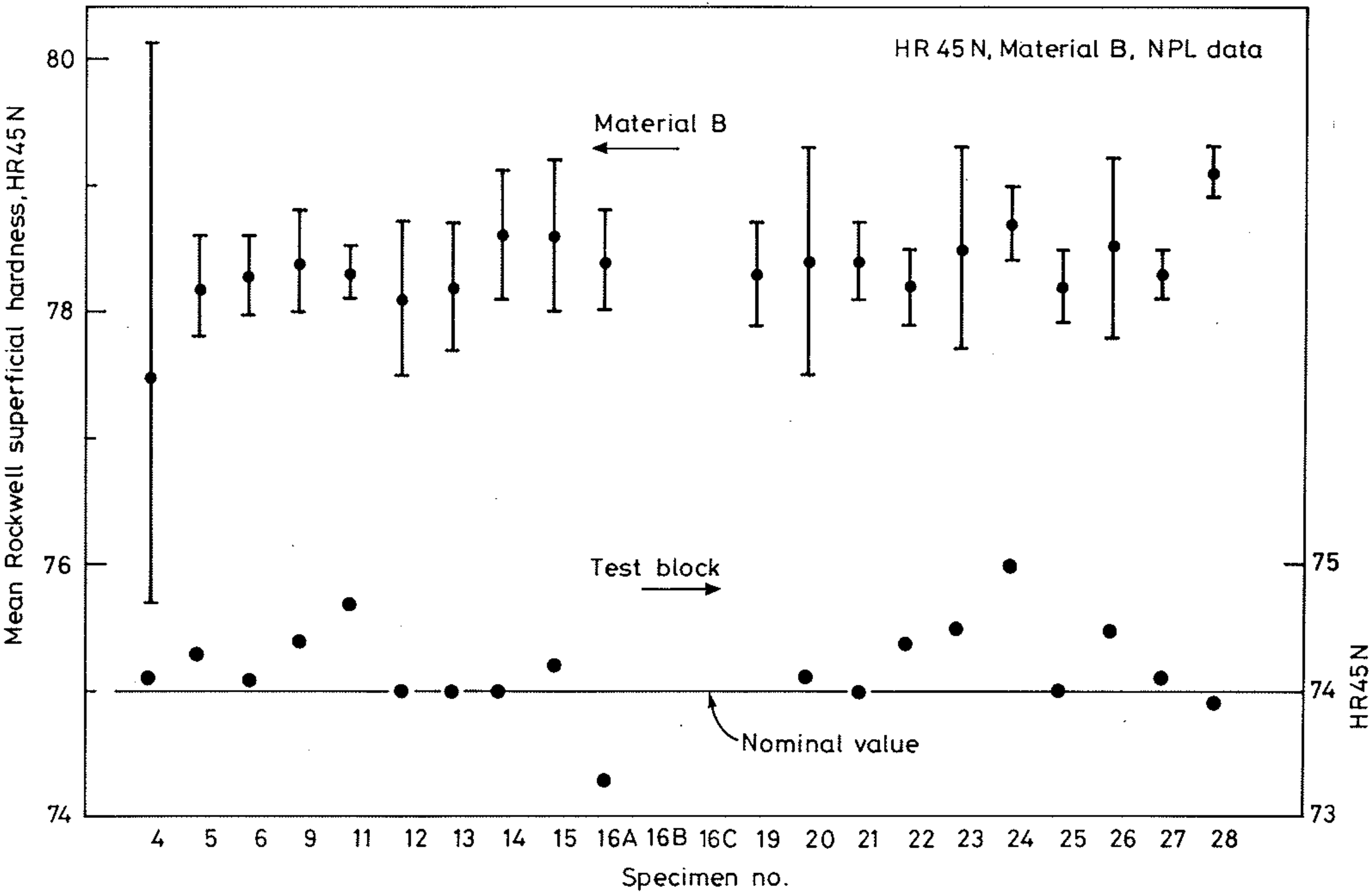

Figure 3 Distribution of mean HR45N results on material B produced by NPL, with single intervening 74 HR45N standard test block indentations.

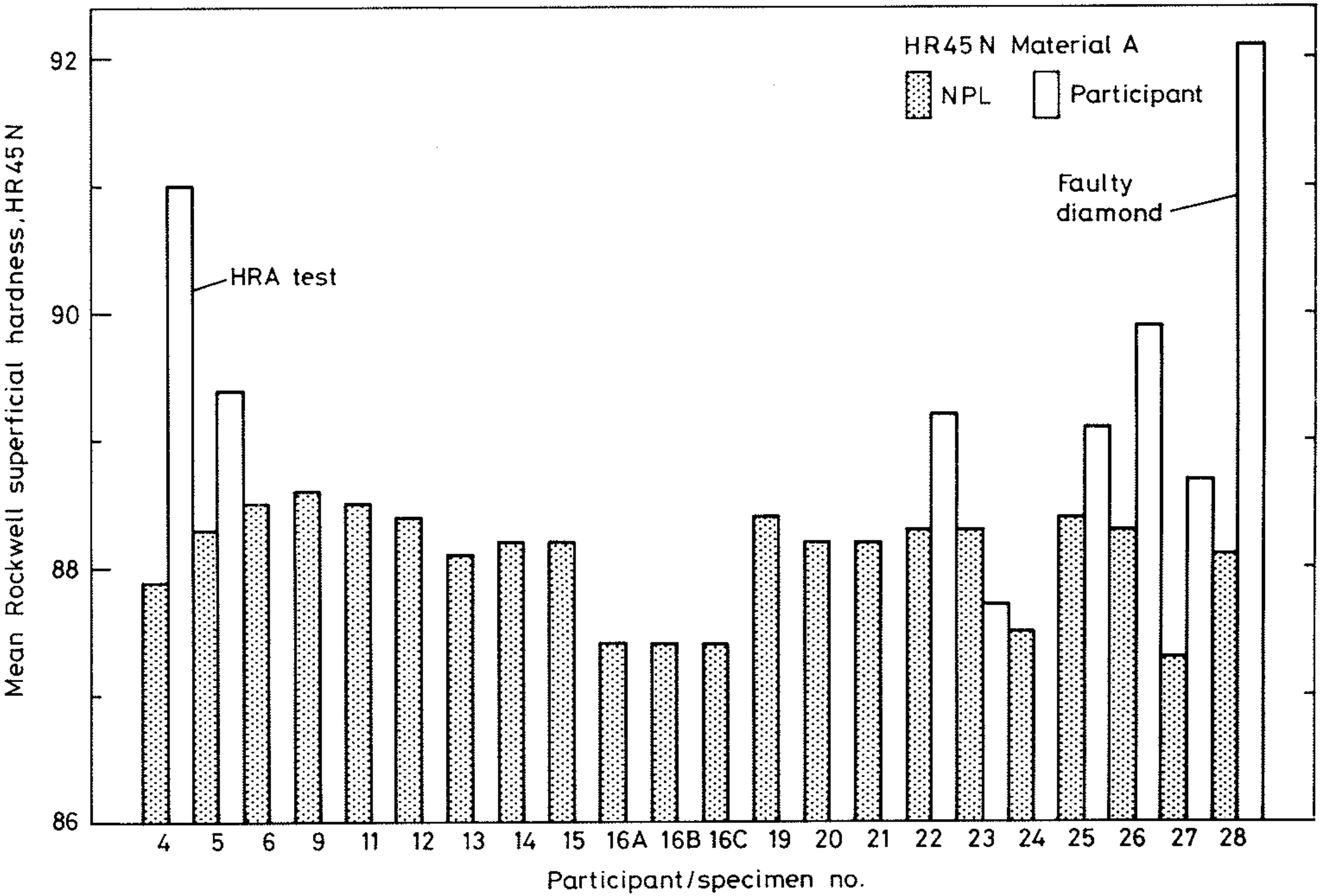

Figure 4 Comparison of mean HR45N results on material A produced by NPL and individual participants.

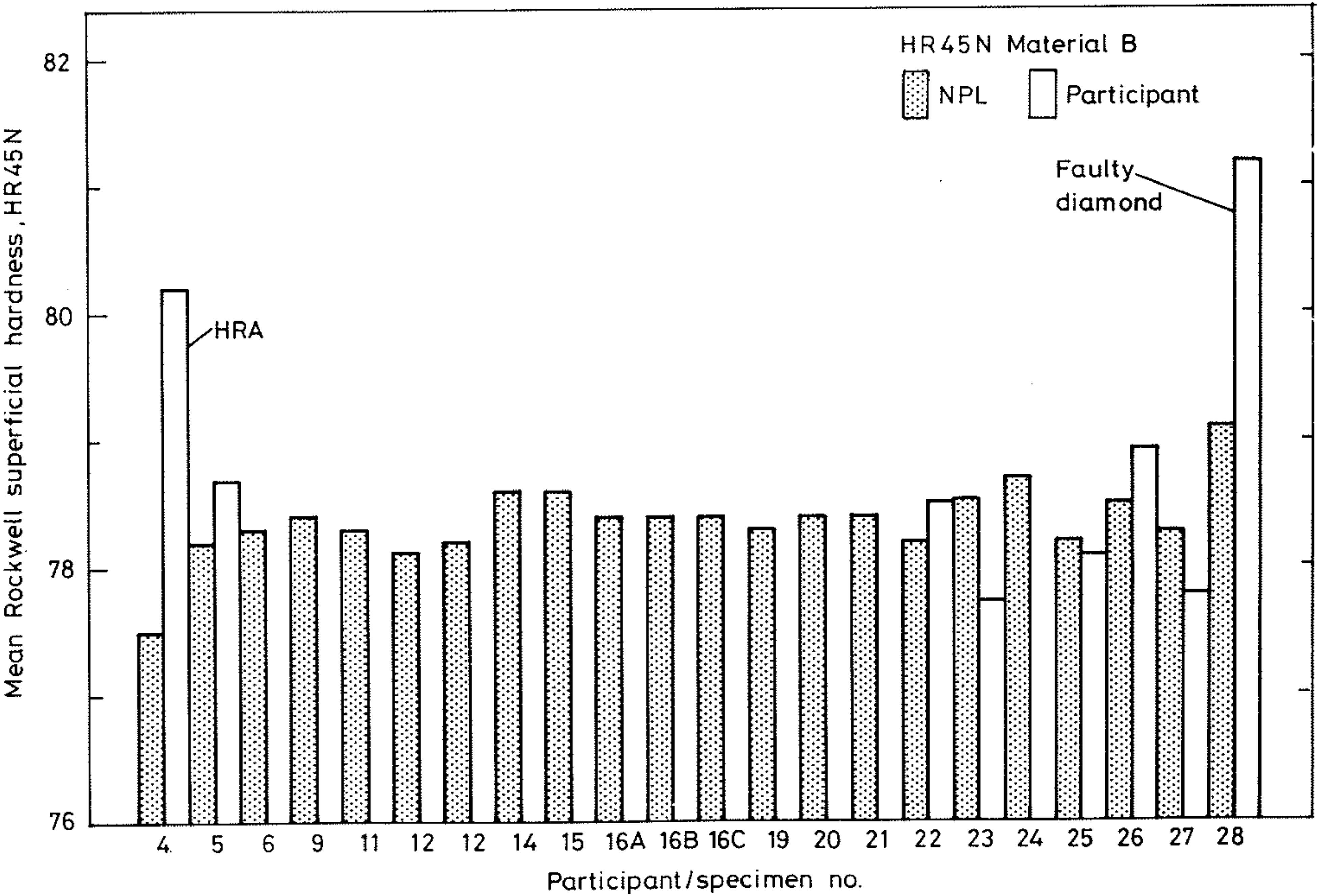

Figure 5 Comparison of mean HR45N results on material B produced by NPL and individual participants.

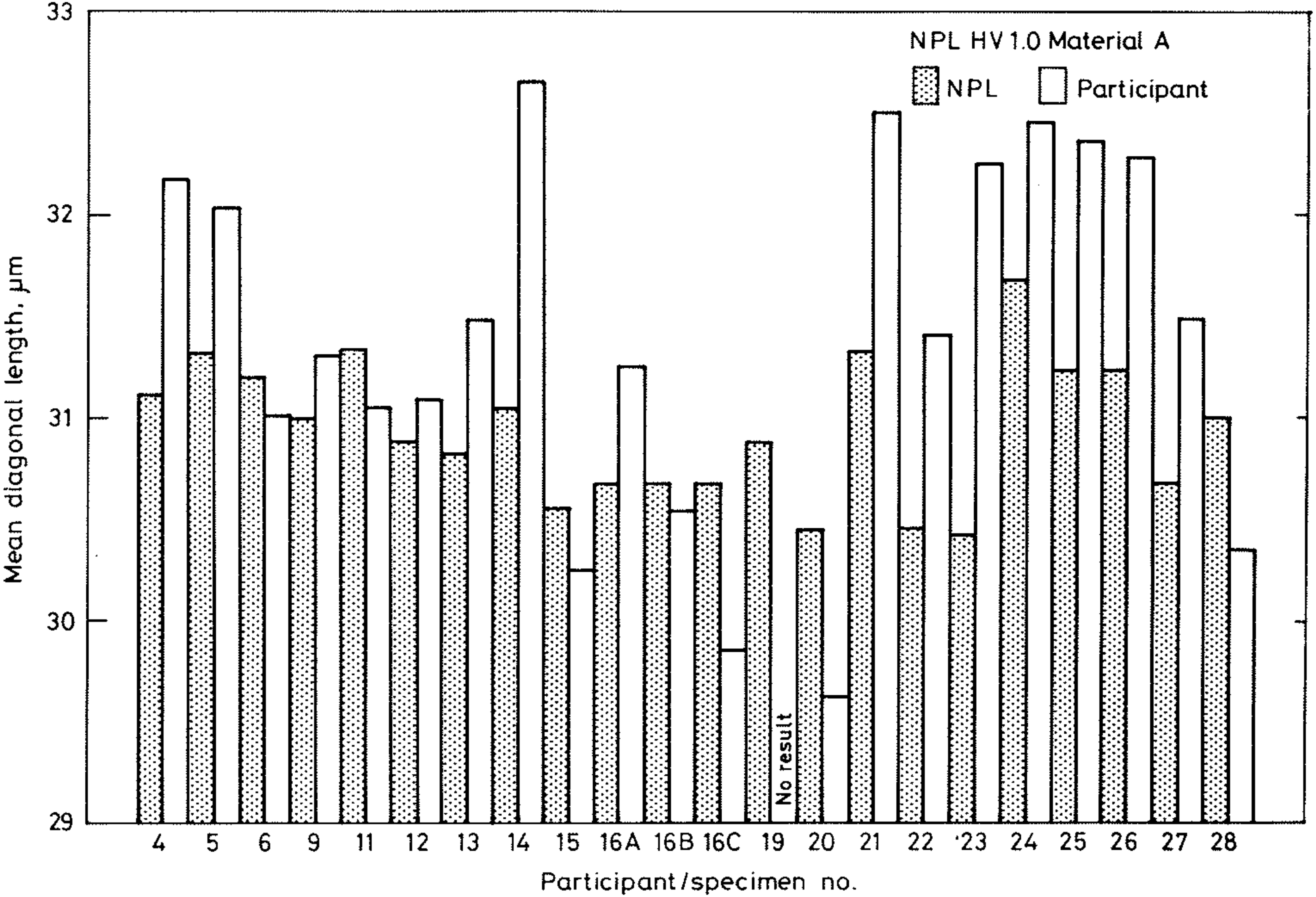

Figure 6 Comparison of mean HV1.0 indentation diagonal lengths in material A placed by NPL as measured by NPL and by participants.

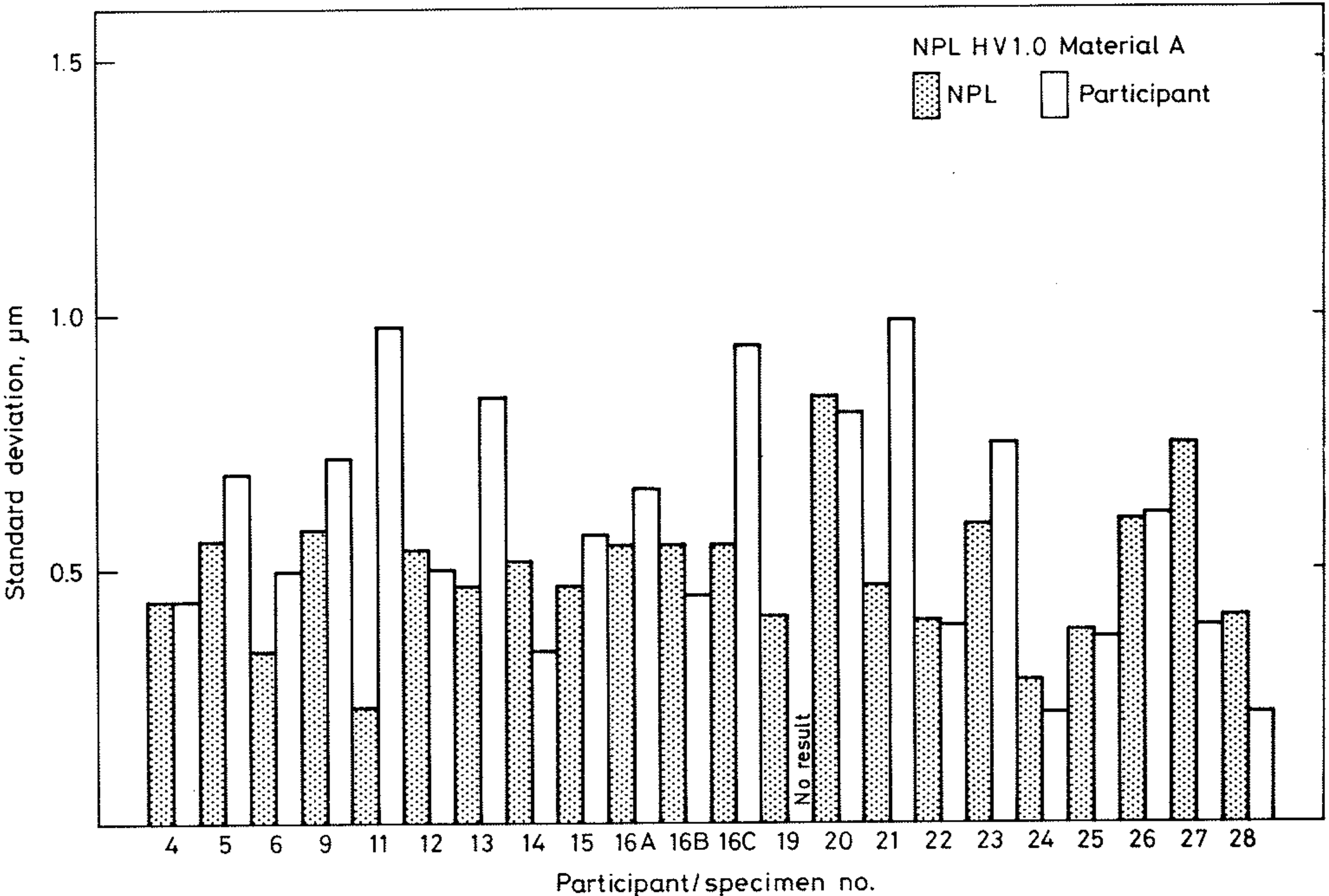

Figure 7 Comparison of standard deviations of diagonal lengths of HV1.0 indentations in material A placed by NPL as measured by NPL and by participants.

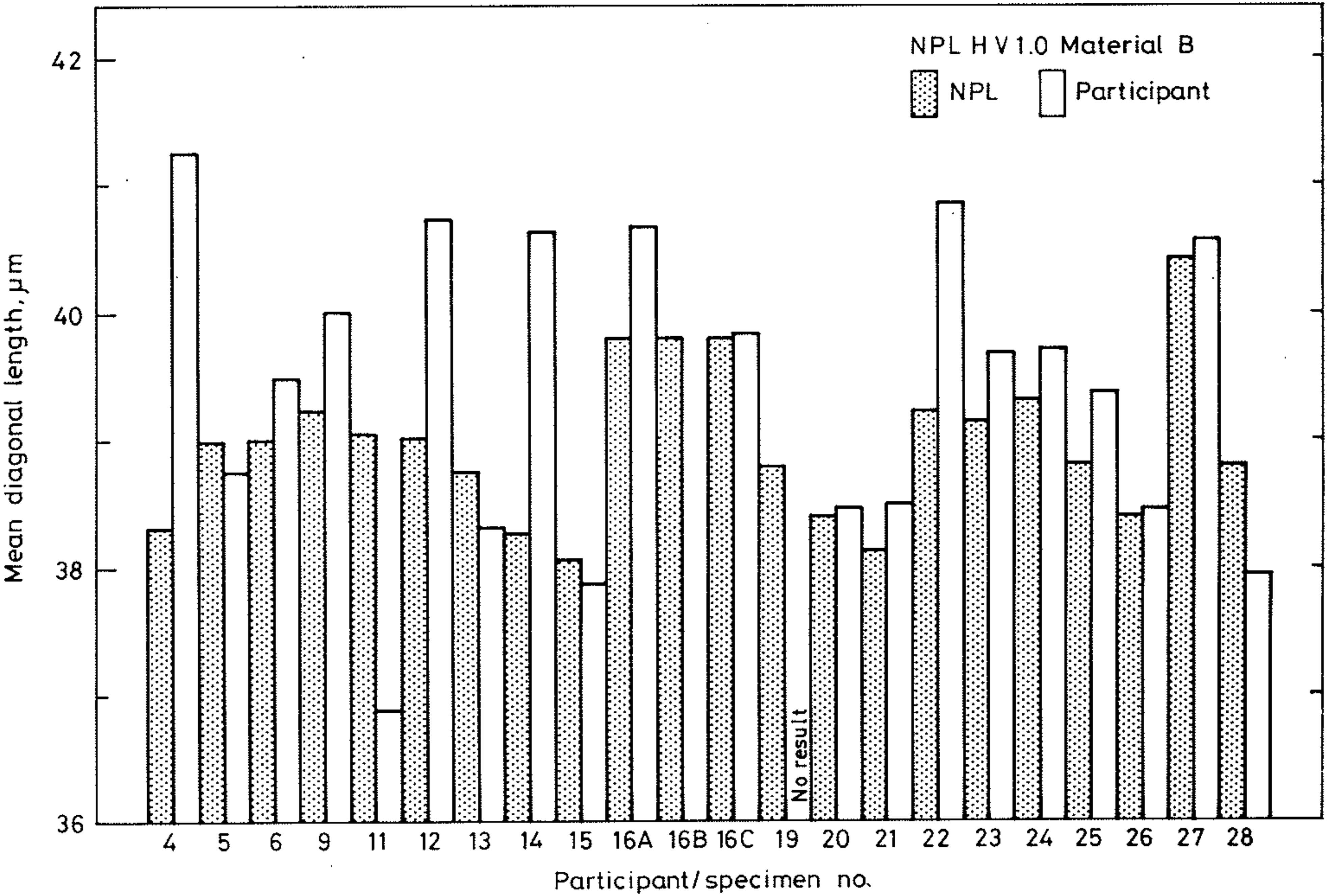

Figure 8 Comparison of mean HV1.0 indentation diagonal lengths in material B placed by NPL as measured by NPL and by participants.

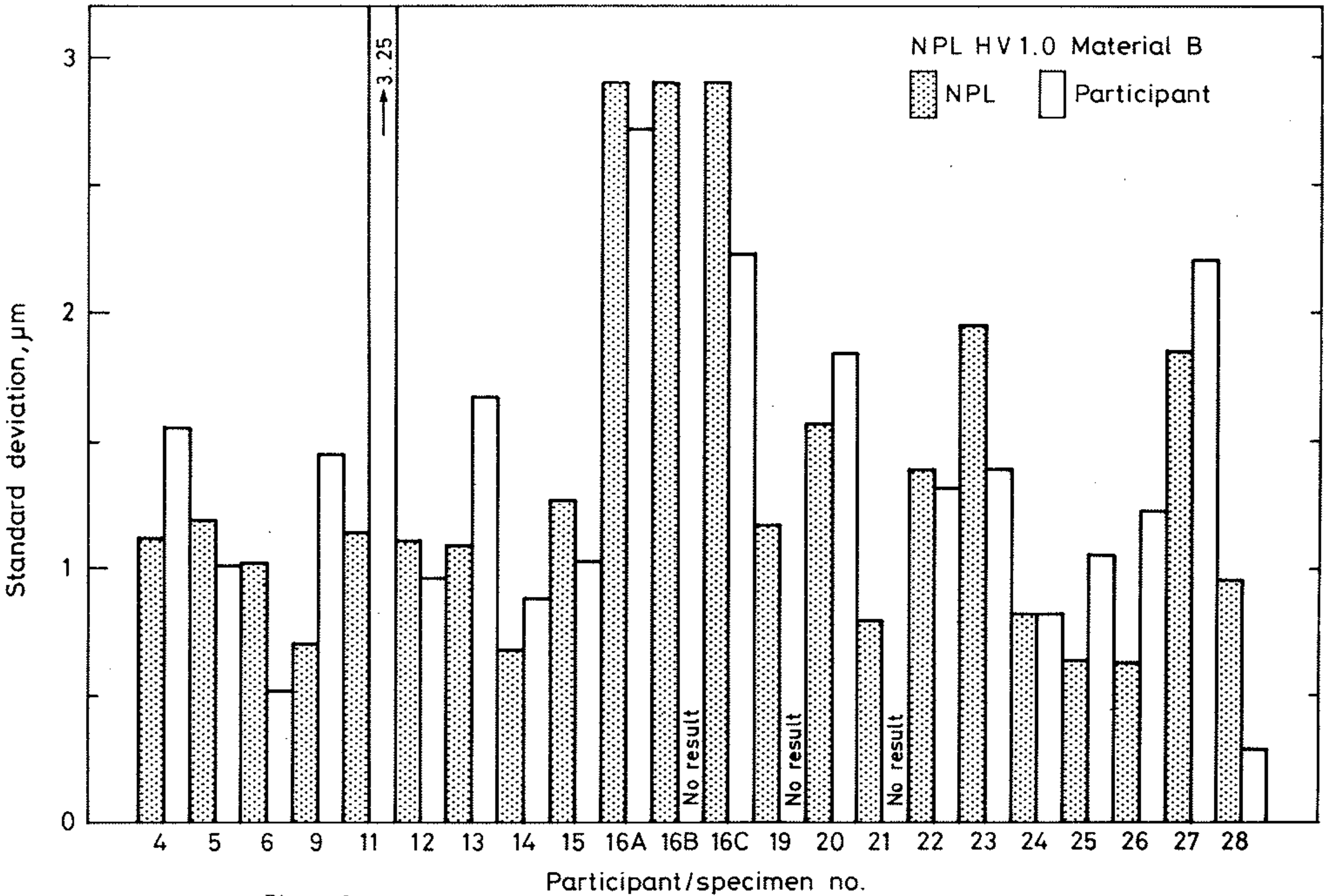

Figure 9 Comparison of standard deviations of diagonal lengths of HV1.0 indentations in material B placed by NPL as measured by NPL and by participants.

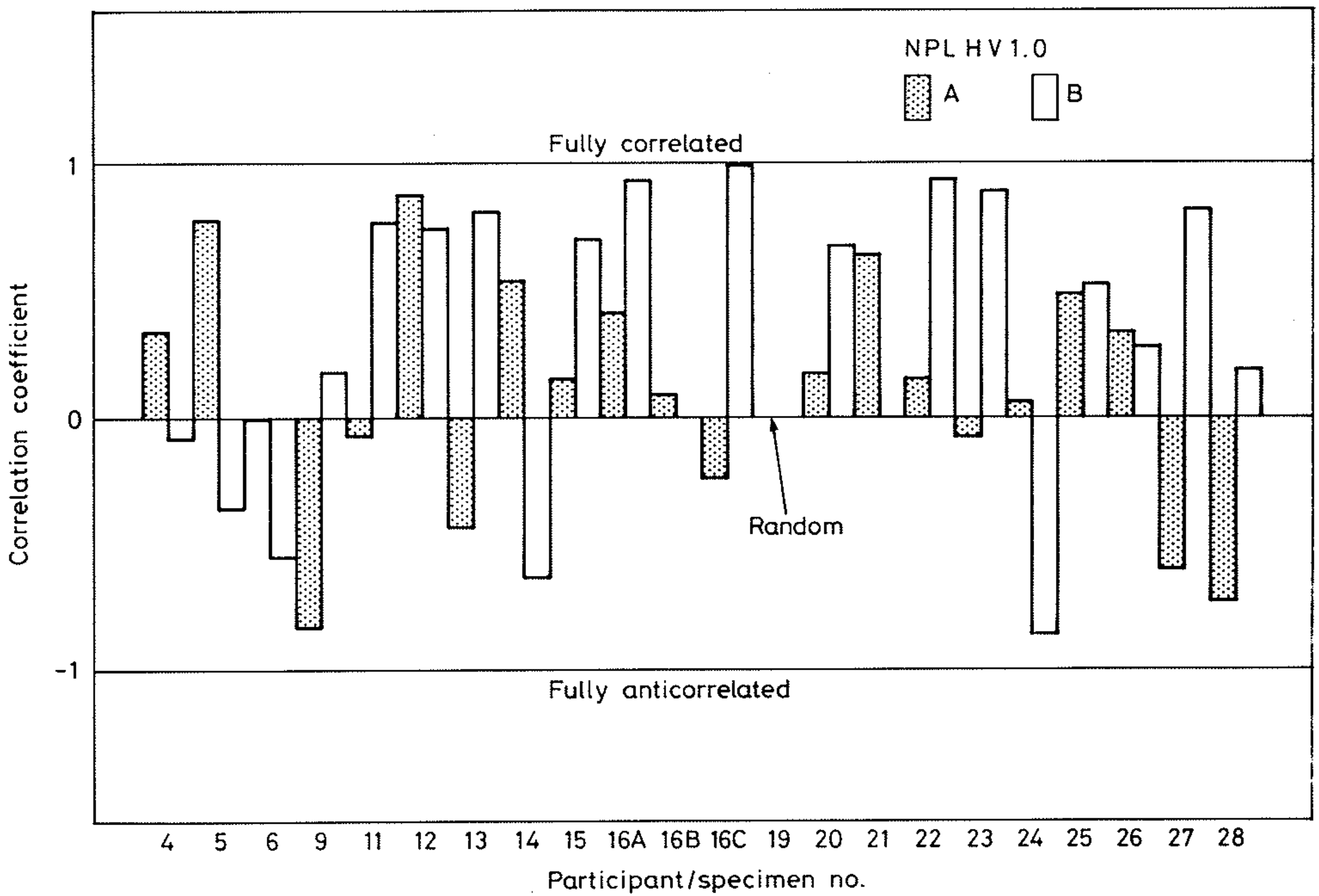

Figure 10 Comparison of correlation coefficients for sets of NPL and participants' measurements of NPL HV1.0 indentations in materials A and B.

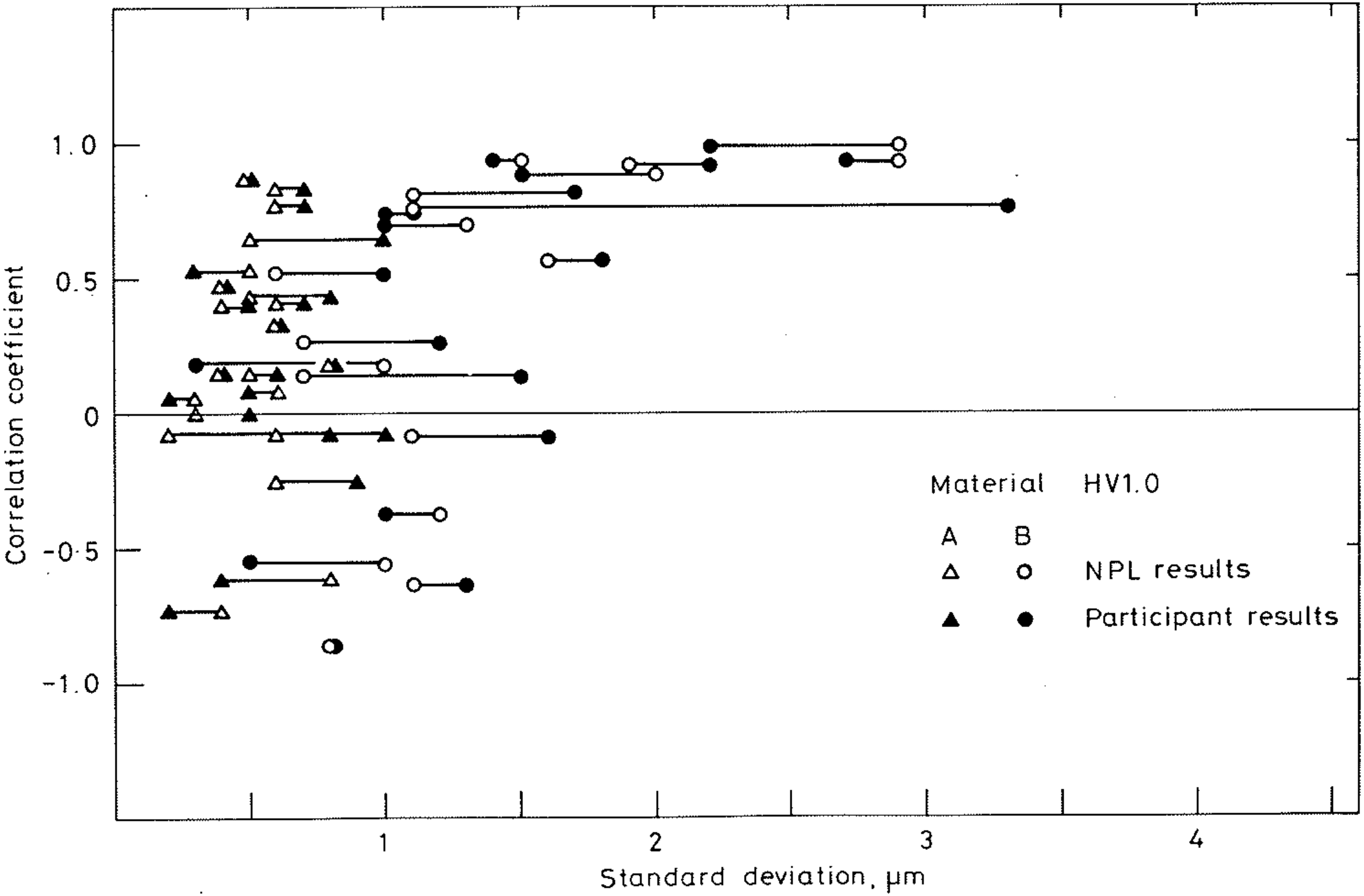

Figure 11 Relationship between correlation coefficient and standard deviation for sets of individual measurements of mean diagonal length of NPL HV1.0 indentations as measured by NPL and by participants.

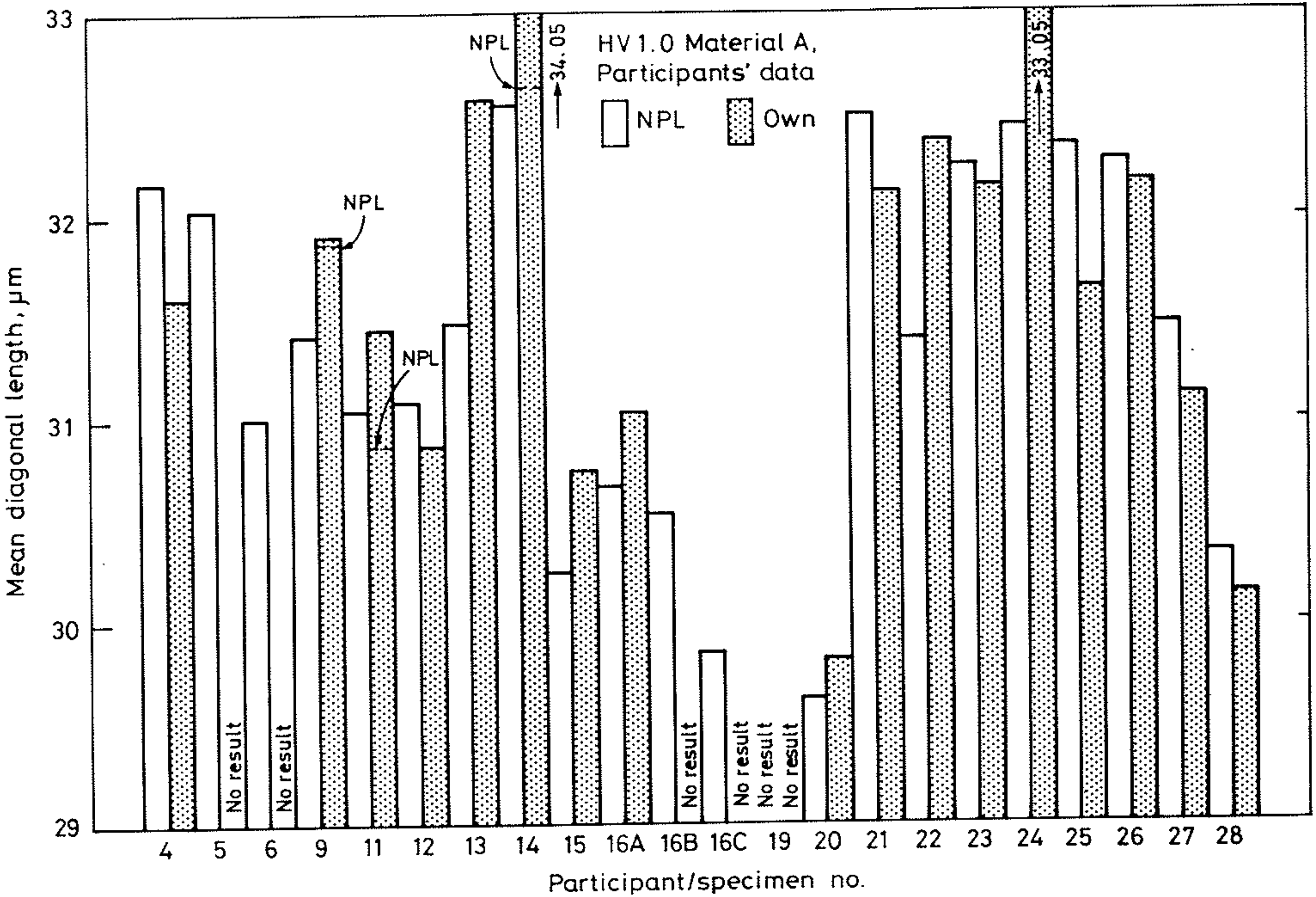

Figure 12 Comparison of mean diagonal lengths of HV1.0 indentations in material as placed by NPL and by the participants, as measured by the participants. Check measurements by NPL are shown as broken lines.

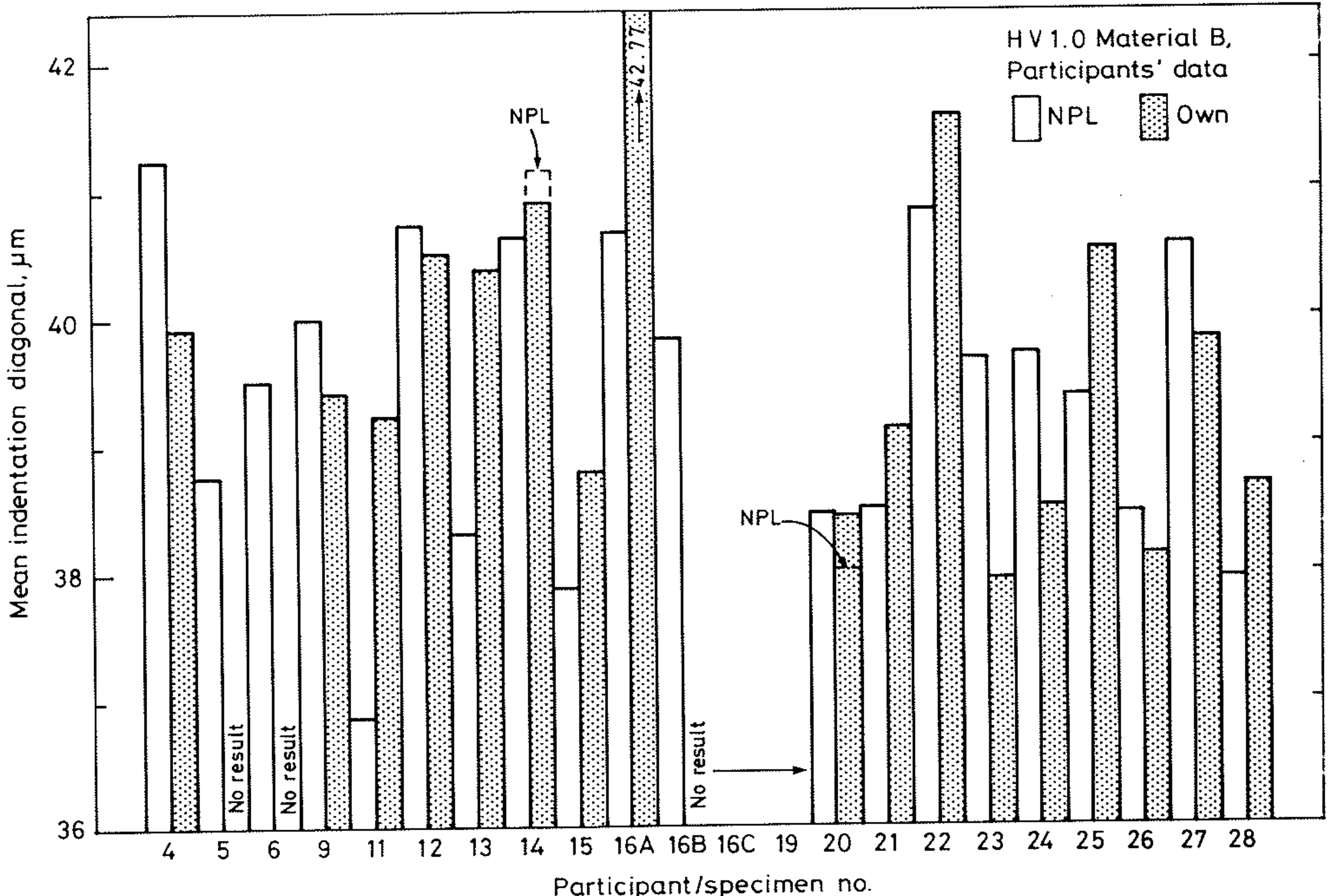

Figure 13 Comparison of mean diagonal lengths of HV1.0 indentations in material B placed by NPL and by the participants, as measured by the participants. Check measurements by NPL are shown as broken lines.

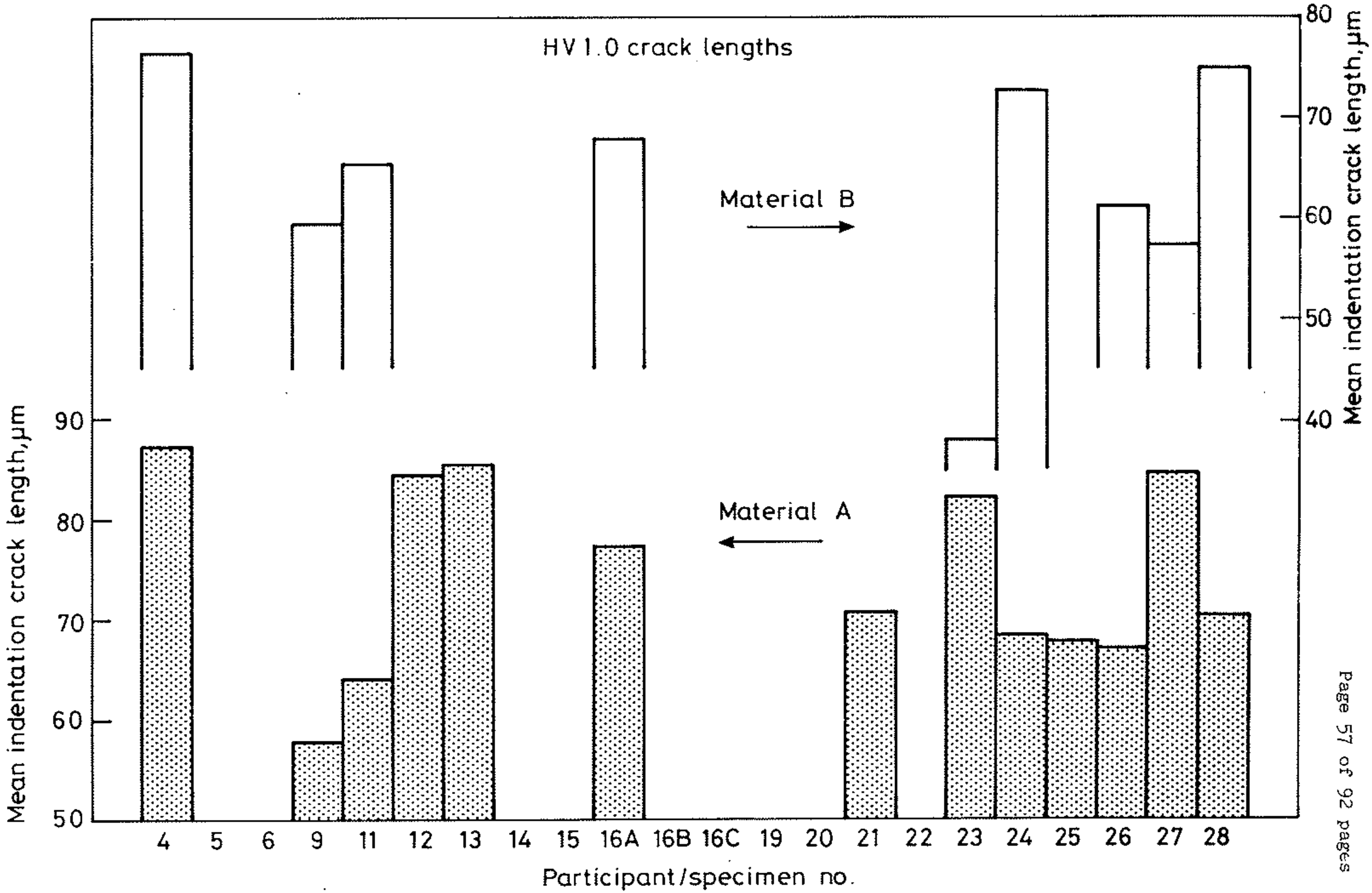

Figure 14 Measurements of total radial crack lengths emanating from corners of HV1.0 indentations placed by the participants in both materials.

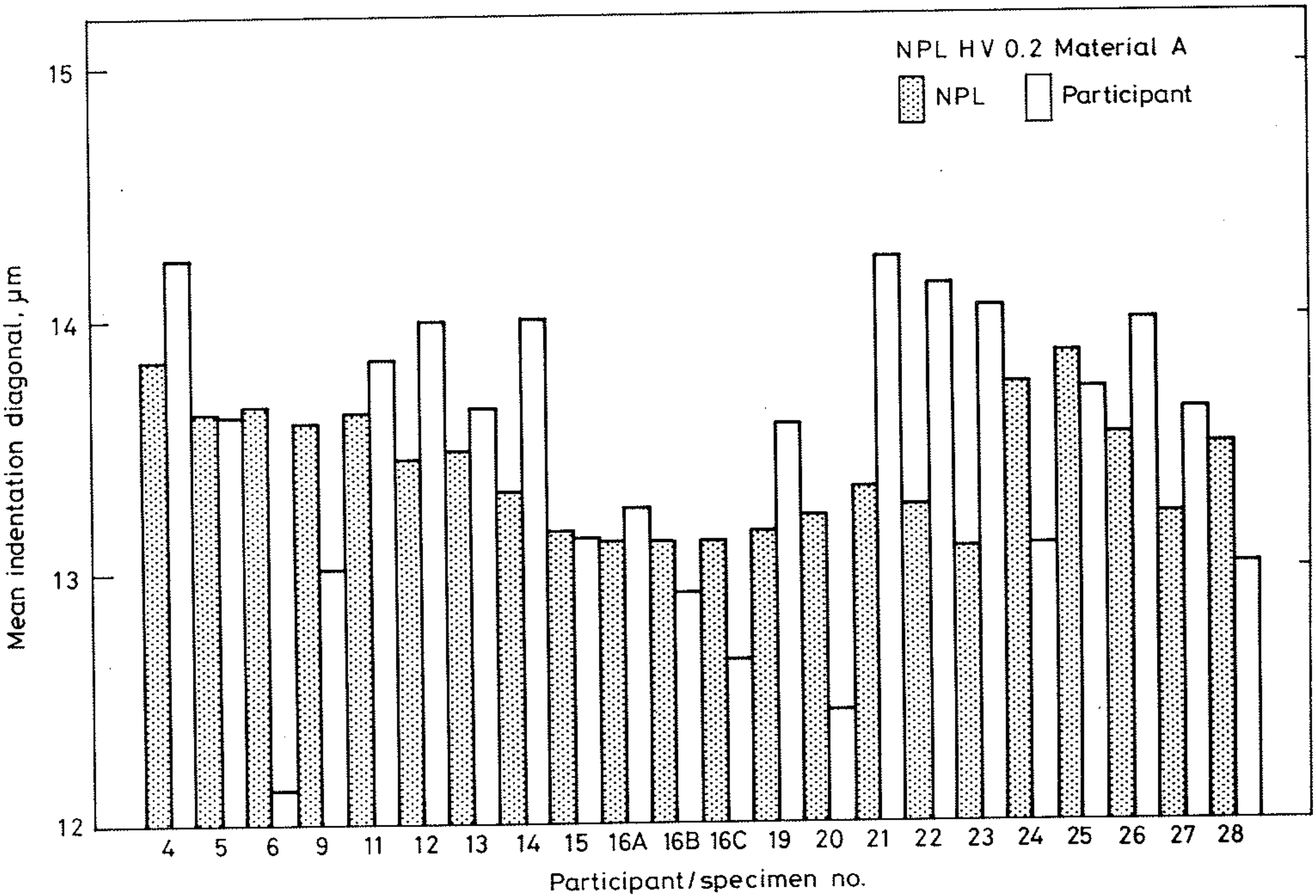

Figure 15 Comparison of mean HV0.2 indentation diagonal lengths in material A placed by NPL as measured by NPL and by participants.

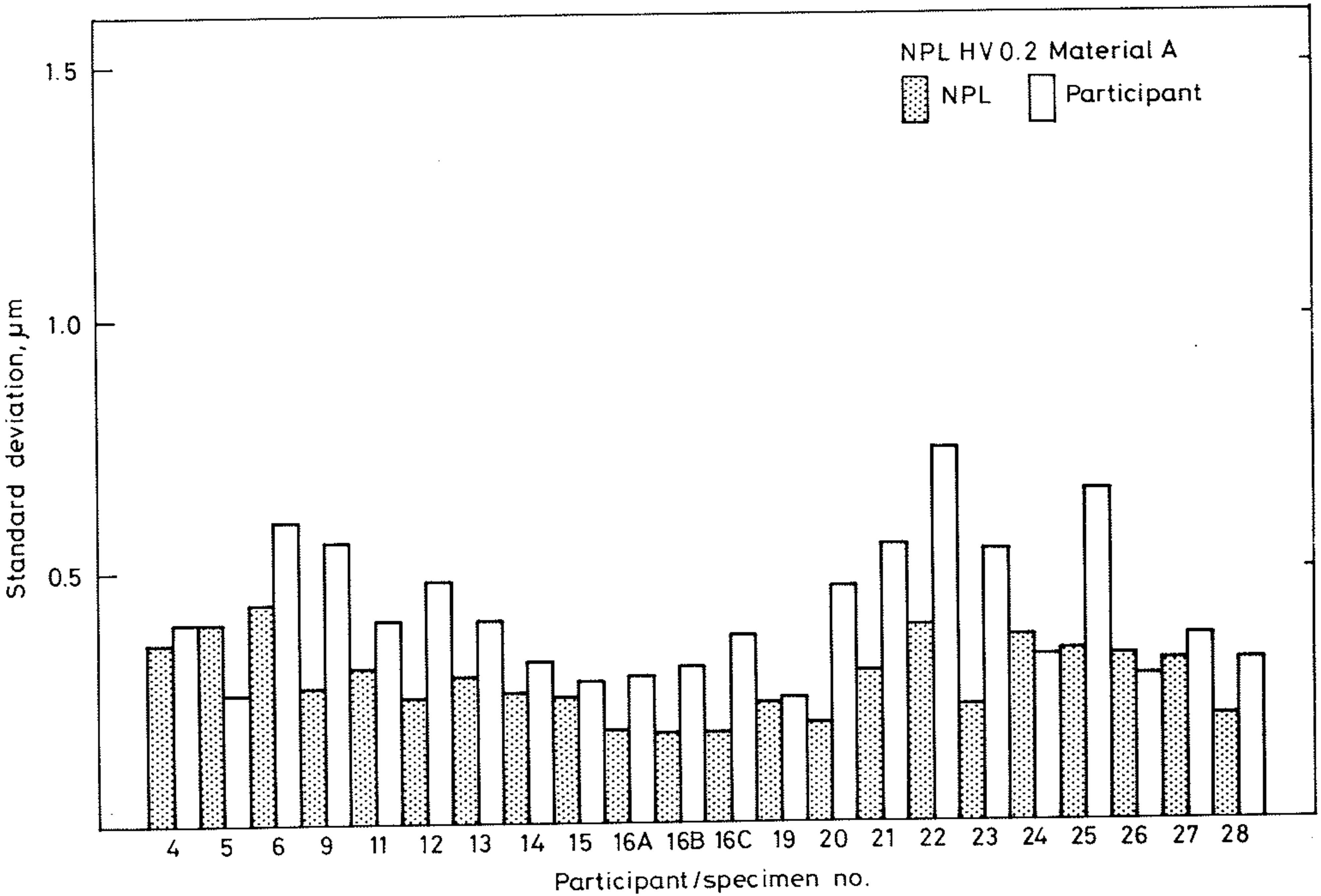

Figure 16 Comparison of standard deviations of diagonal lengths of HV0.2 indentations in material A placed by NPL as measured by NPL and by participants.

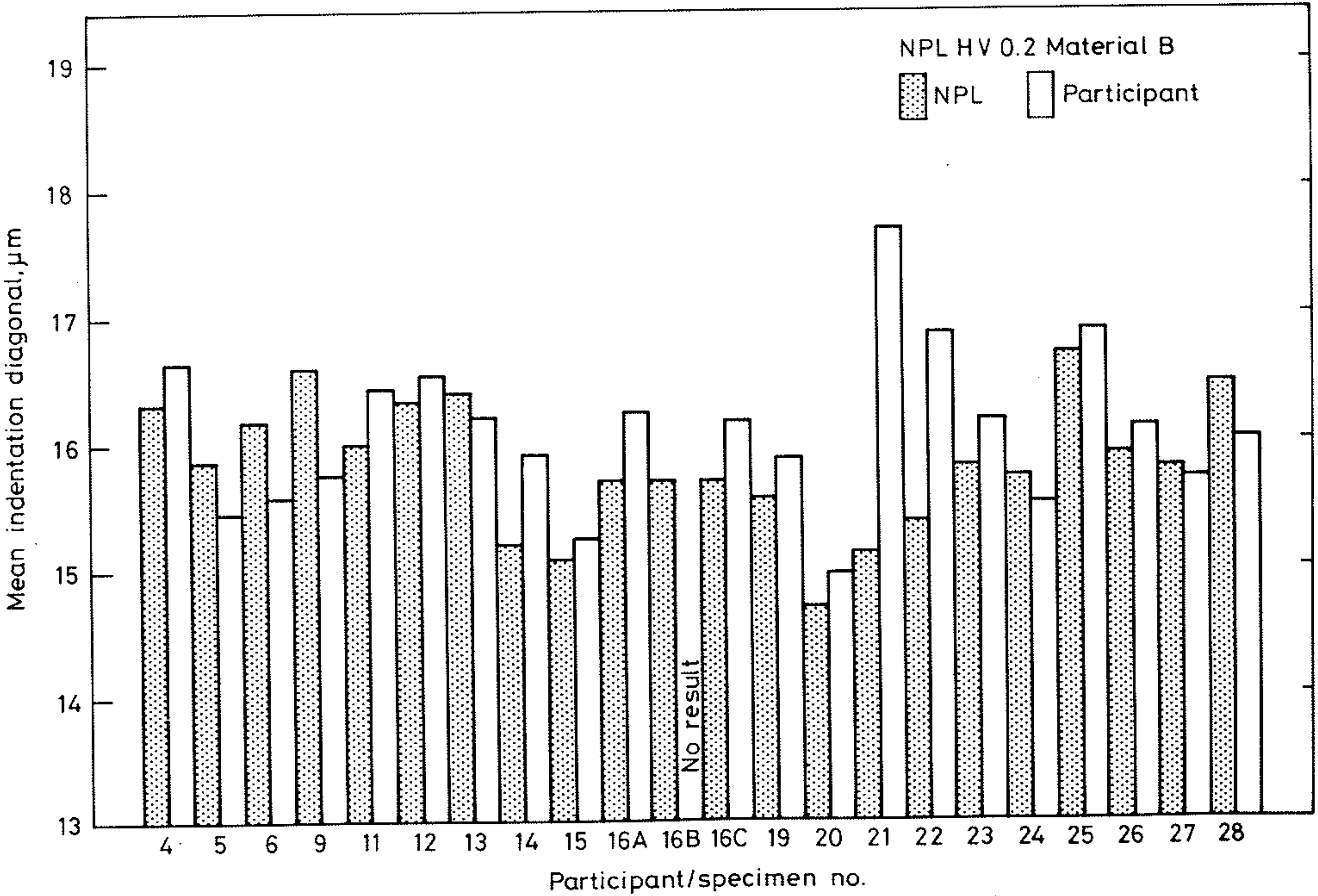

Figure 17 Comparison of mean HV0.2 indentation diagonal lengths in material B placed by NPL as measured by NPL and by participants.

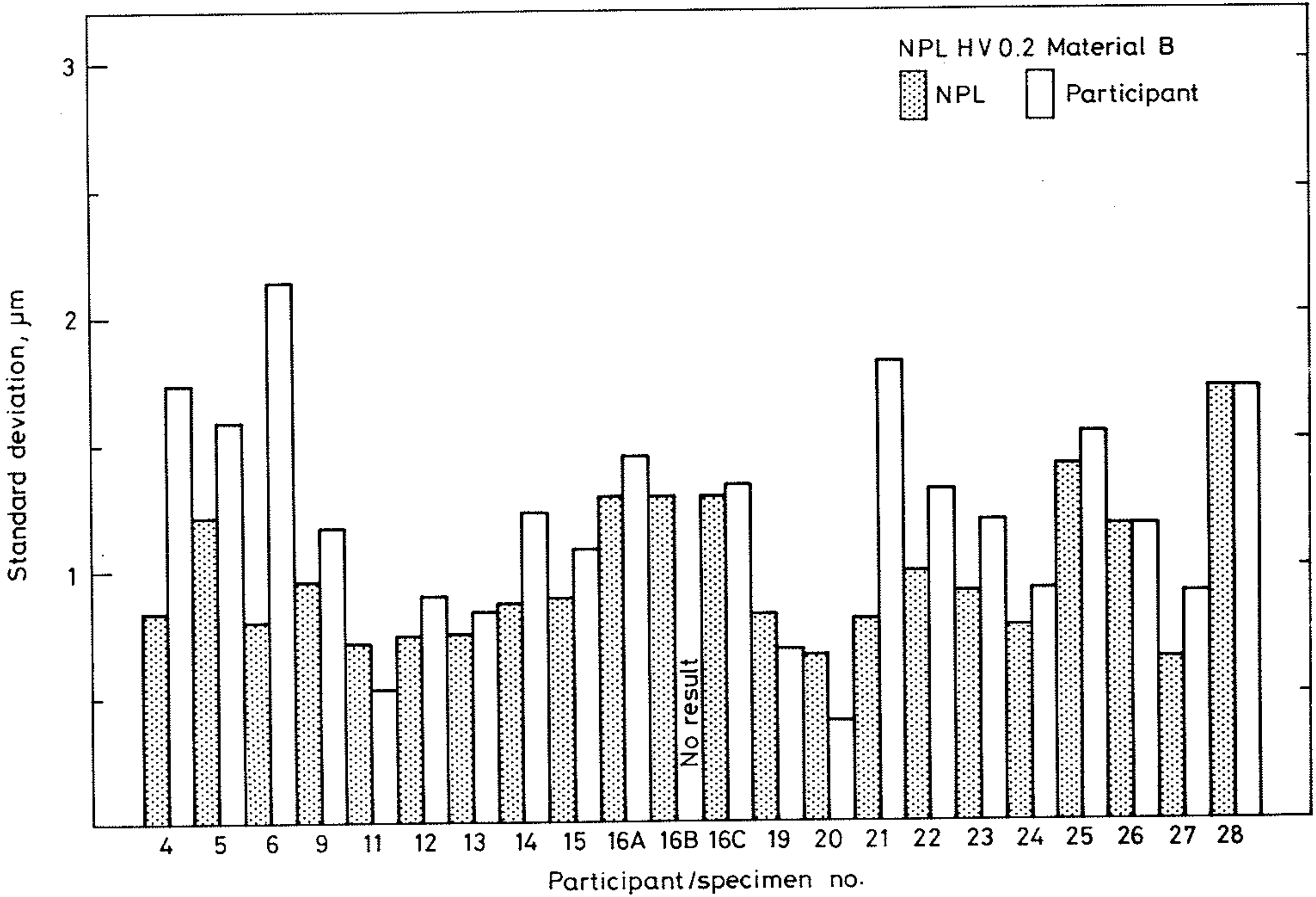

Figure 18 Comparison of standard deviations of diagonal lengths of HV0.2 indentations in material B placed by NPL as measured by NPL and by participants.

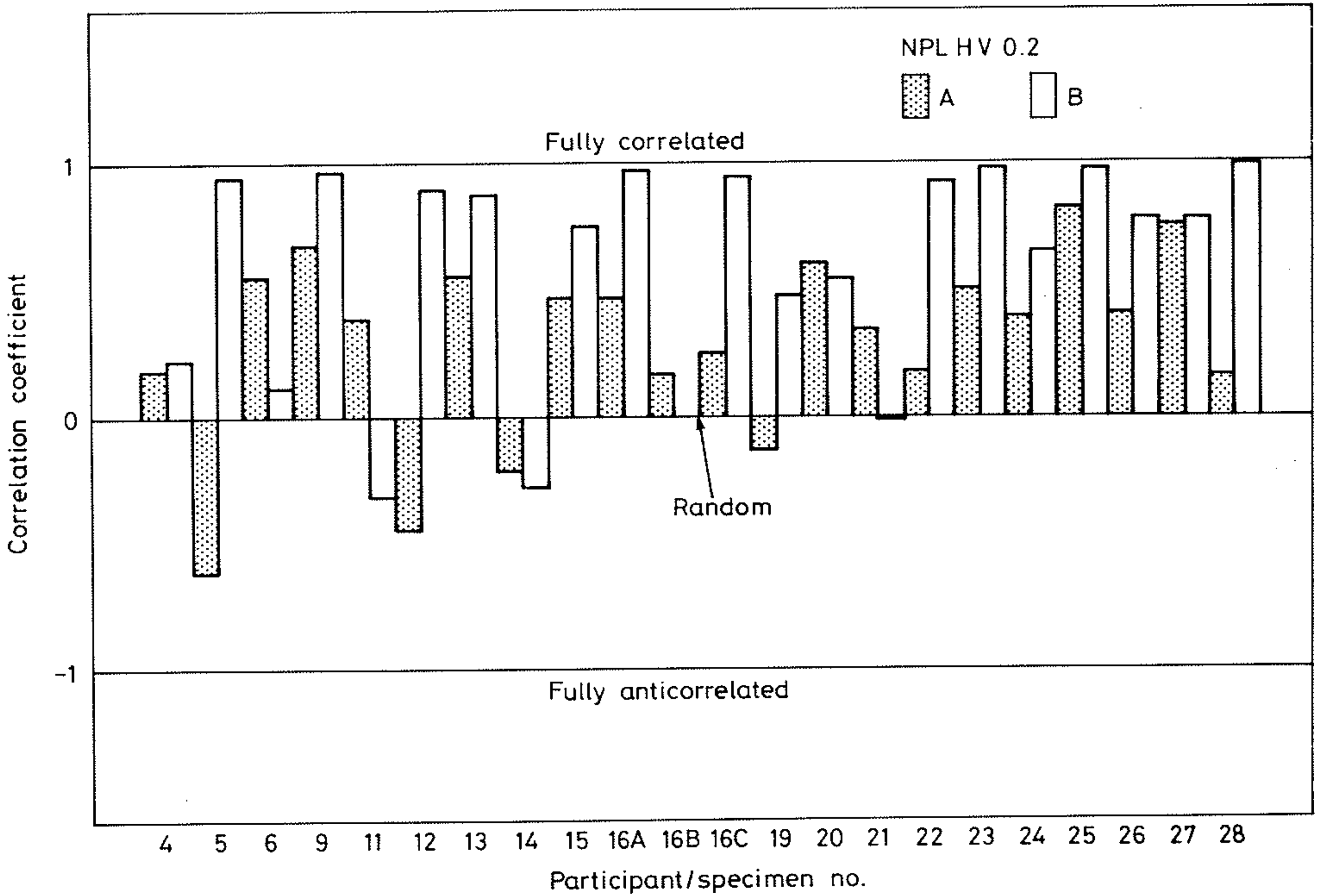

Figure 19 Comparison of correlation coefficients for sets of NPL and participants' measurements of NPL HV0.2 indentations in materials A and B.

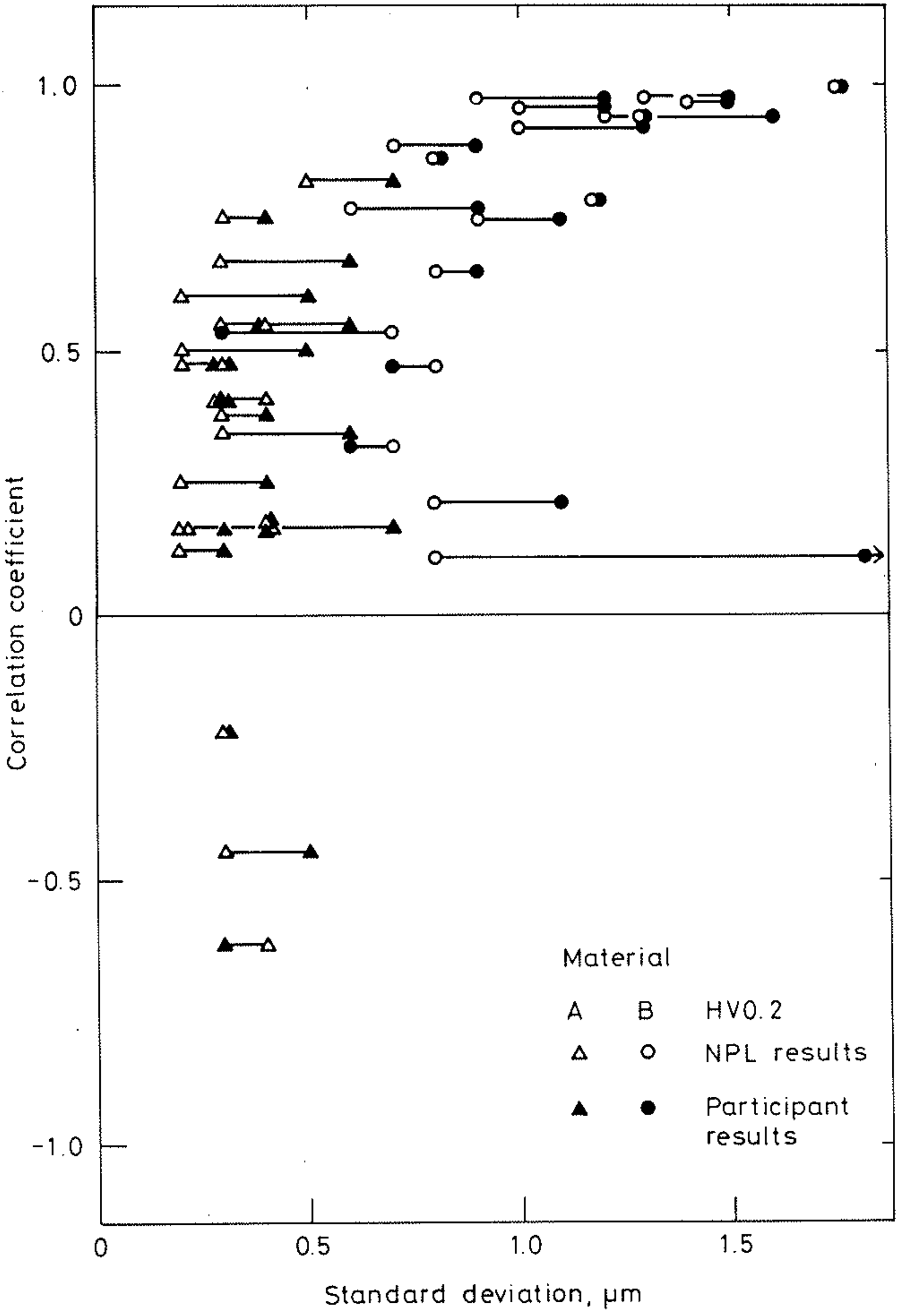

Figure 20 Relationship between correlation coefficient and standard deviation for sets of individual measurements of mean diagonal length of NPL HV0.2 indentations as measured by NPL and by participants.

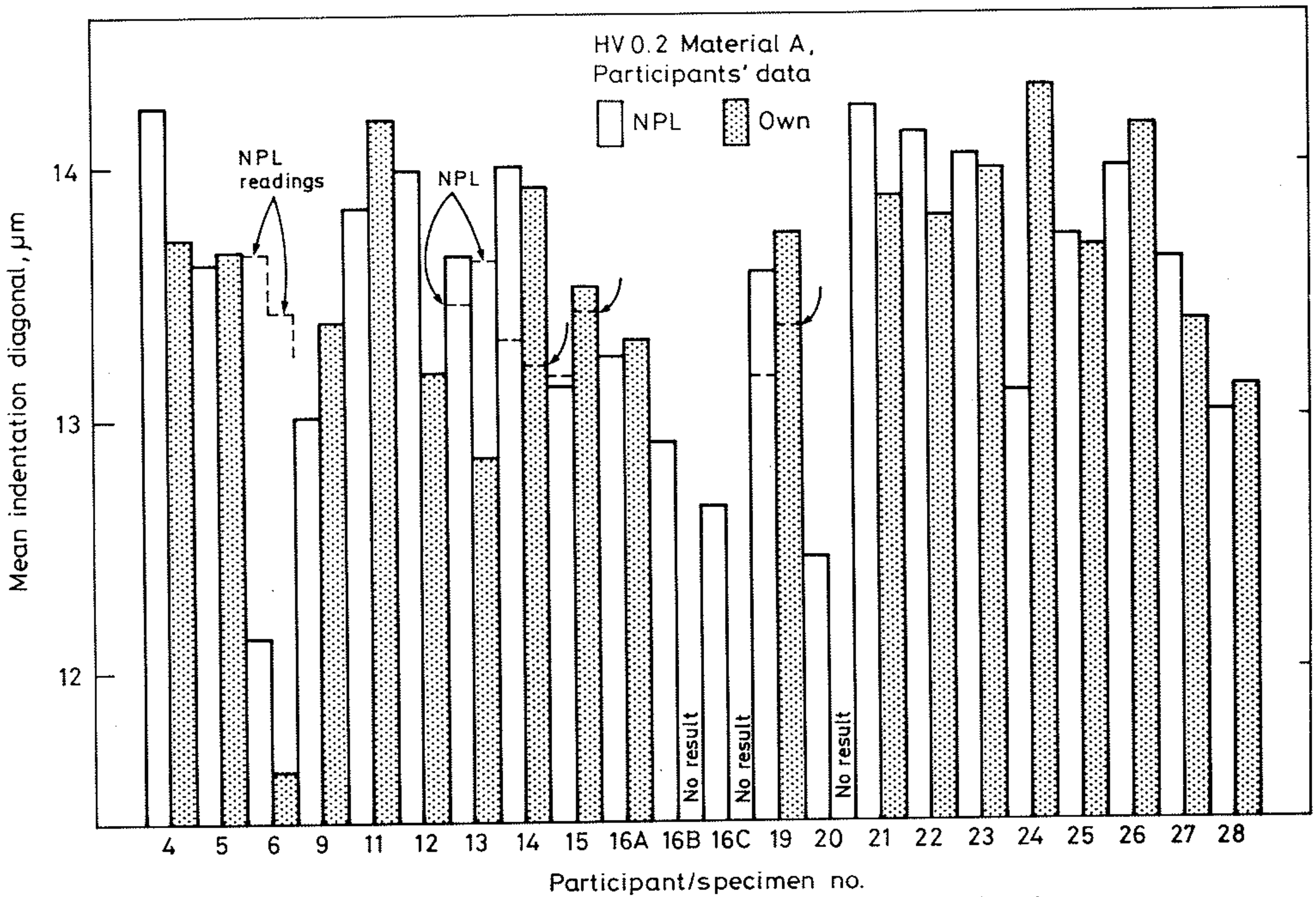

Figure 21 Comparison of mean diagonal lengths of HV0.2 indentations in material A placed by NPL and by the participants, as measured by the participants. Check measurements by NPL are shown as broken lines.

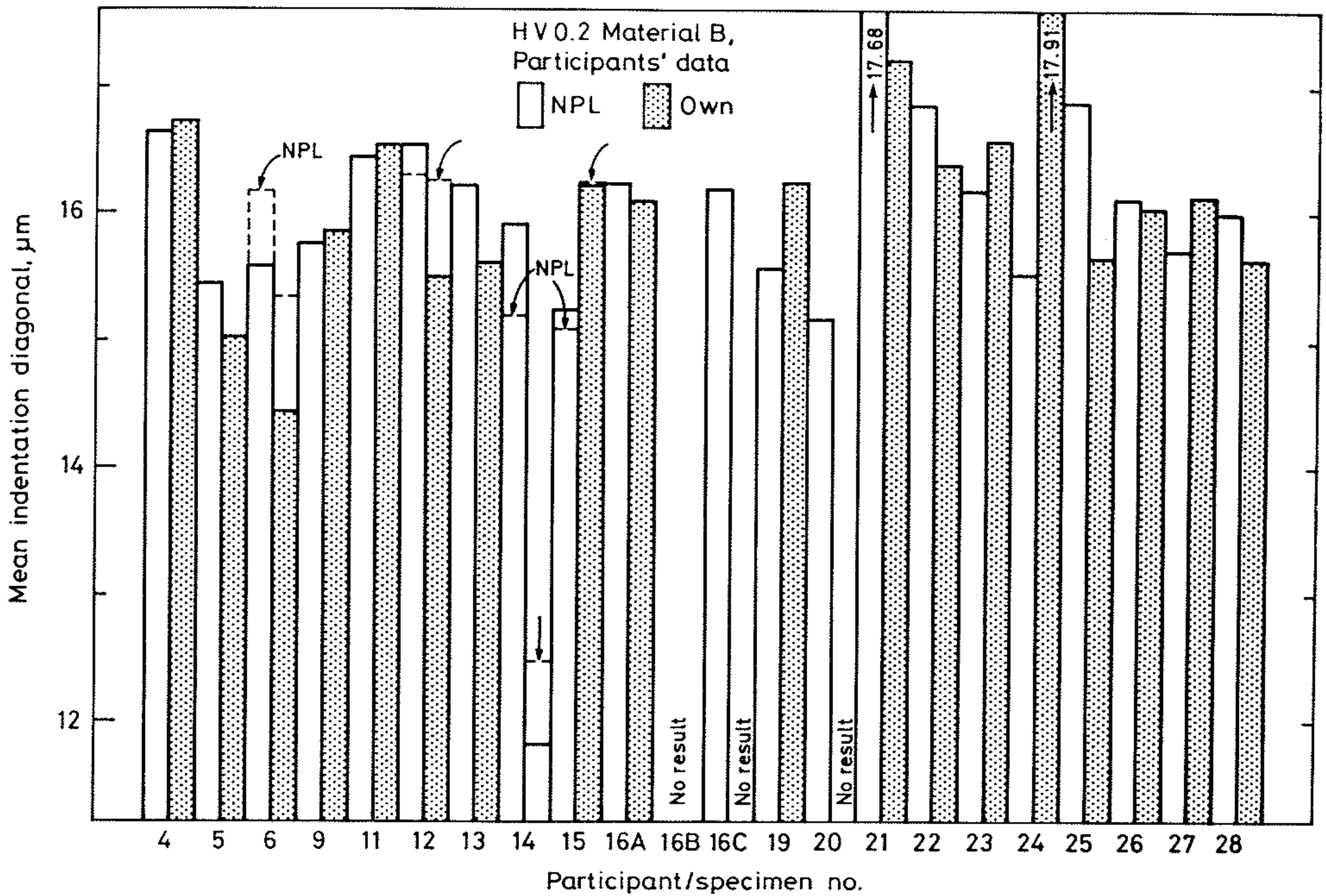

Figure 22 Comparison of mean diagonal lengths of HV0.2 indentations in material B placed by NPL and by the participants, as measured by the participants. Check measurements by NPL are shown as broken lines.

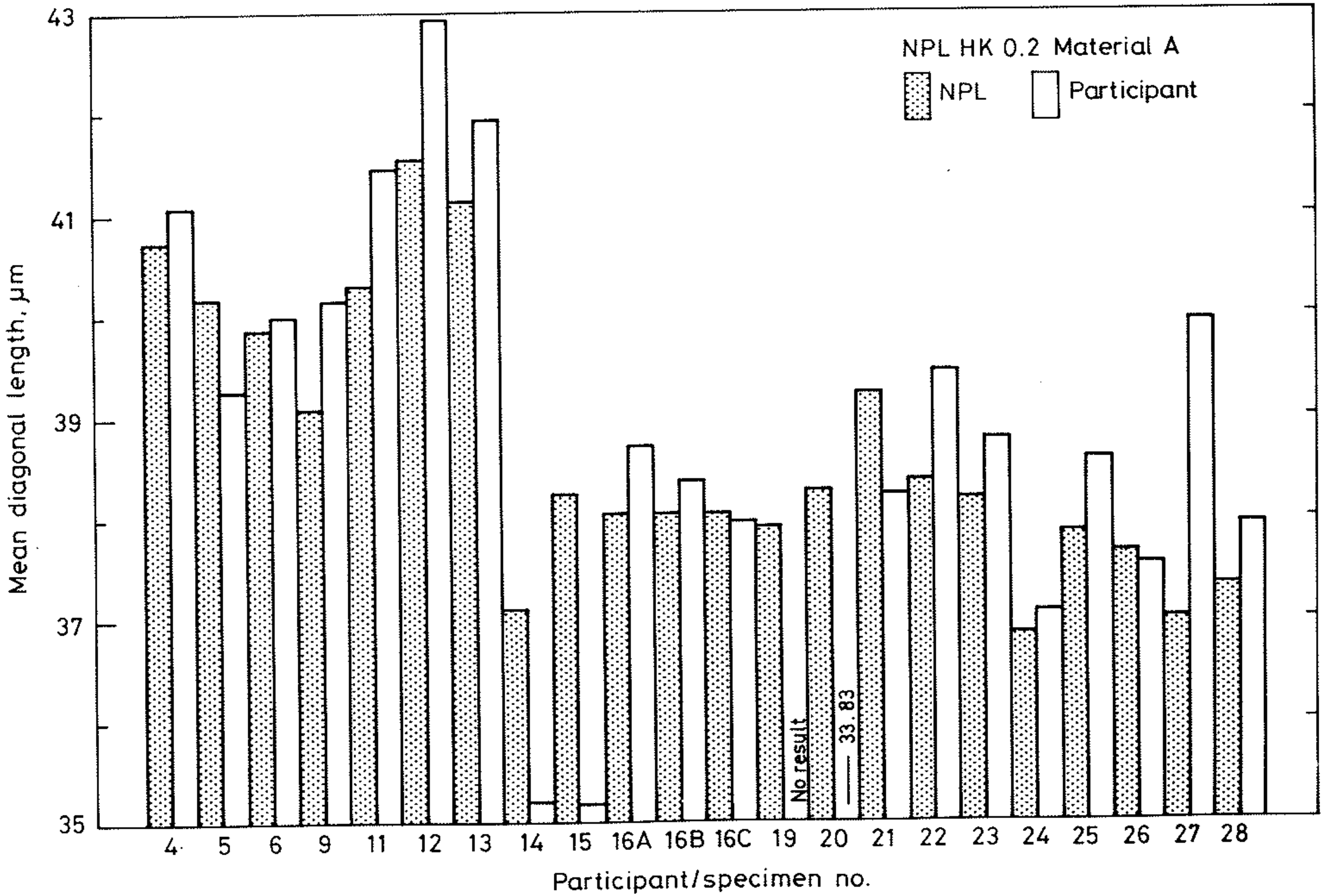

Figure 23 Comparison of HK0.2 indentation diagonal lengths placed by NPL in material A as measured by NPL and by the participants.

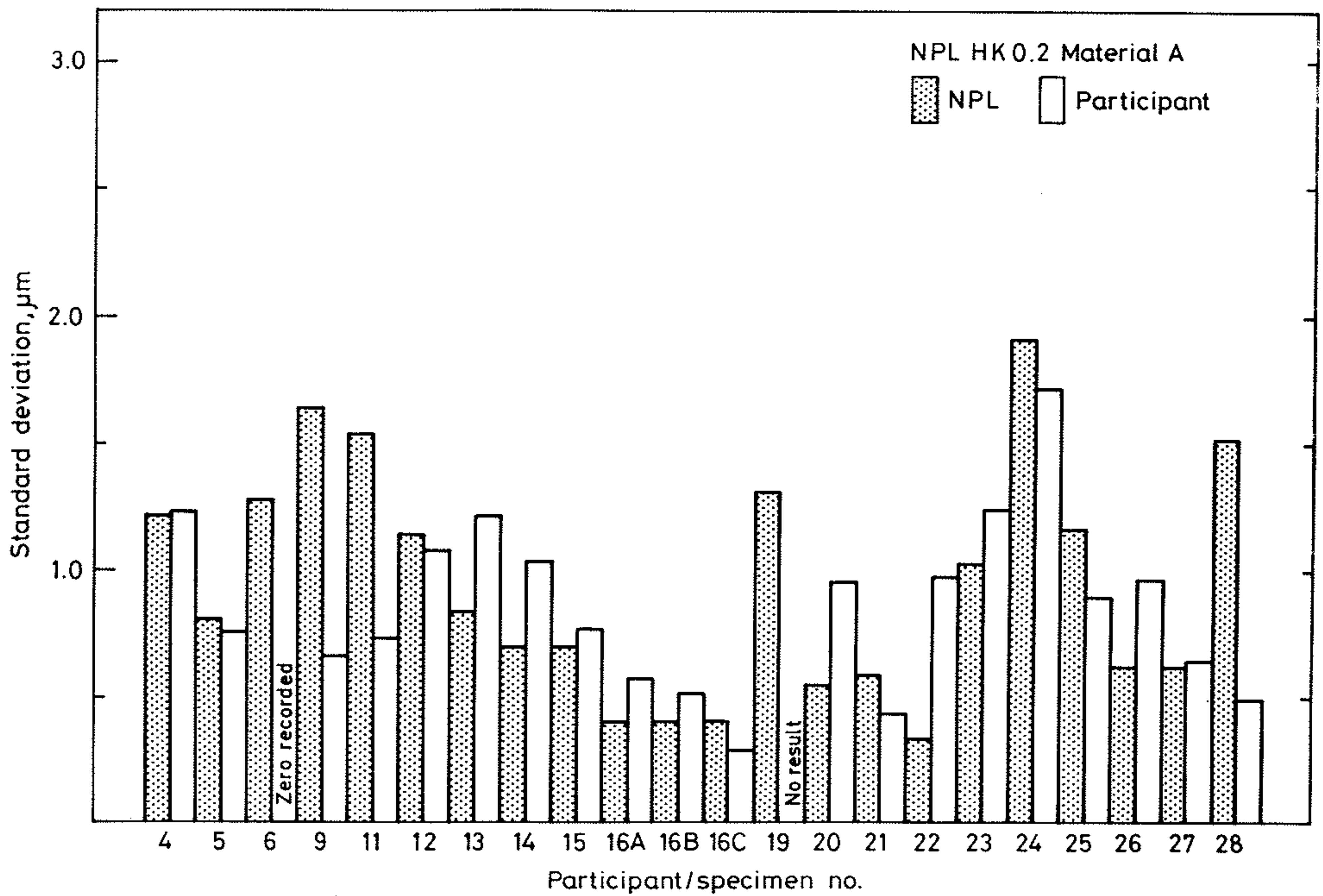

Figure 24 Comparison of standard deviations of diagonal lengths of HK0.2 indentations placed by NPL in material A as measured by NPL and by the participants.

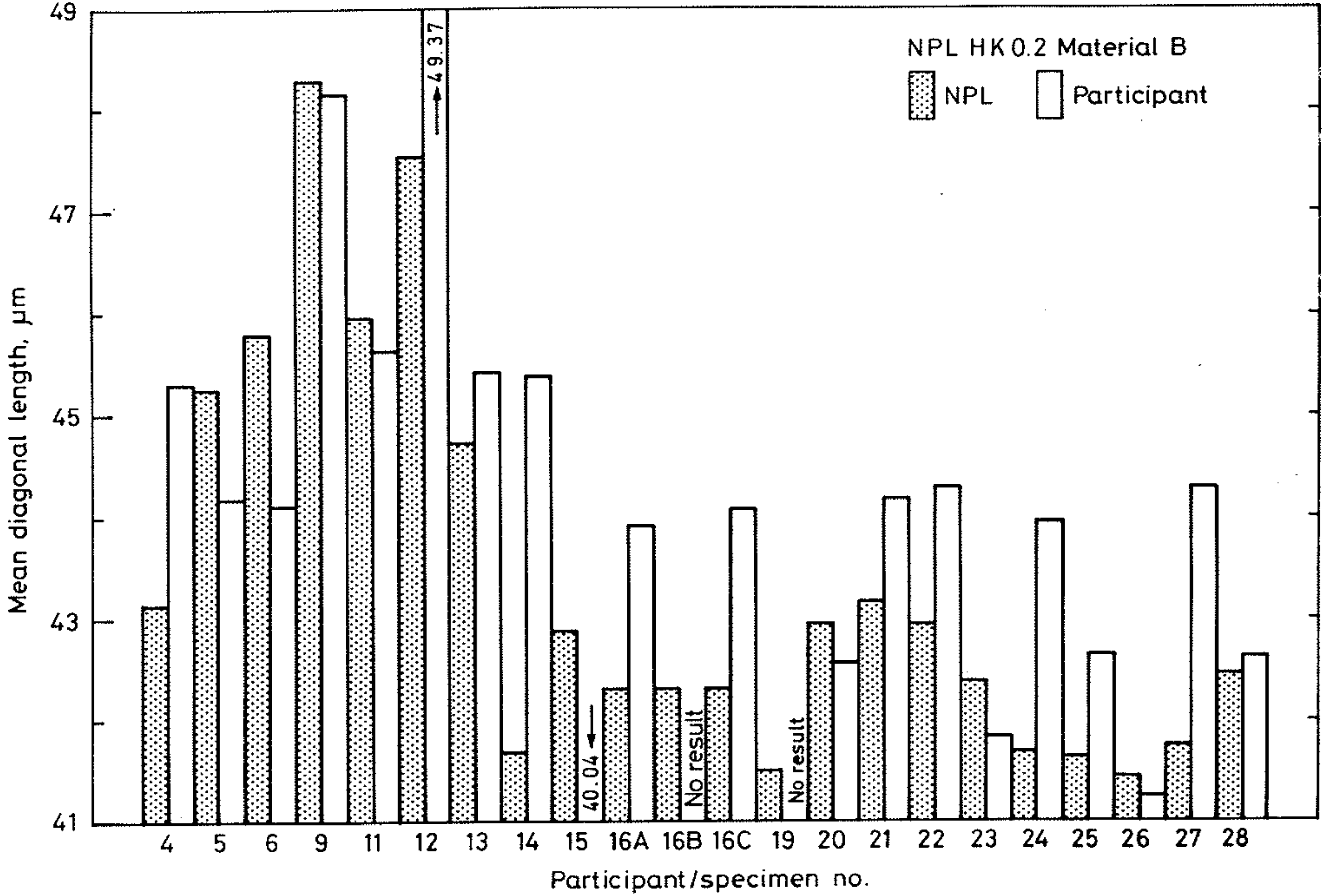

Figure 25 Comparison of mean HK0.2 indentation diagonal lengths placed by NPL in material B as measured by NPL and by the participants.

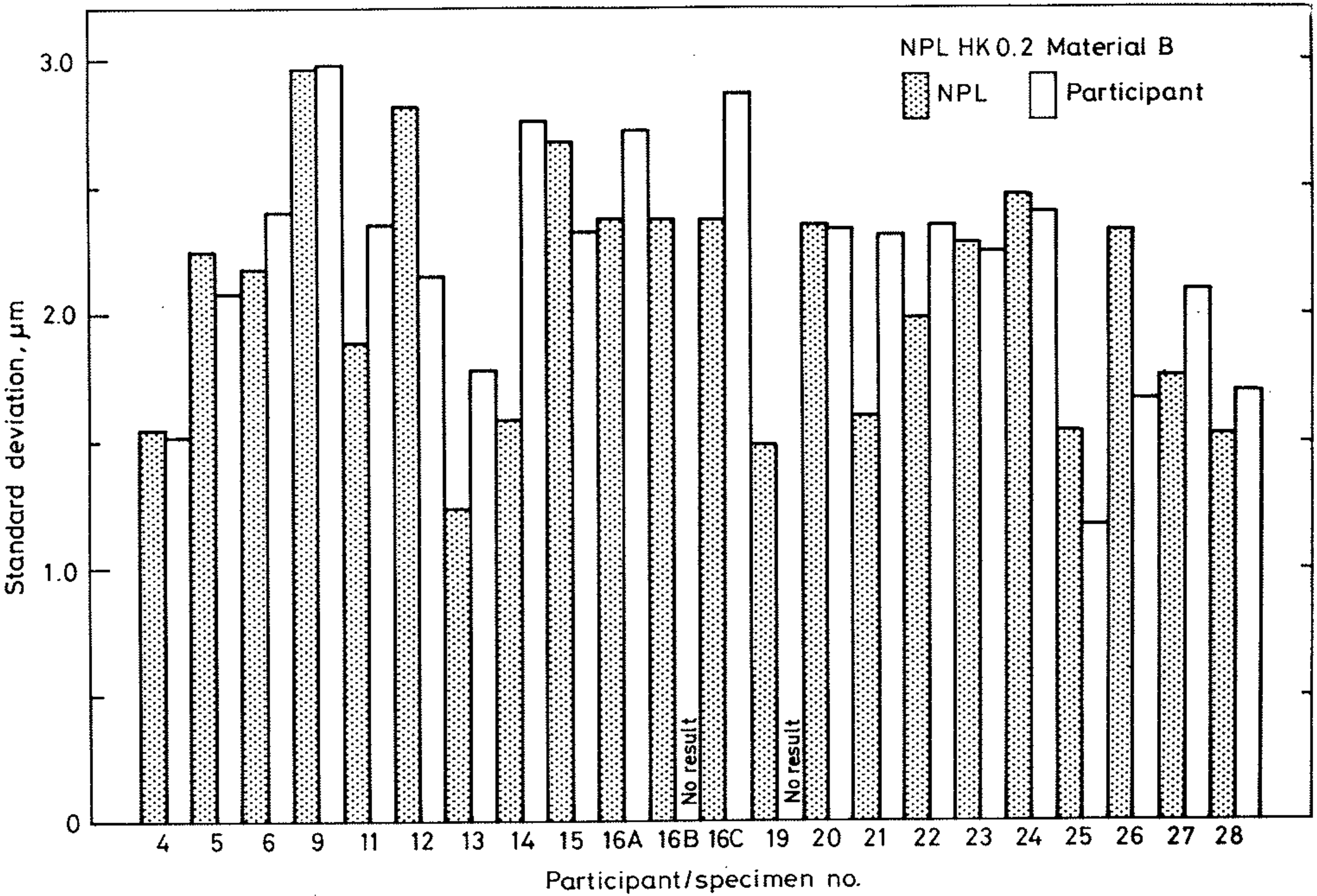

Figure 26 Comparison of standard deviations of diagonal lengths of HK0.2 indentations placed by NPL in material B as measured by NPL and by the participants.

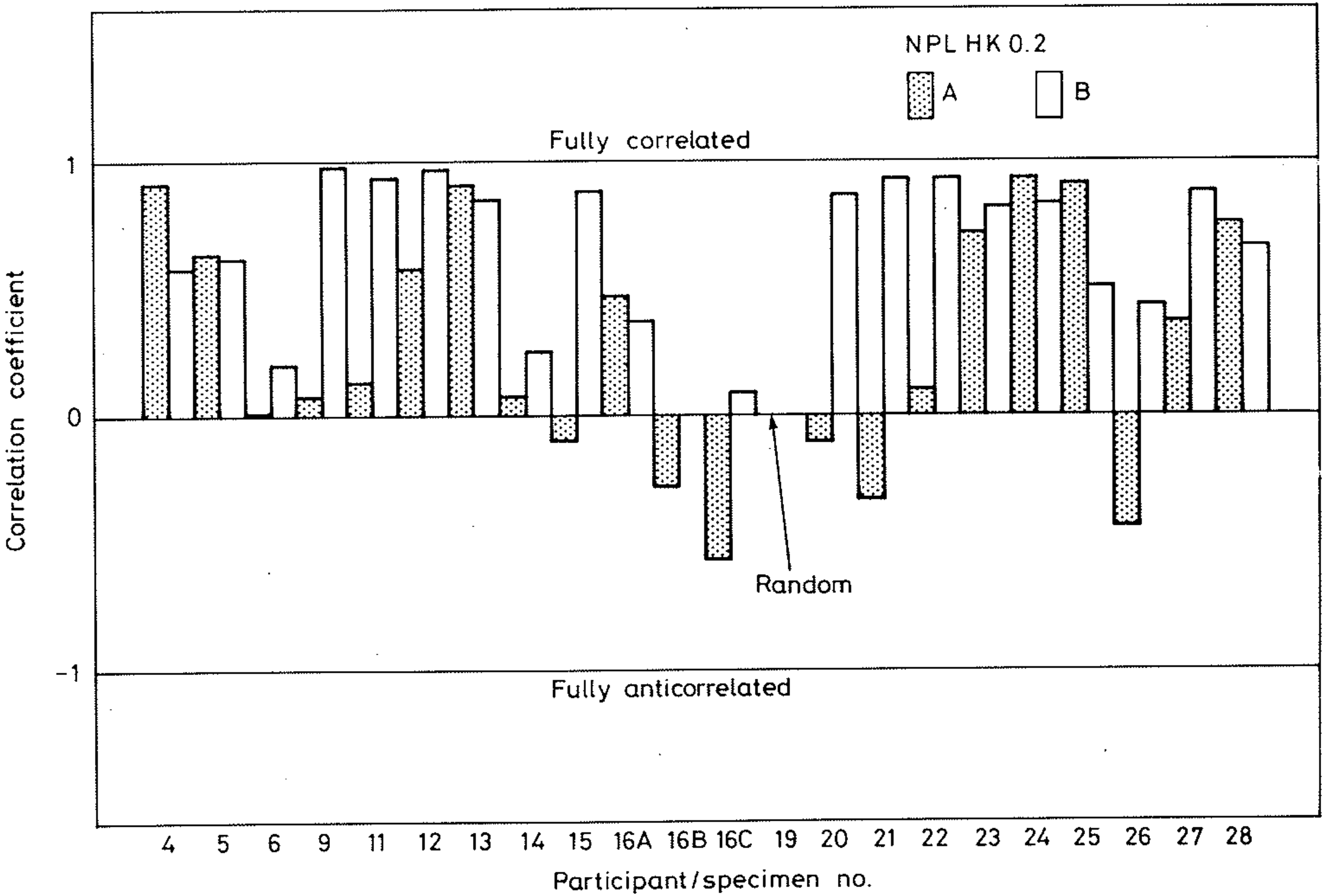

Figure 27 Comparison of correlation coefficients for sets of NPL and participants' measurements of NPL HK0.2 indentations in materials A and B.

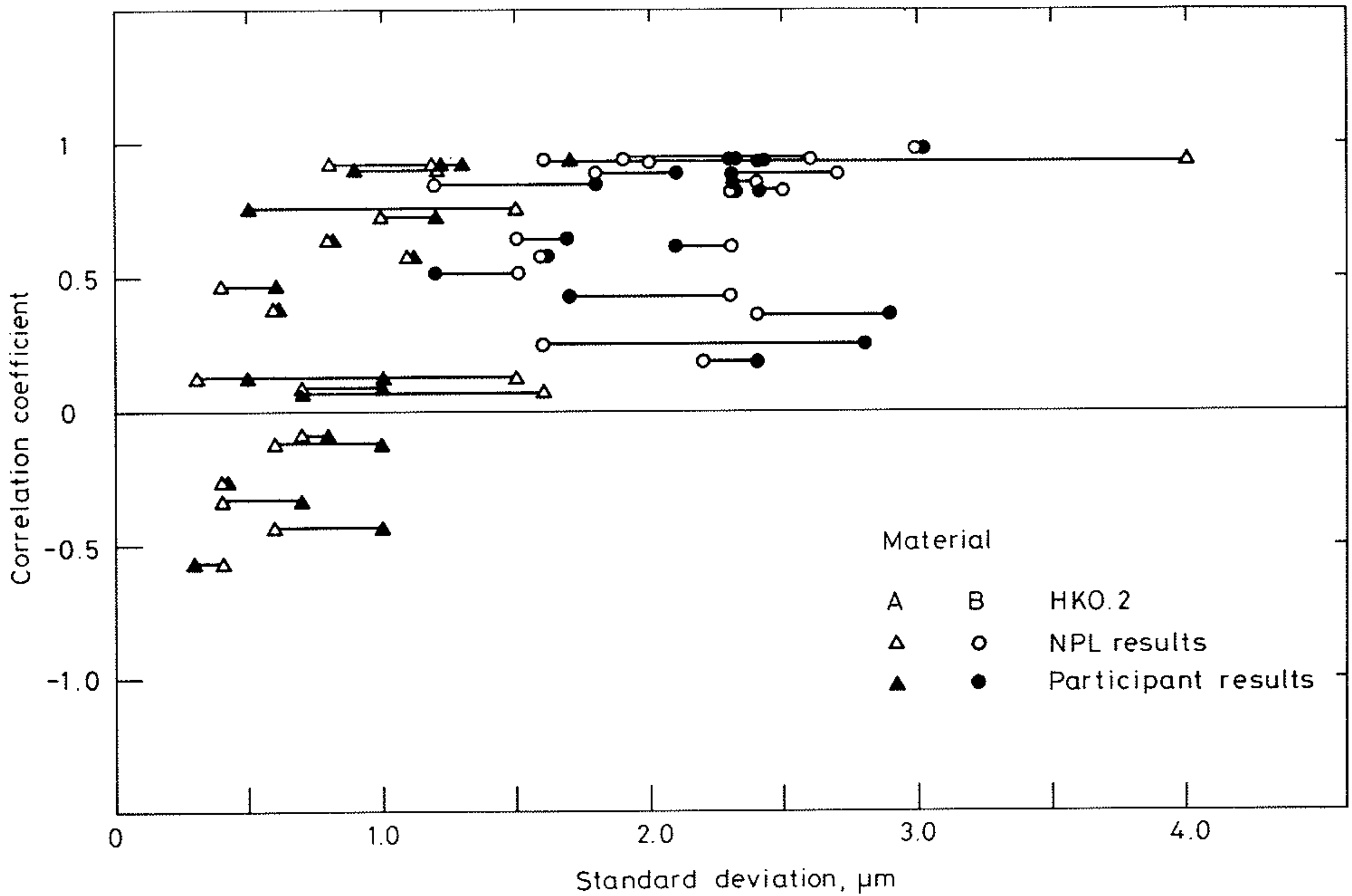

Figure 28 Relationship between correlation coefficient and standard deviation for sets of individual measurements of mean diagonal length of NPL HKO.2 indentations as measured by NPL and by participants.

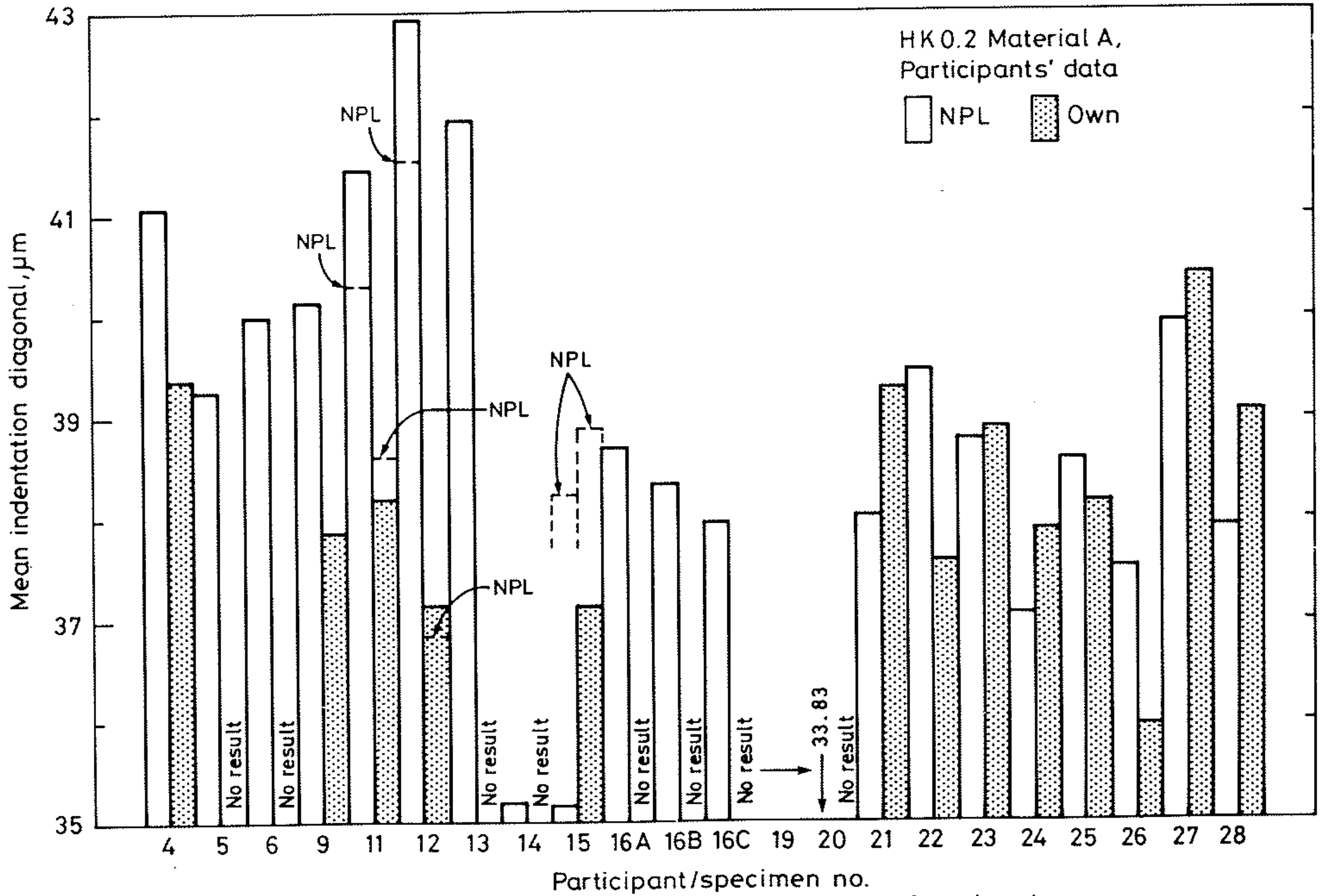

Figure 29 Comparison of mean diagonal lengths of HK0.2 indentations in material A placed by NPL and by the participants, as measured by the participants. Check measurements by NPL are shown as broken lines.

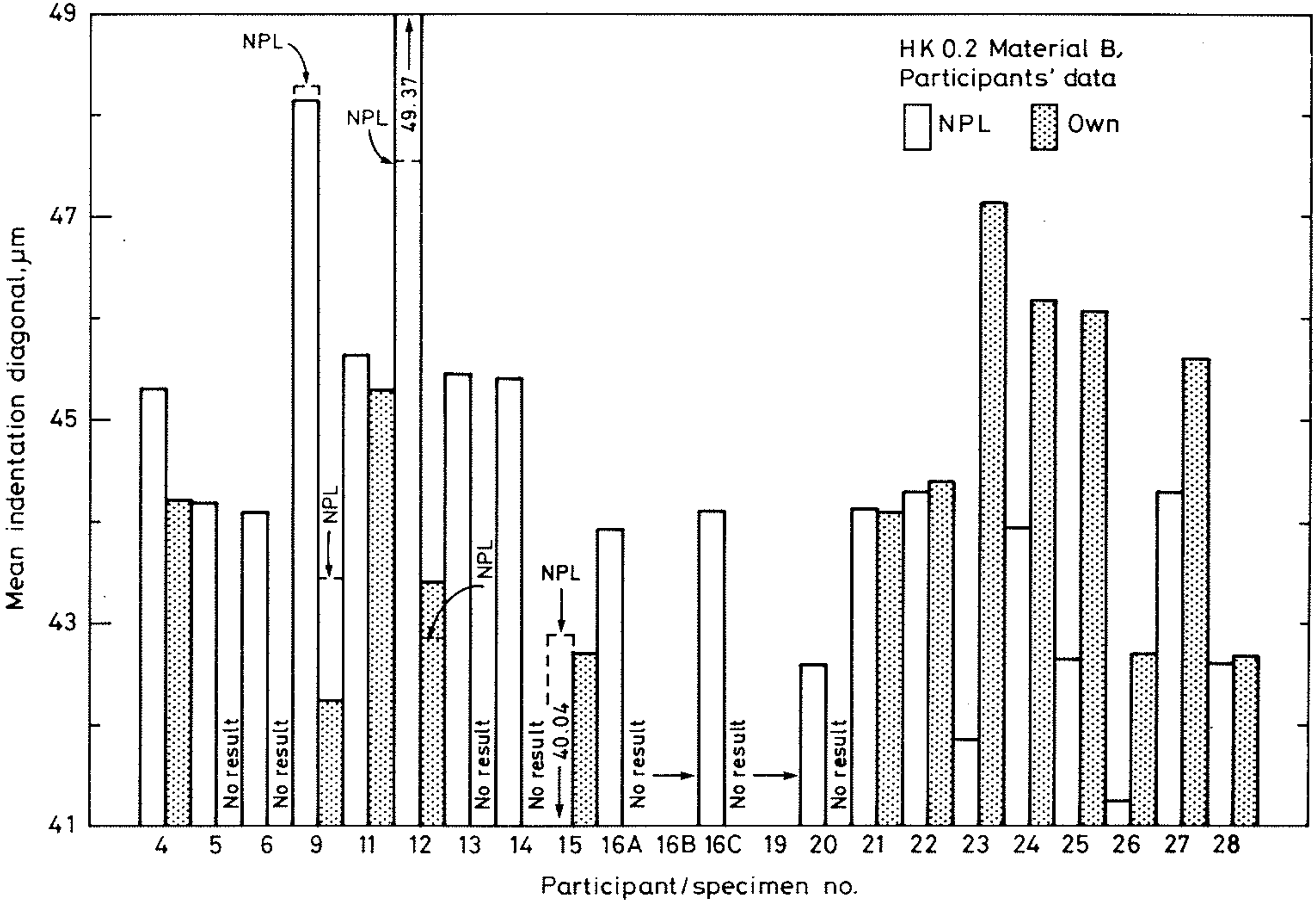

Figure 30 Comparison of mean diagonal lengths of HK0.2 indentations in material B placed by NPL and by the participants, as measured by the participants. Check measurements by NPL are shown as broken lines.

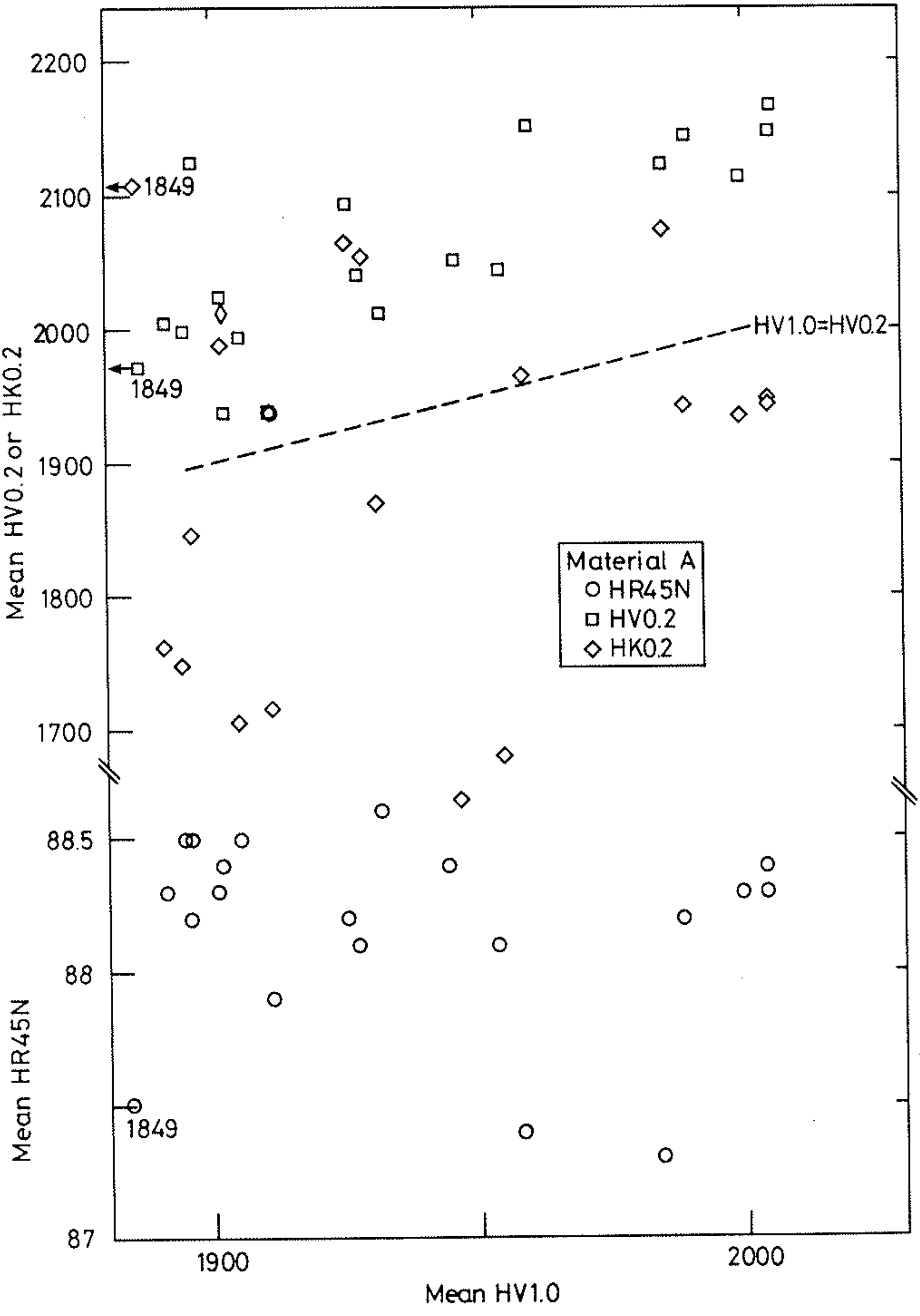

Figure 31 Relationships between HV1.0 hardness numbers and HR45N, HV0.2 and HK0.2 hardness numbers from NPL results on material A. Only HV0.2 shows a significant correlation with HV1.0.

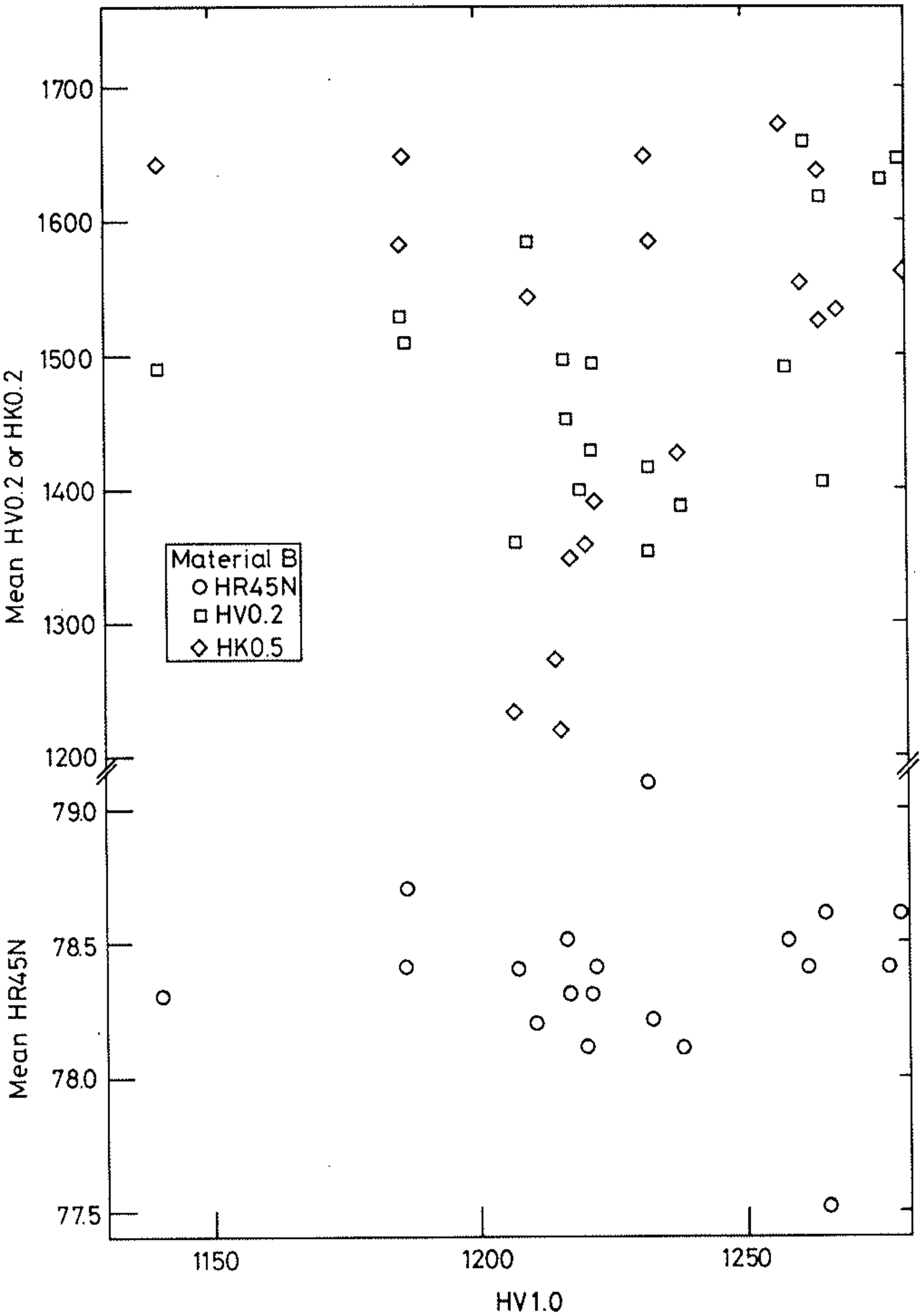

Figure 32 Relationships between HV1.0 hardness numbers and HR45N, HV0.2 and HK0.2 hardness numbers from NPL results on material B. There is no clear correlation.

Appendix I

VAMAS

Test Procedure for Hardness Testing of Ceramics Programme

1 MATERIALS

Two test specimens are supplied in the form of polished discs.

(1) a 99.9% Al_2O_3, 2 µm grain size, density typically > 3.95 Mgm^{-3} ("Vitox", Morgan Matroc Ltd, Anderman Division, UK) - "white".

(2) a 95% Al_2O_3, 5 µm grain size, density typically 3.70 Mgm^{-3}, porosity ~ 5% by volume, second-phase content ~ 15% by volume ("Sintox FA", Lodge Ceramics Ltd, UK) - "pink".

2 PURPOSE

Hardness testing of hard ceramics has not been standardised. The purpose of this exercise is to evaluate the reproducibility with which different laboratories using different testers can measure hardness. The materials have been chosen to represent two types of behaviour. The 99.9% Al_2O_3 white material has high hardness, little load-dependence of hardness, low porosity, and a fine grain size. Testing this material should reveal the limits of precision attainable. On the other hand, the 95% Al_2O_3 pink material is two-phase, has a higher level of porosity and is generally more difficult to test satisfactorily. Testing this material should reveal the difficulties that conventional multiphase materials pose. The exercise covers Vickers, Knoop and Rockwell superficial hardness tests.

3 RATIONALE

The supplied specimens have a set of Vickers and Knoop indentations in them. Participants are asked to measure the size of the indentations according to a set procedure. These indentations will already have been measured by NPL. The purpose is to establish reproducibility of reading.

Participants are then asked to make their own hardness measurements according to set procedures, and to return the blocks to NPL for remeasurement. The purpose is to establish machine reproducibility. Participants are asked to supply details of their test machines and whether they have been able to follow all aspects of the test procedure recommended.

4 OUTLINE TEST PROCEDURE

4.1 Layout of indentations

Figure 1 shows the layout of the NPL-produced indentations, placed in defined arrays on the specimens. Also shown are areas in which participants are asked to make their own indentations in order that they can be located later by NPL. Note that the NPL-introduced HV1.0 indentations act as locators for the line of NPL microhardness indentations. Participants are asked to make a similar set of indentations in the areas indicated in Figure 1.

DO NOT ATTEMPT TO RE-POLISH OR RE-GRIND THE SPECIMENS. THEY MAY BE CLEANED, BUT USE ONLY ETHANOL.

4.2 Measurement of NPL-produced Vickers and Knoop indentations

Participants are asked to use a microscope fitted with a filar measuring device (moveable cross-wires) or a microhardness tester with a purpose-built measuring device. The overall magnification of the measuring device must be checked by use of a reference graticule, and the calibration obtained must be applied to all the measurements made.

Illumination of the microscope is an important factor in making measurements. Best illumination can be obtained by following the procedure given in Reference 1. In particular the aperture stop should be restricted to the field of view (or smaller) in order to reduce light scattering around and beneath the indentations. The illumination may not be adjustable in microhardness testers. The system should be focussed to bring the indentation corners to their sharpest appearance.

Participants are asked to measure both diagonals of the Vickers indentations and the long diagonal of the Knoop indentations, and to record their measurements on the attached sheet. The set of Vickers and Knoop indentations shown schematically in Figure 1 are most readily located by focussing the microscope or measuring system on the test-piece edge and traversing the test-piece for a distance of 3 mm along the centre-line of the indicated band. This should locate the first of the set of HV1.0 indentations. The next indentation is 0.25 mm further on, and so on across the specimen. Microhardness indentations are 0.1 mm apart along the same line.

IMPORTANT NOTE

The sequences of indentations inserted by NPL will include some which are considered to be unacceptable because of excessive cracking, fragmentation, or chipping. The test sheets attached to these instructions will refer to identified indentations numbered in sequence from the edge of the test-piece, including those which are unacceptable. Thus the participant will be asked to measure, for example, indentation numbers 2,4,7,8 and 9 out of a total of, say, 9 indentations of a particular type numbered in sequence from the one nearest the specimen edge. Similarly, when participants make and record their own indentations, the same scheme should be adopted in order that NPL can identify the participants indentations for remeasurement later.

4.3 Participants' indentations

Participants are asked to place their own indentations in the areas indicated in Figure 1 in the sequence shown. The following preferred indentation types are suggested, with alternatives in brackets if the preferred types are not available:

Rockwell Superficial	HR45N	(Rockwell A)
Vickers macrohardness	HV1.0	(HV2.5)
Vickers microhardness	HV0.2	(HV0.1)
Knoop microhardness	HK0.2	

Minimum numbers of 5 of each macrohardness determination and 10 of each microhardness are required, with measurements recorded on the attached sheet. These can be made either before or after measuring the NPL-introduced indentations.

5 CALIBRATION CHECKS ON PARTICIPANTS' MACHINES

5.1 Rockwell machines

The standard calibration procedure as recommended by the manufacturer should be employed. The machine should have recently been serviced and checked. A high hardness test block should be used to ensure the machine is reading accurately. A minimum of 5 test indentations should be made. Any discrepancy between the test-block nominal value and that obtained on the machine should be noted. The test block should be used before and after undertaking tests on the supplied specimens. It is preferred that the calibrations are made on the same scale as used for the tests.

The indenter used should be a single crystal or a polycrystalline diamond, and should be inspected before and after the test for any damage.

The preferred test scale is the superficial N scale, 45 kg load, since this load can be tolerated by most strong ceramics. If a superficial tester is not available, the A scale may be used on the 99.9% Al_2O_3 white material (the 95% Al_2O_3 pink material may crack).

5.2 Vickers macrohardness

The preferred load is 1.0 kg, considered to be the highest practical load at which cracking is not excessive. If this load is not available, a higher load, such as 2.5 kg, may be used.

If the tests are to be made on a standard Vickers (30 kg) machine with reduced load, the optical measuring system should not be used since it is not sufficiently accurate for measuring small indentations. Measurements should be made by calibrated optical microscope (see 4.2 above) or using the optical system of a microhardness tester (see 5.3 below). (This does not apply if the tests are made on a microhardness tester.)

Make an indentation in a metal test block and check that the indentation diagonals are of equal length. If not, follow the normal adjustment procedure to align the diamond. When symmetrical indentations are produced, use a standard test block of as high hardness as possible, preferably greater than 700 HV, and make at least 5 indentations.

Measure the diagonal lengths, calculate hardness and standard deviation, and report on the attached form.

If an electronic load cell is available, check the load applied to the specimen during indentation. Use the calibrated load to make hardness calculations. [This is intended particularly to check high-load machines not normally calibrated or used at low loads where frictional effects may be significant.]

5.3 Vickers and Knoop microhardness

Set up the optical illumination as recommended above, or according to the manufacturer's recommended procedure. Make some trial indentations in a standard test block. Check that the Vickers indentation diagonals are of equal length and that the indentation is not skewed. Adjust if necessary. With the Knoop indenter check the indentation is of a regular kite shape, and is not skewed. Use an objective magnification of at least x25 and an overall magnification of at least x400. Use a graticule to calibrate the filar or other measuring device to check the magnification accuracy.

Check the machine calibration and the operator's measurement criterion by placing at least 5 indentations in a standard test block of at least 700 HV or 700 HK. Record the results on the attached forms, and also record any unresolved problems of measurement.

If possible, check the force being applied by use of a load cell. Record any differences between nominal and actual force.

If possible, inspect the diamond indenters before and after testing and report any chipping or other defects in their appearance. Note any ridge on the apex, or any flats on the edges.

6 TEST INDENTATIONS

Areas of the specimen for indenting by participants are shown in Fig.1. Indentations should be made in the sequence shown. The minimum number and minimum spacing between acceptable indentations must be as follows:

Type	Scale	Number	Minimum Spacing
Rockwell	HR45N (or HRA)	5	3 mm
Vickers	HV1.0 (or HV2.5)	5	0.2 mm (0.5 mm)
Vickers	HV0.2 (or HV0.1)	10	0.1 mm
Knoop	HK0.2	10	0.1 mm

It may well be found that some indentations are not measurable owing to cracking or chipping. Participants are asked to use their own judgement as to the acceptability of each indentation. Even if not acceptable, they should be included on the attached form in order that individual indentations can be identified and checked by NPL if necessary.

Both axes of the Vickers indentations should be measured, the average taken, and the hardness number calculated according to the formula:

$$HV = 1.8544\ P/d_v^2$$

where d_v is the mean diagonal length in mm, and P is the applied force (actual force as determined by a load cell if possible) in 'kg'.

The long axis of the Knoop indentation should be measured, and the hardness number calculated according to the formula:

$$HK = 14.229\ P/d_k^2$$

where d_k is the long diagonal length, and P is the applied force (actual force as determined by a load cell if possible) in 'kg'.

The Rockwell hardness number should be read directly from the scale to the nearest 0.1.

7 REPORTING

The attached results sheets and specimens should be returned to NPL for checking and collation of results.

8 INDENTATION FRACTURE TOUGHNESS

An additional optional exercise is felt to be worthwhile if participants are willing to undertake it. Using the HV1.0 series of indentations introduced by the participants, it should be possible to identify the main cracks emanating from (or from near) the corners of the indentation. Participants are requested to measured the total lengths of the cracks from one side of the indentation to the other (l_1 and l_2 in Fig.2) for each of the indentations in the order in which the indentations were made. The measurements should not be made until at least 1 h after the indentations were made in order to allow crack growth to stabilise. Please note on the results sheet the nature of the illumination or observation technique that was found to be the most appropriate to identify the ends of the cracks.

Evaluation of the data will shed some light on the repeatability with which crack lengths can be measured as a guide to the possible appropriateness of the indentation method for determining fracture toughness.

REFERENCE

Peggs, G N, Leigh, I C, Recommended procedure for micro-indentation Vickers hardness test. NPL Report MOM 62, March 1983. (Copy available on request.)

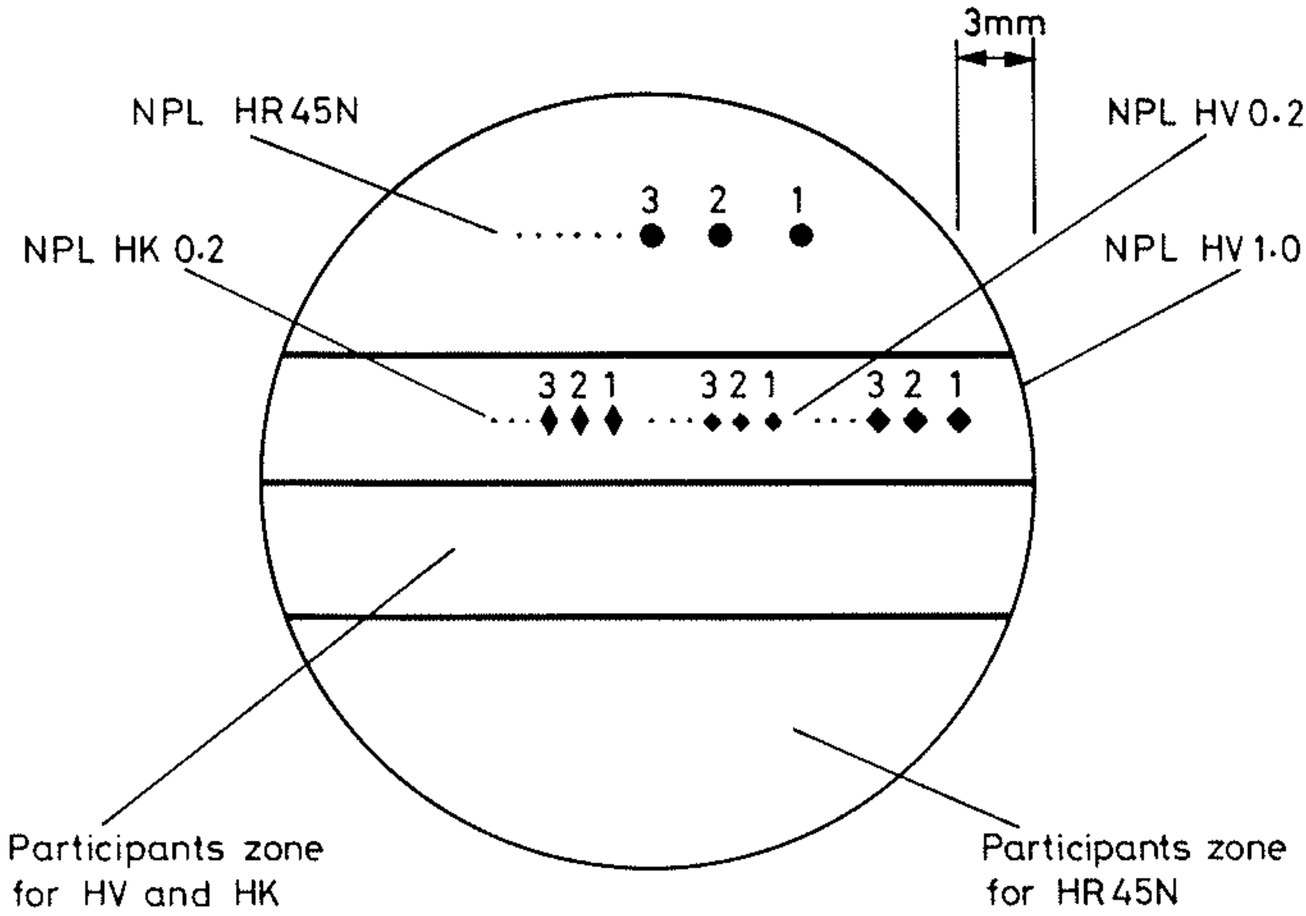

Fig.1 Layout of test specimens showing position of NPL indentations and zones in which participants indentations should be made.

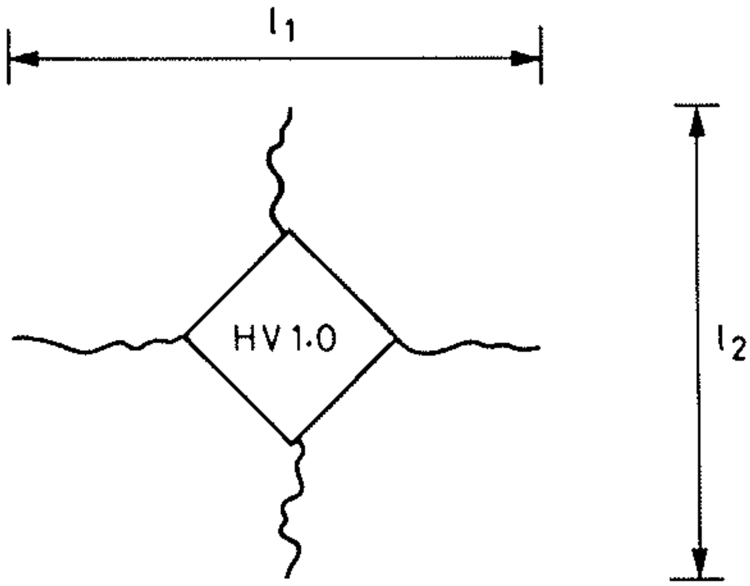

Fig.2 Measurements of crack length of HV1.0 indentations.

Appendix II

V A M A S

Hardness testing round robin

<u>Results Sheets</u>

Name ....

Affiliation ....

Persons undertaking tests (if different)*

	Age	Wearing glasses Y/N	Eyesight**
Rockwell	...	....	....
Vickers	...	....	....
Vickers microhardness	...	....	....
Knoop microhardness	...	....	....

* Details are treated in confidence and are to be used to correlate results with age and condition of eyesight.

** Please use following code.

1 20/20 vision
2 short-sighted
3 long-sighted
4 middle-sighted
5 significantly astigmatic

When completed these results forms should be returned to:

Dr. R. Morrell
Division of Materials Applications
National Physical Laboratory
Teddington
Middlesex TW11 0LW
UK

DO NOT FORGET TO RETURN THE TEST SAMPLES OF ALUMINA!!

RESULTS SHEET 1 - ROCKWELL TESTS

Machine type (make, model)

Scale used: HR45N HRA

Test block hardness ....

Measured values:	before tests	after tests
	....	....
	....	....
	....	....
	....	....
	....	....
	________	________
Means and standard deviations	________	________

Test results

Please record all results in sequence across block, with at least five acceptable ones.

White alumina	(1)	....	Pink alumina	(1)	....
(99.5%)	(2)	....	(95%)	(2)	....
	(3)	....		(3)	....
	(4)	....		(4)	....
Enter "nm" for	(5)	....		(5)	....
indentations	(6)	....		(6)	....
not measured	(7)	....		(7)	....
	(8)	....		(8)	....
		________			________
Means and standard deviations		________			________

Comment on any measurement difficulties, changes in indenter condition, etc.

RESULTS SHEET 2 - VICKERS MACROHARDNESS TESTS

1. Measurement of NPL HV1.0 indentations.

Equipment used for measurement of indentations ....

Nominal magnification ....x Calibrated magnification ....x using graticule

	White alumina			Pink alumina		
	no.*	d_1,mm	d_2,mm	no.*	d_1,mm	d_2,mm
(1)	.....	....	....	.....	....	....
(2)	.....	....	....	.....	....	....
(3)	.....	....	....	.....	....	....
(4)	.....	....	....	.....	....	....
(5)	.....	....	....	.....	....	....
(6)	.....	....	....	.....	....	....
Overall means and std. devs.						

* Number in sequence from indentation nearest the edge of the specimen.

2. Participants indentations.

Machine type (make, model) ....

Scale used: HV1.0 HV2.5

Calibration of load: Actual load ....kg Not done ......

Test block hardness ....

Indentations in the test block: (please make at least five)

Measured values:	d_1, mm	d_2, mm	d_{mean}, mm	HV
(1)	....	....	....	....
(2)	....	....	....	....
(3)	....	....	....	....
(4)	....	....	....	....
(5)	....	....	....	....

Mean and standard deviation

RESULTS SHEET 2 - CONT.

Ceramic test results.

Scale used HV1.0 HV2.5

Please record results in sequence across blocks, including at least 5 you find acceptable. Enter "nm" for those you consider not measurable.

	Indent no.	d_1, mm	d_2, mm	d_{mean}, mm	HV
White alumina:					
	(1)	....	....	....	....
	(2)	....	....	....	....
	(3)	....	....	....	....
nm = not	(4)	....	....	....	....
measured	(5)	....	....	....	....
	(6)	....	....	....	....
	(7)	....	....	....	....
	(8)	....	....	....	....
	(9)	....	....	....	....
	(10)	....	....	....	....

Mean and standard deviation

	Indent no.	d_1, mm	d_2, mm	d_{mean}, mm	HV
Pink alumina:					
	(1)	....	....	....	....
	(2)	....	....	....	....
	(3)	....	....	....	....
nm = not	(4)	....	....	....	....
measured	(5)	....	....	....	....
	(6)	....	....	....	....
	(7)	....	....	....	....
	(8)	....	....	....	....
	(9)	....	....	....	....
	(10)	....	....	....	....

Mean and standard deviation

RESULTS SHEET 2 - CONT.

Indentation crack lengths: (measured as described in Fig.2)

(Insert "nm" for cracks visible but too poorly defined to measure, and "0" if no crack is visible emanating from near the indentation corner on either side of the indentation.)

	White alumina		Pink alumina	
Indent no.	l_1, mm	l_2, mm	l_1, mm	l_2, mm
(1)	....	....	....	....
(2)	....	....	....	....
(3)	....	....	....	....
(4)	....	....	....	....
(5)	....	....	....	....
(6)	....	....	....	....
(7)	....	....	....	....
(8)	....	....	....	....
(9)	....	....	....	....
(10)	....	....	....	....
Means, ± std dev	________		________	

RESULTS SHEET 3 - MICROHARDNESS TESTS

1. Measurement of NPL HV0.2 and HK0.2 indentations.

Equipment used for measurement of indentations ....

Nominal magnification ....x Calibrated magnification x

		Vickers, HV0.2		Knoop, HK0.2	
	Indent no.	d_1, mm	d_2, mm	Indent no.	d_k, mm
White alumina:					
(1)	.....	....	....	.....	....
(2)	.....	....	....	.....	....
(3)	.....	....	....	.....	....
(4)	.....	....	....	.....	....
(5)	.....	....	....	.....	....
(6)	.....	....	....	.....	....
(7)	.....	....	....	.....	....
(8)	.....	....	....	.....	....
(9)	.....	....	....	.....	....
(10)	.....	....	....	.....	....
Means and std. devs.					
Pink alumina:					
(1)	.....	....	....	.....	....
(2)	.....	....	....	.....	....
(3)	.....	....	....	.....	....
(4)	.....	....	....	.....	....
(5)	.....	....	....	.....	....
(6)	.....	....	....	.....	....
(7)	.....	....	....	.....	....
(8)	.....	....	....	.....	....
(9)	.....	....	....	.....	....
(10)	.....	....	....	.....	....
Means and std. devs.					

RESULTS SHEET 3 - CONT

2. Participants indentations.

Machine type (make, model) ....

Last serviced (month/year) ....

Nominal optical magnification x

Calibrated magnification x

Calibration of load:

Actual applied load ....

Not done ....

Calibration:

Test block hardness ...... HV ...... HK

Test block results:

	Vickers d_1,mm	d_2,mm	d_{mean}	HV	Knoop d_k,mm	HK
(1)	....	....	....	....	....	....
(2)	....	....	....	....	....	....
(3)	....	....	....	....	....	....
(4)	....	....	....	....	....	....
(5)	....	....	....	....	....	....
		Means and std devs				

RESULTS SHEET 3 - CONT.

Tests in alumina:

(Please use consecutive numbering for all indentations including those you consider to be unacceptable, recording in the table below the numbers and measurements of at least 10 that you consider acceptable.)

Indent no.	d_1,mm	d_2,mm	d_{mean},mm	HV0.2	Indent no.	d_k,mm	HK0.2
White alumina:							
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
Means and standard deviations				________			________
				________			________
Pink alumina:							
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
Means and standard deviations				________			________
				________			________

<u>Visual examination of indenter</u>

Please comment on chipping or geometry of the diamond indenters used.

Before:

After:

Distribution:

Participants	44	(see p.3)
NIST (VAMAS)	10	
NPL distribution	24	
Reserve	22	
Total	100	

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