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Improving the accuracy of low-load Vickers microhardness testing of hard thin films

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Abstract

Experiments that involved the testing of different steel samples over a wide range of loads were conducted in order to investigate the influence of micro-loads on accuracy of Vickers microindentation hardness testing. We found that for the same material the Vickers microhardness decreases with the decreasing of test load. The measuring error of Vickers microhardness on a steel block sample with known hardness was -25 % when we used 10 gf load force. This is due to the fact that diagonal length values are in the range of micrometers and the precision of reading using optical microscopy is ± 0.5 µm in length for most operators In order to improve accuracy of the reading of indents diagonals length we used two additional methods: scanning electron microscopy and graphical image processing. By this approach the measuring error of $HV_{0.01}$ was reduced to an error of 3.78 %.
On samples coated with nanostructured (TiAlSi)N hard thin films developed by DC unbalanced magnetron sputtering, with thickness of 2...3 µm, we found values around 2500 $HV_{0.01}$ using this improved Vickers microhardness testing methodology.

Keywords: Vickers hardness; low-load microindentation; thin films, hard coatings; (TiAlSi)N coatings.

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1. Introduction

One of the method used to characterize hard coatings mechanical properties is Vickers microindentation hardness testing. The square-based pyramidal shape of indenter with an angle of 136° between two opposite faces assures a constant value for HV at all test loads, over a wide test load range. The Vickers test has the great advantage of one hardness scale being used to test all materials.

In Vickers hardness test the depth of indentation have to be limited to a maximum of 10 % from average diagonal length of indent. This is not a problem for bulk materials, but in the case of thin films it limits the possible load to be applied to very small ones. Usually micro-load range (10-1000) gf is used.

Hard coatings with thickness less than 5 μm have to be tested with 10 gf or 25 gf load. This raised the problem of indentation size effect (ISE) that refers to the fact that at very low loads the test results on a material are not constant over the range of test loads [1-5].

For Vickers tests with an applied force of 100 to 1000 gf, measured hardness values are usually equivalent within statistical precision. The literature shows four trends for load force - hardness Vickers data at low loads [2]:

1. HV decreases as load force decreases;
2. HV increases as load force decreases;
3. HV is essentially constant as load force varies;
4. HV increases, then decreases with decreasing of load force.

Many factors can influence on precision in microindentation hardness testing: instrument factors (accuracy of applied load, inertia effect, speed of loading, lateral moving of the indenter or specimen, intentation time, indenter shape deviations, damage to the indenter, inadequate spacing between indents or form edge, angle of indentation), measurement factors (calibration of the measurement unit, numerical aperture of the objective, magnification, inadequate image quality, uniformity of illumination, distorsion in optics, operator's visual acuity, focusing of the image) and material factors (heterogenity of the specimen, strength of crystallographic texture, if present, quality of specimen preparation, low reflectivity, creep during indentation, fracture during indentation).

Assuming the specimen is properly prepared, Len Samuels stated that ISE problems were not due to the materials, but to difficulties in visual perception of indent tips as indents became smaller [6]. The greatest source of error is in measuring the indent. In general, indents can be measured to a precision of ± 0.5 μm in length for most operators. This leads to substantial hardness variation as the indent size decreases below 20 μm.

The aim of present research is to improve the accuracy of Vickers microhardeness testing using additional methods to measure the values of indent diagonals in order to avoid the major source of errors that is human operators visual perception problem due to light microscope limitations.

2. Methodology

In order to find the influence of load force on Vickers microhardness result we used two steel block tests with already known hardness (718 HV and 466 HV). We applied a wide range of loads (1000, 500, 300, 200, 100, 50, 25, 10 gf) in order to investigate the influence of micro-loads on accuracy of Vickers microindentation hardness testing. On each sample, at each load force, two operators made 5 tests, the results presented in present paper being the average of those measurements. Indentation of samples was made using a CV-AAT 400 Vickers microhardness tester. Firstly the indents were measured using optical microscope (40X magnification) of CV-AAT 400 tester. In the next stage, the indents were visualized and photographed in scanning electron microscope (SEM) JEOL JSM-5200. The images were then processed using the open source Graphical Image Manipulation Program (GIMP).

The same practical approach was followed for nanostructured (TiAlSi)N coatings developed by DC unbalanced magnetron sputtering. After the deposition of 2...3 μm thick coatings on (100)Si and HSS substrates the samples were tested in Vickers tester using 10 gf load force. The reading of indents diagonal length was performed optical (in Vickers tester) and using SEM and GIMP.

3. Results and discussion

Table 1 and figure 1 present the results of performed tests on two samples with known Vickers microhardness: 718 HV and 466 HV. The sample 718 HV has an admissible error of ± 2.0 % (for a load force of 1000 gf) and sample 466 HV an admissible error of ± 3.5 % (for a load force of 200 gf).

Table 1. Vickers microhardness for the samples 718 HV and 466 HV

Load force F [gf]	Sample 718 HV Vickers microhardness HV [kgf/mm^2]	Sample 718 HV HV error [%]	Sample 466 HV Vickers microhardness HV [kgf/mm^2]	Sample 466 HV HV error [%]
1000	727.47	1.32	470.29	0.92
500	733.83	2.20	465.22	-0.17
300	739.00	2.92	457.51	-1.82
200	728.17	1.42	461.00	-1.07
100	730.58	1.75	450.63	-3.30
50	698.65	-2.69	449.50	-3.54
25	630.43	-12.20	412.70	-11.44
10	536.69	-25.25	341.81	-26.65

The results indicate that at 200, 300, 500, 1000 gf force loads the error is negligible and inside the admissible error for sample steel blocks. Using 50 and 100 gf loads results in slightly increased error for sample 466 HV. But when low loads of 25 and 10 gf is used the tests results have high errors. Using 25 gf load the measuring error is 12.20 % for 718 HV sample and 11.44 % for 466 HV sample. Moreover at 10 gf the error is 25.25 % for 718 HV sample and 26.65 % for 466 HV sample.

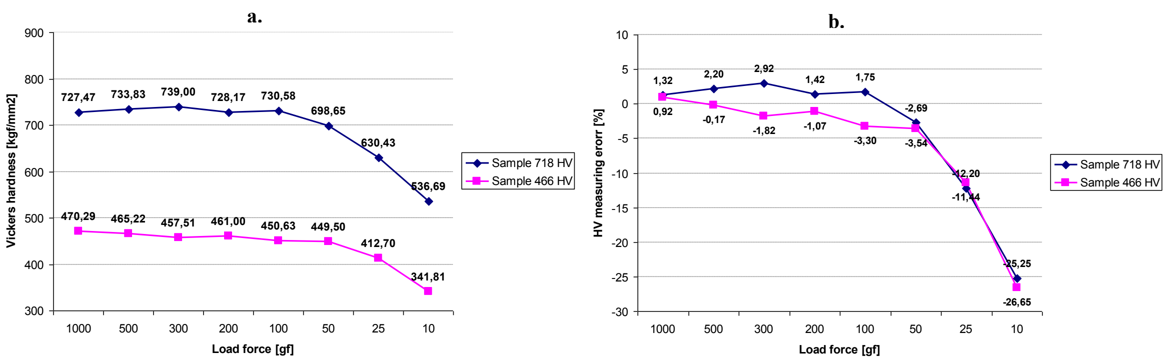

Fig. 1. Influence of load force on Vickers microhardness test results (a – Vickers microhardness, b – measuring error)

Our results confirms the trend mentioned in literature [2] that states that with the decreasing of load force applied on the same material there is a decrease in measured Vickers microhardness.

The diagonals of indents are very small at 10 gf load force. In the case of 718 HV sample we measured an average diagonal of 5.9 μm. In this case, the accepted value of ± 0.5 μm for measuring error due to operators difficulty to read the indent, leads to a ± 25 % error in Vickers microhardness result.

This raises a problem for our researches on hard nanostructured (TiAlSi)N coatings. Coatings are deposited using DC unbalanced magnetron sputtering and have thickness in 2...3 μm range. The expected hardness of these coatings is around 2000 HV. For this value of hardness the average indent diagonal is ~ 3 μm. In order to measure the hardness of these coatings the load force to be applied is limited to 10 gf. We can not accept the high error which can occur in the determination of HV using the optical microscope of Vickers microhardness tester for indents diagonals measurement.

To improve the accuracy of indents diagonals measurements our solution was to use SEM images of indents and process them using a graphical image manipulation program (GIMP). Figure 2 presents the SEM image processed in GIMP of an indent on 718 HV sample obtained in Vickers tester at 10 gf load force. Table 2 presents the comparative results of Vickers microhardness of 718 HV sample, performed at 10 gf load force, using optical and SEM + GIMP methods.

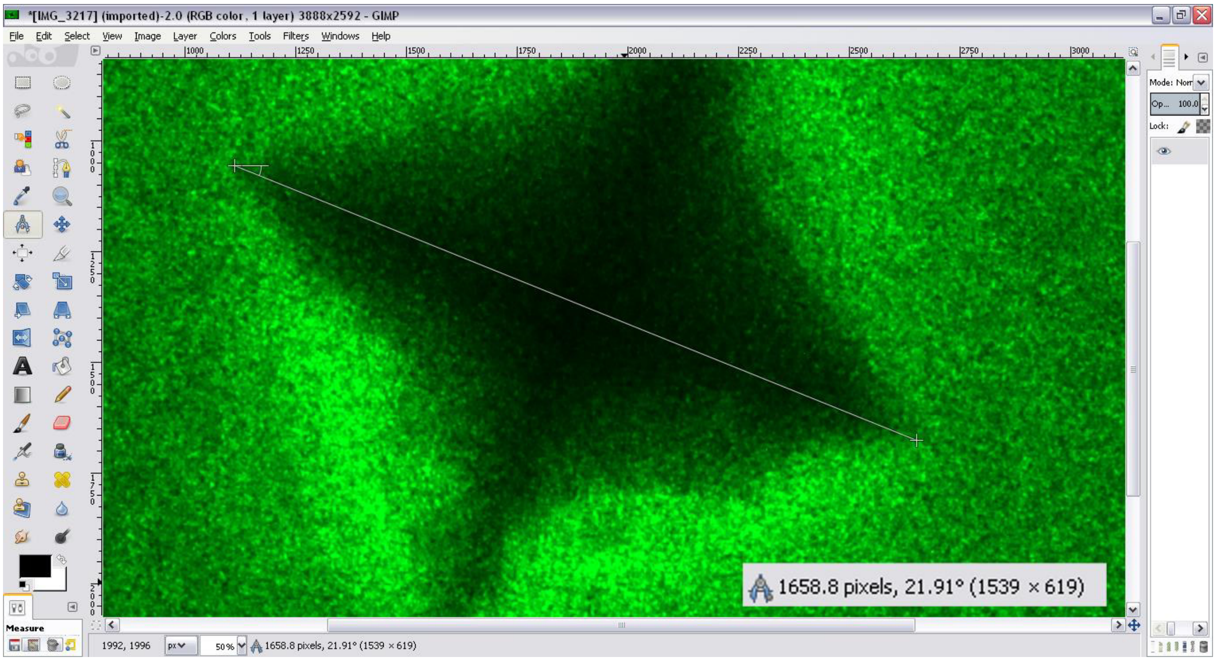

Fig. 2. Indent's diagonal length reading using SEM image processed in GIMP (718 HV sample, load force 10 gf) (screen dump)

Table 2. Vickers microhardness for the sample 718 HV (10 gf load force, indents diagonals measurement: optical, SEM + GIMP)

	Indent's diagonal d_1 [μm]	Indent's diagonal d_2 [μm]	Vickers microhardness HV [kgf/mm^2]	HV [kgf/mm^2]	HV error [%]	Indent's diagonal d_1 [μm]	Indent's diagonal d_2 [μm]	Vickers microhardness HV [kgf/mm^2]	HV [kgf/mm^2]	HV error [%]
	optical					SEM + GIMP				
1	5.93	6.07	515.11			5.02	4.91	752.22		
2	5.78	5.97	537.26			5.08	4.95	736.13		
3	5.95	5.63	553.15	**536.69**	**-25.25**	4.89	5.18	732.12	**745.15**	**3.78**
4	5.95	6.05	515.11			4.96	5.18	721.30		
5	5.78	5.70	562.83			4.85	4.88	783.95		

The results show an important enhancement in the accuracy of Vickers microhardness measurement by using the reading of diagonals length using scanning electron microscopy combined with graphical image processing. The error of HV measurement is 3.78%. Comparing this error with the unacceptable high error if the reading is performed only using optical microscope of Vickers tester, we can conclude that using SEM and GIMP is a practical way to improve the accuracy of Vickers microhardness testing at low loads.

This practical approach that lead to an improved accuracy of microhardness testing was followed by us in the case of nanostructured (TiAlSi)N coatings developed in our DC unbalanced magnetron sputtering unit. The reactive sputtering process was performed using planar rectangular target material of sintered TiAlSi (40 at.% Ti, 40 at.% Al 20 at.% Si) target. Prior to deposition a base pressure of $4.6 \cdot 10^{-6}$ torr was established by operating a 500 l/s turbomolecular pump. The substrates were positioned in a static molybdenum sheet substrate holder, which allowed application of $U_s = -20$ V bias voltage. The Mo sheet was resistively heated to a controlled substrate temperature of $T_s = 500$ ºC. The reactive sputtering process was performed in a mixture of Ar and N_2 atmosphere at $2.9 \cdot 10^{-3}$ torr. During of the reactive sputtering process the nitrogen flow was $q_{N2} = 4.0$ sccm and the argon flow was q_{Ar}=6.0 sccm. A constant sputtering power of 400 W was maintained during of deposition.

After the deposition the samples were tested for Vickers microhardness using low load indentation (10 gf) and optical reading and SEM combined with GIMP reading of indents diagonals length. Figure 3 shows the SEM image processed in GIMP for M22 sample.

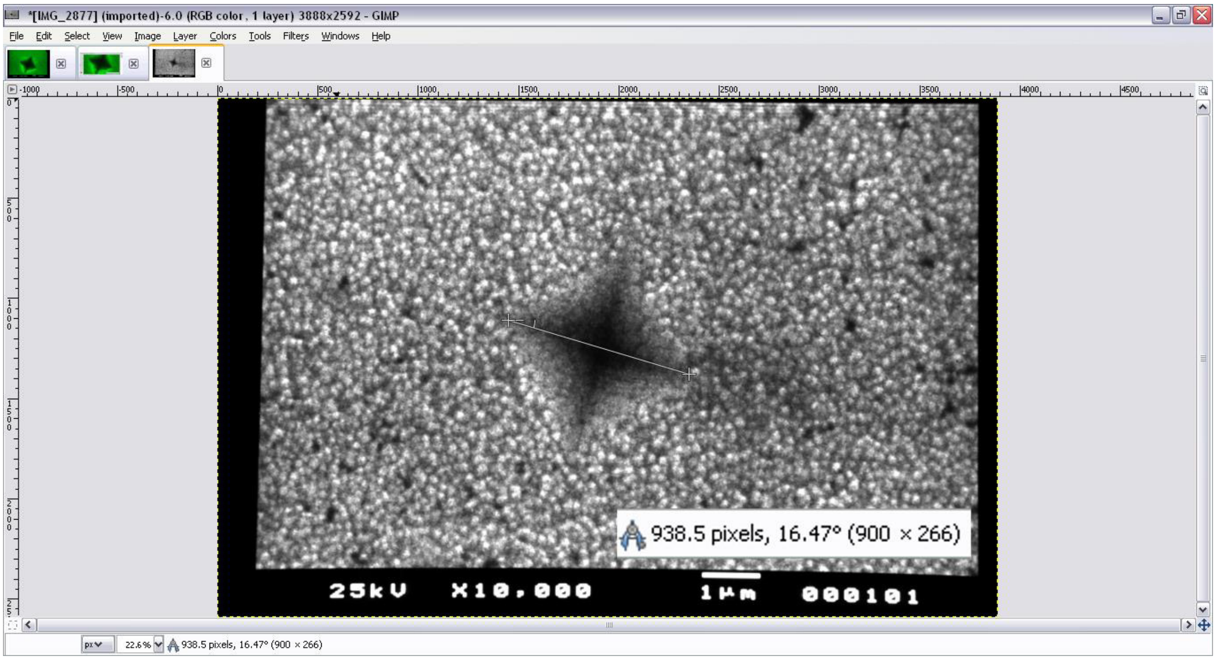

Fig. 3. Indent's diagonal length reading using SEM image processed in GIMP (M22 sample, load force 10 gf) (screen dump)

On M22 sample we measured a 2740 $HV_{0.01}$ using the optical microscope of Vickers tester and 2490 $HV_{0.01}$ using SEM and GIMP. This difference is easily explainable considering the admissible indentation diagonal read error in the optical measurement method, which is ± 0.5 µm, as the average diagonal of the indentations measured with the optical method is 2.6 µm, while measured from the SEM images is 2.73 µm.

4. Conclusions

Present research confirms one of the trends mentioned in specialty literature regarding the relation between load force - Vickers hardness values. Our results show that with the decreasing of load force applied on the same material there is a significant decrease in Vickers hardness results. Moreover, at loads of 10 gf and 25 gf the error is unacceptable high. At 10 gf the error of HV measurement is -25.25%. By reading and processing of indents obtained in Vickers tester (10 gf load force) using scanning electron microscopy and graphical image manipulation program we improve the accuracy of indent diagonal length reading and the error of HV measurement was only 3.78 %.

In the case of hard coatings with low thickness (2...3 μm) where the Vickers microhardness measurements have to be performed with 10 gf load force, we applied this practical approach that very much enhanced the accuracy of testing. On nanostructured (TiAlSi)N coating developed by DC unbalanced magnetron sputtering we measured a 2490 $HV_{0.01}$ Vickers microhardness using SEM combined with GIMP method.

Considering the possible error in diagonal length measurement via optical method, care must be taken when interpreting measurements from low load force Vickers microhardness testing.

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