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Hardness testing on advanced technical ceramics

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Abstract

To validate the European standard ENV 843-4 for hardness measurements on ceramics, three classes of ceramic materials, silicon nitride, silicon carbide, and aluminium oxide, involving 19 ceramics in total, were tested using the traditional techniques Vickers (HV1), Knoop (HK2), and superficial Rockwell (HR45N). The use of new ceramic reference blocks certified according to the standards ISO 4547 and ISO 6507-1 for metallic materials was studied. If the hardness response of the tested materials does not involve chipping and cracking the application of high hardness reference blocks for training users to obtain hardness values comparable with the certified HV1 and HK2 values improves the reproducibility from about 10% to 1 to 3%. The scatter between the laboratories is similar to the scatter within the laboratories. The measurement of the indentation geometry on typical commercial ceramic materials can be made only with higher scatter and reduced reproducibility compared with typical metallic materials, which is caused by the stochastic indentation response. For such materials involving chipping and cracking (for instance SiC), the application of reference blocks with well-shaped indentations does not provide improved comparability of results between the laboratories. The actual indentation response of the ceramic material tested must be considered before selecting the appropriate hardness technique and test force. There is no significant difference between the abilities of the hardness techniques HV1, HK2, and HR45N to discriminate sensitively between materials of closely similar properties.

Keywords: Al_2O_3; Hardness; Standards; SiC; Si_3N_4; Testing

1. Introduction

The measurement of hardness is frequently undertaken but poorly performed on ceramic materials, partly due to experimental issues such as chipping and cracking in the test-piece.[1–5] The main problem is the unsatisfactory reproducibility between the laboratories. The European draft standard ENV 843-4[6] was prepared some years ago with the intention of unifying practices, but there have been many developments in the subject area since that date which need to be taken into account before confirming the standard as an EN or proposing modifications. In particular, the National Institute for Standards and Technology (NIST, USA) has been developing new reference materials for HK1 and HK2 scales (HIPed silicon nitride)[7] and a prototype block for HV1 (HIPed tungsten carbide). Additional ceramics which are appropriate as reference materials have been offered by the Fraunhofer-Institute for Ceramic Technologies and Sintered Materials (IKTS, Germany).[8] The use of such reference blocks is thought to be of considerable value in training users to perform appropriate measurement routines.

The objectives of this paper are to report on the interlaboratory work for evaluating the use of reference blocks as a user guide to measurement, for estimating confidence bands on test results, and for determining the suitability of criteria for acceptance or rejection of

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indentations on a variety of advanced technical ceramics. The work was done as a part of the EC-project CERANORM.[9]

There were two periods of interlaboratory work. In the first period, a study on varied effects of the experimental conditions on the hardness numbers HV1, HK2, and HR45N was performed for typical advanced technical ceramics to validate ENV 843-4. Based on the results of the first period, an interlaboratory exercise was conducted in the second period in order to test the experiences with the indentation response of typical commercial ceramics when the new reference blocks and instructions were used as a guide.

2. Materials selection

The selection of representative ceramic materials is given in Table 1. The two parts of the table concern the two periods of project. The series starts with the reference blocks for HV1, HK2, and HR45N. Detailed information on the silicon nitride ceramics for HK2 can be found in Ref. 7. The WC/Co prototype HV1 reference blocks from NIST were available only for the second period (lower part of Table 1).

Several ceramic materials are typical commercial products from the three widely spread materials classes: silicon nitride, silicon carbide, and aluminium oxide. Materials are included with high porosity of 3% (specimens E), with high hardness (including high tendency to cracking) of 2600 HV1 (specimens H, V), and with medium or enhanced grain size up to 5–8 μm (specimens L or M). For comparison, several materials from a research laboratory (specimens C, D, G, J, R, S, T) with optimized quality for hardness testing have been used.

The preparation of the specimens was mostly performed by one laboratory to obtain unique surface quality. The arithmetic mean roughness of the specimens is $R_a < 0.08$ μm for the typical commercial products but, for the specimens with the quality of reference materials, the roughness has been minimized down to $R_a = 0.002$ μm. The hardness values given in Table 1 are mean results presented below in this paper.

According to the instructions good measurenent practice was required. All participants have to use regularly serviced commercial machines, and normally used

Table 1
Overview of the ceramic materials used in the study

Code	Source/material code[a]	Type	HV1	HK2	HR45N	Reason for use
A	NIST/SRM 2830	HPSN	1580	1430		Reference material, HK2, see Ref. 7.
B		WC/Co			77	Reference material HR45N
C	IKTS	GPSN	1470	1350	87.9	Material of best quality regarding hardness test
D	IKTS	GPSN	1480	1380	88.2	Similar to C, but different hardness
E	Tenmat/Nitrasil R	RBSN	1020	940	80.8	Testing of fine-scale porosity
F	Lucas-Cookson/Syalon 201	SiAlON	1600	1410	88.3	Typical engineering material
G	IKTS	LPS-SiC	2560	1960		High hardness, low porosity
H	CERAMTEC/CD	SiC	2650	1720		High hardness engineering material
I	CERAMTEC/RK	Al_2O_3	1890	1610	88.7	Typical alumina product
J	IKTS	Al_2O_3	2120	1730		Material of best quality regarding hardness test, determination of the force dependence
K	Morgan Matroc/VITOX (white)	Al_2O_3	1990	1710	88.3	Typical ceramic material with low porosity, fine grained, single-phase
L	Morgan Matroc/SINTOX FA	Al_2O_3	1530		78.4	Typical ceramic material with medium porosity, medium grain size, multi-phase
M	Morgan Matroc/VITOX (white) + tempered	Al_2O_3	1800	1570		As L, but heat-treated for investigating effect of grain size
WC	NIST/SRM 2831	WC	1550			Reference material, HV1 Prototype
R	IKTS	GPSN	1500	1380		Reference material for HV1 and HK1
S	IKTS	LPS-SiC	2490	2020	92.1	Reference material for HV1
T	IKTS	Al_2O_3	2070	1740		Reference material for HV1 and HK1
U = E	Tenmat/Nitrasil R	RBSN	1020	930	80.8	Testing of fine-scale porosity
V	IKTS	SiC	2510	2160	91.1	Typical high hardness product
W = I	CERAMTEC/RK	Al_2O_3	1890	1610	88.7	Typical alumina product
X4,X5,X6,X7	IKTS	HPSN	1460–1770	1350–1530	87.2–90.0	Fine grained materials with related properties (by different additives) for sensitivity analysis

[a] It should be noted that the specimens delivered by the producers free of charge are not necessarily identically with the ceramics offered in the catalogues of products.

metallic reference blocks for checking calibrations. The indenters must be certified according to ISO 6507-2, ISO 4546, or EN10109-2.

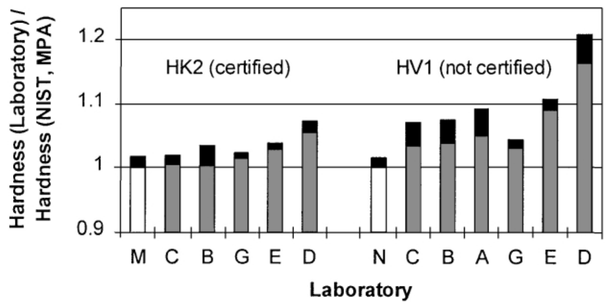

Fig. 1. Hardness results of HK2 (left) and HV1 (right) on HK2 reference blocks. The white bars show the results of the calibration laboratories MPA NRW Dortmund (M) and NIST Gaitherburg (N). All data in this and subsequent similar figures have been normalised, in this case to the means given by the calibration laboratories; the black top to each bar is the positive-going standard deviation of the mean of 10 measurements.

3. Results on reference blocks

At the beginning of the interlaboratory study the hardness measurements of the laboratories were based on NIST reference blocks HK2 (HIPed silicon nitride, specimen A). The results of HK2 and HV1 are gathered in Fig. 1. While the scatter within and between the laboratories concerning Knoop hardness is adequate for most purposes the scatter concerning Vickers hardness is greater. On the one hand the operators of the laboratories with maximal deviation were recommended to train their operators to achieve a better fit to the given calibrated hardness values for improving the comparibility of their data in future. On the other hand the reference blocks HK2 by NIST have not been optimized for Vickers hardness. Slight cracking can be observed at the indentation corners (Fig. 2).

The comparative tests of Rockwell hardness HR45N using the reference blocks 76.95 HR45N (hard metal, specimen B) have given good results. The coefficients of

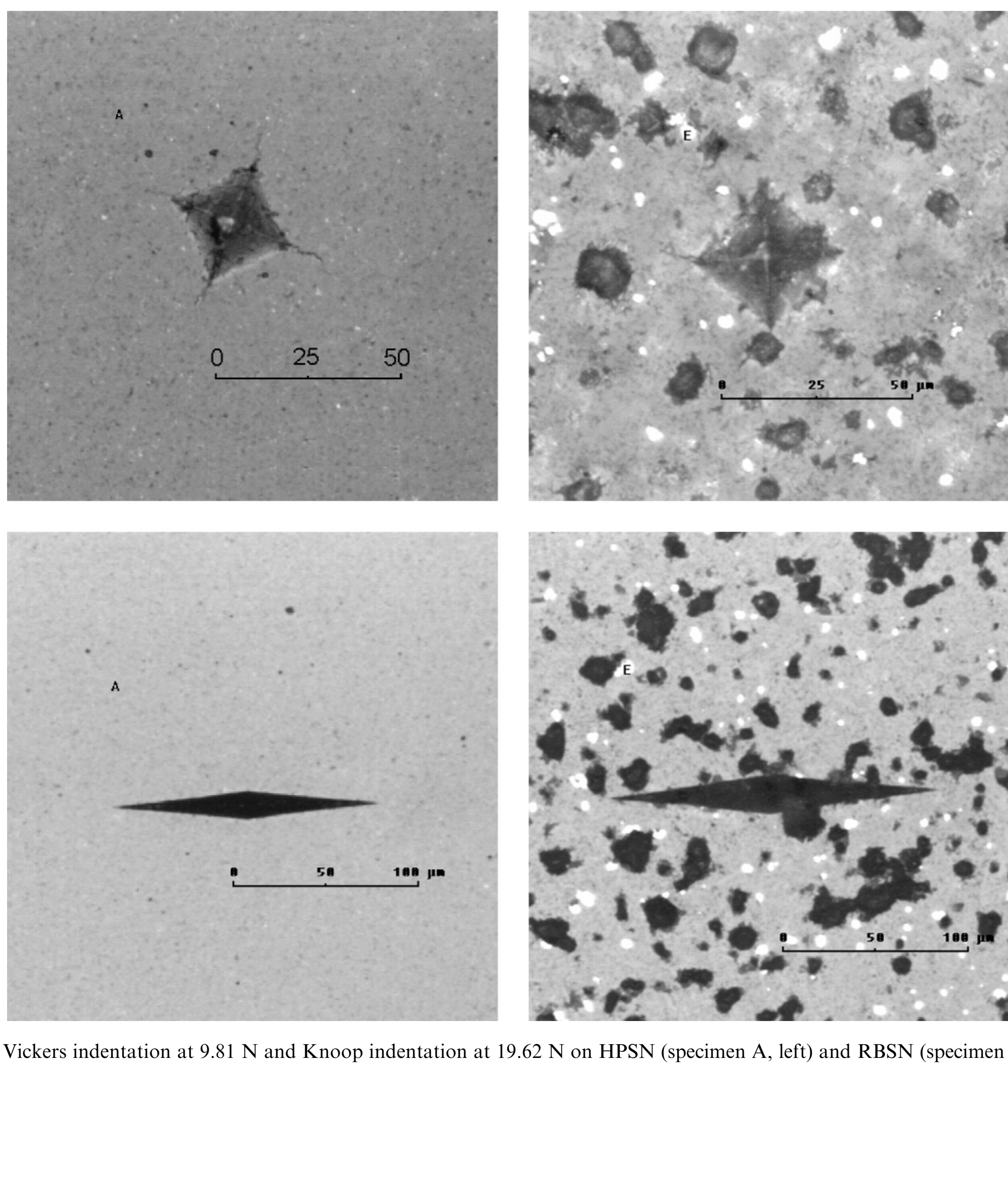

Fig. 2. Vickers indentation at 9.81 N and Knoop indentation at 19.62 N on HPSN (specimen A, left) and RBSN (specimen E, right).

variation within the laboratories as well as the relative deviations to the reference value is less than 1%. However, it should be noted that this corresponds to deviations of 3% in actual indentation depth because of the way Rockwell scales are defined: HR45N = 100−e where e is the indentation depth in micrometres.

The average results on the HV1 blocks (hard metal, specimens WC) obtained in the second period are demonstrated in Fig. 3a and b. Except for two laboratories the results of the participants fit well the calibrated hardness (with the mean tendency to be 2% smaller than the certified values). The standard deviations of the participant's results are similar to the standard deviations of NIST for the measurements on the indentations made by NIST. As is to be expected, the standard deviations are slightly larger if the complete hardness test is performed by the participant, including indenting (right side of Fig. 3a).

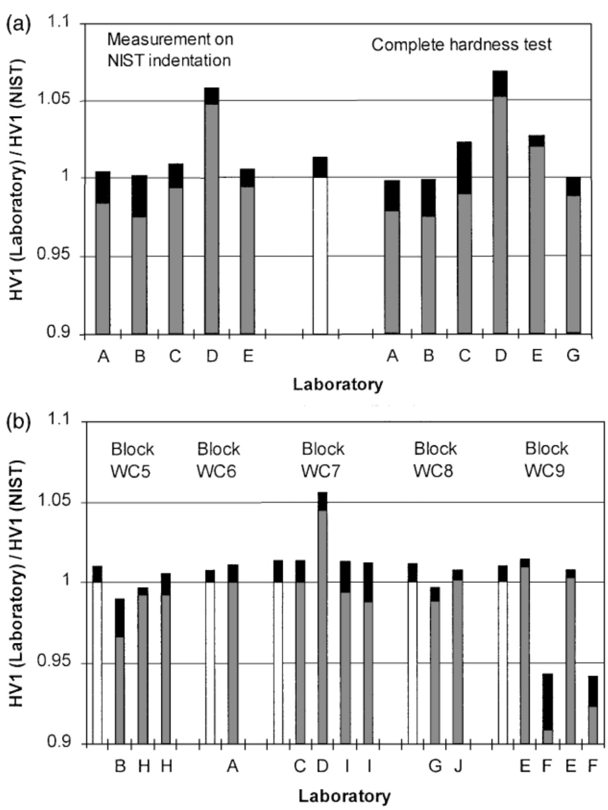

Fig. 3. Hardness results on HV1 WC/Co reference blocks normalised to the NIST means: a. participants did not receive information on calibration data; b. participants received information on calibration data.

The usefulness of the reference materials can be concluded from the simple variance analysis. The result can be seen in Table 2. The variances caused by differently measured indentations (coefficient of variation (COV) of the mean results between laboratories) is separated from the individual laboratory experimental scatter (COV within laboratories).

As seen in Table 2, every coefficient of variation has a similar small value of about 1–2%. There are no significant differences between the deviations from the calibrated values and the scatter between the laboratories. That indicates the consistency of measurement regarding the unknown reference blocks is as good as the consistency of measurement regarding the reference blocks of known calibration. The question arises as to whether the guidance offered by the reference blocks to achieve the correct visual measurement criteria is of essential help in the case of measurements with small scatter. The comparison between Fig. 3a and b shows that two laboratories (D and F) have not taken into account the existing knowledge of the reference hardness. This point has to be taken into account in the interpretation of the HV1 values of these participants on the other specimens.

4. Validation of the standard ENV 843 part 4

To validate the standard several effects of testing conditions on the hardness results HV1, HK2, and HR45N were studied. Basically, the effects to be evaluated are:

- Effect of indenting force hold time;
- Effect of the delay between indentation and the optical measurement;
- Effect of distance between indentations;
- Effect of imperfect alignment of the indenter axis to surface normal;
- Effect of grain size on measurability;
- Effect of surface preparation methods;

Table 2
Separation of the scatter occured in the use of reference blocks

Test	Test block identification	Participant's knowledge of calibration value	COV (%) within labs	COV (%) between labs	Relative deviation of mean results to calibration value (%)
HV1	WC1 (use of NIST indentations)	Not known	1.7	0.8	−1.1
HV1	WC1	Not known	1.9	1.8	−0.9
HV1	WC5 to WC9	Known	1.2	1.4	−0.1
HK2	Si_3N_4	Known	0.7	2.3	2.4

- Effect of cracking from indentation corners on measurement accuracy;
- Effect of loading mode;
- Variance of hardness resulting from lack of perfect homogeneity of microstructure;
- Force dependence of hardness.

The levels for studying the effects were selected according to the restrictions given in the standards.[6,10,11] They are listed in Table 3, under the column headed "levels". The term "long distance" between the neighbouring indentations means the greatest of 5×mean diagonal length, or 5×mean crack length, or 2 mm according to Ref. 6. Short distance means the greater of 3×mean diagonal length or 3×mean crack length.[11] Since the use of the depth sensing hardness technique (or universal hardness, HU) was included in the project CERANORM,[9] the effect of loading mode has also been evaluated. The terms HV1/HU and HK2/HU for the techniques in Table 3 mean that the indentations are performed by the use of an HU-machine and measured optically after indentation by conventional methods. In this way, the results after the relatively fast loading (conventional hardness technique) can be compared with the results after loading by a displacement-controlled table with a slower loading rate (about 2 min up to the maximal force).

Because the study takes into account a number experimental variables and measurement issues, a test matrix was developed for the work coordinated between the participating laboratories. In this way, 53 different tests were agreed and conducted using the hardness techniques HV1, HK2, and HR45N, with the main focus regarding HV1. At least two laboratories contributed to each test. Ten valid indentations per test were required to be performed. The guidelines given in Ref. 10 were used for the acceptance of the generated indentations (see discussion for the modification of the guidance).

The hardness results are collected in Table 4. To see the significant effects the results of the *t*-tests (hypothesis: mean values are equal) are listed in Table 3. The hypothesis is true if the probability for the occurred difference is greater than 10%, is false if the probability for the occurred difference is less than 1%, and is uncertain ("0"), otherwise. In the same way, the table

Table 3
Statistical tests of the effects on the experimental conditions

Effect	Technique	Material	Levels	Results of *F*-test S.D. are equal	Results of *t*-test Mean values are equal
Force hold time	HV1	A	5 vs 60s	True	True
	HK2	A	5 vs 60s	0	0
Delay of observation	HV1	G	60 vs 1day	True	True
	HV1	H	60 vs 1day	True	True
	HV1	J	60 vs 1day	True	True
Distance between indentations	HV1	A	Long vs short	True	True
	HV1	H	Long vs short	True	True
	HK2	A	Long vs short	True	True
	HK2	H	Long vs short	False	False
Alignment	HV1	I	0 vs 1°	True	True
	HV1	K	0 vs 1°	True	0
Grain size	HV1	K vs M	Fine vs coarse	True	False
Surface preparation	HV1/HU	F	Treated vs untreated	True	0
	HV1/HU	K	Treated vs untreated	True	0
Loading mode	HV1	A	HV1 vs HV1/HU	True	True
	HV1	D	HV1 vs HV1/HU	True	False
	HV1	J	HV1 vs HV1/HU	True	False
	HK2	A	HK2 vs HK2/HU	True	0
Sensitivity to small changes in material characteristics	HV1	A vs D		True	False
		C vs D		True	True
		D vs F		True	False
	HK2	A vs D		True	False
		C vs D		True	0
		D vs F		True	False
	HR45N	C vs D		True	0
		D vs F		True	False

Table 4
Selected results to validate the standard ENV 843-4 conditions

Specimen[a]	Technique	Number of laboratories contributing	Number of valid indentations	S.D. of mean within laboratories	S.D. between means of laboratories	S.D. overall, all results	Mean hardness number
A	HV1	5	50	47	35	57	1577
A	HV1/HU	1	10	24		24	1581
A	HV1	2	21	35	13	37	1580
A	HV1	2	22	54	8	55	1586
A	HV1	2	22	57	25	60	1590
C	HV1	5	40	29	15	31	1473
D	HV1	5	40	18	25	31	1483
D	HV1/HU	1	10	19		19	1437
E	HV1	3	39	67	50	75	1019
F	HV1	2	20	40	17	42	1603
F	HV1/HU	1	8	44		44	1562
G	HV1	2	15	93	166	148	2557
G	HV1	2	15	115	235	199	2536
H	HV1	2	10	207		207	2648
H	HV1	2	10	149		149	2724
H	HV1	2	10	180		180	2653
I	HV1	2	19	76	80	96	1886
I	HV0.1	2	20	174	133	199	2035
I	HV1	2	10	60		60	1914
I	HV1	2	17	122	74	133	1893
J	HV1	2	20	34	6	34	2116
J	HV1/HU	1	8	37		37	2027
J	HV1	2	15	22	8	23	2116
K	HV1	2	20	90	37	93	1992
K	HV1	2	21	89	21	91	2067
K	HV1/HU	1	9	52		52	1782
L	HV1	2	15	135	206	196	1531
M	HV1	2	15	66	4	66	1800
M	HV3	1	5	43		43	1673
M	HV10	1	5	36		36	1526
A	HK2	6	49	30	29	38	1434
A	HK2/HU	1	10	7		7	1421
A	HK2	2	19	27	39	39	1497
A	HK2	2	20	17	6	17	1464
A	HK2	2	18	38	25	42	1437
C	HK2	2	10	21	20	26	1351
D	HK2	3	20	25	6	26	1377
E	HK2	2	21	63	38	69	942
F	HK2	2	21	19	7	20	1411
G	HK2	2	15	45	31	50	1955
H	HK2	2	22	94	423	319	1717
H	HK2	2	11	28		28	1259
B	HR45N	3	65	0.1	0.1	0.1	77.2
C	HR45N	2	15	0.2	0.4	0.3	88.0
D	HR45N	2	15	0.2	0.4	0.3	88.2
E	HR45N	2	15	1.0	0.9	1.1	80.5
F	HR45N	2	15	0.3	0.2	0.3	89.7

[a] see Table 1.

contains the *F*-tests because the assumption of the *t*-test is the similarity of the two standard deviations.

Comparing the results in Tables 3 and 4 selected regarding the effect of interest the following conclusion can be drawn:

- In studying the effect of holding time of maximum force ranging from 5 to 60 s, no significant differences of the Vickers hardness and Knoop hardness could be found on the NIST reference blocks (specimens A).

- There are no significant differences if the diagonal length of the indentation is measured one day after loading in comparison to the immediate observation within 60 s after loading.
- For the HPSN reference blocks, no effect was observed if the indentations were placed with the distance between the centres of the greater of 3× mean diagonal length or crack length according to Ref. 11 instead of the greatest of 5× mean diagonal length or crack length, or 2 mm according to Ref. 6. This was observed for both Vickers hardness and Knoop hardness. However, the response of the commercial silicon carbide (specimen H) is different. Whereas no effect was observed for Vickers hardness there was an reduction of the Knoop hardness when the spacing was closer than recommended.
- No effect on the Vickers hardness was observed for the three materials classes if the specimen was inclined by 1° related to the axis of the indenter.
- Higher polish quality reduced the scatter of hardness significantly as is shown on specimens with the quality of reference material, for instance specimen G in comparison to specimen H (Table 4).

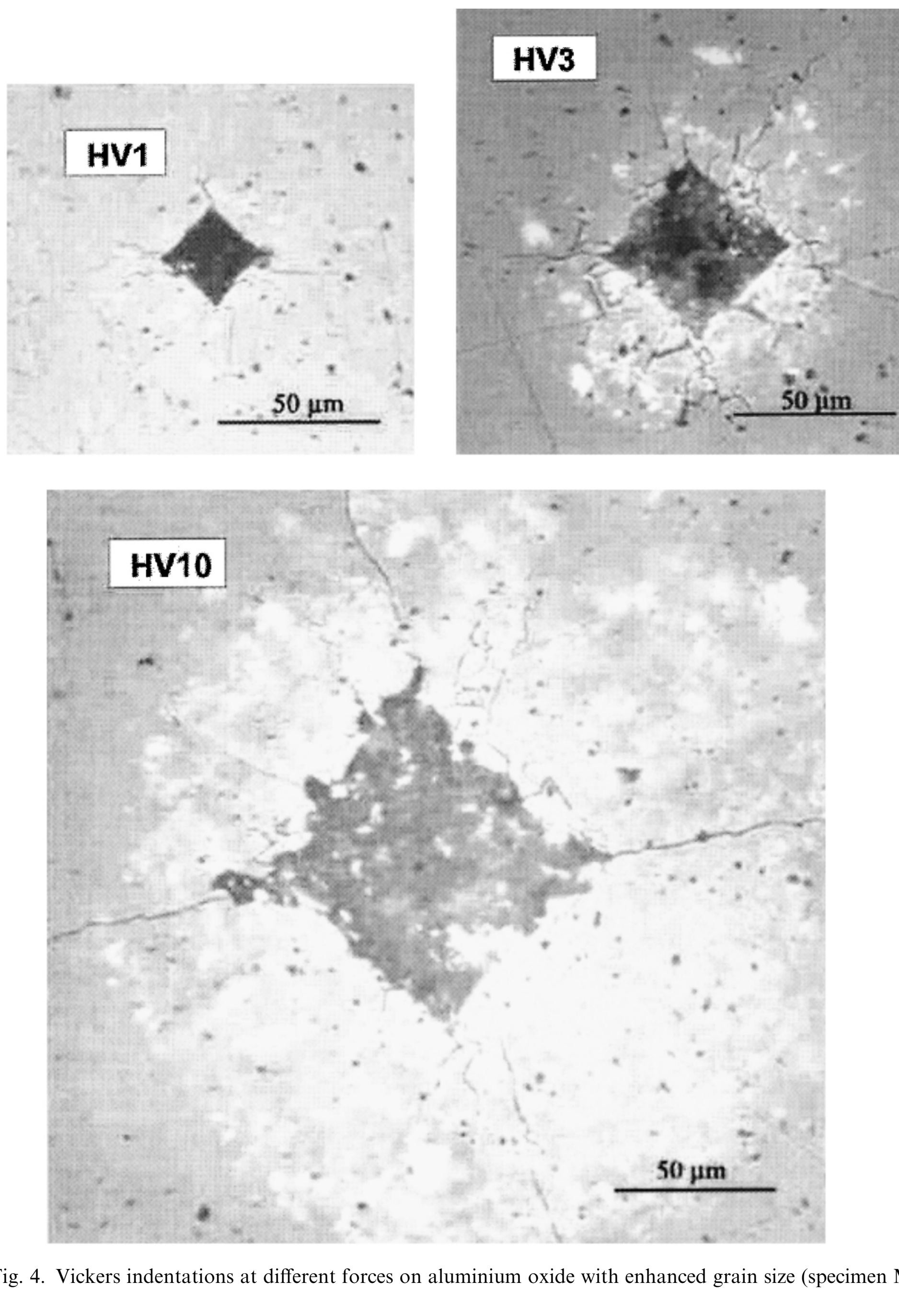

Fig. 4. Vickers indentations at different forces on aluminium oxide with enhanced grain size (specimen M).

However, regarding the influence of the surface preparation technique (i.e. potential for including residual stress) the obtained results do not allow a conclusion due to the scatter of results.

- Using the conventional hardness techniques the separation of the effects of porosity (by Nitrasil R, specimen E), of grain size (by heat-treated Vitox, specimen M), and of cracking (by CeramTec SiC, specimen H) is not possible. A significant reduction of hardness is only observed for the higher porosity of Nitrasil R. The standard deviations are not always increased.
- The diagonal lengths (HV1/HU, HK2/HU) of the indentation after loading by a displacement-controlled table (HU-machine) equal the diagonal lengths after loading by dead-weight hardness machine for both Vickers hardness and Knoop hardness. Only in a few cases did the diagonal length seem to be reduced a little after loading by HU-machine. From this it can be concluded that the plastic indentation size of the specimens tested is essentially machine independent.
- The hardness is reduced with increasing force. However, the standard deviation can increase if the visibility of the indent becomes poorer because of the enhanced radial and lateral cracking, including chipping, as is demonstrated by aluminium oxide (specimen M) in Fig. 4.

5. Results of the interlaboratory exercise

The first set of specimens of the interlaboratory exercise were specimens the hardness of which was to be readily measurable for the laboratories. For this reason reference materials were used, but the laboratories did not receive the calibrated hardness values. In fact, the results on the specimens R, S, and T demonstrated in Fig. 5a–c do not differ more than 4% relative to the (unknown) reference value obtained by the Materialprüfungsamt

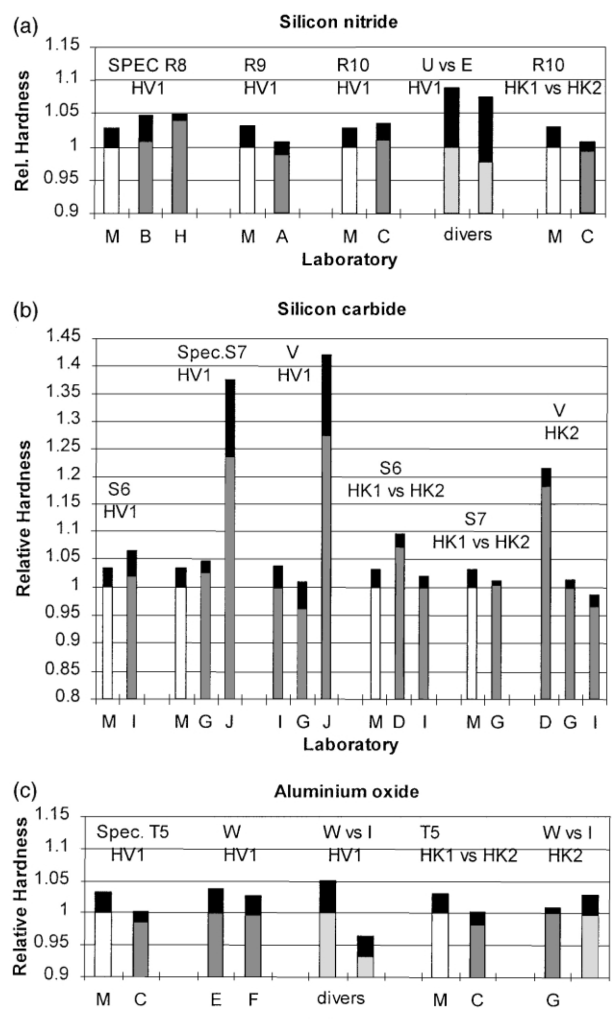

Fig. 5. Hardness results on ceramic materials with the quality of reference blocks (specimens R, S, T) and on typical ceramic materials (specimens U, V, W), normalised to the results for laboratory M: a. silicon nitride; b. silicon carbide; c. aluminium oxide.

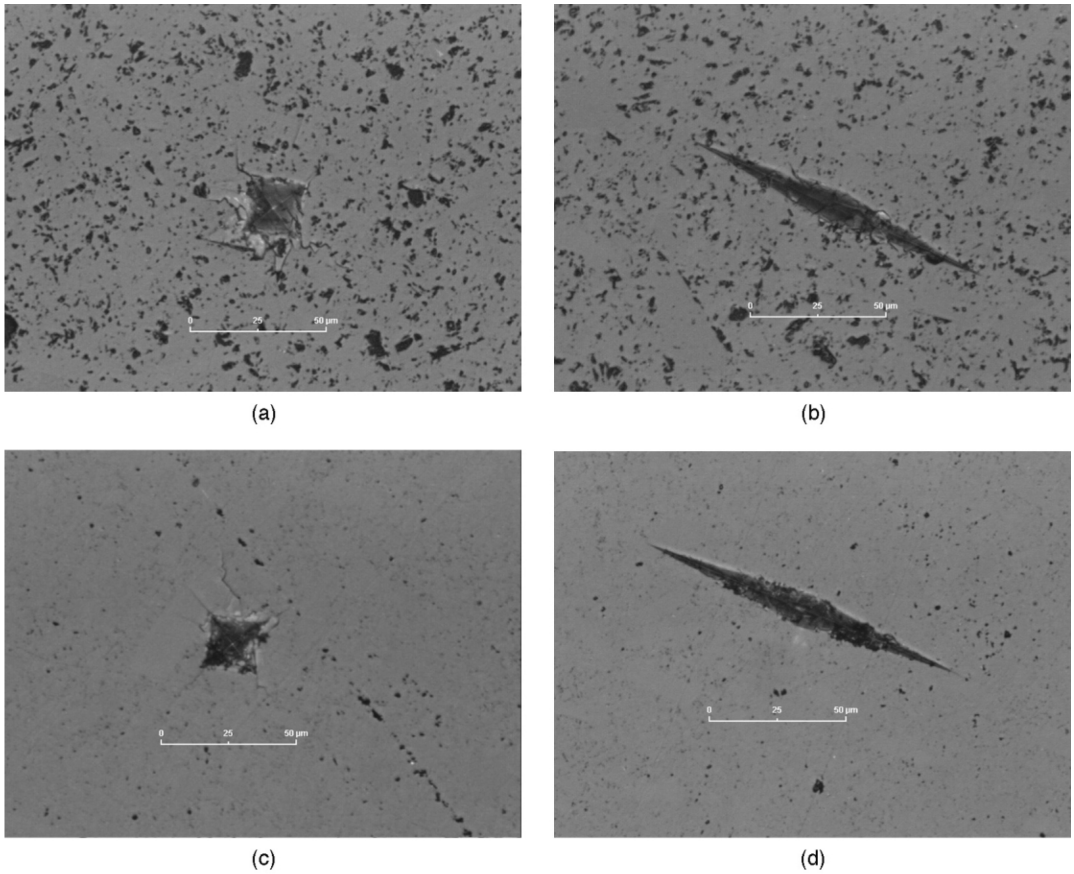

Fig. 6. Demonstration of the critical case for the diagonal measurement involving polish quality on silicon carbide as received (a, b, specimen V1) and after improving polish quality (c, d, specimen V2) for HV1 (a, c) and for HK2 (b, d).

Nordrhein-Westfalen, Dortmund (abbreviated to M in the figures). The coefficients of variation are small (less than 4%). Only two laboratories which have been dealing mainly with metals obtained substantially higher hardness on silicon carbide, specimen S (Fig. 5b).

The second set of specimens of selected ceramics were not so readily measurable. As shown in Fig. 6a and b, the surface quality of the specimen V as received was not optimal. The bad polish state of the very hard material did not allow a precise observation of the indentation. Fig. 6c and d, show the surface with the improved polish state. The Vickers indentations of this silicon carbide are so susceptible to chipping that the tests should be invalid according to the criteria in Ref. 10. However, three laboratories tested specimen V as received to determine the scatter caused by such a bad situation. The results are notable because the coefficients of variation are less than 5% and the mean values of two laboratories do not differ significantly (Fig. 5b). Note, the deviations to the results of the laboratories D and J are not relevant because the consistency of their results on hardness reference blocks is not satisfied. It seems to demonstrate that experts of ceramics can estimate the right location of the missing tips in Figure 6a. As seen in Fig. 6d, the tips of Knoop indentation are more readily visible. Therefore Knoop hardness on specimen V gives smaller standard deviations (Fig. 5b).

The second set of specimens of silicon nitride and aluminium oxide, specimens U and W, have also given hardness values with higher coefficients of variation up to 10% (Fig. 5a and c). Since the specimens U and W have been tested previously as specimens E and I during the first period of project by different laboratories, a comparison with the previous results is of interest. As shown by the white bars in the Fig. 5a and c, the response of the silicon nitride (specimens U and E) and the aluminium oxide (specimens W and I) are different. While the HV1 values of specimens U and E are similar regarding the enhanced scatter, the HV1 values of specimens W and I are significantly different, but the HK2 values of specimens W and I do not differ significantly. Obviously, the selection of the best hardness technique is dependent on the particular response of the tested ceramic material.

6. Sensitivity of the different types of hardness techniques

To demonstrate the ability of Vickers hardness (HV), Knoop hardness (HK), superficial Rockwell hardness

(HRN), as well as the depth-sensing hardness with Vickers pyramid (HU(V)) or a Knoop pyramid (HU(K)), to discriminate between materials, the mean hardness values including the overall standard deviation are plotted in Fig. 7 for the materials classes silicon nitride, silicon carbide, and aluminium oxide. The hardness values were normalised to the material with the smallest scatter within the class, i.e. to material D (silicon nitride), G (silicon carbide), and J (aluminium oxide). The following conclusion can be drawn from these figures:

- For specimens of silicon nitride (A, C, D, E, F) and aluminium oxide (I, J, K, L, M), the hardness rankings are completely the same using HV1, HK2, and HR45N. For silicon carbide (G, H) the different sequences of HV1 and HK2 can not be clearly defined because the scatter of results.
- Even the sequences of the standard deviations are independent of the hardness techniques, implying that a portion of the scatter results from the stochastic response of the ceramic material. Note, in some cases the scatter is much higher than the uncertainty in hardness testing of metals.
- The standard deviations, σ, of the hardness techniques are different with the sequence σ (HV1) > σ (HK2) > σ (HR45N).

If the ability of the hardness techniques to discriminate between materials can be evaluated using the ratio of hardness difference to average standard deviation, σ:

$$D_{ij} = \frac{2(H_i - H_j)}{(\sigma_{Hi} + \sigma_{Hj})}$$

The discrimination abilities, D_{ij}, of the three conventional hardness techniques are very similar for the silicon nitrides as is shown in Table 5. These results have been confirmed using the *t*-test (see Table 3). In particular, the pairs of ceramics C vs D and X5 vs X7 are very close in properties. Significant differences are indicated by $D_{ij} > 1$ in Table 5.

For completing the sensitivity analysis, three laboratories tested four silicon nitride ceramics the processing and properties of which are closely similar. The results are demonstrated in Table 5 and Fig. 8. With the exception of HK2 by laboratory C, all results on the group of silicon nitride ceramics, X4, X5, X5, and X7, are ranked the same independent of the hardness technique. As seen in Table 5, the results of the first tests on the specimens A, C, D, and E are confirmed completely.

Table 5
Discrimination ability, D_{ij}, of the hardness techniques on the silicon nitride ceramics of which processing and properties are closely similar

Comparison between the specimens	Hardness techniques		
	HV1	HK2	HR45N
D vs A	2.4	2.1	
C vs D	0.4	1.2	0.6
D vs F	2.5	1.8	2.5
X7 vs X5	0.6	−0.2	0.5
X5 vs X6	1.1	2.6	2.3
X6 vs X4	1.0	2.8	1.5

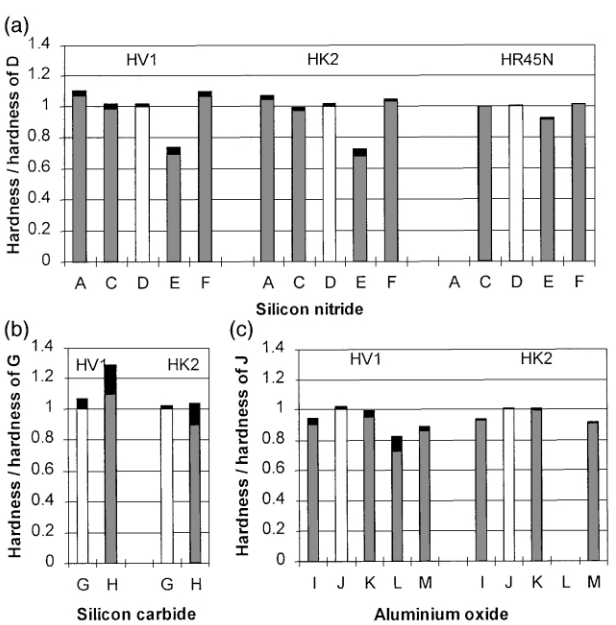

Fig. 7. Hardness results on (a) silicon nitride, normalised to the results from laboratory D, (b) silicon carbide, normalised to the results for laboratory G, and (c) aluminium oxide, normalised to the results for laboratory J.

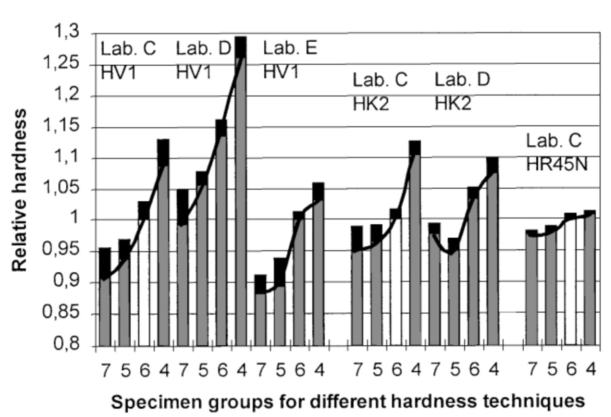

Fig. 8. Results of four closely similar silicon nitride specimens tested by three different methods showing consistency of ranking, normalised to the results for laboratory C on material X6.

7. Discussion and conclusion

A discussion on the reliability of the results has to concern the human observer, the technique, and the response of ceramics.

The application of high-hardness reference blocks for training users to obtain comparable HV1 and HK2 hardness values has been generally successful, resulting in a reduced scatter between the laboratories. If the hardness response of the tested materials does not involve chipping and cracking the coefficients of variation range typically from 1 to 3%.

The small scatter measured on ceramics with optimal hardness response permits discrimination of two materials such as specimens C and D of which HK2 values are different by as little as 2.5% (Table 4). That means the averages of the indentation diagonals vary by 1.8 μm. However, the HV1 values of the two materials cannot be discriminated completely as proved by the *t*-test (Table 3). An explanation is the smaller difference of the mean diagonal lengths of only 0.4 μm. It is less than the optical resolution of about 1 μm.[3,12,13]

Figs. 7 and 8 as well as Table 5 demonstrate that the three hardness techniques used in this study have the similar ability to discriminate between materials of closely similar hardness. This is surprising, particularly concerning HR45N. Differences of less than 1 can be discriminated whereas standard deviations of 0.1 μm are obtained, i.e. a scatter of 0.1 μm at the depth difference of about 12 μm.[6] It is unexpected that the scatter is not increased by cracking caused by the high force of 441 N. A typical pattern of the spherical indentation formed by the Rockwell indenter is given in Fig. 9. Radial cracks appeared which do not seem to influence substantially the plastic deformation.

The smaller the scatter, the better is the ability to detect effects of measurement procedure. For instance, the time dependence of the deformation under load is known to be smaller for ceramics than for metals. In this way, an effect of the holding time has not been detected easily. For the same reason, the statistical tests on Vickers/Knoop hardness results show that all results of various laboratories do not differ significantly from the results obtained by using an HU-machine and measuring optically. That means the results are modified only slightly by the different force-time regime in the test of depth sensing hardness compared with those of the conventional Vickers/Knoop hardness procedure. However, a small reduction in hardness can be detected for the HU machine if the two results are compared within the same laboratory using optical measurement with the same microscope. Such a level of detection can be expected only for the specimens of high quality (specimens A, C, D). Therefore, a hardness standard for typical commercial ceramic materials does not need strong restrictions for the time–force regime.

In the case of typical commercial ceramic materials the scatter of hardness is higher even if great effort is employed to improve the polish quality. The rankings of the standard deviations of different materials are independent of the hardness technique and of the laboratory, implying that a major portion of the scatter results from the stochastic response of the ceramic material, i.e. the variation in microstructure (porosity, grain orientation, grain size, secondary phase distibution, etc.).

Surface roughness caused by the porosity of technical ceramics cannot be reduced by improved polishing. Therefore, a part of the higher scatter seen in these materials is based on the enhanced natural surface roughness caused by the particular microstructure of the ceramics (comparison of the specimens C, D with E, as well as specimen J with K and L in Table 4).

Enhanced crack formation, partly connected with chipping, disturbs the visibility of indentation edges (for instance SiC, specimen V, in Fig. 6). In this way the

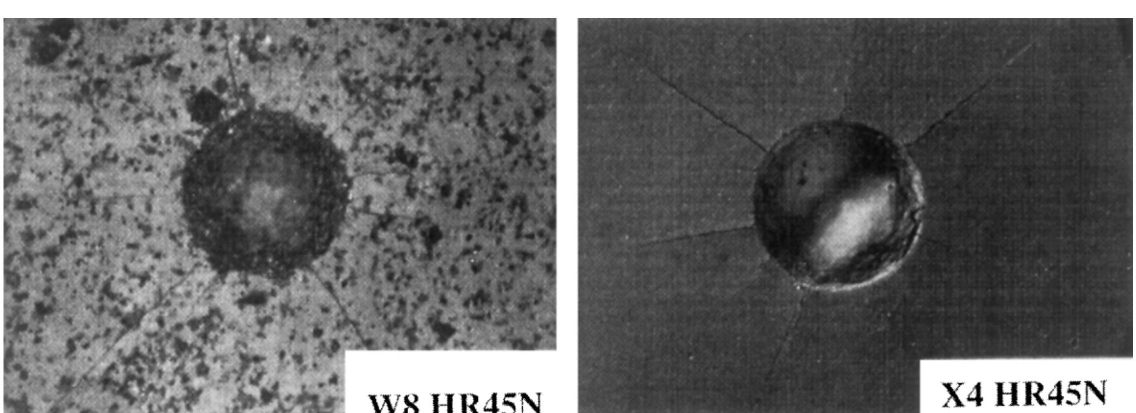

Fig. 9. Rockwell indentation HR45N on aluminium oxide (specimen I or W, left) and on HPSN (specimen X4, right). Radius of impression is about 160 μm.

optical measurement of the indentation geometry becomes more difficult. However, it is noteworthy how reproducibly the tests can be performed by laboratories with experience in ceramics, although the scatter is slightly enhanced by the particular indentation responses (SiC-specimens G, T, V and Al_2O_3-specimens I, K in Table 4 and Fig. 5). It seems to be better to base the acceptance of the generated indentations on the visibility of edges than on the kind of cracking according to the guidelines.[10] Because of the high scatter, the dependence of the hardness on cracking is not significant.

For those materials prone to chipping and cracking (for instance SiC) or for materials with poor visibility of the indentations (for instance Al_2O_3), the application of reference blocks with well-shaped indentations does not provide improved comparability of results between the laboratories. As a consequence, in addition to the use of conventional hardness blocks for checking the calibration of the machine ceramic reference blocks with more realistic hardness response are to be used. The hardness response of such a reference material has to demonstrate better the real pattern of the indentations even if the scatter is enhanced. Therefore, the mean hardness of the ceramic reference material should be determined by a round robin of laboratories with the experiences on ceramics. It is also recommendated to use reference blocks with the same type of optical contrast. For instance, a silicon nitride block can be used for darker or relatively opaque ceramics, but a white block should perhaps be used for optically white or translucent materials in which the corners of the indentation are much more difficult to see.

It also appears that the selection of the optimal hardness techniques and an appropriate test forces must be varied in accordance to the particular hardness response.

It is quite clear that with increasing test force resulting in more enhanced cracking, the scatter of the hardness test becomes so high that the measurement must be rejected. There is a balance between sufficiently large indentations (the high hardness of ceramics gives small indentations) and sufficiently little cracking. For instance, the coefficients of variation for the Vickers hardness on specimen I (aluminium oxide) at 9.8, 29.4, and 98.1 N are 3.0, 1.4, and 3.2%, respectively. In fact, the test at 29.4 N with the smallest coefficient of variation seems to be optimal for this material. Because such a best test force is dependent on the material's response, a precise comparison of different technical ceramics requires the selection of an appropriate test force which in principle cannot necessarily be defined by a standard force.

The same point should be taken into account regarding the observed tendency that the standard deviations of the different hardness techniques decrease in the order HV1, HK2, and HR45N. For different testing forces, a variation of ranking can be expected.

The standard ENV 843-4 has been confirmed as a useful tool for the determination of hardness on advanced technical ceramics. Some conditions of the standard are stronger than necessary. There is no effect of the holding time (5–60 s), of the distance between the indentations (more than the threefold diagonal length), of the inclination related to the axis of the indenter (less than 1°, only proved for Vickers indenter), and only a narrow effect of the kind of loading (including HU-machines).

The separation of the effects of porosity, of grain size, and of cracking is not possible using traditional hardness techniques. More information on the elastic–plastic behaviour is expected to be obtainable from the use of the depth sensing hardness test.[9]

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