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VNIIFTRI / PTB BILATERAL COMPARISON ON MARTENS AND INDENTATION HARDNESS SCALES

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Abstract

The present article contains the results of bilateral comparisons in the field of nanoindentation conducted in 2014 in the framework of cooperation between PTB and metrological institutes of Russia. The comparisons were held using primary standard machines applied in PTB and VNIIFTRI. Hardness numbers of Martens and indentation hardness scales obtained by the two laboratories were compared with due consideration of expanded uncertainties of measurements.

Keywords: nanoindentation, Martens hardness, indentation hardness, bilateral comparison, micro- and nano-range

1. INTRODUCTION

The comparisons were held using primary standard machines belonging to PTB and VNIIFTRI. Hardness measurements were conducted on samples made of sapphire monocrystal, fused quartz and polycarbonate. The range of loads applied to indenters vary between 0,1 mN and 500 mN. Hardness numbers on Martens and indentation hardness scales obtained by the two laboratories were compared with due consideration of expanded uncertainties of measurements. The components of hardness measurement uncertainties on Martens and indentation hardness scales were studied and their traceability to primary standard machines of mass, length and acceleration of gravity was provided. The materials for hardness test blocks were selected on the basis of the studies [1, 2]. The main advantage of these test blocks lies in the fact that they are homogeneous in hardness, do not oxidize with time and isotropy axis of sapphire monocrystal coincides with the direction of the indenter penetration.

2. MAIN PART

Hardness on Martens and indentation scales were measured using methods specified in the standard [3], on the machines corresponding to the requirements of the standard [4]. The hardness of polycarbonate was determined at the loads of 0,1 mN; 0,5 mN; 1 mN; 5 mN; 10 mN; 50 mN; 100 mN. The hardness of fused quartz was determined at the loads of 0,5 mN; 1 mN; 5 mN, 10 mN, 50 mN; 100 mN. The hardness of sapphire was determined at the loads of 5 mN; 10 mN; 50 mN; 100 mN, 500 mN. 20 measurements were conducted on each of the samples at the above mentioned loads. The machines used standard Berkovich indenters with the angle between the vertical axis and a side facet equaling $(65{,}0 \pm 0{,}3)^0$.

The air temperature in the laboratory during measurements equaled $(21 \pm 2)\ ^0$C, the relative humidity of the air in the laboratory was (45 ± 5) %. The drift velocity of the samples surface due to a temperature change was less than 0,05 nm/s. The approach velocity of the indenter to the surface was no more than 100 nm/s. The time of the load application and removal equaled 30 s, the time of exposure to the load was 10 s.

During the comparisons the following points were determined:

-mean hardness value on Martens and indentation scales for each test block at each load;

-root-mean-square deviation of hardness measurement results;

-mean value of maximum depth of the indenter penetration for each test block at each load;

-mean value of contact depth of the indenter penetration for each test block at each load;

-functions of cross section area and surface area of the indenter;

-expanded uncertainties of hardness measurement results on Martens and indentation scales.

Tables 1 and 2 show differences in hardness measurements performed on indentation and Martens scales on a sample made of fused quartz. Tables 3 and 4 show differences in hardness measurements carried out on indentation and Martens scales on a sample made of polycarbonate. Tables 5 and 6 show differences in hardness measurements conducted on indentation and Martens scales on a sample made of sapphire.

Table 1. Difference in results of indentation hardness measurements on fused quartz between PTB and VNIIFTRI

Fmax, mN	Difference in H_{IT}, GPa	$u(H_{IT})_{PTB}$, GPa	$u(H_{IT})_{VNIIFTRI}$, GPa
0,5	0,5	0,9	0,60
1,0	0,12	0,9	0,61
5,0	0,12	0,9	0,61
10,0	0,43	0,9	0,64
500,0	0,68	0,6	0,58

Table 2. Difference in results of Martens hardness measurements on fused quartz between PTB and VNIIFTRI

Fmax, mN	Difference in HM, GPa	$u(HM)_{PTB}$, GPa	$u(HM)_{VNIIFTRI}$, GPa
1,0	1,42	1,5	0,1
5,0	1,35	1,5	0,1
10,0	1,40	1,4	0,1
500,0	0,65	1,2	0,1

Table 3. Difference in results of indentation hardness measurements on polycarbonate between PTB and VNIIFTRI

Fmax, mN	Difference in H_{IT}, GPa	$u(H_{IT})_{PTB}$, GPa	$u(H_{IT})_{VNIIFTRI}$, GPa
0,1	0,05	0,03	0,02
0,5	0,03	0,02	0,01
1,0	0,02	0,02	0,01
5,0	0,02	0,02	0,01
10,0	0,05	0,02	0,01
50,0	0,01	0,007	0,01
100,0	0,01	0,006	0,01

Table 4. Difference in results of Martens hardness measurements on polycarbonate between PTB and VNIIFTRI

Fmax, mN	Difference in HM, GPa	$u(HM)_{PTB}$, GPa	$u(HM)_{VNIIFTRI}$, GPa
1,0	0,01	0,035	0,01
5,0	0,00	0,025	0,01
10,0	0,01	0,020	0,01
50,0	0,02	0,015	0,01
100,0	0,01	0,015	0,01

Table 5. Difference in results of indentation hardness measurements on sapphire between PTB and VNIIFTRI

Fmax, mN	Difference in H_{IT}, GPa	$u(H_{IT})_{PTB}$, GPa	$u(H_{IT})_{VNIIFTRI}$, GPa
5,0	0,22	2,8	1,9
10,0	0,47	2,8	1,9
50,0	4,18	3,4	1,8
100,0	3,89	3,2	1,8
500,0	4,02	2,5	1,8

Table 6. Difference in results of Martens hardness measurements on sapphire between PTB and VNIIFTRI

Fmax, mN	Difference in HM, GPa	$u(HM)_{PTB}$, GPa	$u(HM)_{VNIIFTRI}$, GPa
5,0	6,85	2,8	0,4
10,0	6,59	2,6	0,4
50,0	5,18	2,5	0,4
100,0	4,12	2,0	0,4
500,0	3,31	2,0	0,3

Tables 7 and 8 show maximum and minimum deviations in hardness numbers on Martens and indentation scales between the two laboratories on the measured test blocks.

Table 7. Maximum and minimum deviations in modulus between VNIIFTRI and PTB laboratories during hardness measurements on Martens scales.

Sample	Maximum deviation, GPa	Minimum deviation, GPa
polycarbonate	0,02	0,00
fused quartz	1,42	0,65
sapphire	6,85	3,31

Table 8. Maximum and minimum deviations in modulus between VNIIFTRI and PTB laboratories during hardness measurements on indentation scales.

Sample	Maximum deviation, GPa	Minimum deviation, GPa
polycarbonate	0,05	0,01
fused quartz	0,68	0,12
sapphire	4,18	0,22

It is seen from the analysis of Tables 7 and 8 that the maximum relative deviation of indentation hardness measurement results was observed on the polycarbonate sample. The maximum relative deviation of Martens hardness measurement was observed on the sapphire sample. In general, the results of measurements conducted on the three samples are in good agreement. However, a difference has been revealed in the results of measurements performed on the sample made of sapphire on Martens and indentation hardness scales at loads between 50 mN and 500 mN, as well as on the sample made of polycarbonate on indentation hardness scales at loads between 0,1 mN and 10 mN, which require further adjustment.

3. CONCLUSIONS

The results of bilateral comparisons in the field of nanoindentation between PTB and VNIIFTRI are in agreement.

It is planned to further continue the comparisons as regional additional ones in the line of COOMET.

4. REFERENCES

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