Indentation Velocity effect on Martens Hardness Measurement

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1 Introduction

The Martens Hardness (*HM*) is an important parameter characterising the elastic-plastic properties of the to be investigated sample material which is derived from the instrumented indentation test. At present the standardisation of the instrumented indentation test in the framework of ISO/DIS 14577-1, -2, -3 is underway [1]. This standard addresses the macro-, micro- and nanorange of the indentation test. The peculiarities of the nanoindentation test when measuring samples with thin coatings (coating thickness d < 2 µm) will be considered in ISO/CD 14577-4 for which inputs came from CEN TC 184/WG 5 [2] and from the EU-project „INDICOAT“ [3].
Martens Hardness presents a number of advantages, but, as any newly defined method, requires a general analysis of influence quantities to determine the sensitivity coefficients necessary for the uncertainty evaluation.
Indentation velocity was found to be one of the main influence quantities for Rockwell and Vickers scales, therefore its effect was evaluated in a previous work that indicated an effect much higher than expected. In that work some warning was given, because the analysis was based on the results obtained on a single Hardness Standard Machine, moreover based on a simple experimental plan that did not guarantee any separation of the effects of time and velocities.
In the present work these drawbacks are overcome. The analysis is based on an experimental plan that takes into account the load increasing time, the velocity of the initial part of indentation and the velocity of the last part of indentation, that is for the Rockwell and the Vickers method the most important influence factor.
Moreover, following the resolution adopted within the recent ISO TC 164/SC3 meeting during which Martens Hardness was extended to cover from nano to macro ranges, experiments are performed on each of these ranges and with different machines, delivering in that way more significant results.

2 Investigation of the indentation velocity effect on Martens Hardness in micro and nano ranges

The micro and the nano range of the instrumented indentation test are mainly applied in order to assess the mechanical properties of thin coatings. Therefore, the investigations of the indentation velocity in the micro and nano range were carried out on samples with thin coatings.

2.1 Experimental plan

In order to assess the influence of the indentation velocity v_{ind} on the Martens Hardness *HM* when measuring samples with thin coatings the following experimental plan was set up.

If one considers that coatings and substrate can be either soft or hard, one gets the four coating/substrate combinations soft/soft, hard/soft, soft/hard and hard/hard. In the framework of these investigations the following coating/substrate combinations with the corresponding coating thicknesses as shown in Table 1 had been investigated. (The samples belonged to the EU project „INDICOAT“ [3]).

Table 1

Coating/substrate combination	**Material of coating and substrate**	**Coating thicknesses *d* of the samples, µm**
Soft/soft	Al on BK 7	310; 1050; 3400 nm
Hard/soft	Al_2O_3 on Ni	200; 800; 2000 nm
Soft/hard	Au on Ni	0; 300; 1200; 5000 nm
Hard/hard	DLC on M2 tool steel	0; 200; 1000; 2000 nm

The indentation measurements were carried out with the micro hardness measuring device Fischerscope H100 using a Vickers and a Berkovich indenter. The measurement conditions are comprised in Table 2.

Table 2
Measurement conditions for the investigation of coated samples

Test force *F*, mN	**Force application rate $F_t = \partial F/\partial t$, mN/s**			
	Au on Ni	**Al_2O_3 on Ni**	**Al on BK7**	**DLC on steel**
1	0,01	0,01	0,01	
1	0,033	0,033	0,033	0,033
1	0,1	0,1	0,1	0,1
3	0,1	0,1	0,1	0,01
10	0,01	0,01	0,01	
10	0,1	0,1	0,1	0,1
10	0,33	0,33	0,33	0,33
10	1	1	1	1
30				1
100				0,1
100				1
100				3,33
100				10

Because the real tip shape of the used indenters remarkably influences the measurement results, the area functions describing the indenter tip geometry in dependence on the indentation depth were determined with a metrological Scanning Force Microscope with laser interferometers fitted in the movement axes *x*,*y*,*z* [4]. The area functions of the indenters were used for correction of the hardness measuring values.

Moreover, the frame compliance C_f of the used micro hardness measuring device was determined according to the method of Oliver and Pharr [5] yielding a value C_f = 17,5 nm/N. The measurement results were corrected by this value of C_f.

2.2 Measurement results and data analysis

The indentation velocity v_{ind} was determined from the following relationship:

$$v_{ind} = \frac{h_{\max} F_t}{F} \qquad (1)$$

Therefore, in the investigation of coated samples the mean indentation velocity is evaluated.

The measurement results given in the following figures are mean values obtained each from 10 single measurements. By regression analysis trends between HM and vind are derived. The large scatter of the measurement values cannot be attributed only to the uncertainty of measurement, but also to other influence quantities, like the test force etc.

Fig. 1 and 2 show the results for the dependence of Martens Hardness *HM* on the indentation velocity for the investigated coating/substrate combinations using the Berkovich indenter.

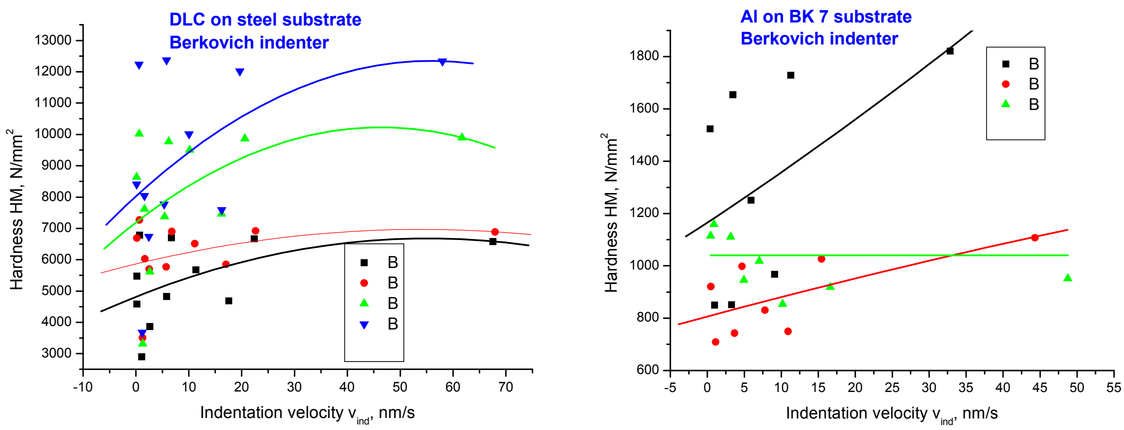

Fig. 1 Dependence of Martens Hardness *HM* on the indentation velocity v_{ind} for the coating/substrate combinations Al coating on BK 7 substrate and DLC coating on steel substrate using a Berkovich indenter

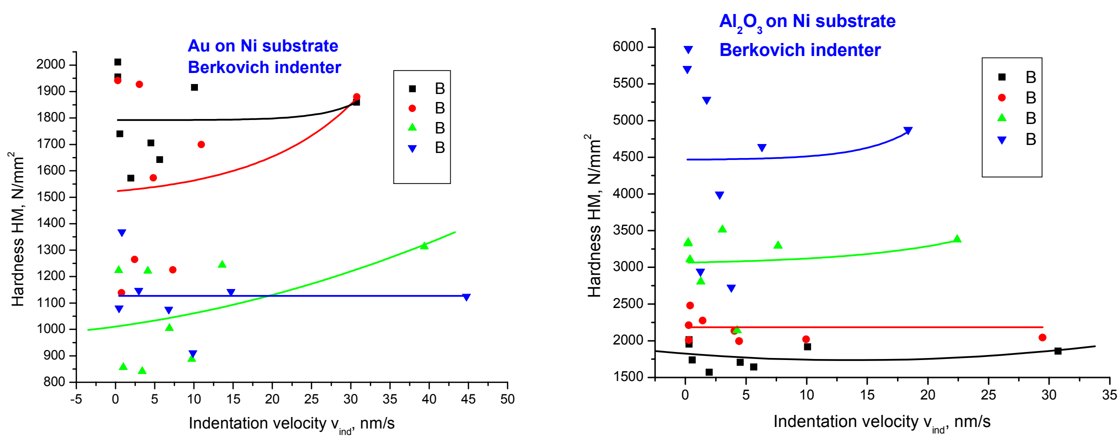

Fig. 2 Dependence of Martens Hardness *HM* on the indentation velocity v_{ind} for the coating/substrate combinations Au coating on Ni substrate and Al_2O_3 coating on Ni substrate using a Berkovich indenter

2.3 Discussion

From Fig. 1 and 2 one can see that as a general trend the hardness *HM* rises with increasing indentation velocity v_{ind}. But the effect of the indentation velocity also depends on the coating thickness. This effect is summarised as a trend in Table 3.

Table 3
Trend for of the dependence of the indentation velocity v_{ind} on the coating thickness *d*

Coating/substrate combination	Trend for relationship $v_{ind} = f(d)$
Al on BK7	-
DLC on steel	++
Al_2O_3 on Ni	+
Au on Ni	-

An important result of the EU project „INDICOAT“ [3] regarding the determination of hardness and elasticity from nanoindentation was that instead of a classification between hard and soft coating materials the classification between brittle and ductile coating materials is more relevant. This seems also to be the case when evaluating the influence of the indentation velocity. Table 3 reveals that for the ductile coating materials Al and Au the influence of v_{ind} decreases with increasing coating thickness *d*. On the other side, for the brittle coating materials Al_2O_3 and DLC v_{ind} increases with increasing coating thickness *d*. But for Al_2O_3 this effect is much smaller than for DLC.
Fig. 3 represents the relative change of Martens Hardness *ΔHM* in dependence on the coating thickness of the different coatings.

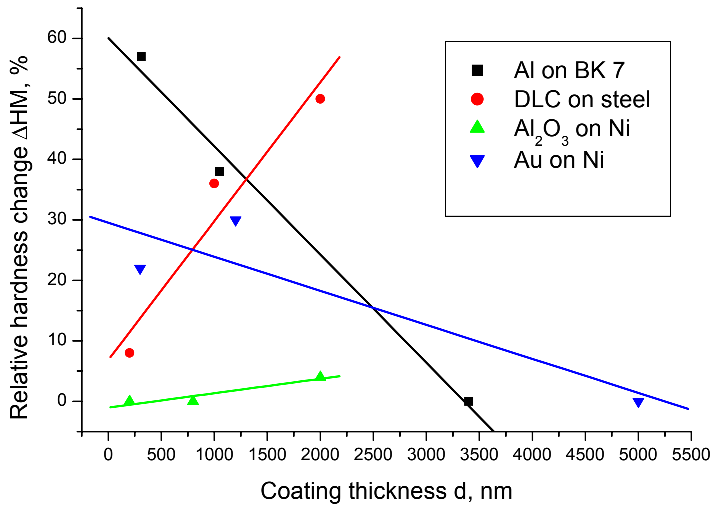

Fig. 3 Relative change of Martens Hardness *ΔHM* in dependence on the coating thickness of the different coatings

Fig. 3 shows that the largest relative changes *ΔHM* happen for ductile materials with small coating thickness and for brittle materials with large coating thickness.

Under these conditions the conclusion should be drawn that for comparable measurements of *HM* it is necessary to stipulate restricted ranges of the indentation velocity v_{ind}. Under the aspect of practicability a range of v_{ind} = (10 ... 20) nm/s is proposed.
As the measurement results for Berkovich and Vickers indenter do not show significant differences they are not discussed in detail.

3 Investigation of the indentation velocity effect on Martens Hardness in the macro range

As shown in the field of Rockwell and Vickers hardness, indentation velocity is sensed mainly in the last part of indentation. This allows to decouple loading time, that is naturally correlated with velocity in the common laws of motion. Even in this case this method, consisting in a first part of indentation produced with an initial velocity and a last part of indentation with a final velocity, the correlation coefficient between loading time and final velocity can be reduced to acceptable levels of about 0,25. This can be done using the feedback controlled Standard Hardness Machine of IMGC that allows velocity changes during indentation. It should be evident, in any case, that it is not possible to reduce correlation level between loading time and initial velocity, as, just for reasons of elementary physics, the correlation between loading time and mean velocity is very strict.

3.1 Experimental plan

Measurements have been done with a Vickers indenter over blocks of medium (about 40 HRC) and high (about 69 HRC) level of hardness, being this range that shoved the maximum sensitivity to final velocity for Rockwell and Vickers measurement. For the medium hardness level tests were made with the application of forces of 30 kgf, 60 kgf and 100 kgf, loading times of about 10 s, 20 s and 50 s and final velocities ranging from about 1 µm/s to 25 µm/s. For the high hardness level only the forces of 60 kgf and 100 kgf were tested in a velocity range between about 0,7 µm/s to 16 µm/s, while the loading time was kept at the same previous levels.

3.2 Measurement results and data analysis

The best way for presenting the results and for giving a correct information is to give the graph of the Martens Hardness obtained for the different conditions of loading time and velocity.
To save space only the plots obtained on the hardest block are presented, as for the medium block no significant effect was shown. Lines of tendency have been added, even if regression analysis shows that the visually acceptable tendency is at the limit of significance for velocity (slope of about -16 (N/mm^2)/(µm/s) with a standard deviation of 11 (N/mm^2)/(µm/s), for the load of 60 kgf and about –19 (N/mm^2)/(µm/s) with a standard deviation of 8 (N/mm^2)/(µm/s), for the load of 100 kgf) and not at all significant for the time (slope of about -3,6 (N/mm^2)/(µm/s) with a standard deviation of 4,0 (N/mm^2)/(µm/s), for the load of 60 kgf and about +3,1 (N/mm^2)/(µm/s) with a standard deviation of 2,2 (N/mm^2)/(µm/s), for the load of 100 kgf).

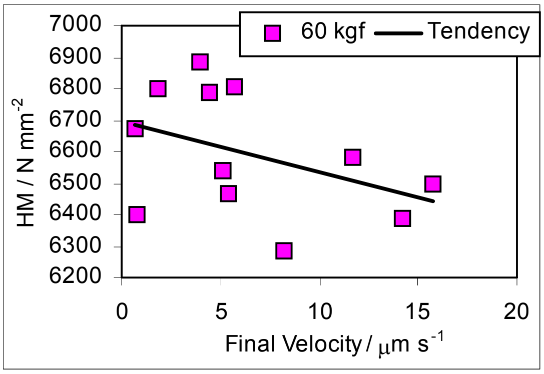

Fig. 4 Effect of final velocity on Martens Hardness. Load 60 kgf

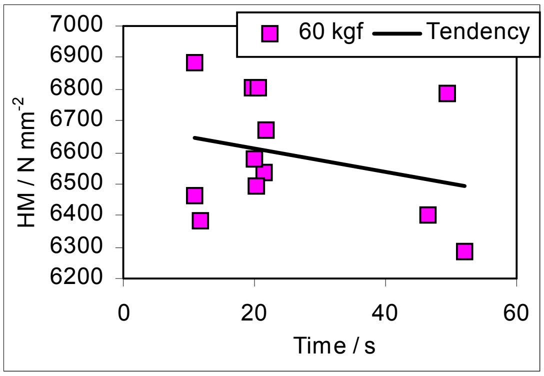

Fig. 5 Effect of loading time on Martens Hardness. Load 60 kgf

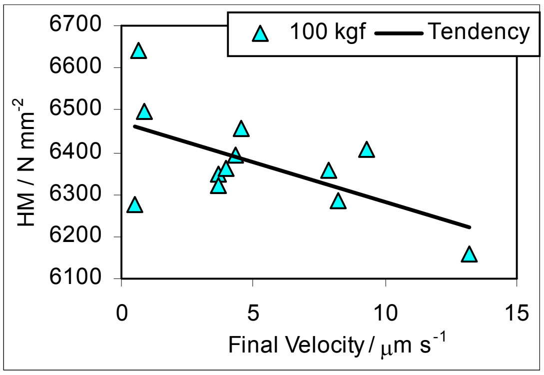

Fig. 6 Effect of final velocity on Martens Hardness. Load 100 kgf

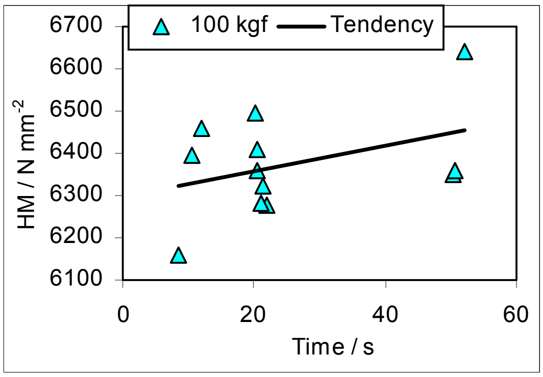

Fig. 7 Effect of loading time on Martens Hardness. Load 100 kgf

3.3 Discussion

Tests made show that in case of high level of hardness a significant effect of the final velocity of indentation is present, having the same slope direction as that evidenced by Marriner [6] for Rockwell C and for Vickers. The velocity effect on Martens Hardness is of about 0,6% $\mu m^{-1}s$ for both forces of 60 kgf and 100 kgf, that is about 6% for a velocity variation from 3 to 12 µm/s, significantly larger than the effect of the same velocity change on Vickers measurement. One shall say that the characteristic curve path, as observed for Rockwell and Vickers measurement, is not evidenced, but this could be due to the large dispersion of HM results.
The effect of loading time seems to be not significant and the same happens to both final velocity and loading time effects for medium or low hardness levels.

4 Summary

The indentation test in the micro- and nanorange is analysed for practical indentation velocities in the range between (2 ... 60 nm/s). This is one decisive peculiarity when measuring coated samples with a coating thickness d < 2 µm as compared with measurements in the macro range where typical indentation velocities are of the order > 1 µm/s.
The influence of the indentation velocity v_{ind} for coated samples yields the largest relative changes of Martens Hardness ΔHM for ductile materials with small coating thickness and for brittle materials with large coating thickness. In order to assure comparable measurement results of *HM* in the draft standard ISO/CD 14577-4 should be stipulated a range of indentation velocity v_{ind} = (10 ... 20) nm/s.

The investigation of the effect of the indentation velocity on coated samples should be continued in the direction to reveal further influence quantities, like the indentation velocity in different parts of the indentation process.
For the macro range a sensitivity to final velocity of indentation has been confirmed, but only for high hardness level.

5 References

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