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Technical Working Area 3
CERAMICS

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Final Report

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Technical Working Area 3
Ceramics

VAMAS Round Robin on Conventional Hardness Test on Whisker Reinforced Ceramics

by

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Summary

This report describes the results of a VAMAS round robin to examine the conventional hardness test methods to whisker reinforced ceramics. Silicon nitride ceramics with 0vol%, 10vol% and 20vol% silicon carbide whiskers were tested, using Vickers and Knoop hardness test methods, by 21 participants from 9 countries. The hardness values increases with the increase of whisker content on both of Vickers and Knoop tests. The scattering of the data was not so different on monolithic and composite samples. Indenting surface and direction did not affect the hardness values so much, compare to the scattering of the data. We can conclude that Vickers and Knoop hardness tests are useful for the characterization of whisker reinforced ceramics.

Keywords: ceramics, round robin, hardness, composites, silicon nitride, silicon carbide, whiskers, Vickers, Knoop

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Participants List

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	Dr. M. Mizuno	Japan Fine Ceramics Center
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	Mr. T. Nose	Nippon Steel Co.
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UK	Dr. R. Morrell	National Physical Laboratory
USA	Dr. J. R. Varner	New York State College of Ceramics at Alfred University
	Mr. G. Quinn	National Institute of Standard and Technology

1. Introduction

Hardness is one of the most common parameters for the evaluation of structural ceramics. In VAMAS TWA#3 project, hardness round robin test was conducted by National Physical Laboratory of UK, in 1988. In this round robin, Vickers, Knoop and Rockwell superficial hardness were discussed. After this round robin, standard test methods of hardness for engineering ceramics were published in Japan (JIS R 1610 (1991)), in EU (CEN/TC184 ENV843-4 (1994)) and in USA (ASTM C1326 and C1327 (1996)). In ISO/TC206 "Fine Ceramics" committee, test method of hardness has been discussed since 1994, for making a draft for ISO standard. Therefore, we can say, we already have a sort of basic agreement, for testing the hardness of engineering ceramics.

However, we are not sure that these test methods can be applied for the test of ceramic composites. In ISO/TC206, Vickers and Knoop hardness tests are discussed. Knoop hardness may show some anisotropy of the data with indenting directions, so, it is reasonable to test it on ceramic composites, for confirming the usage is reasonable or not.

In this round robin test, silicon carbide whisker reinforced silicon nitride is tested.

(note) In the committee draft of the standard on test method for hardness on fine ceramics in ISO/TC206 (ISO/CD14705), it is written that the usage of the unit of GPa is preferred, for expressing the hardness. However in this report, conventional hardness number (test force, in kgf, divided by defined area, in mm^2, and no unit specified) is used, as following the existing ISO standards (for example, ISO 4545, ISO 6507-2).

2. Materials

Tested ceramic composites were commercially supplied ceramics by Japan Metals &Chemicals Co.ltd., named Kryptonite. It is formed by slip casting, and pressureless sintered body. Three kinds of whisker content, 0vol%, 10vol% and 20vol% were prepared. The shape of the test piece was rectangular bar of 4.5 x 3.5 x 19 mm. One surface of 4.5 x 19 mm and one surface of 3.5 x 19 mm were polished. JFCC machined and polished the samples. The symbols were A (0%), C (10%) and D (20%). For distinguishing the surfaces, 4.5 x 19 mm surface was called "top", and 3.5 x 19 mm surface was called "side". Properties table for sample D ,supplied by the manufacturer, is shown in table.

Photo 1 shows the optical micrographs of the polished surfaces of the samples.

Each participant received 2 pieces of "A" sample, and 3 pieces of "C" and "D" samples.

Table Properties of $SiC_{(w)}/Si_3N_4$, Kryptonite

Manufacturer	Japan Metals & Chemicals Co.,Ltd.
Main Component	Silicon Nitride
SiC-whisker	20 vol%
Density	3.20×10^3 kg/m^3
Porosity	less than 0.1%
Flexural Strength	880 MPa (RT)
	880 MPa (1000°C)
	490 MPa (1100°C)
Fracture Toughness	6.2 MPa. $m^{0.5}$
Thermal Expansion Coefficient	4.1×10^{-6} °C^{-1} (RT to 1200°C)
Thermal Conductivity	19.2 W/m· K (RT)
	14.0 W/m. K (1200°C)
Thermal Shock	950 °C

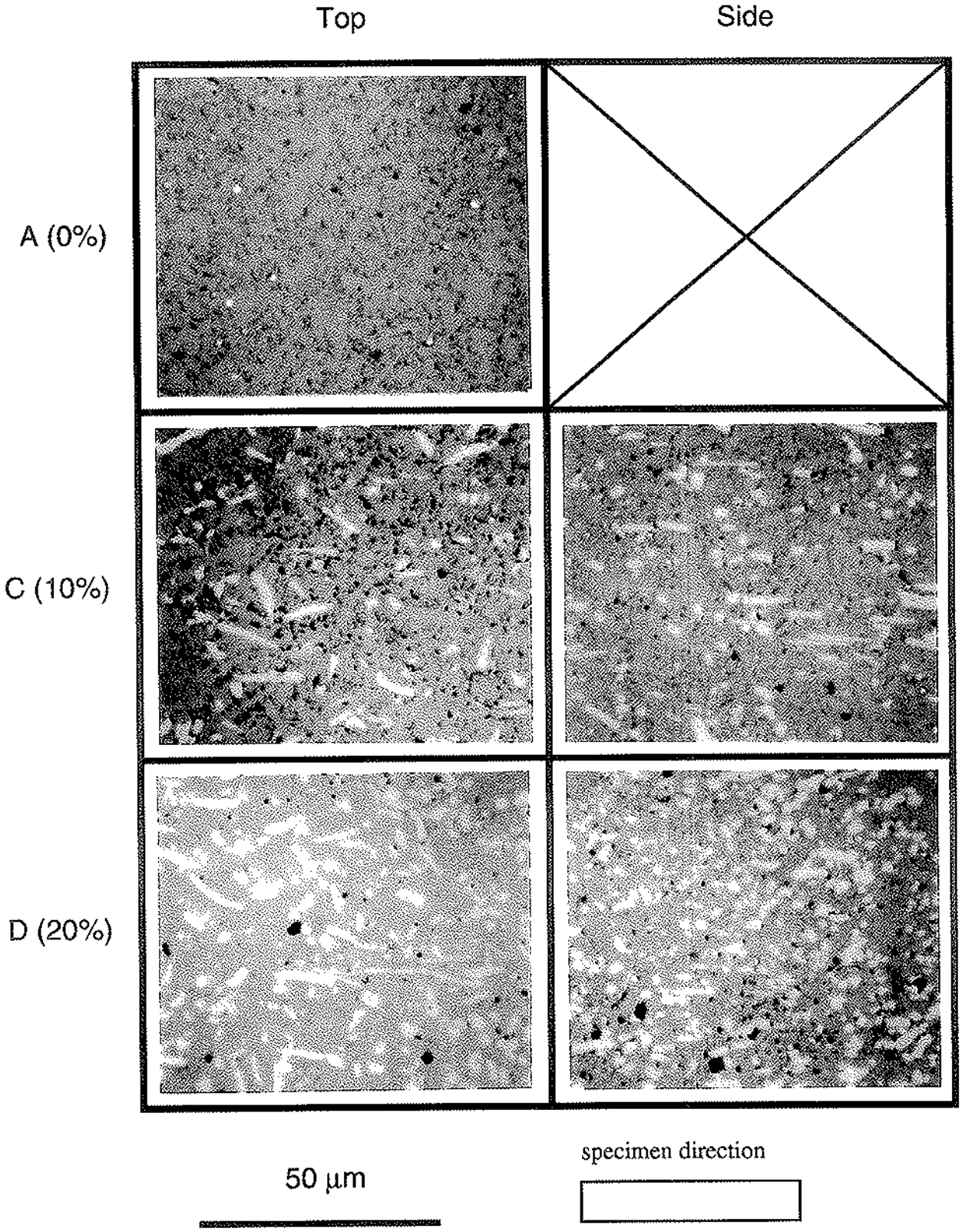


Photo 1 Optical micrographs of the polished top and side surfaces of each sample. Light phases on sample C and D are SiC whiskers.

3. Experiments

Experimental procedures were informed by the instruction sheets, which are attached in the Appendix. Five indents were made for one test condition, and the mean values and the standard deviations were calculated.

3.1 Vickers hardness

For Vickers hardness, test forces of 1kgf and 10kgf were used. In the case of Vickers, indenting direction does not affect the results, then the test conditions were;
(1) For A sample, tests were done only on the top surface. Indent number was 10 (= 5 indents for 1 sample in 2 test forces).
(2) For C and D samples, tests were done on the top and the side surfaces. Indent number was 40 (= 5 indents for 2 samples for 2 test forces on 2 surfaces).

3.2 Knoop hardness

For Knoop hardness, test forces of 1kgf and 2kgf were used. In the case of Knoop, the direction of the indent and the direction of the whiskers may affect the results. Then, test conditions were;
(1) For A sample, tests were done only on the top surface. Indent number was 10 (= 5 indents for 1 sample in 2 test forces).
(2) For C and D samples, tests were done on the top and the side surfaces. On the side surface, 2 different test direction, parallel or perpendicular to the long direction of the specimens, were tested. Indent number was 60 (= 5 indents for 2 samples in 2 test forces on 3 surfaces and directions).

Then, the maximum number of the indents at one participant became 120. As written in the instruction, the test on sample C was optional, and participants can skip some test conditions if their equipment did not fit for some test conditions, mainly on test forces. So, some of the participants test the indents less than 120.

Photo 2 shows some examples of the indentations on sample D in each test condition.

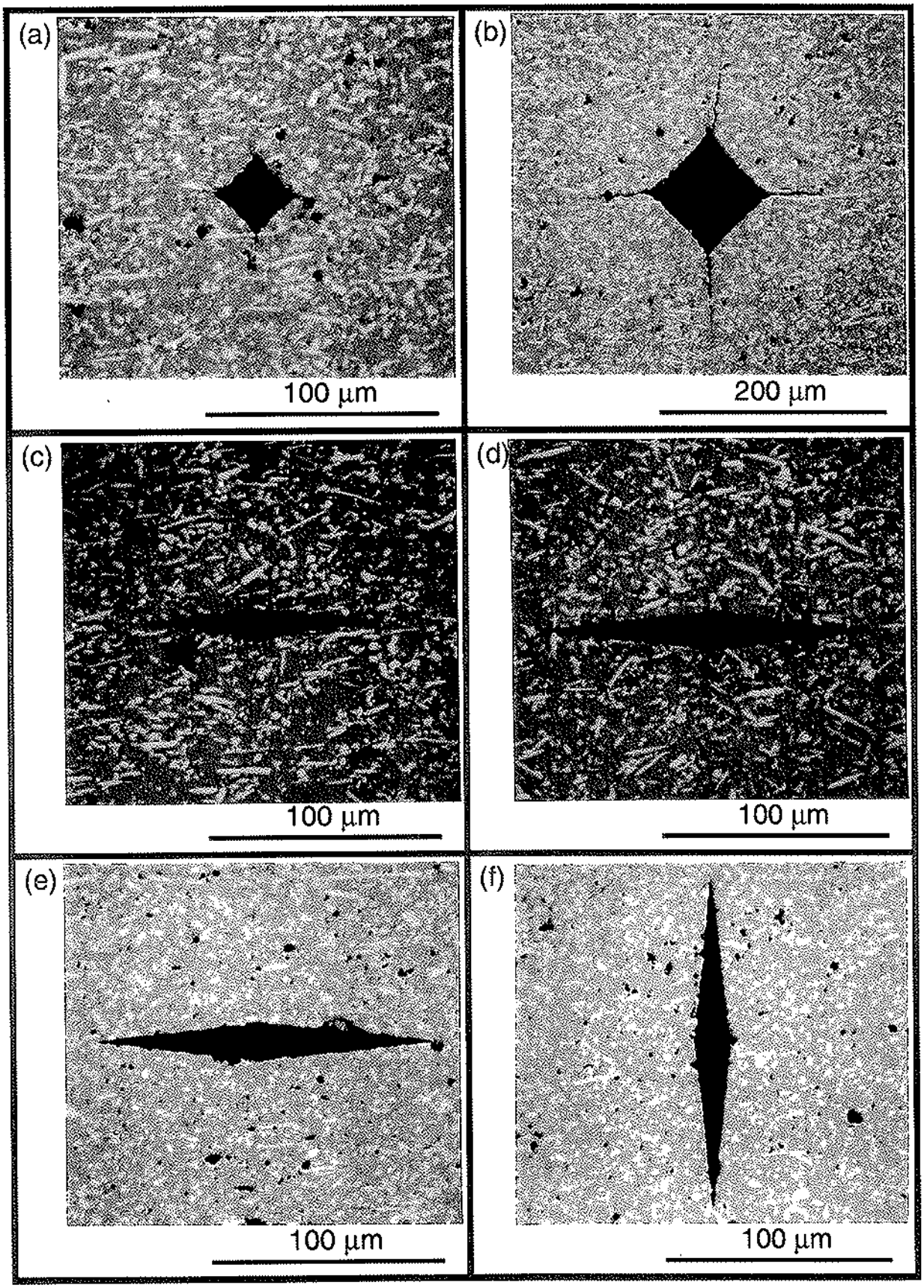


Photo 2 Examples of the indentations on sample D.
(a) HV1 on top surface, (b) HV10 on top surface (note the scale difference),
(c) HK1 on top surface, (d) HK2 on top surface,
(e) HK2 on side surface parallel to the long direction of the specimen,
(f) HK2 on side surface perpendicular to the long direction of the specimen.

4. Results and Discussion

Figure 1-1 shows the average values measured at the top surface from all the participants. The error bar indicates the standard deviation of the average values from all the participants. We can see the results as follows;
(1) Hardness values, both Vickers and Knoop, increase with whisker content. Hardness of D sample is 7 to 10% larger than A sample on all test conditions.
(2) Test force dependence was found on both Vickers and Knoop hardness. Hardness value becomes smaller with higher test force.

Figure 1-2 shows the average of standard deviation from each participant. That means this figure shows the tendency of the scattering of the data in each participant. The tendency of the values are not so different on A, C and D samples. That means the scattering was not so different on monolithic silicon nitride (sample A) and whisker composites (sample C and D). The standard deviation values are smaller on higher test force results on both of Vickers and Knoop tests. Larger indents were made by higher test force, and it can reduce the scattering of the hardness values.

Figure 1-3 shows the standard deviation of the average values, obtained from each participant. That means this figure indicates the scattering among the participants. The tendency is similar to Figure 1-2. Composite samples show a little larger scattering, but the difference is not so large compare to the scattering on monolithic sample. The scattering becomes smaller in larger test force data, and scattering of HK2 test was very small compare to other test conditions.

Figure 2-1 to 2-4 show the surface and indent direction dependence of the Vickers and Knoop hardness. As you can see, the difference of the data on each condition is smaller than the standard deviation (shown in error bar). we can conclude that the surface and the direction did not affect the hardness values of the whisker composites. However, in Figure 2-3 and 2-4, you can see the average values of the "side perpen" condition are always larger than the "side para" results. It is interesting even though the difference is small enough, compare to the standard deviation.

Form Figure 3-1 to 3-24, you can overview the results from each participants. You can see some tendency exist in some laboratories, some always report high or low values in HV or in HK. What is interesting is, those who reports high HV values do not always report high HK values. It was a mistake of me, round-robin organizer, that I did not ask returning the samples with the results, for confirming the reason of this kind of tendency. However, we can think of the usage of standard blocks for understanding this kind of scattering.

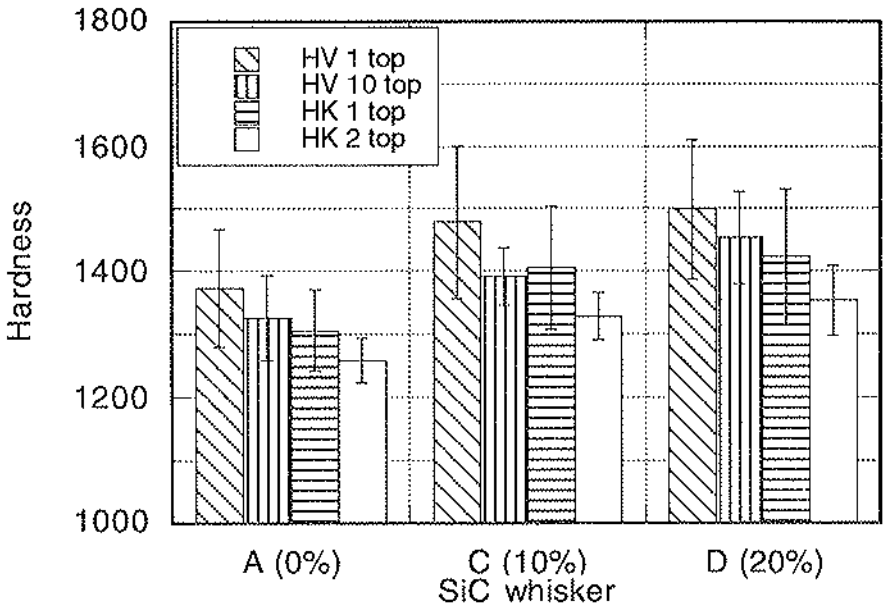


Fig. 1-1 Average of all the data on top surface.

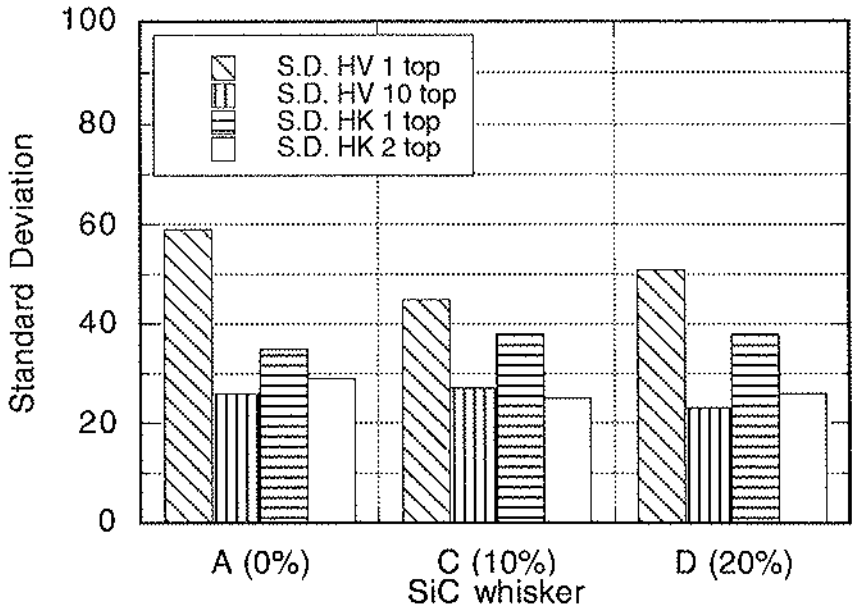


Fig. 1-2 Average of the standard deviation from all participants on each test condition.

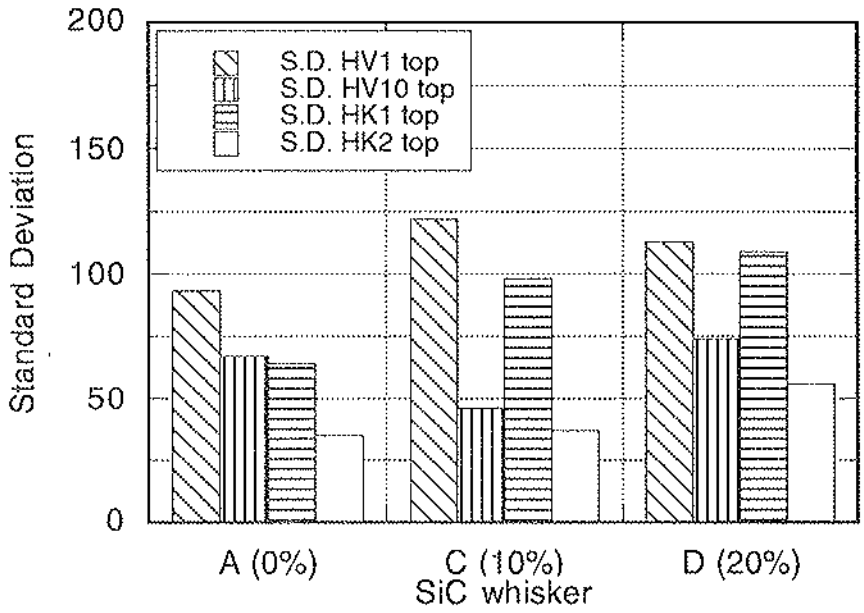


Fig. 1-3 Standard deviation of the average of all participants on each test conditions.

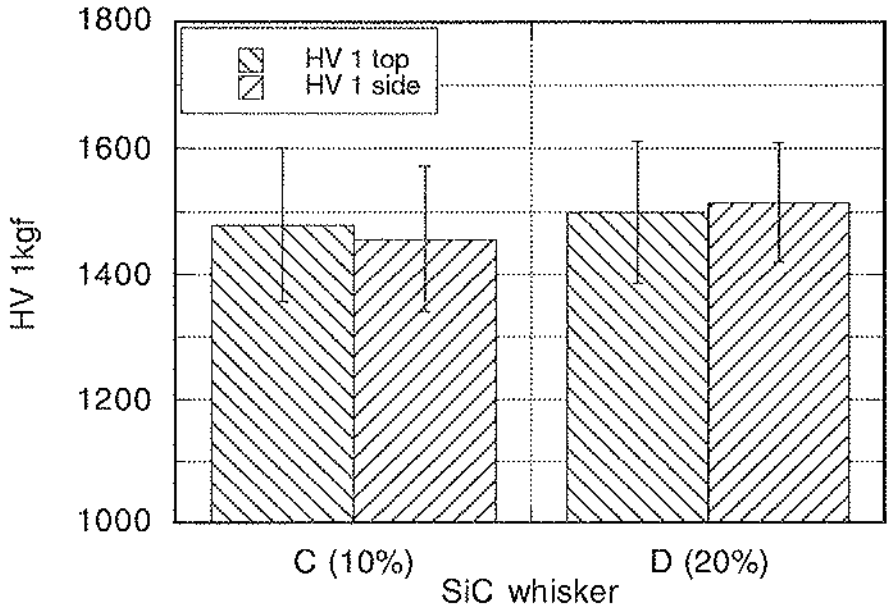


Fig. 2-1 Average of HV1 hardness from top and side surface on sample C and D.

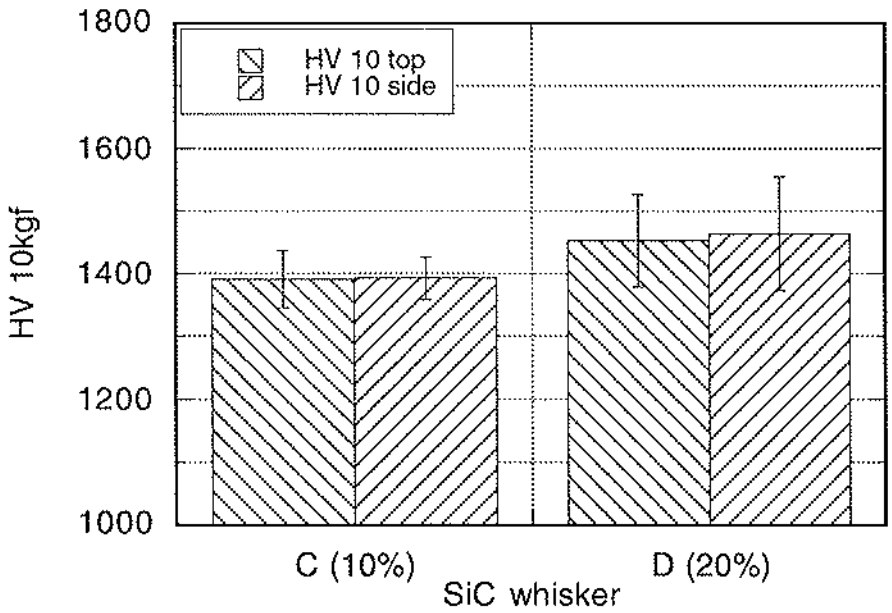


Fig. 2-2 Average of HV10 hardness from top and side surface on sample C and D.

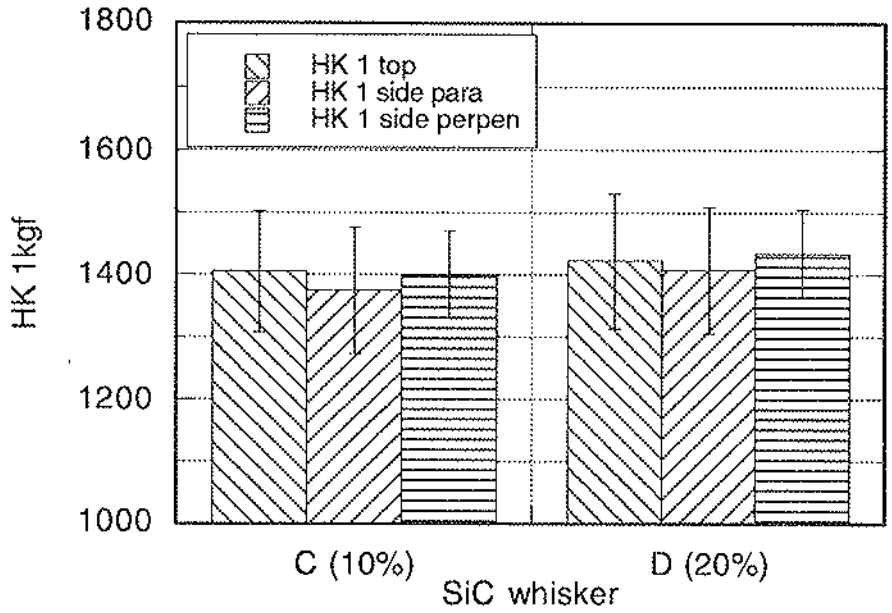


Fig. 2-3 Average of HK1 hardness from top and side surface on sample C and D.

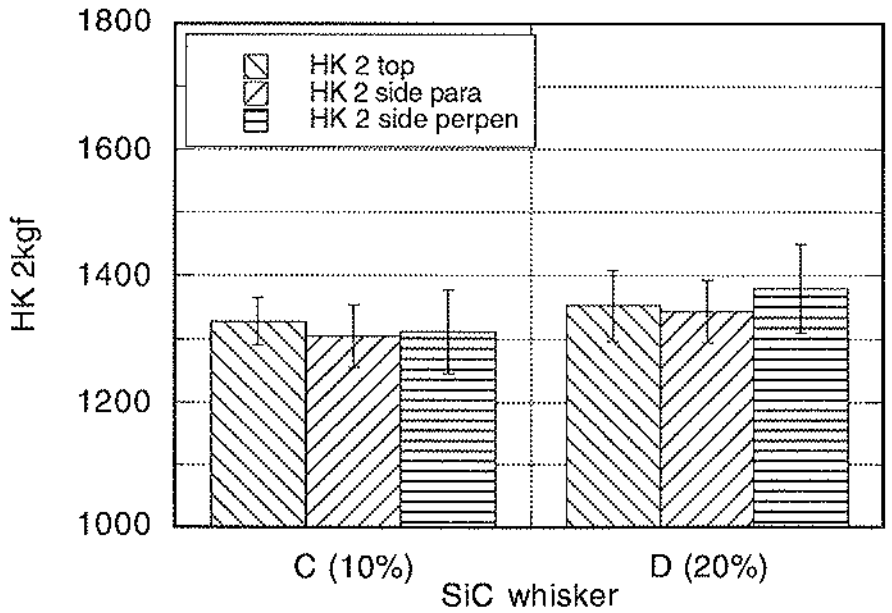


Fig. 2-4 Average of HK2 hardness from top and side surface on sample C and D.

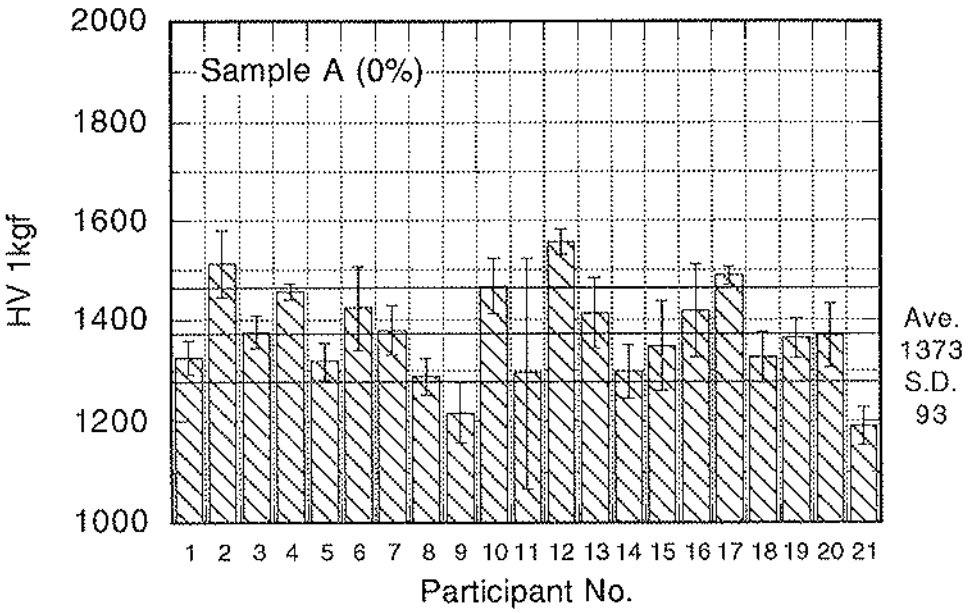


Fig. 3-1 HV1 for top on sample A from each participant.

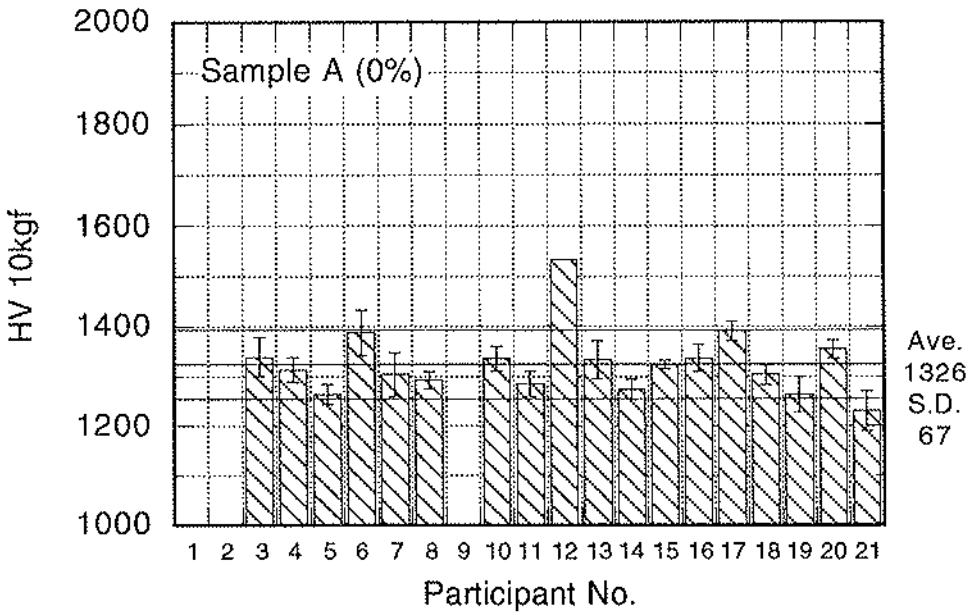


Fig. 3-2 HV10 for top on sample A from each participant.

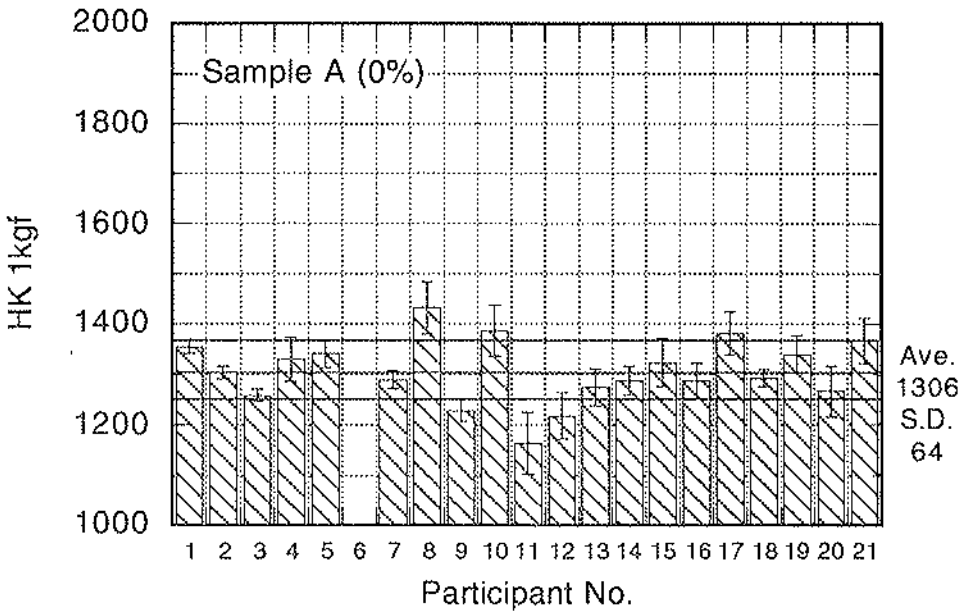


Fig. 3-3 HK1 for top on sample A from each participant.

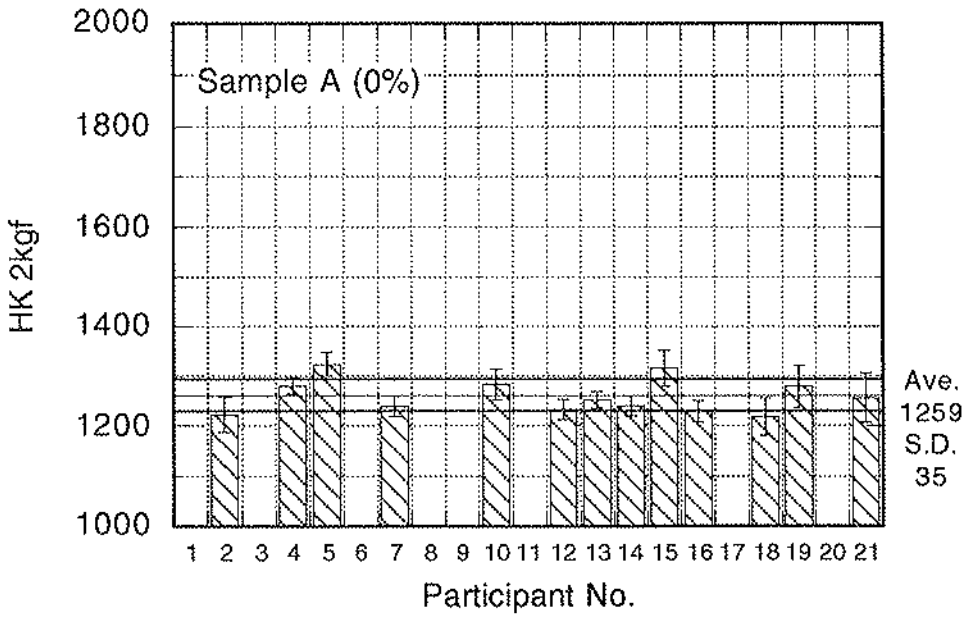


Fig. 3-4 HK2 for top on sample A from each participant.

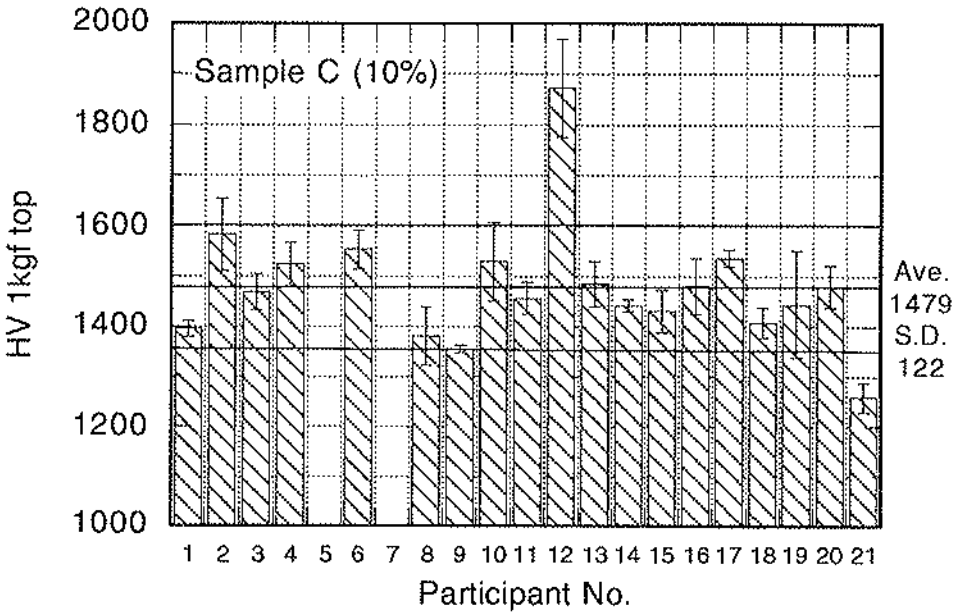


Fig. 3-5 HV1 for top on sample C from each participant.

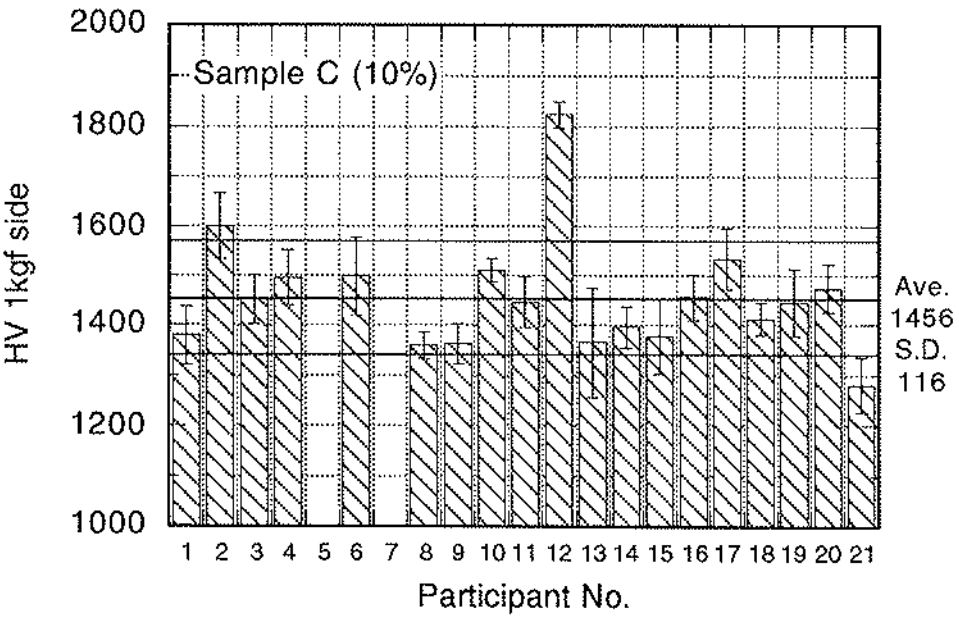


Fig. 3-6 HV1 for side on sample C from each participant.

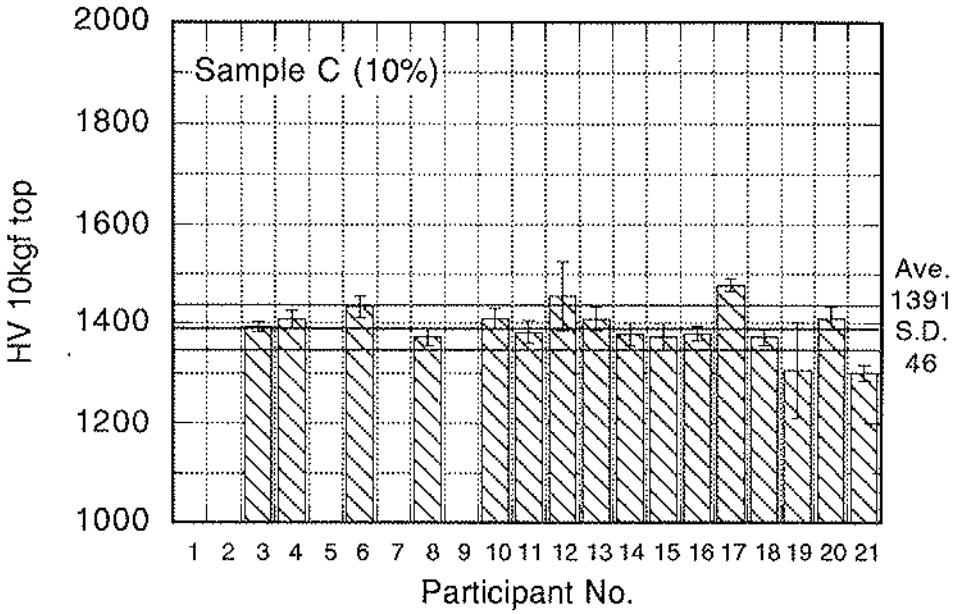


Fig. 3-7 HV10 for top on sample C from each participant.

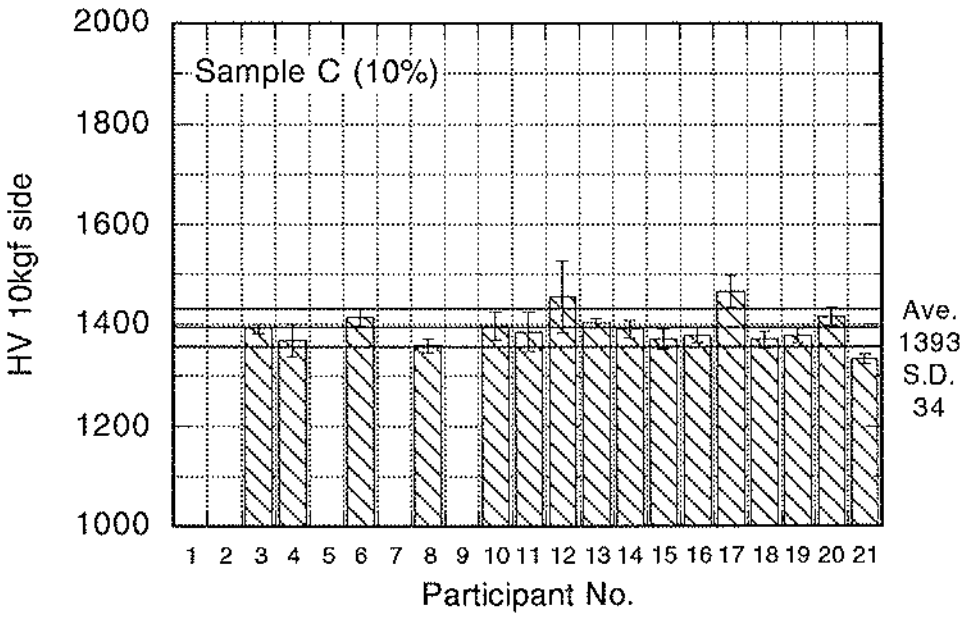


Fig. 3-8 HV10 for side on sample C from each participant.

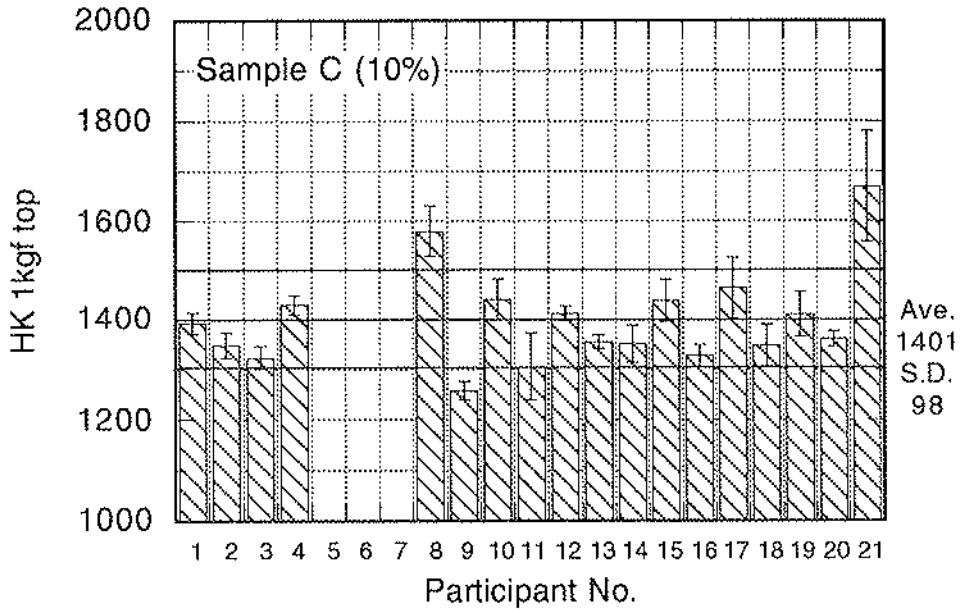


Fig. 3-9 HK1 for top on sample C from each participant.

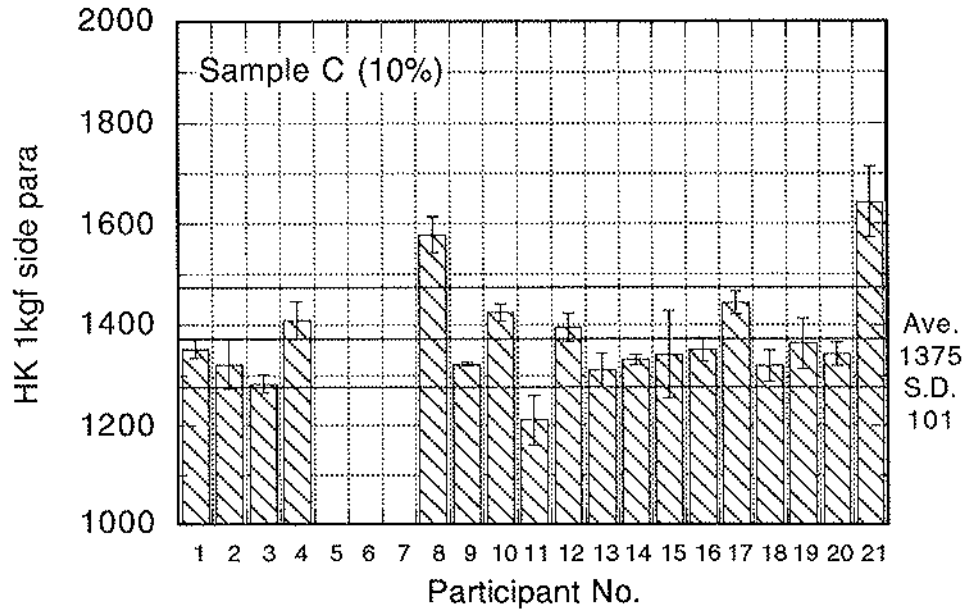
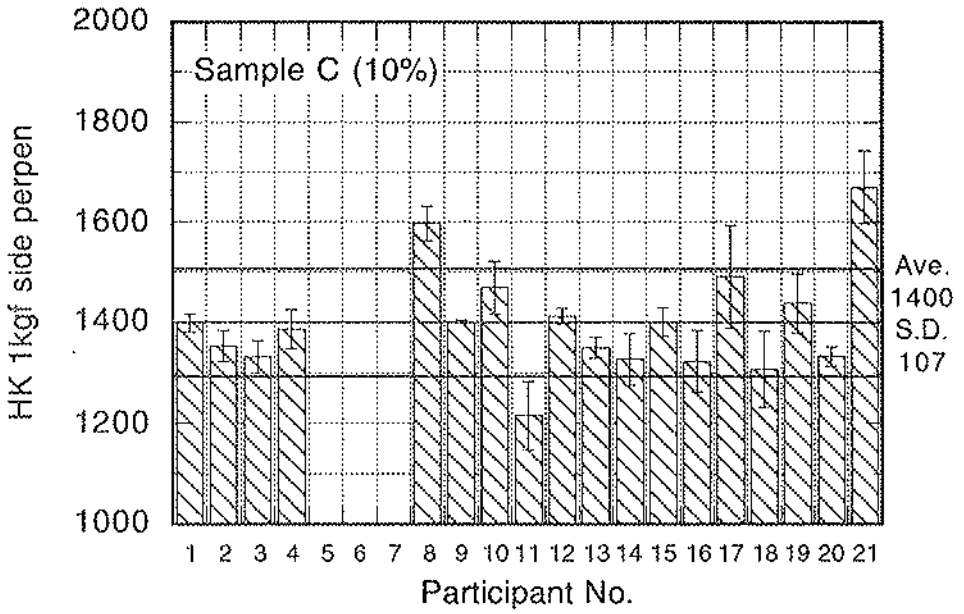


Fig. 3-10 HK1 for side and parallel to the sample on sample C from each participant.



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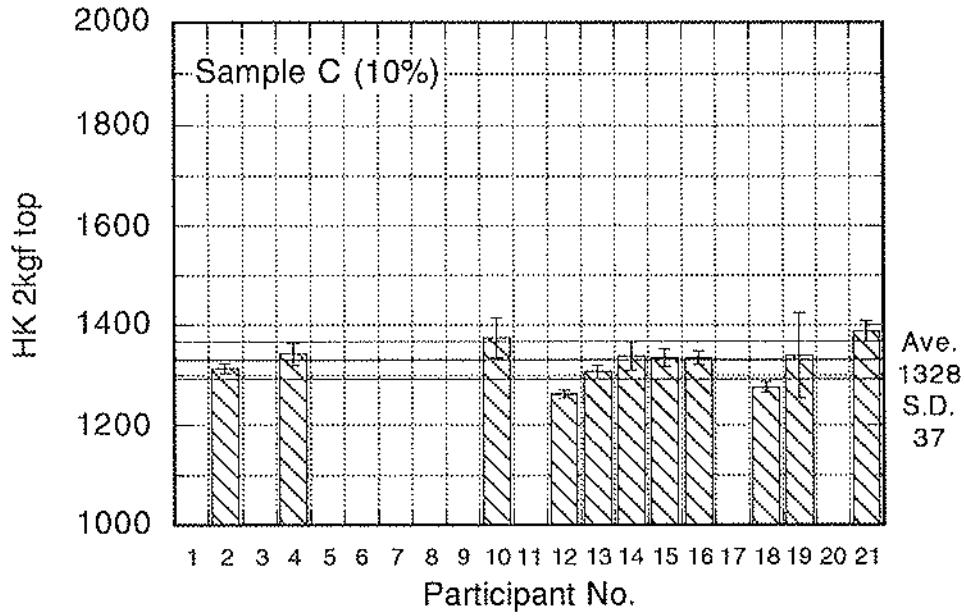

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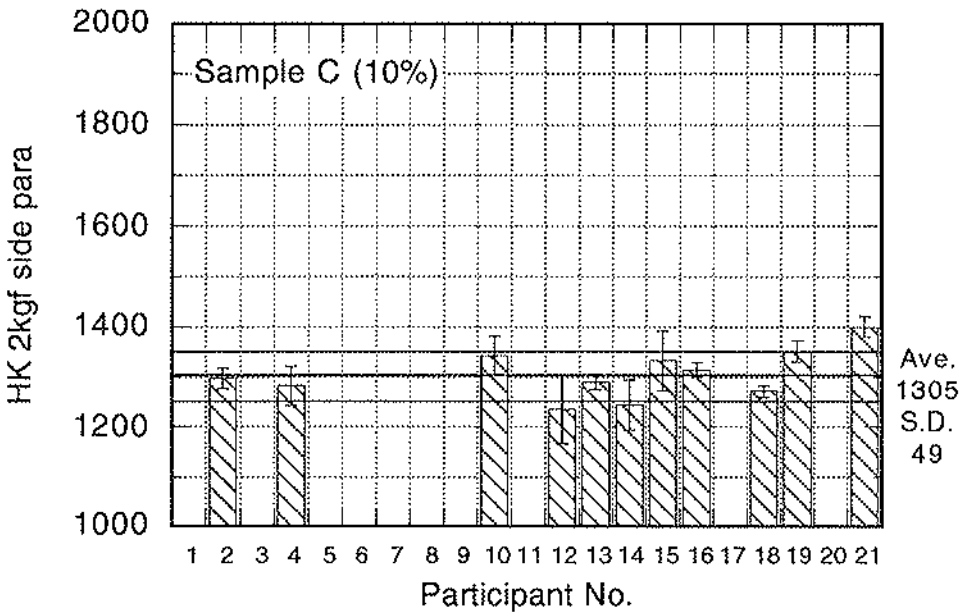


Fig. 3-13 HK2 for side and parallel to the sample on sample C from each participant.

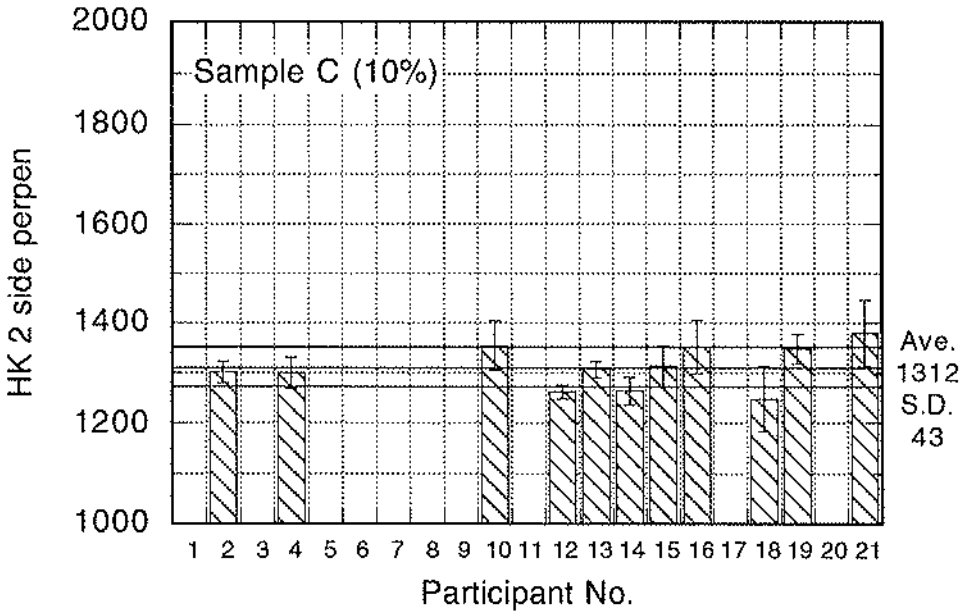


Fig. 3-14 HK2 for side and perpendicular to the sample on sample C from each participant.

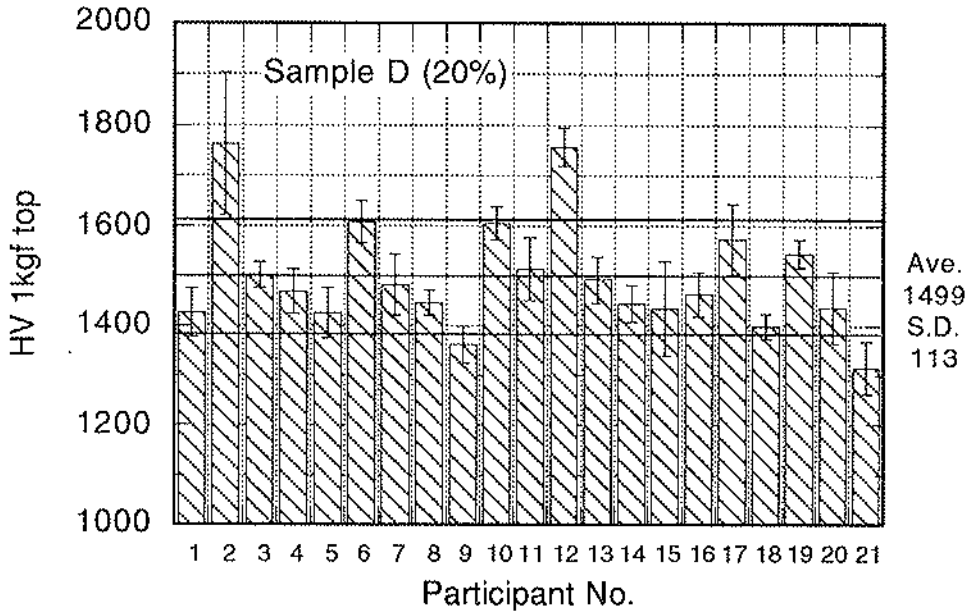


Fig. 3-15 HV1 for top on sample D from each participant.

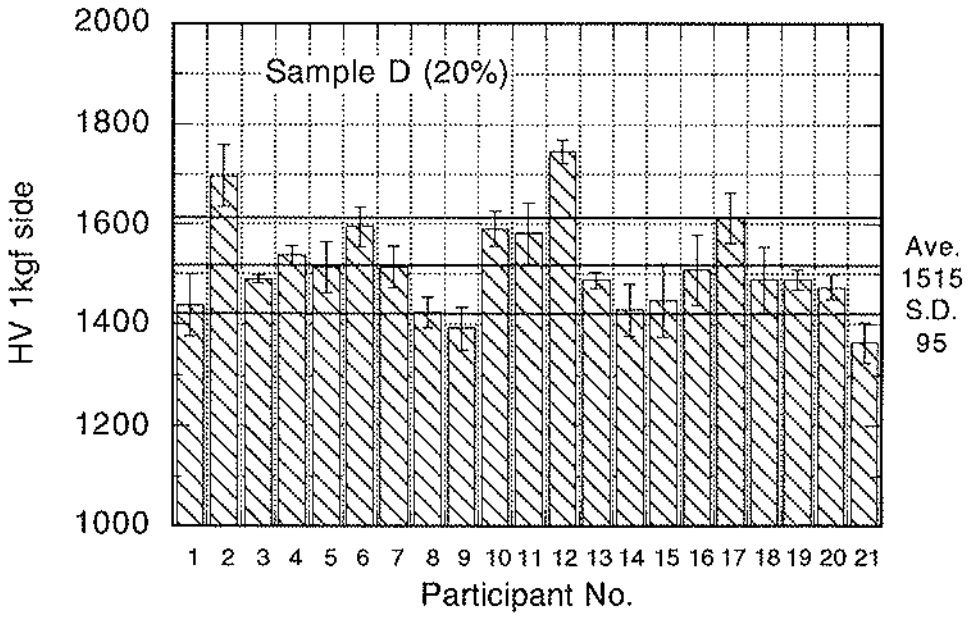


Fig. 3-16 HV1 for side on sample D from each participant.

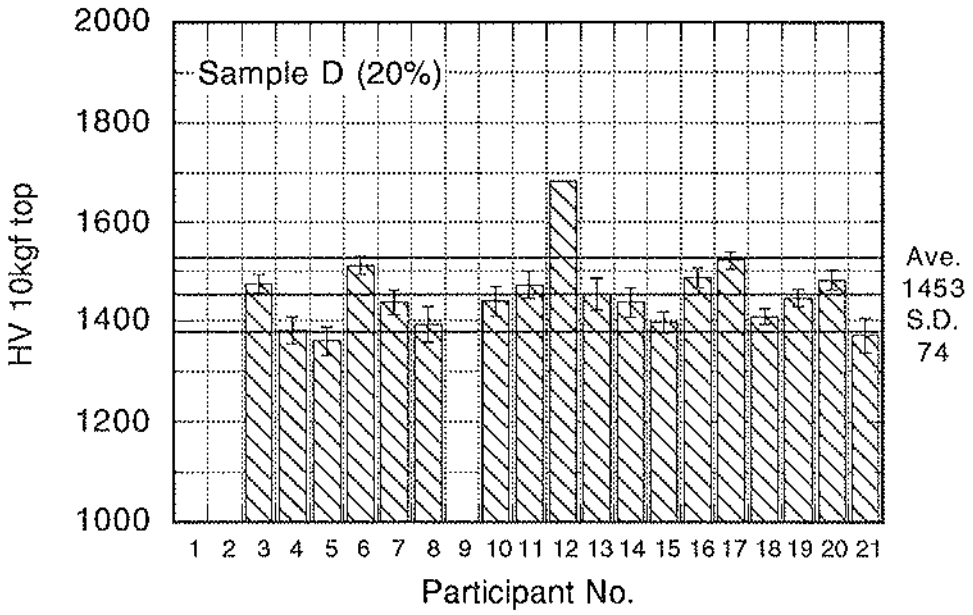


Fig. 3-17 HV10 for top on sample D from each participant.

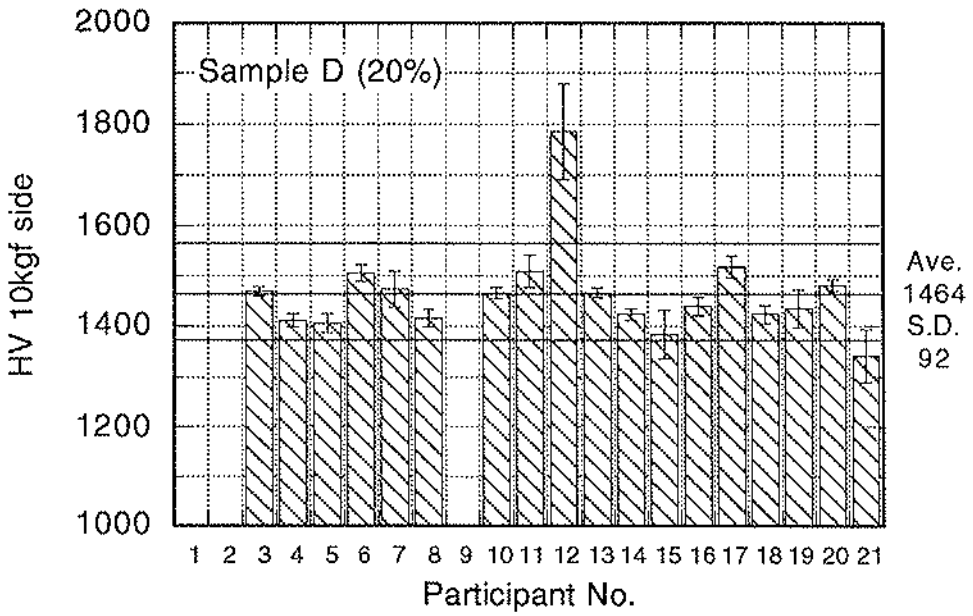


Fig. 3-18 HV10 for side on sample D from each participant.

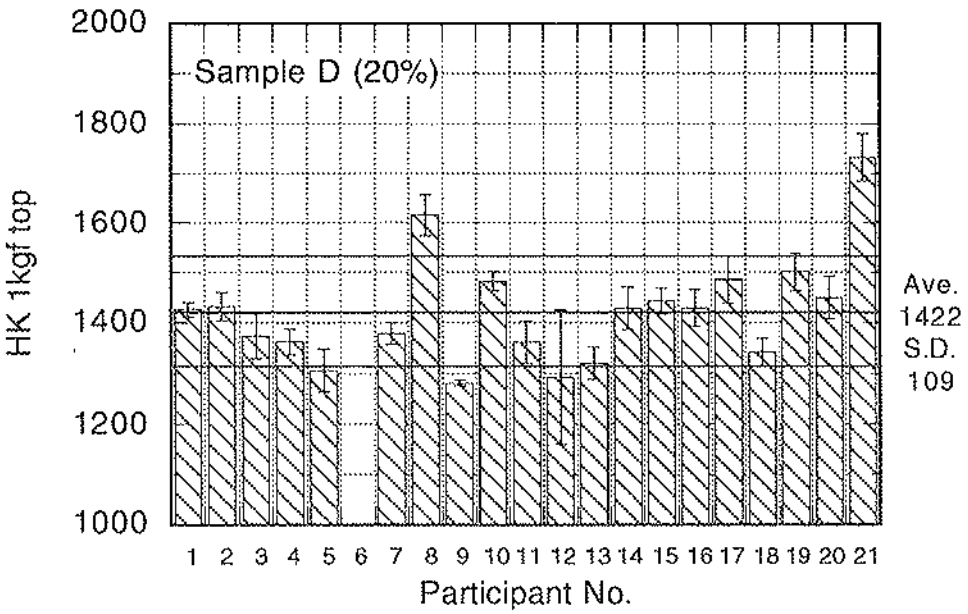


Fig. 3-19 HK1 for top on sample D from each participant.

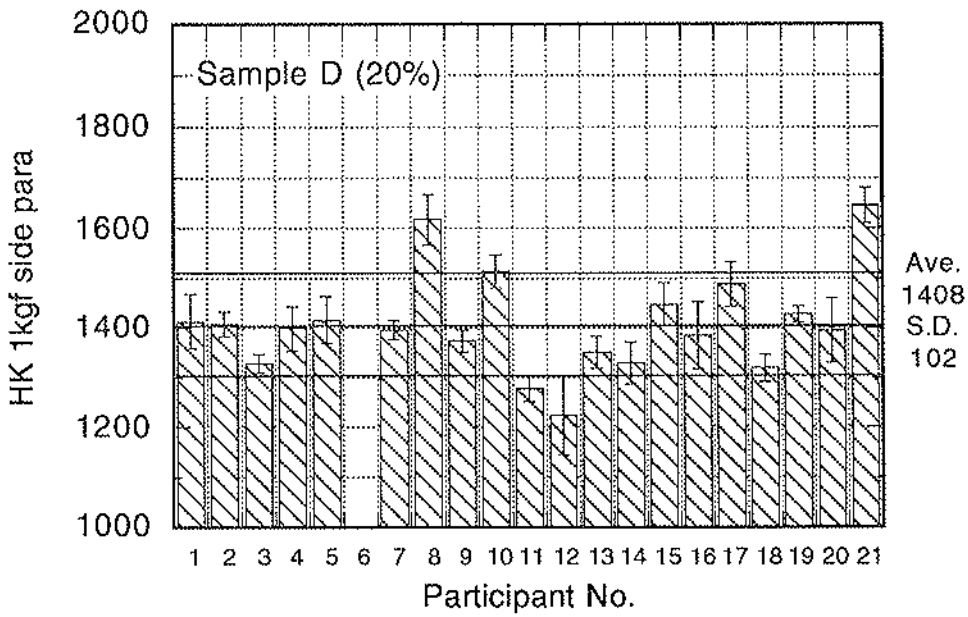


Fig. 3-20 HK1 for side and parallel to the sample on sample D from each participant.

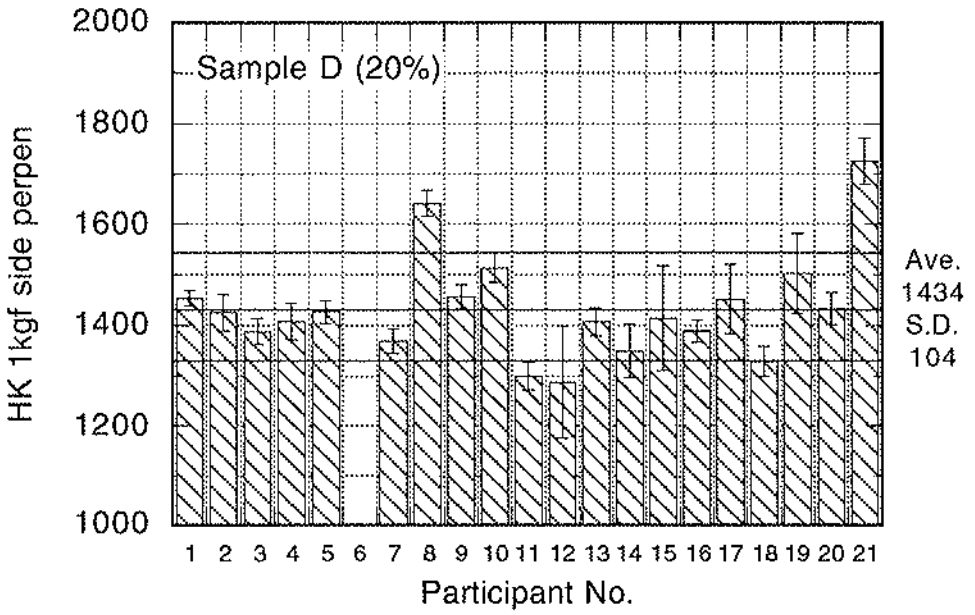


Fig. 3-21 HK1 for side and perpendicular to the sample on sample D from each participant.

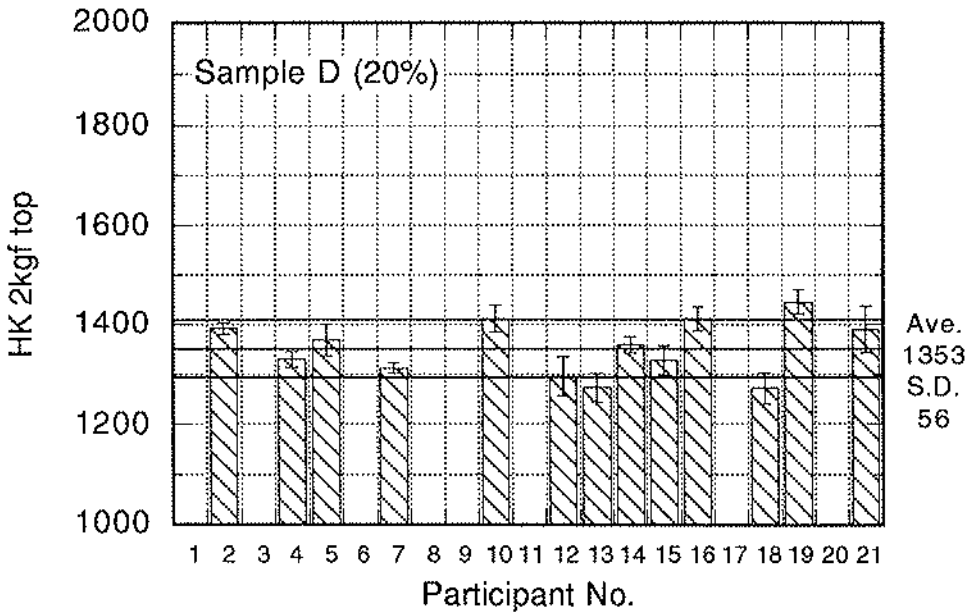


Fig. 3-22 HK2 for top on sample D from each participant.

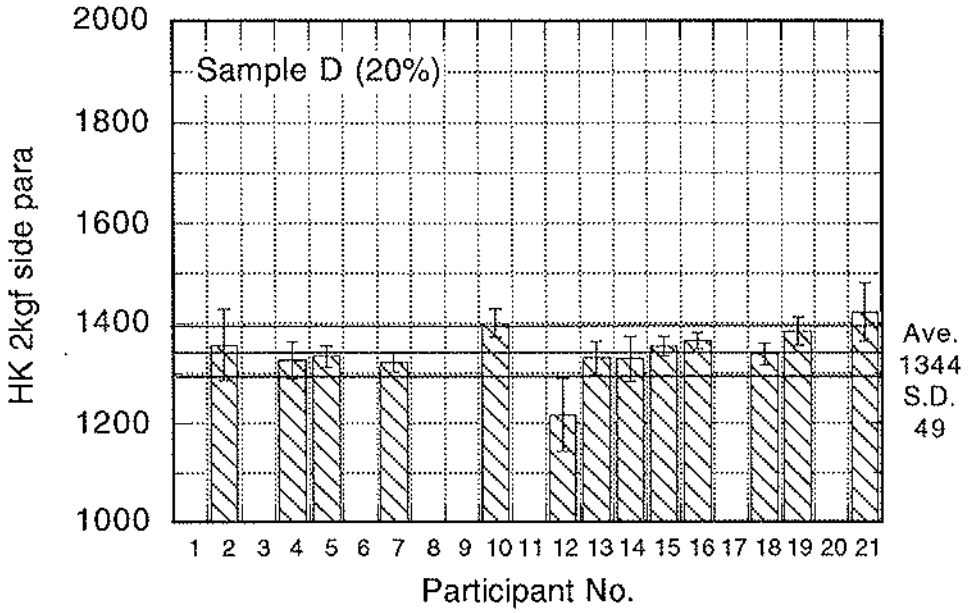


Fig. 3-23 HK2 for side and parallel to the sample on sample D from each participant.

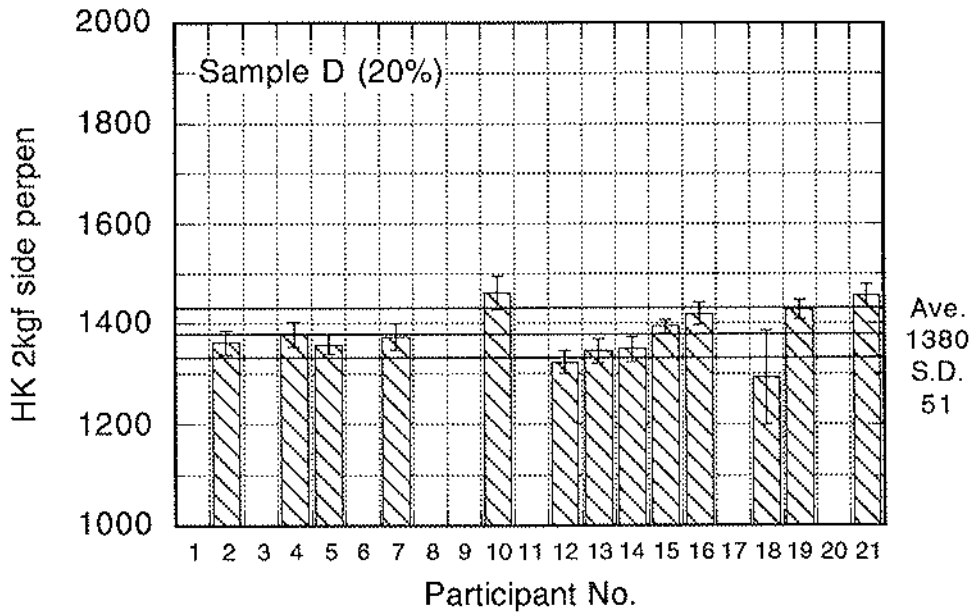


Fig. 3-24 HK2 for side and perpendicular to the sample on sample D from each participant.

5. Conclusions

We tried a round robin test for Vickers and Knoop hardness on whisker reinforced ceramics. The conclusions are;
(1) As shown in Figure 1-1, we can measure the increase of the hardness of these samples with whisker content, and the scattering of the data in each participant and among the participants are not different on monolithic sample (sample A) and the whisker composites (sample C and D). We can conclude the conventional Vickers and Knoop hardness tests are useful and meaningful for testing the whisker reinforced ceramics.
(2) We had results that the hardness values show test force dependence. It is required confirming the test force for comparing the hardness results. It is better to have a fixed test force for standard hardness test method of ceramic composites.
(3) In larger test force, the scattering, both in each participant and among the participants, are reduced. Large enough indent may be required for diminishing the scattering of the hardness test on ceramic composites.

In ISO/TC206 "Fine ceramics" technical committee, the standard test method for testing the monolithic ceramics is under discussion. In the draft of it, the recommended test forces are 1kgf for Vickers and 1 and 2kgf for Knoop. In this round robin test, we could confirm the results of scattering were not so different in monolithic sample (sample A) and composites samples (sample C and D). We can say, these recommendations can be expanded to the whisker reinforced composites. However, we confirmed that the higher test force can give us small scatterings, so HK2 can be a better choice than HK1, and HV10 is also a good choice, besides the recommendation of HV1.

Appendix I Tables

Table I to III (6 pages) are the average and standard deviation values, for summarizing the results. In the last two columns of the table, Average and two S.D. values appear. S.D. value under the average is the average of the standard deviations obtained in each participant, that corresponds to the value in Figure 1-2. The value on the right of the average is the standard deviation of the average calculated in each participant, that corresponds to the values in Figure 1-3.

Following 21 pages are the data table from each participant.

Table I

Sample A (0%)											
Participant No.	1	2	3	4	5	6	7	8	9	10	11
HV 1kgf	1326	1513	1377	1456	1319	1424	1379	1288	1216	1467	1295
S. D.	33	67	33	16	37	83	49	37	59	55	228
HV 10kgf			1340	1314	1264	1388	1304	1293		1336	1286
S. D.			40	26	21	45	45	17		24	26
HK 1kgf	1356	1304	1258	1330	1342		1289	1432	1228	1387	1164
S. D.	14	12	12	44	28		18	52	22	50	61
HK 2kgf		1223		1280	1324		1239			1283	
S. D.		35		16	25		21			31	

Table I (continued)

Sample A (0%)												
Participant No.	12	13	14	15	16	17	18	19	20	21	Average	S.D.
HV 1kgf	1556	1414	1298	1349	1419	1488	1326	1364	1369	1190	1373	93
S. D.	25	70	53	88	93	19	51	39	63	38	59	
HV 10kgf	1533	1334	1274	1324	1337	1392	1304	1263	1355	1230	1326	67
S. D.	0	39	20	8	27	19	20	36	19	40	26	
HK 1kgf	1217	1274	1288	1324	1287	1382	1293	1340	1266	1367	1306	64
S. D.	46	37	29	48	37	43	17	38	51	45	35	
HK 2kgf	1232	1251	1240	1316	1229		1218	1279		1256	1259	35
S. D.	21	17	21	37	21		37	43		50	29	

Table II

Sample C (10%)												
Participant No.		1	2	3	4	5	6	7	8	9	10	11
HV 1kgf	Top	1396	1582	1469	1524		1553		1382	1356	1530	1457
S. D.		16	71	36	42		38		59	7	78	31
HV 1kgf	Side	1380	1600	1452	1495		1499		1359	1363	1511	1446
S. D.		58	66	50	55		79		27	39	24	51
HV 10kgf	Top			1393	1408		1433		1372		1410	1382
S. D.				10	18		22		16		21	23
HV 10kgf	Side			1392	1369		1415		1359		1397	1386
S. D.				9	31		17		13		27	38
HK 1kgf	Top	1393	1349	1324	1430				1579	1257	1441	1305
S. D.		22	25	23	20				51	18	42	67
HK 1kgf	Side //	1353	1322	1284	1410				1578	1323	1425	1211
S. D.		18	49	18	37				35	4	17	50
HK 1kgf	Side ⊥	1399	1354	1331	1387				1596	1401	1468	1215
S. D.		18	30	32	38				35	3	52	68
HK 2kgf	Top		1314		1343						1375	
S. D.			11		22						40	
HK 2kgf	Side //		1297		1282						1343	
S. D.			21		40						40	
HK 2kgf	Side ⊥		1302		1299						1354	
S. D.			22		31						50	

Table II (continued)

Sample C (10%)													
Participant No.		12	13	14	15	16	17	18	19	20	21	Average	
HV 1kgf	Top	1872	1486	1443	1432	1481	1536	1409	1445	1480	1259	1479	122
S. D.		98	44	12	42	55	17	30	106	41	30	45	
HV 1kgf	Side	1823	1366	1396	1378	1456	1534	1413	1446	1474	1281	1456	116
S. D.		26	109	41	76	46	62	33	68	48	54	53	
HV 10kgf	Top	1455	1408	1378	1373	1379	1478	1372	1306	1410	1301	1391	46
S. D.		71	24	23	28	15	13	15	96	23	16	27	
HV 10kgf	Side	1455	1403	1391	1372	1379	1466	1372	1379	1417	1334	1393	34
S. D.		71	10	17	19	15	31	17	14	18	9	22	
HK 1kgf	Top	1412	1355	1351	1438	1327	1464	1348	1411	1361	1669	1401	98
S. D.		15	13	37	43	23	62	42	46	15	111	38	
HK 1kgf	Side //	1395	1311	1332	1342	1352	1444	1320	1364	1343	1643	1375	101
S. D.		28	34	10	86	23	23	31	50	23	70	34	
HK 1kgf	Side ⊥	1412	1350	1327	1401	1323	1490	1307	1437	1333	1669	1400	107
S. D.		15	21	51	28	61	102	76	58	19	73	43	
HK 2kgf	Top	1262	1307	1338	1335	1335		1276	1339		1388	1328	37
S. D.		8	14	28	17	13		11	85		21	25	
HK 2kgf	Side //	1234	1290	1244	1332	1314		1271	1352		1401	1305	49
S. D.		68	16	50	61	15		11	22		20	33	
HK 2kgf	Side ⊥	1262	1307	1264	1314	1352		1248	1348		1380	1312	43
S. D.		14	17	28	42	54		65	30		67	38	

Table III

Sample D (20%)												
Participant No.		1	2	3	4	5	6	7	8	9	10	11
HV 1kgf	Top	1427	1762	1503	1470	1426	1608	1483	1447	1362	1606	1515
S. D.		49	141	25	45	51	41	61	26	37	32	63
HV 1kgf	Side	1438	1696	1489	1537	1513	1594	1514	1423	1392	1590	1581
S. D.		62	62	8	19	51	41	41	30	42	35	62
HV 10kgf	Top			1475	1382	1360	1512	1437	1393		1440	1473
S. D.				19	26	29	19	24	35		30	27
HV 10kgf	Side			1469	1412	1406	1505	1474	1416		1466	1510
S. D.				10	13	18	16	36	17		11	32
HK 1kgf	Top	1427	1432	1374	1363	1306		1379	1615	1281	1482	1362
S. D.		14	28	45	26	43		21	41	6	19	41
HK 1kgf	Side //	1414	1409	1329	1400	1417		1397	1617	1375	1515	1278
S. D.		55	24	19	44	47		19	50	22	33	27
HK 1kgf	Side ⊥	1454	1426	1389	1409	1427		1370	1640	1457	1514	1299
S. D.		15	36	26	36	23		24	26	24	29	28
HK 2kgf	Top		1392		1330	1368		1312			1413	
S. D.			12		16	32		12			27	
HK 2kgf	Side //		1357		1326	1334		1322			1400	
S. D.			71		37	21		20			28	
HK 2kgf	Side ⊥		1361		1378	1357		1373			1461	
S. D.			25		25	19		27			34	

Table III (continued)

Sample D (20%)													
Participant No.		12	13	14	15	16	17	18	19	20	21	Average	
HV 1kgf	Top	1756	1493	1446	1435	1464	1574	1399	1546	1437	1315	1499	113
S. D.		38	46	37	95	44	70	25	28	72	53	51	
HV 1kgf	Side	1745	1487	1428	1447	1508	1612	1488	1488	1473	1364	1515	95
S. D.		24	16	52	72	71	51	65	20	24	39	42	
HV 10kgf	Top	1682	1453	1437	1397	1487	1522	1408	1446	1481	1371	1453	74
S. D.		0	32	30	22	20	18	16	17	20	34	23	
HV 10kgf	Side	1785	1466	1424	1384	1440	1518	1424	1435	1481	1342	1464	92
S. D.		94	10	11	48	18	22	18	37	11	52	26	
HK 1kgf	Top	1293	1321	1429	1443	1429	1486	1342	1501	1450	1731	1422	109
S. D.		133	32	42	25	37	48	28	38	42	48	38	
HK 1kgf	Side //	1224	1352	1330	1448	1386	1488	1320	1429	1395	1645	1408	102
S. D.		80	33	43	43	68	44	27	17	65	35	40	
HK 1kgf	Side ⊥	1287	1408	1350	1415	1390	1452	1329	1503	1434	1725	1434	104
S. D.		112	27	53	103	23	68	30	78	32	46	42	
HK 2kgf	Top	1296	1272	1359	1328	1412		1271	1446		1390	1353	56
S. D.		39	28	16	30	24		31	25		47	26	
HK 2kgf	Side //	1216	1330	1329	1354	1365		1338	1385		1422	1344	49
S. D.		74	33	45	20	17		21	28		59	36	
HK 2kgf	Side ⊥	1322	1344	1348	1394	1420		1293	1428		1456	1380	51
S. D.		23	24	24	13	23		93	20		24	29	

Participant No.1									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1281				1362					
1312				1344					
1326				1362					
1369				1339					
1344				1373					
1326				1356					
33				14					
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1403	1407			1389	1323	1420			
1411	1333			1362	1349	1384			
1396	1411			1423	1365	1414			
1403	1305			1392	1368	1378			
1369	1442			1398	1362	1400			
1396	1380			1393	1353	1399			
16	58			22	18	18			
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1463	1369			1423	1344	1440			
1423	1455			1412	1426	1464			
1459	1377			1449	1497	1440			
1344	1484			1429	1398	1473			
1447	1505			1420	1406	1452			
1427	1438			1427	1414	1454			
49	62			14	55	15			
Machine Type:									
HV1	AKASHI MVK-E								
HV10									
HK1	AKASHI MVK-E								
HK2									

Participant No.2									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1544				1306			1177		
1399				1316			1253		
1544				1316			1252		
1509				1288			1237		
1567				1296			1195		
1513				1304			1223		
67				12			35		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1662	1677			1349	1387	1390	1319	1301	1306
1571	1662			1360	1326	1373	1322	1292	1294
1647	1531			1376	1250	1311	1319	1277	1279
1531	1553			1308	1311	1349	1294	1285	1337
1501	1576			1354	1336	1349	1314	1332	1294
1582	1600			1349	1322	1354	1314	1297	1302
71	66			25	49	30	11	21	22
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1880	1772			1412	1432	1370	1390	1240	1321
1604	1628			1412	1415	1435	1388	1388	1382
1917	1637			1479	1368	1437	1409	1421	1380
1777	1713			1437	1412	1467	1396	1348	1359
1633	1729			1420	1417	1420	1376	1390	1365
1762	1696			1432	1409	1426	1392	1357	1361
141	62			28	24	36	12	71	25
Machine Type:									
HV1	Shimadzu HMV-2000								
HV10									
HK1	Shimadzu HMV-2000								
HK2	Shimadzu HMV-2000								

Participant No.3									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1392		1341		1262					
1340		1271		1239					
1344		1374		1272					
1411		1348		1262					
1400		1364		1253					
1377		1340		1258					
33		40		12					
Sample C (10%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1480	1505	1405	1384	1342	1284	1360			
1439	1455	1403	1392	1311	1264	1311			
1431	1384	1382	1400	1291	1284	1289			
1522	1492	1391	1382	1336	1276	1331			
1471	1423	1386	1401	1342	1313	1365			
1469	1452	1393	1392	1324	1284	1331			
36	50	10	9	23	18	32			
Sample D (20%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1505	1501	1478	1467	1301	1316	1409			
1527	1492	1501	1476	1371	1331	1395			
1463	1484	1472	1470	1401	1352	1412			
1518	1480	1474	1480	1379	1306	1382			
1501	1488	1448	1454	1418	1342	1347			
1503	1489	1475	1469	1374	1329	1389			
25	8	19	10	45	19	26			
Machine Type:									
HV1	Brickers 220 / Durimet								
HV10	Brickers 220 / Durimet								
HK1	Brickers 220 / Durimet								
HK2									

Participant No.4

Sample A (0%)

HV1	HV10	HK1	HK2
1435	1304	1406	1289
1468	1339	1316	1258
1443	1324	1311	1296
1472	1273	1321	1270
1460	1328	1296	1287
1456	1314	1330	1280
16	26	44	16

Sample C (10%)

HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1586	1484	1436	1417	1446	1365	1352	1354	1281	1314
1493	1489	1408	1374	1435	1379	1387	1339	1218	1260
1527	1586	1392	1374	1426	1452	1357	1375	1301	1291
1480	1480	1394	1341	1446	1438	1395	1325	1286	1286
1536	1435	1411	1341	1398	1415	1446	1321	1326	1343
1524	1495	1408	1369	1430	1410	1387	1343	1282	1299
42	55	18	31	20	37	38	22	40	31

Sample D (20%)

HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1489	1541	1383	1434	1326	1467	1420	1334	1265	1352
1536	1527	1359	1405	1357	1406	1349	1316	1352	1367
1464	1568	1398	1406	1382	1347	1446	1354	1319	1371
1447	1532	1415	1404	1355	1384	1423	1314	1347	1418
1416	1519	1355	1412	1393	1395	1406	1332	1348	1384
1470	1537	1382	1412	1363	1400	1409	1330	1326	1378
45	19	26	13	26	44	36	16	37	25

Machine Type:

HV1	ZWICK 3212
HV10	ZWICK 3212
HK1	ZWICK 3212
HK2	ZWICK 3212

Participant No.5									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1377		1301		1326			1306		
1281		1252		1389			1306		
1301		1255		1336			1361		
1305		1252		1316			1306		
1333		1258		1341			1339		
1319		1264		1342			1324		
37		21		28			25		
Sample C (10%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
Sample D (20%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1471	1536	1399	1407	1247	1412	1455	1417	1341	1333
1459	1545	1339	1413	1281	1461	1429	1374	1354	1350
1447	1455	1356	1389	1357	1409	1423	1341	1337	1386
1347	1567	1328	1389	1316	1346	1392	1339	1299	1361
1403	1463	1376	1433	1331	1458	1437	1369	1337	1357
1425	1513	1360	1406	1306	1417	1427	1368	1334	1357
51	51	29	18	43	47	23	32	21	19
Machine Type:									
HV1	ZWICK 3212								
HV10	ZWICK 3212								
HK1	ZWICK 3212								
HK2	ZWICK 3212								

Participant No.6									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1459		1430							
1526		1430							
1338		1398							
1459		1340							
1338		1340							
1424		1388							
83		45							
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1595	1564	1433	1433						
1525	1497	1433	1402						
1525	1564	1402	1402						
1525	1371	1465	1402						
1595	1497	1433	1433						
1553	1499	1433	1414						
38	79	22	17						
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1638	1564	1533	1498						
1564	1564	1533	1498						
1638	1638	1498	1533						
1564	1564	1498	1498						
1638	1638	1498	1498						
1608	1594	1512	1505						
41	41	19	16						
Machine Type:									
HV1	TIRA TEST 2300								
HV10	TIRA TEST 2300								
HK1									
HK2									

Participant No.7									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1362		1309		1303			1256		
1463		1277		1259			1227		
1344		1321		1303			1228		
1347		1246		1295			1217		
1377		1366		1283			1265		
1379		1304		1289			1239		
49		45		18			21		
Sample C (10%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
Sample D (20%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1501	1476	1452	1519	1344	1387	1368	1294	1310	1365
1463	1509	1439	1427	1378	1392	1368	1310	1342	1394
1484	1531	1402	1478	1387	1406	1389	1317	1342	1407
1567	1576	1427	1492	1395	1376	1392	1312	1297	1346
1400	1480	1465	1452	1392	1426	1333	1328	1320	1352
1483	1514	1437	1474	1379	1397	1370	1312	1322	1373
61	41	24	36	21	19	24	12	20	27
Machine Type:									
HV1	LEITZ MINILOAD 2								
HV10	HTM 1234								
HK1	LEITZ MINILOAD 2								
HK2	LEITZ MINILOAD 2								

Participant No.8									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1251		1277		1522					
1336		1293		1414					
1301		1321		1402					
1251		1288		1428					
1301		1288		1396					
1288		1293		1432					
37		17		52					
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1431	1355	1378	1366	1640	1585	1627			
1284	1373	1355	1366	1612	1572	1560			
1411	1319	1355	1360	1517	1524	1582			
1373	1392	1390	1366	1539	1620	1572			
1411	1355	1384	1337	1588	1591	1640			
1382	1359	1372	1359	1579	1578	1596			
59	27	16	13	51	35	35			
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1492	1471	1384	1408	1600	1660	1635			
1431	1392	1355	1439	1579	1640	1629			
1431	1431	1427	1396	1584	1533	1612			
1431	1411	1433	1408	1632	1614	1640			
1451	1411	1366	1427	1678	1638	1683			
1447	1423	1393	1416	1615	1617	1640			
26	30	35	17	41	50	26			
Machine Type:									
HV1	VICKERS-ARMSTRONGS LTD. V.P.H.T.M. SN255184								
HV10	VICKERS-ARMSTRONGS LTD. V.P.H.T.M. SN255184								
HK1	LEITZ DURIMET Nr 776956								
HK2									

Participant No.9									
Sample A (0%)									
HV1	HV10		HK1			HK2			
1238			1222						
1125			1202						
1210			1218						
1288			1243						
1219			1257						
1216			1228						
59			22						
Sample C (10%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1354	1392			1259	1328	1406			
1365	1369			1245	1318	1398			
1347	1329			1288	1321	1403			
1354	1318			1250	1326	1398			
1358	1407			1245	1320	1400			
1356	1363			1257	1323	1401			
7	39			18	4	3			
Sample D (20%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1329	1319			1286	1336	1464			
1384	1411			1278	1381	1467			
1315	1411			1288	1382	1471			
1392	1396			1279	1382	1414			
1392	1422			1274	1392	1467			
1362	1392			1281	1375	1457			
37	42			6	22	24			
Machine Type:									
HV1	Matsuzawa M&T 70								
HV10									
HK1	Matsuzawa M&T 70								
HK2									

Participant No.10									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1454		1308		1420			1281		
1477		1316		1419			1248		
1525		1363		1424			1261		
1499		1336		1312			1328		
1381		1358		1359			1296		
1467		1336		1387			1283		
55		24		50			31		
Sample C (10%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1554	1495	1433	1411	1371	1410	1427	1394	1351	1384
1524	1546	1419	1384	1439	1414	1494	1389	1338	1414
1410	1493	1387	1433	1464	1429	1544	1304	1383	1297
1627	1527	1422	1393	1481	1452	1417	1401	1278	1366
1533	1493	1389	1363	1448	1421	1460	1389	1367	1307
1530	1511	1410	1397	1441	1425	1468	1375	1343	1354
78	24	21	27	42	17	52	40	40	50
Sample D (20%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1618	1556	1434	1462	1490	1496	1555	1430	1383	1517
1575	1632	1466	1485	1475	1534	1528	1378	1361	1428
1594	1618	1397	1467	1458	1466	1506	1443	1434	1446
1656	1589	1433	1464	1510	1535	1477	1393	1409	1465
1587	1557	1472	1454	1479	1546	1505	1423	1413	1447
1606	1590	1440	1466	1482	1515	1514	1413	1400	1461
32	35	30	11	19	33	29	27	28	34
Machine Type:									
HV1	Zwick Model Z323								
HV10	Zwick Model Z323								
HK1	Zwick Model Z323								
HK2	Zwick Model Z323								

Participant No.11									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1407		1288		1180					
924		1312		1224					
1232		1242		1065					
1423		1288		1195					
1488		1299		1157					
1295		1286		1164					
228		26		61					
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1431	1423	1362	1330	1341	1276	1106			
1439	1488	1405	1371	1354	1250	1247			
1471	1463	1407	1417	1321	1163	1286			
1439	1488	1359	1422	1187	1195	1204			
1505	1369	1376	1392	1323	1169	1233			
1457	1446	1382	1386	1305	1211	1215			
31	51	23	38	67	50	68			
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1415	1614	1455	1494	1344	1286	1281			
1497	1497	1494	1468	1403	1295	1326			
1540	1604	1447	1555	1409	1259	1333			
1576	1652	1460	1521	1326	1308	1276			
1549	1540	1508	1513	1326	1243	1278			
1515	1581	1473	1510	1362	1278	1299			
63	62	27	32	41	27	28			
Machine Type:									
HV1	AKASHI AVK-A								
HV10	AKASHI AVK-A								
HK1	AKASHI AVK-A								
HK2									

Participant No.12									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1558		1533		1210			1216		
1514		1533		1165			1238		
1576		1533		1291			1224		
1558		1533		1210			1265		
1576		1533		1210			1216		
1556		1533		1217			1232		
25		0		46			21		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1930	1811	1533	1533	1395	1423	1395	1265	1265	1265
1930	1869	1533	1533	1423	1423	1423	1265	1265	1248
1930	1811	1403	1403	1423	1368	1423	1265	1112	1248
1703	1811	1403	1403	1423	1395	1395	1265	1265	1265
1869	1811	1403	1403	1395	1368	1423	1248	1265	1282
1872	1823	1455	1455	1412	1395	1412	1262	1234	1262
98	26	71	71	15	28	15	8	68	14
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1811	1703	1682	1854	1291	1120	1115	1265	1098	1316
1756	1756	1682	1682	1423	1220	1243	1248	1265	1291
1703	1756	1682	1682	1078	1315	1368	1299	1185	1352
1756	1756	1682	1854	1291	1176	1395	1334	1265	1316
1756	1756	1682	1854	1380	1291	1315	1334	1265	1334
1756	1745	1682	1785	1293	1224	1287	1296	1216	1322
38	24	0	94	133	80	112	39	74	23
Machine Type:									
HV1	GALILEO type MICROSCAN								
HV10	WOLPERT type TESTOR								
HK1	LEITZ type DURIMET								
HK2	LEITZ type DURIMET								

Participant No.13									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1492		1311		1217			1264		
1442		1292		1261			1244		
1307		1323		1290			1269		
1391		1352		1315			1226		
1438		1393		1285			1254		
1414		1334		1274			1251		
70		39		37			17		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1462	1522	1424	1414	1340	1285	1335	1298	1286	1300
1513	1403	1369	1399	1356	1268	1386	1327	1273	1323
1539	1267	1430	1392	1346	1343	1346	1309	1316	1322
1426	1380	1407	1398	1375	1315	1346	1309	1281	1281
1492	1257	1411	1414	1359	1343	1335	1291	1292	1307
1486	1366	1408	1403	1355	1311	1350	1307	1290	1307
44	109	24	10	13	34	21	14	16	17
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1548	1504	1424	1462	1300	1383	1405	1247	1364	1314
1522	1466	1448	1451	1359	1333	1380	1285	1306	1351
1504	1492	1507	1471	1351	1378	1386	1266	1355	1343
1450	1475	1448	1470	1305	1305	1445	1247	1338	1379
1442	1496	1437	1476	1290	1362	1425	1313	1286	1331
1493	1487	1453	1466	1321	1352	1408	1272	1330	1344
46	16	32	10	32	33	27	28	33	24
Machine Type:									
HV1	Wilson Model 300								
HV10	Wilson Model 300								
HK1	Wilson Model 300								
HK2	Wilson Model 300								

Participant No.14									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1219		1275		1278			1272		
1281		1281		1323			1235		
1362		1303		1250			1225		
1315		1264		1310			1219		
1315		1249		1281			1250		
1298		1274		1288			1240		
53		20		29			21		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1459	1388	1402	1401	1321	1328	1238	1330	1282	1268
1431	1415	1400	1379	1341	1321	1336	1378	1296	1233
1431	1373	1359	1371	1365	1336	1349	1326	1250	1285
1443	1347	1380	1413	1409	1346	1360	1352	1171	1297
1451	1455	1351	1391	1321	1328	1354	1304	1219	1238
1443	1396	1378	1391	1351	1332	1327	1338	1244	1264
12	41	23	17	37	10	51	28	50	28
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1476	1455	1443	1412	1440	1323	1349	1352	1363	1376
1419	1505	1447	1429	1472	1378	1384	1376	1350	1328
1492	1377	1477	1412	1458	1321	1412	1365	1253	1319
1435	1411	1419	1436	1406	1362	1274	1335	1326	1348
1407	1392	1397	1432	1368	1266	1333	1365	1353	1367
1446	1428	1437	1424	1429	1330	1350	1359	1329	1348
37	52	30	11	42	43	53	16	45	24
Machine Type:									
HV1	AKASHI AVK-C1								
HV10	AKASHI AVK-C1								
HK1	AKASHI AVK-C1								
HK2	AKASHI AVK-C1								

Participant No.15									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1328		1324		1354			1291		
1355		1324		1381			1382		
1364		1324		1328			1299		
1472		1335		1303			1308		
1227		1312		1255			1299		
1349		1324		1324			1316		
88		8		48			37		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1472	1243	1381	1363	1452	1231	1368	1326	1224	1326
1482	1421	1399	1387	1368	1395	1373		1354	1265
1402	1392	1343	1381	1482	1409	1422	1344	1354	1326
1411	1421	1399	1343	1452	1266	1423	1354	1354	1282
1392	1411	1343	1387	1437	1409	1420	1317	1372	1372
1432	1378	1373	1372	1438	1342	1401	1335	1332	1314
42	76	28	19	43	86	28	17	61	42
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1435	1411	1395	1411	1482	1429	1266	1317	1326	1392
1492	1451	1377	1437	1437	1452	1482	1363	1372	1376
1461	1569	1434	1369	1423	1395	1452	1291	1372	1411
1514	1411	1389	1393	1452	1452	1519	1354	1344	1392
1274	1392	1389	1312	1423	1512	1354	1317	1354	1401
1435	1447	1397	1384	1443	1448	1415	1328	1354	1394
95	72	22	48	25	43	103	30	20	13
Machine Type:									
HV1	Frank-Härteprüfgerät FINOWTEST 38536 + NEOPHOT 32								
HV10	Frank-Härteprüfgerät FINOWTEST 38536 + NEOPHOT 32								
HK1	Frank-Härteprüfgerät FINOWTEST 38536								
HK2	Frank-Härteprüfgerät FINOWTEST 38536								

Participant No.16									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1558		1321		1342			1216		
1355		1355		1243			1248		
1472		1367		1291			1232		
1355		1344		1291			1248		
1355		1299		1267			1200		
1419		1337		1287			1229		
93		27		37			21		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1514	1392	1355	1379	1291	1368	1316	1354	1300	1265
1558	1472	1379	1355	1316	1342	1342	1335	1317	1392
1431	1431	1379	1391	1342	1316	1368	1317	1335	1373
1431	1514	1391	1391	1342	1368	1368	1335	1317	1392
1472	1472	1391	1379	1342	1368	1220	1335	1300	1335
1481	1456	1379	1379	1327	1352	1323	1335	1314	1351
55	46	15	15	23	23	61	13	15	54
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1472	1605	1506	1466	1452	1316	1423	1373	1373	1412
1392	1558	1492	1440	1452	1316	1395	1432	1354	1412
1472	1431	1466	1415	1423	1452	1368	1412	1392	1432
1472	1472	1506	1440	1368	1452	1368	1432	1354	1392
1514	1472	1466	1440	1452	1395	1395	1412	1354	1452
1464	1508	1487	1440	1429	1386	1390	1412	1365	1420
44	71	20	18	37	68	23	24	17	23
Machine Type:									
HV1	Albert GNEHM Type 200 nr. 6/14 H/H Dia testor UR-UZ 237								
HV10	Albert GNEHM Type 200 nr. 6/14 H/H Dia testor UR-UZ 237								
HK1	Albert GNEHM Type 200 nr. 6/14 H/H Dia testor UZ 7070								
HK2	Otto Wolpert Dia testor 2 Rc Testor UZ 7070								

Participant No.17

Sample A (0%)

HV1	HV10	HK1	HK2
1510	1400	1380	
1500	1380	1330	
1480	1370	1450	
1460	1390	1370	
1490	1420	1380	
1488	1392	1382	
19	19	43	

Sample C (10%)

HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1560	1590	1490	1460	1520	1440	1560			
1520	1560	1480	1490	1520	1450	1450			
1540	1530	1460	1500	1380	1480	1330			
1540	1430	1490	1420	1480	1420	1580			
1520	1560	1470	1460	1420	1430	1530			
1536	1534	1478	1466	1464	1444	1490			
17	62	13	31	62	23	102			

Sample D (20%)

HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1610	1630	1540	1540	1540	1500	1500			
1490	1650	1500	1540	1430	1500	1340			
1510	1660	1520	1490	1450	1410	1510			
1610	1540	1510	1510	1480	1510	1450			
1650	1580	1540	1510	1530	1520	1460			
1574	1612	1522	1518	1486	1488	1452			
70	51	18	22	48	44	68			

Machine Type:

HV1	Matsuzawa Seiki Co.Ltd., Hardness Tester DVK-2
HV10	Matsuzawa Seiki Co.Ltd., Hardness Tester DVK-2
HK1	Matsuzawa Seiki Co.Ltd., Hardness Tester DVK-2
HK2	

Participant No.18									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1238		1298		1299			1187		
1361		1298		1267			1172		
1355		1278		1288			1241		
1343		1329		1302			1234		
1334		1319		1310			1258		
1326		1304		1293			1218		
50		20		17			37		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1381	1429	1387	1348	1377	1370	1325	1268	1284	1238
1456	1397	1386	1393	1394	1318	1348	1282	1277	1283
1417	1384	1360	1371	1305	1299	1174	1282	1262	1141
1384	1464	1371	1383	1360	1320	1324	1261	1258	1305
1407	1390	1354	1366	1302	1291	1364	1287	1276	1274
1409	1413	1372	1372	1348	1320	1307	1276	1271	1248
30	33	15	17	42	31	76	11	11	65
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1404	1509	1431	1432	1307	1326	1311	1229	1332	1346
1373	1536	1387	1425	1329	1353	1335	1316	1318	1323
1421	1448	1405	1446	1346	1278	1332	1269	1325	1336
1372	1397	1401	1418	1383	1329	1294	1266	1372	1331
1425	1552	1415	1397	1343	1314	1375	1275	1342	1128
1399	1488	1408	1424	1342	1320	1329	1271	1338	1293
25	65	16	18	28	27	30	31	21	93
Machine Type:									
HV1	V10								
HV10	V10								
HK1	V10								
HK2	V10								

Participant No.19									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1400		1223		1301			1297		
1301		1288		1301			1206		
1392		1229		1357			1278		
1368		1303		1357			1297		
1361		1273		1386			1316		
1364		1263		1340			1279		
39		36		38			43		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1263	1512	1385	1386	1417	1386	1386	1356	1336	1316
1459	1486	1248	1374	1357	1386	1480	1376	1376	1356
1485	1468	1164	1380	1480	1386	1417	1189	1336	1376
1540	1425	1357	1358	1386	1274	1386	1397	1336	1316
1476	1337	1374	1397	1417	1386	1514	1376	1376	1376
1445	1446	1306	1379	1411	1364	1437	1339	1352	1348
106	68	96	14	46	50	58	85	22	30
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1531	1459	1463	1488	1448	1417	1448	1419	1356	1419
1504	1485	1421	1445	1514	1417	1417	1464	1419	1419
1568	1485	1439	1386	1514	1448	1584	1419	1356	1419
1559	1513	1458	1427	1480	1448	1480	1464	1397	1464
1568	1496	1451	1427	1548	1417	1584	1464	1397	1419
1546	1488	1446	1435	1501	1429	1503	1446	1385	1428
28	20	17	37	38	17	78	25	28	20
Machine Type:									
HV1	Shimadzu HMV 2000								
HV10	Akashi AVK50								
HK1	Shimadzu HMV 2000								
HK2	Shimadzu HMV 2000								

Participant No.20									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1329		1366		1293					
1454		1355		1252					
1418		1366		1204					
1308		1366		1245					
1336		1321		1338					
1369		1355		1266					
63		19		51					
Sample C (10%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1505	1403	1378	1440	1356	1343	1338			
1467	1522	1440	1415	1378	1330	1305			
1415	1505	1415	1415	1373	1333	1330			
1518	1488	1415	1390	1359	1328	1333			
1496	1450	1402	1427	1341	1383	1359			
1480	1474	1410	1417	1361	1343	1333			
41	48	23	18	15	23	19			
Sample D (20%)									
HV1	HV1	HV10	HV10	HK1	HK1	HK1	HK2	HK2	HK2
top	side	top	side	top	side //	side ⊥	top	side //	side ⊥
1336	1434	1452	1478	1414	1375	1381			
1475	1479	1505	1492	1434	1434	1434			
1513	1492	1492	1492	1493	1487	1440			
1391	1492	1478	1478	1496	1323	1451			
1471	1467	1478	1465	1411	1356	1463			
1437	1473	1481	1481	1450	1395	1434			
72	24	20	11	42	65	32			
Machine Type:									
HV1	AKASHI MVK-EL								
HV10	AKASHI AVK-A								
HK1	AKASHI MVK-EL								
HK2									

Participant No.21									
Sample A (0%)									
HV1		HV10		HK1			HK2		
1189		1206		1424			1313		
1235		1177		1358			1266		
1189		1277		1374			1180		
1204		1236		1299			1239		
1131		1256		1380			1280		
1190		1230		1367			1256		
38		40		45			50		
Sample C (10%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1207	1284	1321	1343	1513	1632	1708	1397	1391	1387
1281	1366	1277	1321	1777	1548	1615	1353	1430	1273
1271	1261	1299	1343	1769	1712	1689	1410	1412	1383
1267	1277	1310	1332	1615	1611	1574	1389	1379	1459
1271	1219	1299	1332	1671	1711	1757	1389	1395	1397
1259	1281	1301	1334	1669	1643	1669	1388	1401	1380
30	54	16	9	111	70	73	21	20	67
Sample D (20%)									
HV1 top	HV1 side	HV10 top	HV10 side	HK1 top	HK1 side //	HK1 side ⊥	HK2 top	HK2 side //	HK2 side ⊥
1291	1298	1332	1267	1668	1639	1693	1340	1506	1438
1238	1366	1378	1390	1704	1686	1671	1358	1404	1497
1354	1396	1414	1390	1757	1608	1727	1457	1422	1455
1369	1388	1343	1343	1734	1615	1789	1416	1438	1441
1325	1373	1390	1321	1793	1675	1746	1381	1342	1449
1315	1364	1371	1342	1731	1645	1725	1390	1422	1456
53	39	34	52	48	35	46	47	59	24
Machine Type:									
HV1	AKASHI AVK-A								
HV10	AKASHI AVK-A								
HK1	AKASHI AVK-C2								
HK2	AKASHI AVK-C2								

Appendix II Related documents

1. Project Proposal Form (1 page)
2. Project Initiation Form (2 pages)
3. Instructions (8 pages)

Proposed Advanced Ceramics Project

- VAMAS TWA#3 CERAMICS -

Proposed by
National Industrial Research Institute of Nagoya and
Japan Fine Ceramics Center
(Nagoya, JAPAN)

Conventional Hardness Test on Whisker Reinforced Ceramics

Objectives:	Ensuring the adoption of conventional (Vickers and Knoop) hardness tests to whisker reinforced ceramics	
factors:	methods (Vickers, Knoop)	
	testing force (1, 2, 10kgf; if possible)	
Materials:	Si_3N_4/SiC-whisker (20vol%), Si_3N_4 (optional);Si_3N_4/SiC-whisker (10vol%)	
Samples	Three plate samples (45 x 10 x 3.5mm) for one material	
Methods:	Vickers hardness test (1kgf, 10kgf)	
	surface (top or side surface)	
	Knoop hardness test (1kgf, 2kgf)	
	test direction (parallel or perpendicular to the whisker direction)	
Conditions	Temperature	Room Temperature
	Number of indents	5 for one condition
	Total indents;	20 for monolithic Si_3N_4 (10 for Vickers and 10 for Knoop) 50 for 20vol% whisker composites (20 for Vickers and 30 for Knoop) (optional); 50 for 10vol% whisker composites
Projected Time	8 hours	
Participants	15 to 20 participants	

Proposed Schedule

Nov. 1995.	Distribution of specimens and instructions.
Apr. 1996.	Deadline for sending results back to NIRIN.
June 1996	Distribution of preliminary analysis of the results.
Aug. 1996	Deadline for participants review of analysis.
Dec. 1996	Publication of final report.

VERSAILLES PROJECT ON ADVANCED MATERIALS AND STANDARDS
TECHNICAL WORKING AREA PROJECT INITIATION FORM

0 TWA Number

TWA #3 Advanced Ceramics

1 Theme

III Ceramics and CMC

2 Title

Conventional Hardness Test on Whisker Reinforced Ceramics

3 Objectives

Hardness test is one of the most common and basic tests on engineering ceramics for discussing mechanical properties. Standard of hardness testing for fine ceramics is being discussed in ISO/TC206/WG3, but it is for monolithic ceramics. Then we should have some common data for discussing the hardness on ceramics matrix composites, for discussing the pre-standardization for ceramic matrix composites.

This proposed VAMAS round-robin will focus on conventional hardness tests (Vickers and Knoop tests) for whisker reinforced ceramics. It will be prepared by Nataonal Industrial Research Institute of Naoya (NIRIN: Nagoya, Japan) and Japan Fine Ceramics Center (JFCC: Nagoya Japan).

4 Deliverables

Results and analysis will be used for discussing the standardize procedure of the hardness tests for ceramic matrix composites.

5 Prestandardization Needs

The standard testing method of hardness for monolithic fine ceramics is discussed in ISO/TC206/WG3. However, it is not for testing ceramics matrix composites. It is important to know that the discussed standard document can fit for ceramics matrix composites. Then, we perform a round-robin test for taking a basic data for discussing the standard testing method for hardniss testing, and it will help for prestandardize discussion on ceramic matrix composites.

6 Plan

NIRIN and JFCC are coordinating this program in cooperation. Testing techniques are Vickers and Knoop hardness tests. We will discuss the effect of indenting force and indent direction to the hardness values, from the round-robin results. The projected time for one participant is about 8-10 hours, that follows the guideline in TWA #3.

7 Timescales

Specimens are now being prepared by JFCC, and registration for participation is already informed from several research institutes.

<Planned Schedule>

Dec. 1995	Distribution of specimens and instructions.
Mar. 1996	Deadline for sending results back to NIRIN.
June 1996	Distribution of preliminary analysis of the results.
Sep. 1996	Deadline for participants' review of analysis.
Dec. 1996	Publication of final report.

8 Funding

The specimen cost has been paid by JFCC, from the budget of VAMAS research program that is supplied from Science and Technology Agency, Japan.

9 Industry and International Participation

Break down of the prospected participants are as follows;

Canada	0	France	0	Germany	3
Italy	4	Japan	6	U.K.	1
USA	2	CEC	2		
Others	2	(Australia	1	Switzerland	1)

Industry, Private labs	3
Academia	2
Government labs	15

10 Dissemination

A final VAMAS report will be prepared by NIRIN and JFCC, available for public dissemination.

11 Identification of Proposal Source

Name : Shuji SAKAGUCHI
Organization : National Industrial Research Institute of Nagoya
Address : 1-1, Hirate-cho, Kita-ku, Nagoya 462 JAPAN
Phone No. : +81-52-911-2111
Fax No. : +81-52-916-2802

Name : Mineo MIZUNO
Organization : Japan Fine Ceramics Center
Address : 2-4-1, Mutsuno, Atsuta-ku, Nagoya 456 JAPAN
Phone No. : +81-52-871-3500
Fax No. : +81-52-871-3599

INSTRUCTIONS FOR VAMAS ROUND ROBIN ON VICKERS/KNOOP HARDNESS OF CERAMIC MATRIX COMPOSITES

(Revised: 1996-02-02)

1. Introduction

This round robin is for applying the conventional hardness testing methods to ceramic matrix composites. Vickers and Knoop hardness tests are adopted for this round robin. The procedure of the both tests are appeared in ISO standards, ISO 6507 and 4545, basically for metals. The sample is silicon carbide whisker reinforced silicon nitride. So, it is not for a discussion for long fiber composites. The sample is prepared by slip casting, so, it may have an anisotropy, especially on the side surface.

2.Specimen

2.1 Material

The material used for this round robin is silicon carbide whisker reinforced silicon nitride, Kryptonite, which is manufactured by Japan Metals and Chemicals Co. Ltd., Tokyo, Japan.

Three kinds of samples are supplied which has 20, 10 and 0vol% of SiC whiskers.

2.2 Shape and dimensions of the Specimen

The specimens distributed to participants are rectangular bars of 19x 4.5x 3.5mm.

The 19x 4.5mm surface is named "top" surface.

The 19x 3.5mm surface is named "side" surface.

3.Testing machine

Testing machine should follow ISO standards of Vickers/Knoop testing machine, ISO 146 and ISO 4546.

4.Distribution of Specimens

The number of supplied specimens is four for each of 0, 10 and 20 vol% specimen. One top surface and one side surface on each specimen are polished for testing.

5.Testing conditions

For 0vol% sample, the following tests should be made;

(1) Five Vickers indents by 1kgf on top surface.

(2) Five Vickers indents by 10kgf on top surface.

((1) and (2) correspond to (a) in figure.)

(3) Five Knoop indents by 1kgf on top surface.

(4) Five Knoop indents by 2kgf on top surface.

((3) and (4) correspond to (c) in figure.)

If some of the testing force are not available on the equipment, it can be neglected.

For 20vol% sample, the following tests should be made;
(1) Five Vickers indents by 1kgf on top surface.
(2) Five Vickers indents by 10kgf on top surface.
((1) and (2) correspond to (a) in figure.)
(3) Five Vickers indents by 1kgf on side surface.
(4) Five Vickers indents by 10kgf on side surface.
((3) and (4) correspond to (b) in figure.)
(5) Five Knoop indents by 1kgf on top surface.
(6) Five Knoop indents by 2kgf on top surface.
((5) and (6) correspond to (c) in figure.)
(7) Five Knoop indents by 1kgf on side surface, parallel to the long direction of the bar.
(8) Five Knoop indents by 2kgf on side surface, parallel to the long direction of the bar.
((7) and (8) correspond to (d) in figure.)
(9) Five Knoop indents by 1kgf on side surface, perpendicular to the long direction of the bar.
(10) Five Knoop indents by 2kgf on side surface, perpendicular to the long direction of the bar.
((9) and (10) correspond to (e) in figure.)
If some of the testing force are not available on the equipment, it can be neglected.

For 10vol% specimens, the testing procedures are same as for 20vol%, but the testing is optional. Participants must get data on 0vol% and 20vol%, but need not on 10vol% if the the participants think the experiments take too much time.

See the attached page of table and figure for testing conditions.

The average and standard deviation of each testing condition should be reported. See the attached report form.

5.1 Distance of indents

The distance of the indent must be kept the following figures.

6. Calculations

Vickers hardness is calculated by equation (1).

$$HV=0.18909\frac{F}{d^2} \qquad (1)$$

where, HV: Vickers hardness,
F: Test force (N),
d: Average of diagonal lengths (mm).

Knoop hardness is calculated by equation (2).

$$HK=1.4509\frac{F}{d^2} \qquad (2)$$

where, HK: Knoop hardness,
F: Test force (N),
d: length of longer diagonal (mm).

(Note that HV and HK have no unit specified, and these equations correspond to the value in kgf/mm^2, not in GPa.)

7. Deadline for returning data

All the data sheets should be sent back to the following contact address by 1996-03-31.

<Schedule>

Dec. 1995	Distribution of specimens and instructions.
Mar. 1996	Deadline for sending results back to NIRIN.
June 1996	Distribution of preliminary analysis of the results.
Sep. 1996	Deadline for participants' review of analysis.
Dec. 1996	Publication of final report.

<Contact address>

Shuji SAKAGUCHI
National Industrial Research Institute of Nagoya
1-1, Hirate-cho, Kita-ku, Nagoya 462 JAPAN
Phone: +81-52-911-2111 Fax: +81-52-916-2802

VAMAS Round Robin
Vickers/Knoop hardness of ceramic matrix composites
Report form

Conditions:

Si3N4 / Si3N4-10%SiC(w) / Si3N4-20%SiC(w)
Vickers / Knoop
1kgf / 2kgf / 10kgf
Top (4.5 x 19.5mm) / Side (3.5 x 19.5mm)
Parallel (d) / Perpendicular (e)

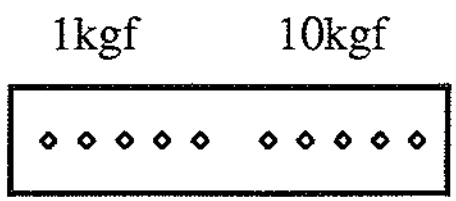
Machine Type (Maker, Model No.) _____

Temperature _____ Humidity _____

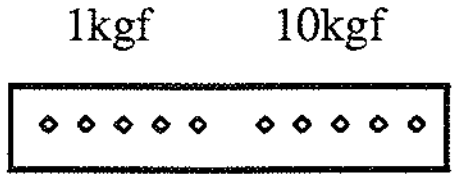
No.	Dia(1)(μm)	Dia(2)(μm)	Dia-Average	Hardness
1				
2				
3				
4				
5				
*Dia(2) and Dia-Average are only for Vickers.				Average
				Standard Deviation

Number of Data for Each Condition: Conventional Hardness Round Robin

Material	Vickers	Knoop	
	Force : Surface : Number	Force : Surface : Number	
Si_3N_4	1kgf : Top (a) : 5 indents	1kgf : Top (c) : 5 indents	If some of the testing force are not available on the equipment, it can be neglected. In total; Vickers: 15 (1kgf), 15 (10kgf) Knoop : 20 (1kgf), 20 (2kgf)
	10kgf : Top (a) : 5 indents	2kgf : Top (c) : 5 indents	
Si_3N_4-SiC(w)20%	1kgf : Top (a) : 5 indents Side (b) : 5 indents	1kgf : Top (c) : 5 indents Side (d) : 5 indents Side (e) : 5 indents	
	10kgf : Top (a) : 5 indents Side (b) : 5 indents	2kgf : Top (c) : 5 indents Side (d) : 5 indents Side (e) : 5 indents	
Si_3N_4-SiC(w)10%	1kgf : Top (a) : 5 indents Side (b) : 5 indents	1kgf : Top (c) : 5 indents Side (d) : 5 indents Side (e) : 5 indents	Testing is Optional In total; Vickers: 10 (1kgf), 10 (10kgf) Knoop : 15 (1kgf), 15 (2kgf)
	10kgf : Top (a) : 5 indents Side (b) : 5 indents	2kgf : Top (c) : 5 indents Side (d) : 5 indents Side (e) : 5 indents	

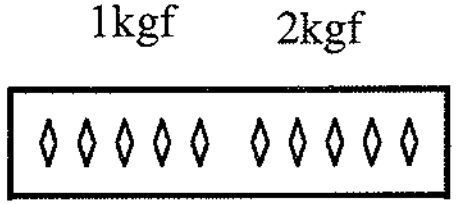


(a)

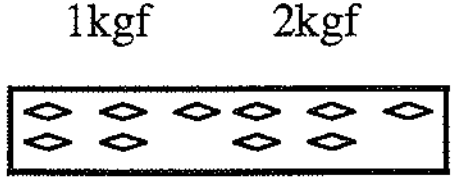


(b)

Vickers

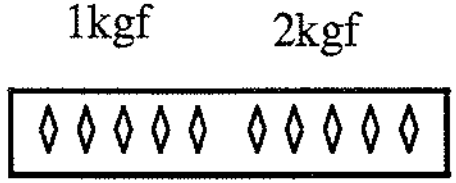


(c)

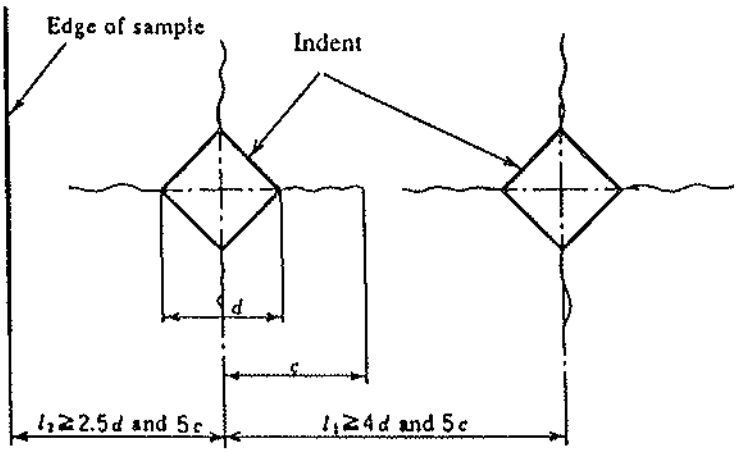


(d)

Knoop



(e)



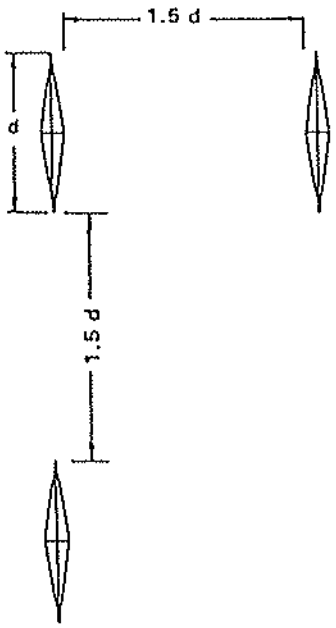
c: length from the center of indent to the end of crack

d: length of indent diagonal

l_1: distance between centers of indents

l_2: distance from center of indent to the edge of sample

Distance between indents and the distance from indent to the sample edge
(from JIS R 1610)



Closest permitted spacing for Knoop indentations
(from ASTM draft)

Appendix III Statistical Analysis of Data by ISO 5725 part 2 (G. Quinn)

This part was prepared by Mr. George Quinn of NIST, USA. He discussed the repeatability and the reproducibility of this round robin data, according to the ISO procedure. This discussion is quite meaningful for understanding this round robin results. Then, the authors agreed with Mr. Quinn that this document shall be attached in this report as this appendix part. The authors acknowledge his effort for preparing this analysis.

Appendix III

Statistical Analysis of Data by ISO 5725 part 2

G. Quinn

The laboratory average and standard deviation results were analyzed according to ISO standard 5725-2:

> "Accuracy (Trueness and Precision) of Measurement Methods and Results, Part 2: Basic Method for Determination of Repeatability and Reproducibility of a Standard Measurement" First Edition, 1994.

Part 1 of ISO 5725 defines the terms. Precision refers to the closeness of agreement between test results and it is the general term for variability between repeated measurements. There are two conditions of precision: "repeatability" and "reproducibility."

Repeatability is defined as the "precision under repeatability conditions" which are tests conducted by the same laboratory on a homogeneous material in the shortest practical time. Repeatability is also known as the "within-lab" variability. The *repeatability standard deviation* is defined in ISO 5725-1 as:

"The standard deviation of test results under repeatability conditions."

Note: It is a measure of dispersion of the distribution of test dispersion results under repeatability conditions.

Note: Similarly, "repeatability variance" and "repeatability coefficients of variation could be defined and used as measures of dispersion of test results under repeatability conditions.

Reproducibility is defined as the "precision under reproducibility conditions" which are tests performed by different laboratories on the same material. Reproducibility is also known as the "between-lab" variability. The definition of the *reproducibility standard deviation* is:

"The standard deviation of test results under reproducibility conditions."

Note: It is a measure of dispersion of the distribution of test dispersion results under reproducibility conditions.

Note: Similarly, "reproducibility variance" and "reproducibility coefficients of variation could be defined and used as measures of dispersion of test results under reproducibility conditions.

Mathematical formulae for both the repeatability and reproducibility standard deviations are given in ISO 5725-part 2. The repeatability standard deviation for one test condition (e.g. Material A, Vickers, 1 kg, top surface) is simply the root mean square of the individual laboratory standard deviations. The reproducibility standard deviation is a little more complicated and depends upon the within-lab standard deviations and the deviations of the laboratory means from the grand mean.

Table III-1 shows the repeatability and reproducibility standard deviations and the grand mean for all test conditions and materials in this round robin. The laboratory averages and standard deviations were used for these calculations. ISO 5725-2 has two possible procedures to identify

Table III-1
VAMAS Conventional Hardness of Ceramic Composites Round Robin

Repeatability and Reproducibility by ISO 5725 part 2

Test	Material		Within - lab Repeatability Std. Dev.	Coef. of Var.	Between - Lab Reproducibility Std. Dev.	Coef of Var.	# of labs.	Grand Mean	Comments (See notes below)
Vickers 1 kg	A		55	4.0%	106	7.7%	20	1377	Lab 11 deleted, scatter too high
	C - top		43	2.9%	90	6.1%	17	1457	Lab 12 deleted, mean and scatter too high; Lab 19 deleted, scatter too high
	C - side		54	3.7%	91	6.3%	17	1440	Lab 12 deleted, mean too high; Lab 13 deleted, scatter too high
	D - top		50	3.4%	108	7.3%	20	1486	Lab 2 deleted, mean and scatter too high
	D - side		47	3.1%	92	6.1%	20	1503	Lab 12 deleted, mean too high
Vickers 10 kg	A		30	2.3%	51	3.9%	17	1314	Lab 12 deleted, mean too high
	C - top		20	1.4%	43	3.1%	14	1393	Lab 12 deleted, scatter too high; Lab 19 deleted, scatter too high
	C - side		21	1.5%	36	2.6%	15	1389	Lab 12 deleted, scatter too high
	D - top		25	1.7%	54	3.7%	17	1440	Lab 12 deleted, mean too high. Scatter = 0?
	D - side		26	1.8%	53	3.6%	17	1445	Lab 12 deleted, mean and scatter are too high
Knoop 1 kg	A		38	2.9%	73	5.6%	20	1306	no outliers
	C - top		37	2.7%	81	5.9%	17	1385	Lab 21 deleted, mean and scatter too high
	C -side	parallel	31	2.3%	86	6.3%	16	1360	Lab 21 deleted, mean and scatter too high; Lab 12 deleted, scatter too high
	C -side	perpendicular	43	3.1%	92	6.7%	16	1378	Lab 21 deleted, mean too high; Lab 17 deleted, scatter too high
	D - top		34	2.4%	87	6.1%	18	1412	Lab 21 deleted, mean too high; Lab 12 deleted, scatter too high
	D - side	parallel	41	2.9%	87	6.2%	18	1406	Lab 21 deleted, mean too high; Labs 12 deleted, scatter too high
	D - side	perpendicular	38	2.6%	86	6.0%	17	1427	Lab 21 deleted, mean too high; Labs 12 and 15 deleted, scatter too high
Knoop 2 kg	A		31	2.4%	44	3.5%	13	1259	no outliers
	C - top		21	1.6%	44	3.3%	10	1327	Lab 19 deleted, scatter too high
	C -side	parallel	34	2.6%	55	4.2%	10	1313	Lab 12 deleted, scatter too high
	C -side	perpendicular	42	3.2%	57	4.4%	11	1312	no outliers
	D - top		28	2.1%	62	4.6%	13	1353	no outliers
	D - side	parallel	37	2.7%	46	3.4%	12	1355	Lab 12 deleted, mean too low and scatter too high
	D - side	perpendicular	24	1.7%	50	3.6%	12	1387	Lab 18 deleted, scatter too high.

Notes: "Deleted" = the data set is an outlier and is not included in the Repeatability or Reproducibility estimates, nor in ithe Grand Average
"Scatter too high" = the standard deviation of the lab data set is an outlier and is too large.
"Mean too high or too low" = the mean of the lab data set is an outlier since it deviates too much from the other labs means.

"outlier" or "straggler" data sets which differ from the overall results at a 1% (outlier) or 5% (straggler) confidence levels. Mandel's *h* and *k* statistics were used in the present study. (Note: this procedure is very similar to that used in ASTM E 691-92, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method" except that the latter uses a 0.5% significance level for outliers.)

The "k-statistic" is the within-lab consistency statistic and is calculated for each laboratory data set. Values greater than 1.0 indicate a greater than average amount of scatter within a laboratory's data set. $k_{critical}$, which is from the F test at a 1.0% significance level, depends upon the number of replicates per test condition (5 in the present round robin) and the number of participating laboratories (up to 21 in the present round robin.) $k_{critical}$ values for outliers are given in Table 6 of ISO 5725-2 and $k_{critical}$ values for stragglers in Table 7.

The "h-statistic" is the between-lab consistency statistic is also calculated for each laboratory's data set. It quantifies the difference of one laboratory's result is from the grand mean. Values greater than 1.0 indicate the laboratory's result has greater than the usual deviation. $h_{critical}$, which is from the two-tailed student t distribution at a 1.0% significance level, also depends upon the number of replicates per test condition (5) and the number of participating laboratories (up to 21). $h_{critical}$ values for outliers and stragglers are also given in Tables 6 and 7 of ISO 5725-2, respectively.

An example of the application of ISO 5725-2 to one test condition, material A, Vickers 1 kg, is shown in Table III-2a. The values of $h_{critical}$ and $k_{critical}$ are shown on the bottom of the spreadsheet and each laboratory's data set is compared to these statistics. Laboratory 11's data set is identified as an outlier (the k statistic is higher than $k_{critical}$) which means that the data set standard deviation is too high. Two other data sets (laboratories 12 and 21) were identified as stragglers since their mean values had a relatively high deviancy from the grand mean. Table III-2b shows the adjusted statistics when the outlier data set is deleted.

Another example is Table III-3, which shows the analysis for Material D, top surface, Knoop 2 kg. No outliers were identified in this case.

The last example is Table III-4a, which shows the analysis for Material D, side surface, perpendicular orientation, Knoop 2 kg. In this case, laboratory 18's within-lab scatter (standard deviation) was high and was identified as an outlier. Table III-4b shows the statistics with this laboratory set deleted.

All the laboratory test conditions were analyzed in a similar fashion and the summary results are shown in Table III-1. The following conclusion may be drawn from Table III-1:

1. The within-laboratory precision is always smaller than the between-laboratory precision.

2. Usually only one or two laboratory data sets were identified as outliers. In four conditions with the Knoop hardness method, no outliers were identified. Data from laboratories 12 and 21 were often outliers.

Table III-2a

VAMAS COMPOSITE HARDNESS ROUND ROBIN — MATERIAL A — VICKERS 1 kg - All lab sets

Quatro Pro for Windows spreadsheet for Statistical Analysis of Data by:
ISO 5725 part 2 Using Mandel's analysis for h and k

Report as your output, the bold numbers.

Run on : 10/20/97

Lab/Group #	Number of Replicates	Lab/Group Average (units)	Within-lab/group "REPEATABILITY (r)" Lab/Group Std Dev (Units)	Mandel's k statistic		Between-lab/group "REPRODUCIBILITY (R)" Dev. from Average (Units)	Mandel's h statistic		(std dev)^2	(Dev. from avg)^2
1	5	1326	33	0.45		-47.00	-0.50	1	1089.0	2209.0
2	5	1513	67	0.92		140.00	1.50	2	4489.0	19600.0
3	5	1377	33	0.45		4.00	0.04	3	1089.0	16.0
4	5	1456	16	0.22		83.00	0.89	4	256.0	6889.0
5	5	1319	37	0.51		-54.00	-0.58	5	1369.0	2916.0
6	5	1424	83	1.14		51.00	0.55	6	6889.0	2601.0
7	5	1379	49	0.67		6.00	0.06	7	2401.0	36.0
8	5	1288	37	0.51		-85.00	-0.91	8	1369.0	7225.0
9	5	1216	59	0.81		-157.00	-1.69	9	3481.0	24649.0
10	5	1467	55	0.75		94.00	1.01	10	3025.0	8836.0
11	5	1295	228	3.12	<< ** outlier	-78.00	-0.84	11	51984.0	6084.0
12	5	1556	25	0.34	* straggler >	183.00	1.96	12	625.0	33489.0
13	5	1414	70	0.96		41.00	0.44	13	4900.0	1681.0
14	5	1298	53	0.73		-75.00	-0.81	14	2809.0	5625.0
15	5	1349	88	1.21		-24.00	-0.26	15	7744.0	576.0
16	5	1419	93	1.27		46.00	0.49	16	8649.0	2116.0
17	5	1488	19	0.26		115.00	1.23	17	361.0	13225.0
18	5	1326	50	0.68		-47.00	-0.50	18	2500.0	2209.0
19	5	1364	39	0.53		-9.00	-0.10	19	1521.0	81.0
20	5	1369	63	0.86		-4.00	-0.04	20	3969.0	16.0
21	5	1190	38	0.52	* straggler >	-183.00	-1.96	21	1444.0	33489.0
21	**5**	**1373.0**	Sr = **73.0**			SR= **113.8**				
# of Labs/Grou	n (avg)	avg of the lab avgs.	Repeatability std. dev.			Reproducibility std. dev.				
	Sxbar = 93.2		CV (%) = **5.3**			CV (%) = **8.3**				
	std dev. of avgs. above		Repeatability Coef. of Variation			Reproducibility Coef. of variation				
			** Outlier k crit = **1.78**			** Outlier h crit = **2.39**				
			* Straggler k crit = 1.52			* Straggler h crit 1.89				

(Look up both h crit and k crit in Table 6 for 1% Outliers and Table 7 for 5% Stragglers. Values of h or k greater than crit suggest Outliers or Stragglers. CHECK LAB RESULTS.

k is the "within-lab consistency statistic." Values greater than 1 indicate greater than average amount of scatter within a lab set. k crit is from the F test at a 1.0% significance level.

h is the "between-lab consistency statistic." It shows how deviant one lab's average is from the "grand mean." Values > 1 indicate greater than the average deviation. Two tailed student t test at 1.0% significance level.

Table III-2b

VAMAS COMPOSITE HARDNESS ROUND ROBIN — MATERIAL A — VICKERS 1 kg - DELETE LAB 11

Quatro Pro for Windows spreadsheet for Statistical Analysis of Data by:
ISO 5725 part 2 Using Mandel's analysis for h and k

Report as your output, the bold numbers.

Run on : 10/20/97

Lab/Group #	Number of Replicates	Lab/Group Average (units)	Within-lab/group "REPEATABILITY (r)" Lab/Group Std Dev (Units)	Mandel's k statistic		Between-lab/group "REPRODUCIBILITY (R)" Dev. from Average (Units)	Mandel's h statistic
1	5	1326	33	0.60		-50.90	-0.54
2	5	1513	67	1.22		136.10	1.45
3	5	1377	33	0.60		0.10	0.00
4	5	1456	16	0.29		79.10	0.84
5	5	1319	37	0.68		-57.90	-0.62
6	5	1424	83	1.52		47.10	0.50
7	5	1379	49	0.89		2.10	0.02
8	5	1288	37	0.68		-88.90	-0.95
9	5	1216	59	1.08		-160.90	-1.72
10	5	1467	55	1.00		90.10	0.96
12	5	1556	25	0.46	* straggler >	179.10	1.91
13	5	1414	70	1.28		37.10	0.40
14	5	1298	53	0.97		-78.90	-0.84
15	5	1349	88	1.61	< new straggler *	-27.90	-0.30
16	5	1419	93	1.70	< new straggler *	42.10	0.45
17	5	1488	19	0.35		111.10	1.18
18	5	1326	50	0.91		-50.90	-0.54
19	5	1364	39	0.71		-12.90	-0.14
20	5	1369	63	1.15		-7.90	-0.08
21	5	1190	38	0.69	* straggler >	-185.90	-1.99
20	**5**	**1376.9**	Sr = **64.8**			SR= **105.8**	
# of Labs/Grou	n (avg)	avg of the lab avgs.	Repeatability std. dev.			Reproducibility std. dev.	
	Sxbar =	93.8	CV (%) = **4.0**			CV (%) = **7.7**	
	std dev. of avgs. above		Repeatability Coef. of Variation			Reproducibility Coef. of variation	
			** Outlier k crit =	1.78		** Outlier h crit =	2.39
			* Straggler k crit =	1.52		* Straggler h crit	1.89

Lab	(std dev)^2	(Dev. from avg)^2
1	1089.0	2590.8
2	4489.0	18523.2
3	1089.0	0.0
4	256.0	6256.8
5	1369.0	3352.4
6	6889.0	2218.4
7	2401.0	4.4
8	1369.0	7903.2
9	3481.0	25888.8
10	3025.0	8118.0
12	625.0	32076.8
13	4900.0	1376.4
14	2809.0	6225.2
15	7744.0	778.4
16	8649.0	1772.4
17	361.0	12343.2
18	2500.0	2590.8
19	1521.0	166.4
20	3969.0	62.4
21	1444.0	34931.6

(Look up both h crit and k crit in Table 6 for 1% Outliers and Table 7 for 5% Stragglers. Values of h or k greater than crit suggest Outliers or Stragglers. CHECK LAB RESULTS.

k is the "within-lab consistency statistic." Values greater than 1 indicate greater than average amount of scatter within a lab set. k crit is from the F test at a 1.0% significance level.

h is the "between-lab consistency statistic." It shows how deviant one lab's average is from the "grand mean." Values > 1 indicate greater than the average deviation. Two tailed student t test at 1.0% significance level.

Table III-3

VAMAS COMPOSITE HARDNESS ROUND ROBIN — MATERIAL D - top — KNOOP 2 kg

all lab sets

Quatro Pro for Windows spreadsheet for Statistical Analysis of Data by:
ISO 5725 part 2 Using Mandel's analysis for h and k

Report as your output, the bold numbers.

Run on : 10/20/97

Lab/Group #	Number of Replicates	Lab/Group Average (units)	Within-lab/group "REPEATABILITY (r)" Lab/Group Std Dev (Units)	k statistic		Between-lab/group "REPRODUCIBILITY (R)" Dev. from Average (Units)	h statistic		(std dev)^2	(Dev. from avg)^2
2	5	1392	12	0.43		39.00	0.69	2	144.0	1521.0
4	5	1330	16	0.57		-23.00	-0.41	4	256.0	529.0
5	5	1368	32	1.15		15.00	0.27	5	1024.0	225.0
7	5	1312	12	0.43		-41.00	-0.73	7	144.0	1681.0
10	5	1413	27	0.97		60.00	1.06	10	729.0	3600.0
12	5	1296	39	1.40		-57.00	-1.01	12	1521.0	3249.0
13	5	1272	28	1.00		-81.00	-1.43	13	784.0	6561.0
14	5	1359	16	0.57		6.00	0.11	14	256.0	36.0
15	5	1328	30	1.07		-25.00	-0.44	15	900.0	625.0
16	5	1412	24	0.86		59.00	1.05	16	576.0	3481.0
18	5	1271	31	1.11		-82.00	-1.45	18	961.0	6724.0
19	5	1445	25	0.90		93.00	1.65	19	625.0	8649.0
21	5	1390	47	1.68	<* straggler	37.00	0.66	21	2209.0	1369.0
13	**5**	**1353.0**	Sr = **27.9**	Repeatability std. dev.		SR= **61.7**	Reproducibility std. dev.			
# of Labs/Grou	n (avg)	avg of the lab avgs.								
	Sxbar = 56.5	std dev. of avgs. above	CV (%) = **2.1**	Repeatability Coef. of Variation		CV (%) = **4.6**	Reproducibility Coef. of variation			

** Outlier k crit	1.76	** Outlier h crit =	2.27
* Straggler k crit	1.51	* Straggler h crit	1.84

(Look up both h crit and k crit in Table 8 for 1% Outliers and Table 7 for 5% Stragglers. Values of h or k greater than crit suggest Outliers or Stragglers. CHECK LAB RESULTS.

k is the "within-lab consistency statistic." Values greater than 1 indicate greater than average amount of scatter within a lab set. k crit is from the F test at a 1.0% significance level.

h is the "between-lab consistency statistic." It shows how deviant one lab's average is from the "grand mean." Values > 1 indicate greater than the average deviation Two tailed student t test at 1.0% significance level.

Table III-4a

VAMAS COMPOSITE HARDNESS ROUND ROBIN — MATERIAL D -side perpendicular — KNOOP 2 kg all lab sets

Quatro Pro for Windows spreadsheet for Statistical Analysis of Data by:
ISO 5725 part 2 Using Mandel's analysis for h and k

Report as your output, the bold numbers.

Run on : 10/20/97

Lab/Group #	Number of Replicates	Lab/Group Average (units)	Within-lab/group "REPEATABILITY (r)" Lab/Group Std Dev (Units)	k statistic		Between-lab/group "REPRODUCIBILITY (R)" Dev. from Average (Units)	h statistic		(std dev)^2	(Dev. from avg)^2
2	5	1361	25	0.72		-18.62	-0.37		2	625.0	346.5
4	5	1378	25	0.72		-1.62	-0.03		4	625.0	2.6
5	5	1357	19	0.55		-22.62	-0.45		5	361.0	511.5
7	5	1373	27	0.78		-6.62	-0.13		7	729.0	43.8
10	5	1461	34	0.98		81.38	1.61		10	1156.0	6623.5
12	5	1322	23	0.67		-57.62	-1.14		12	529.0	3319.5
13	5	1344	24	0.70		-35.62	-0.70		13	576.0	1268.5
14	5	1348	24	0.70		-31.62	-0.63		14	576.0	999.5
15	5	1394	13	0.38		14.38	0.28		15	169.0	206.9
16	5	1420	23	0.67		40.38	0.80		16	529.0	1630.9
18	5	1293	93	2.69	< outlier **	-86.62	-1.71		18	8649.0	7502.2
19	5	1428	20	0.58		48.38	0.96		19	400.0	2341.1
21	5	1456	24	0.70		76.38	1.51		21	576.0	5834.6
13	**5**	**1379.6**	Sr = **34.5**	Repeatability std. dev.		SR= **59.2**	Reproducibility std. dev.			
# of Labs/Grou	n (avg)	avg of the lab avgs.								
	Sxbar = 50.5		CV (%) = **2.5**	Repeatability Coef. of Variation		CV (%) = **4.3**	Reproducibility Coef. of variation			
	std dev. of avgs. above									
			** Outlier k crit	1.76		** Outlier h crit =	2.27			
			* Straggler k crit	1.51		* Straggler h crit	1.84			

(Look up both h crit and k crit in Table 6 for 1% Outliers and Table 7 for 5% Stragglers. Values of h or k greater than crit suggest Outliers or Stragglers. CHECK LAB RESULTS.

k is the "within-lab consistency statistic." Values greater than 1 indicate greater than average amount of scatter within a lab set. k crit is from the F test at a 1.0% significance level.

h is the "between-lab consistency statistic." It shows how deviant one lab's average is from the "grand mean." Values > 1 indicate greater than the average deviation. Two tailed student t test at 1.0% significance level.

Table III-4b

VAMAS COMPOSITE HARDNESS ROUND ROBIN — MATERIAL D -side perpendicular — KNOOP 2 kg

Delete Lab 18

Quatro Pro for Windows spreadsheet for Statistical Analysis of Data by:
ISO 5725 part 2 Using Mandel's analysis for h and k

Report as your output, the bold numbers.

Run on : 10/20/97

Lab/Group #	Number of Replicates	Lab/Group Average (units)	Within-lab/group "REPEATABILITY (r)" Lab/Group Std Dev (Units)	k statistic	Between-lab/group "REPRODUCIBILITY (R)" Dev. from Average (Units)	h statistic	(std dev)^2	(Dev. from avg)^2
2	5	1361	25	1.05	-25.83	-0.57	625.0	667.4
4	5	1378	25	1.05	-8.83	-0.20	625.0	78.0
5	5	1357	19	0.80	-29.83	-0.66	361.0	890.0
7	5	1373	27	1.13	-13.83	-0.31	729.0	191.4
10	5	1461	34	1.42	74.17	1.64	1156.0	5500.7
12	5	1322	23	0.96	-64.83	-1.43	529.0	4203.4
13	5	1344	24	1.00	-42.83	-0.95	576.0	1834.7
14	5	1348	24	1.00	-38.83	-0.86	576.0	1508.0
15	5	1394	13	0.54	7.17	0.16	169.0	51.4
16	5	1420	23	0.96	33.17	0.73	529.0	1100.0
19	5	1428	20	0.84	41.17	0.91	400.0	1694.7
21	5	1456	24	1.00	69.17	1.53	576.0	4784.0
12	**5**	**1386.8**	Sr = **23.9**	Repeatability std. dev.	SR= **50.0**	Reproducibility std. dev.		
# of Labs/Grou	n (avg)	avg of the lab avgs.						
	Sxbar =	45.2	CV (%) = **1.7**	Repeatability Coef. of Variation	CV (%) = **3.6**	Reproducibility Coef. of variation		
	std dev. of avgs. above							
			** Outlier k crit	1.75	** Outlier h crit =	2.25		
			* Straggler k crit	1.51	* Straggler h crit	1.83		

(Look up both h crit and k crit in Table 6 for 1% Outliers and Table 7 for 5% Stragglers. Values of h or k greater than crit suggest Outliers or Stragglers. CHECK LAB RESULTS.

k is the "within-lab consistency statistic." Values greater than 1 indicate greater than average amount of scatter within a lab set. k crit is from the *F* test at a 1.0% significance level.

h is the "between-lab consistency statistic." It shows how deviant one lab's average is from the "grand mean." Values > 1 indicate greater than the average deviation. Two tailed student t test at 1.0% significance level.

3. Comparisons of the grand means show that the indentation size effect is present for all materials and orientations.

4. The hardness of the three materials was ranked: $H_D > H_C > H_A$, for all conditions. The differences may or may not be statistically significant, however.

5. There appears to be no difference in HV with orientation for materials C and D.

6. The Knoop hardness of the top and sides of materials C and D are similar.

7. The Knoop hardness, perpendicular, is usually greater than the Knoop hardness, parallel for materials C and D, but the differences are probably not statistically significant.

8. The lowest variability (highest precision) was for higher indentation loads: HV10 and HK2. The precision estimates did not vary very much for a given test procedure! Table III-5 shows the average repeatability and reproducibilty precision estimates for the test methods used in this round robin.

Table III-5
Grand precision estimates by test method.

Test Method	Repeatability (Within-lab) Precision Coefficient of Variation	Reproducibility (Between-lab) Precision Coefficient of Variation
Vickers, 1 kg	3.4%	6.7%
Vickers, 10 kg	1.7%	3.4%
Knoop, 1 kg	2.7%	6.1%
Knoop, 2 kg	2.3%	3.9%

This analysis is a shortened version of a fully comprehensive analysis that could have been conducted according to ISO 5725-2. The comprehensive analysis would include an analysis of all *individual test results* and not simply the laboratory average and standard deviations. Graphs of the h and k statistics also could reveal systematic trends in the laboratory data, e.g., if one laboratory's mean hardness was systematically too high or too low, or the scatter was systematically too high or low.

Finally, we observe that the precision estimates are specific to the three materials used in this study. It is assumed that the specimens furnished to each laboratory were as uniform as possible. Any variability from specimen to specimen is included in the statistics. Furthermore, the precision estimates are pertinent to the specific test methods used as specified in the instructions (Appendix II). Any differences in the procedures used in the test methods may change the precision estimates.