

INFLUENCE OF MEASUREMENT CYCLE PARAMETERS ON MARTENS AND INDENTATION HARDNESS VALUES

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Abstract: The present paper explores the influence of various testing cycle parameters of the instrumental indentation method on the hardness measurement result. The measurements were held using the Russian national primary standard machine on Martens and indentation hardness scales. The study used a wide range of materials at different loads and obtained hardness measurement results in the micro- and nano-ranges with due consideration of the influence of testing cycle parameters.

Keywords: instrumental indentation, national primary standard machine, hardness, nano-range.

1. INTRODUCTION

The present paper was compiled in the framework of metrological studies of the Russian national primary standard machine on Martens and indentation hardness scales [1].

The studies described in the present paper aimed at finding a measurement mode that would minimize the influence of testing cycle parameters on the hardness measurement result.

ISO 14577-3:2015, which specifies a method for the calibration of test blocks, defines testing cycle parameters for calibration hardness testers. The current paper demonstrates a change in hardness of the samples made of various materials on indentation scales depending on the approach velocity of the indenter, time of loading, unloading, as well as exposure to the load.

2. EXPERIMENTAL PROCEDURE

Experiments were held on samples made of polymethylmethacrylate, polycarbonate, gold, brass, aluminum monocrystal, tungsten monocrystal, fused quartz, sapphire monocrystal. Hardness on indentation scales was determined according to [2]. Berkovich indenter with a $65,27^\circ$ angle between the vertical axis and the side facet was used during measurements.

Frame compliance and indenter area function were determined according to [3] before measurements. After that, heterogeneity of the samples was defined. Dispersion of results (difference between the maximum and minimum hardness values) as well as relative standard deviation of 15 measurements were considered the measures of heterogeneity.

The air temperature in the room during the use of the primary standard machine is maintained at $(21.4 \pm 0.2)^\circ\text{C}$,

and the relative humidity of the air is $(43 \pm 2)\%$. The instrumental indentation installations that make part of the standard machine are placed on vibro-isolated foundations, which minimizes the influence of vibration of measurements [4].

Hardness on indentation scales was tested at different forces. Table 1 shows the maximum applied forces for each sample.

Table 1. Tested samples and applied forces.

Sample materials	Applied force, mN
Polymethylmethacrylate	0,1; 0,2; 0,5; 0,8; 1;
Polycarbonate	0,1; 0,2; 0,5; 1;
Gold	0,5; 1; 5
Brass	0,5; 1; 5; 10;
Aluminum monocrystal	0,5; 1; 5;
Tungsten monocrystal	1; 5; 10; 50;
Fused quartz	0,5; 1; 5; 10; 50
Sapphire monocrystal	2; 5; 10; 50;

Testing cycle parameters varied for each sample and force. The approach velocity of the indenter towards the sample surface changed from 5 nm/s up to 100 nm/s. The time of load application and exposure varied from 10 to 40 s, while the unloading time changed from 5 to 20 s. When one of the parameters varied, the others remained constant.

As a result of measurements, mean hardness values on indentation scales were determined for each applied force.

Figure 1 shows the dependence of hardness on indentation and Martens scales at the maximum load of 1 mN on the approach velocity of the indenter. The time of loading and exposure to the load equals 30 s. The unloading time equals 10 s. The material of the sample is aluminum monocrystal.

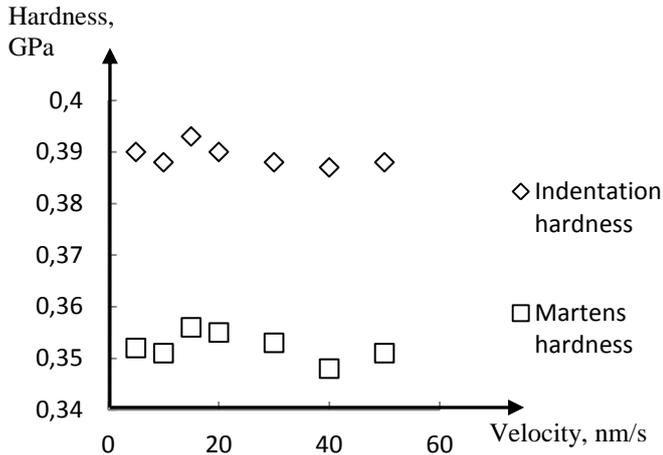


Fig. 1 Dependence of H_{IT} and HM on indenter velocity for aluminum monocrystal at 1 mN load.

Figure 2 shows the dependence of hardness on indentation and Martens scales at the maximum load of 5 mN on the approach velocity of the indenter.

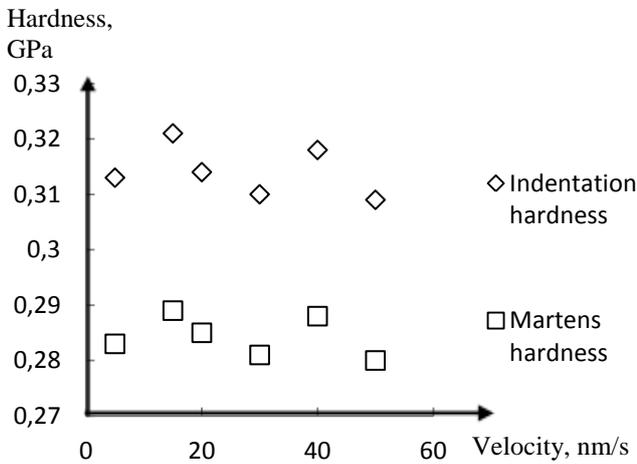


Fig. 2 Dependence of H_{IT} and HM on indenter velocity for aluminum monocrystal at 5 mN load.

Figure 3 shows the dependence of hardness on indentation and Martens scales at the maximum load of 1 mN on the approach velocity of the indenter. The material of the sample is gold.

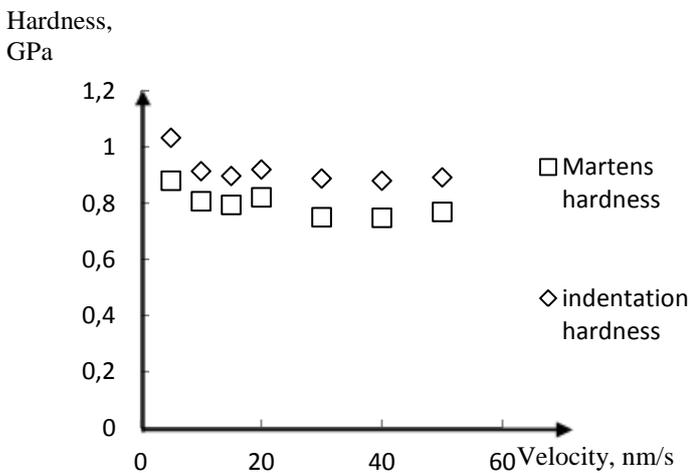


Fig. 3 Dependence of H_{IT} and HM on indenter velocity for gold.

Figure 4 shows the dependence of hardness on indentation and Martens scales at the maximum load of 0.5 mN on the approach velocity of the indenter. The material of the sample is brass.

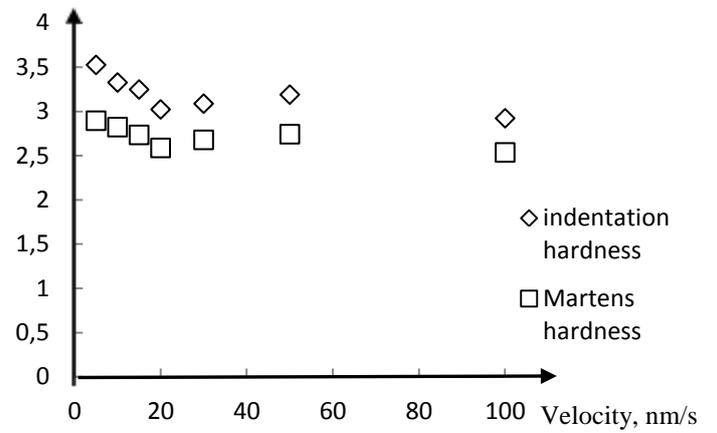


Fig. 4 Dependence of H_{IT} and HM on indenter velocity for brass.

Figure 5 shows the dependence of hardness on indentation and Martens scales at the maximum load of 1 mN on the approach velocity of the indenter. The material of the sample is tungsten.

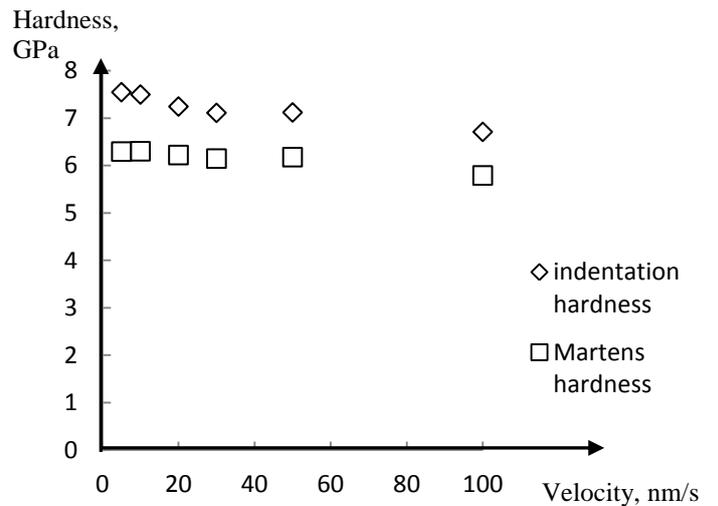


Fig. 5 Dependence of H_{IT} and HM on indenter velocity for tungsten at 1 mN load.

Figure 6 shows the dependence of hardness on indentation and Martens scales at the maximum load of 50 mN on the approach velocity of the indenter. The material of the sample is tungsten.

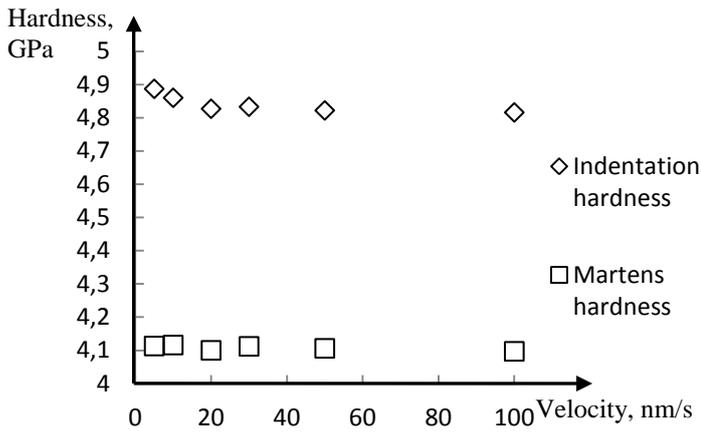


Fig. 6 Dependence of H_{IT} and HM on indenter velocity for tungsten at 50 mN load.

Figure 7 shows the dependence of hardness on indentation and Martens scales at the maximum load of 1 mN on the approach velocity of the indenter. The material of the sample is fused quartz.

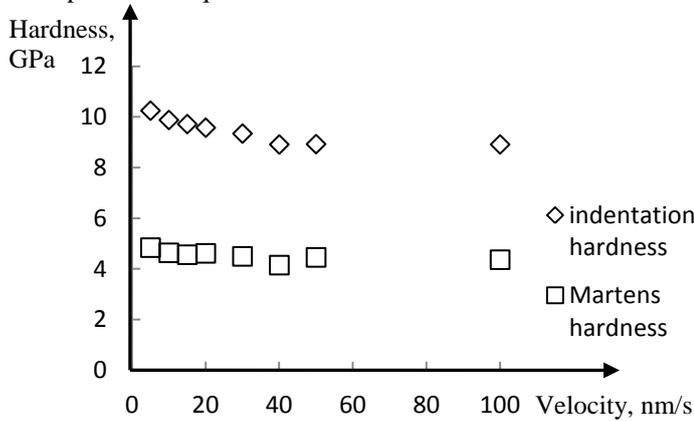


Fig. 7 Dependence of H_{IT} and HM on indenter velocity for fused quartz at 1 mN load.

Figure 8 shows the dependence of hardness on indentation and Martens scales at the maximum load of 50 mN on the approach velocity of the indenter. The material of the sample is fused quartz.

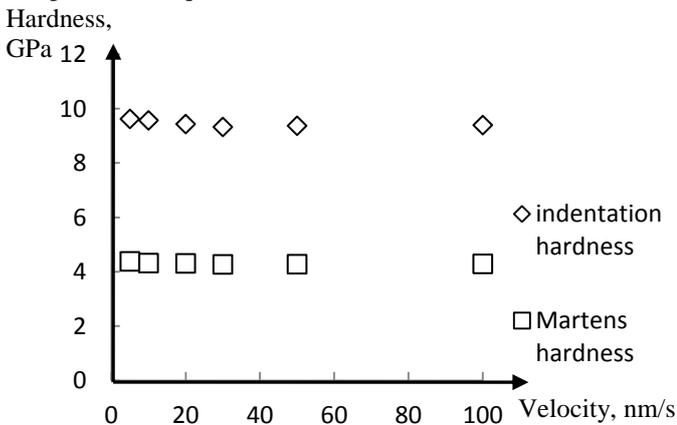


Fig. 8 Dependence of H_{IT} and HM on indenter velocity for fused quartz at 50 mN load.

Figure 9 shows the dependence of hardness on indentation and Martens scales at the maximum load of 2 mN on

the approach velocity of the indenter. The material of the sample is sapphire monocrystal.

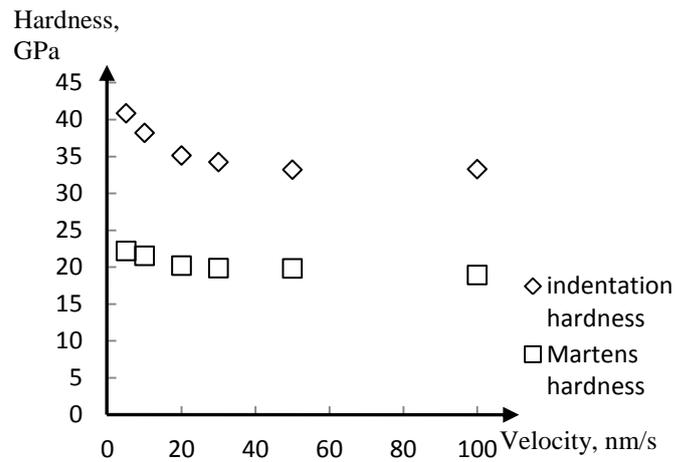


Fig. 9 Dependence of H_{IT} and HM on indenter velocity for sapphire monocrystal at 2 mN load.

Figure 10 shows the dependence of hardness on indentation and Martens scales at the maximum load of 100 mN on the approach velocity of the indenter. The material of the sample is sapphire monocrystal.

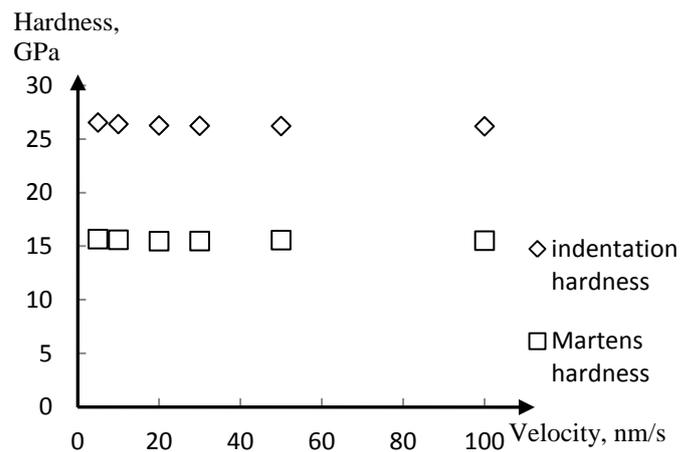


Fig. 10 Dependence of H_{IT} and HM on indenter velocity for sapphire monocrystal at 100 mN load.

Figures 11, 12 show the dependence of hardness on indentation and Martens scales at the maximum load of 0,1 mN and 0,2 mN on the approach velocity of the indenter. The material of the sample is polycarbonate.

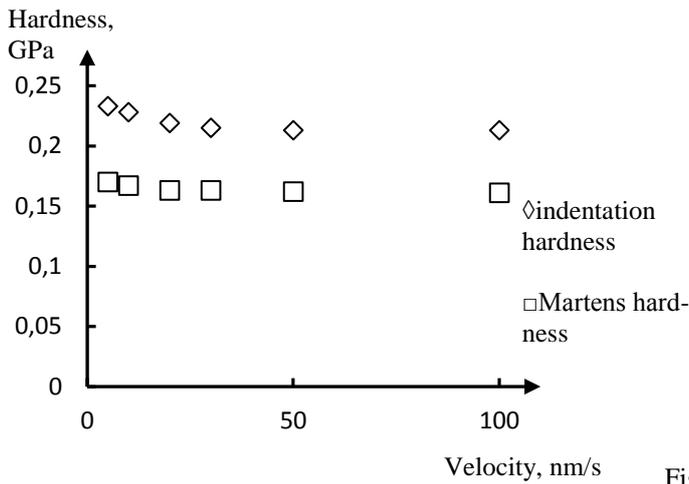


Fig. 11 Dependence of H_{IT} and HM on indenter velocity for polycarbonate at 0,1 mN load.

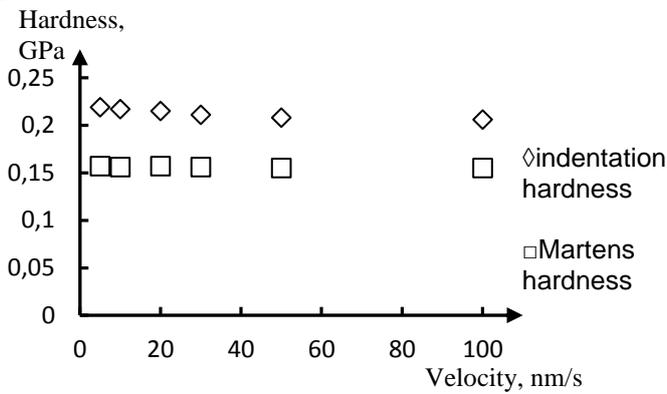


Fig. 12 Dependence of H_{IT} and HM on indenter velocity for polycarbonate at 0,2 mN load

Figures 13, 14, 15 show the dependence of hardness on indentation and Martens scales at the maximum load of 0,1 mN on the time parameters of the testing cycle. The material of the sample is polycarbonate.

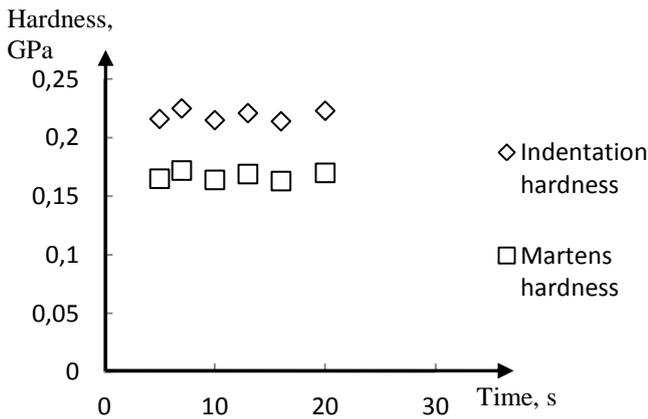


Fig. 13 Dependence of H_{IT} and HM on unloading time. Loading time and exposure time are 30 s.

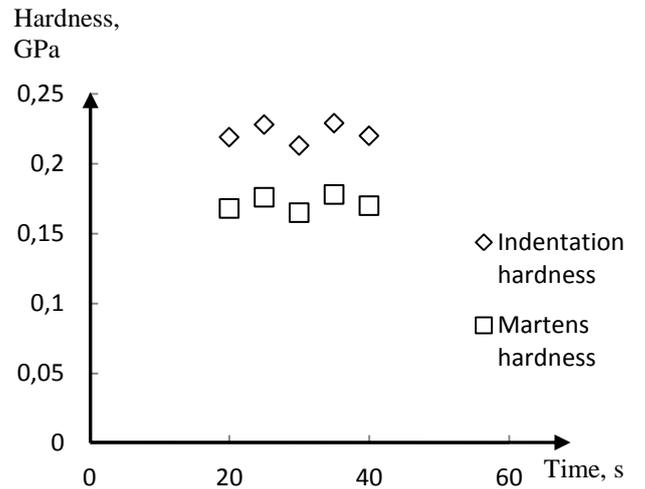


Fig. 14 Dependence of H_{IT} and HM on exposure time. Loading time is 30 s and unloading time is 10 s.

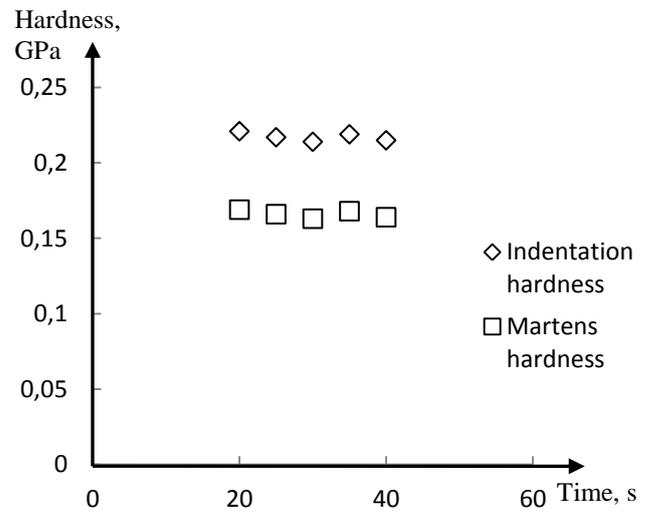


Fig. 15 Dependence of H_{IT} and HM on loading time. Exposure time is 30 s and unloading time is 10 s.

Figures 16, 17, 18 show the dependence of hardness on indentation and Martens scales at the maximum load of 0,1 mN on the time parameters of the testing cycle. The material of the sample is aluminum monocrystal.

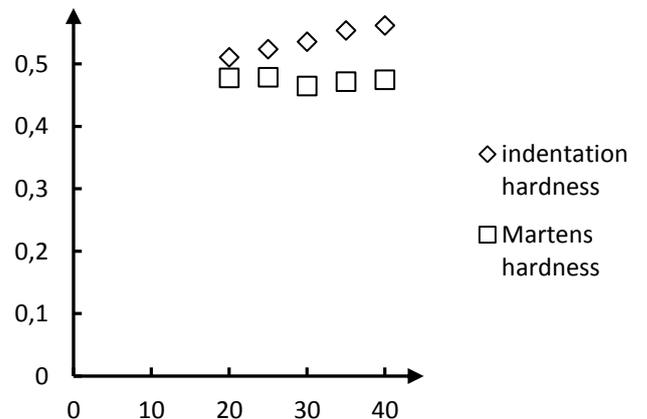


Fig. 16 Dependence of H_{IT} and HM on loading time. Exposure time is 30 s and unloading time is 10 s.

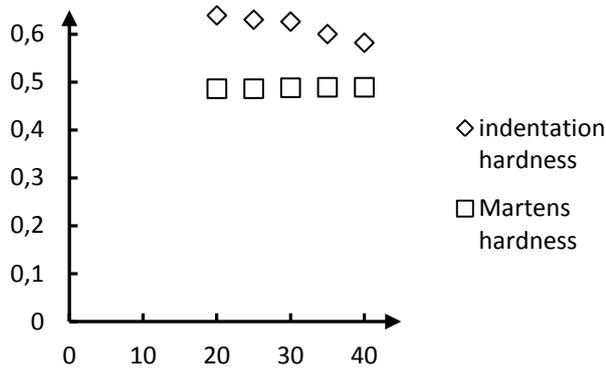


Fig. 16 Dependence of H_{IT} and HM on exposure time. Loading time is 30 s and unloading time is 10 s.

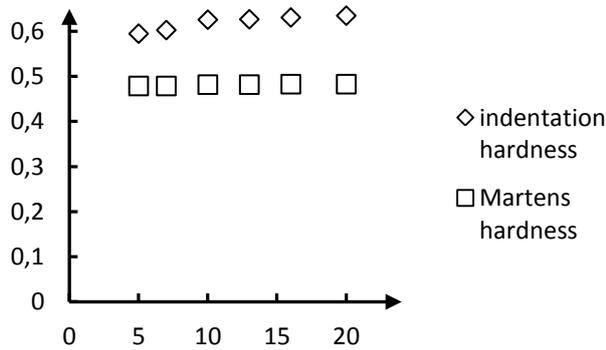


Fig. 17 Dependence of H_{IT} and HM on unloading time. Loading time and exposure time are 30 s.

For all materials, except highly plastic like aluminum, it was observed that there is a decrease in hardness dependence on the approach speed of the indenter with an increase in the maximum applied force. A decrease in hardness values with increasing approach speed of the indenter are quite noticeable when the maximum penetration depth of the tip is less than 400 nm. A strong dependence of hardness values was observed while testing materials (such as sapphire and fused quartz) for which the ratio between elastic work and plastic work approximately equals to or is above 1.

We would also like to point out an experimental fact, namely that the ratio between elastic work and plastic work in the material decreases with increasing approach speed of the indenter.

3. CONCLUSIONS

The obtained results show that hardness numbers on indentation scales depend on testing cycle parameters. Martens hardness numbers are almost independent of testing cycle parameters.

Hardness measurements in the nano-range show a change in hardness numbers depending on the approach velocity of the indenter towards the sample surface. The use of an approach velocity higher than or equal to 20 nm/s and less than 100 nm/s in the indentation test cycle can minimize the influence of this speed on hardness. At speeds close to 100 nm/s an increase in the repeatability of measurements was observed. Therefore, the optimal approach speed of the indenter is in the range between 20 and 50 nm/s. The au-

thors suggest amending ISO 14577-3 by regulating the approach velocity of the indenter in the range between 20 and 50 nm/s. Hardness numbers, apart from aluminum, are almost independent of loading, unloading and exposure time if they change from 10 s to 40 s (for loading and unloading time) and from 5 s to 20 s (for exposure time). While measuring indentation hardness on aluminum, it was observed that there is a weak dependence of hardness on the time of exposure to the load and the time of load application.

The results obtained in the present paper allow to take into consideration the influence of testing cycle parameters during hardness measurements on indentation scales in the micro- and nano-ranges using the national primary standard machine.

4. REFERENCES

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