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HARDNESS MEASUREMENT OF COATING THIN FILM BY INDENTATION PROFILE OBSERVATION USING SCANNING PROBE MICROSCOPY

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Abstract – According to the principle of a conventional Vickers (Berkovich) hardness measurement, hardness is calculated from diagonal lengths or side lengths of indentation. The diagonal length, the conjunction line length of pyramidal indentation, and the distance from the vertex to the base of trigonal pyramidal indentation are measured accurately by observing a vertical cross section. Scanning probe microscopy (SPM) is a good method for observing the vertical and horizontal cross sections of the indentation. However, such parameters in the cross section profile must be defined as the principle of the method to obtain a reliable nano-hardness value..

Keywords: nano-indentation, scanning probe microscopy, nano-haradness

1. INTRODUCTION

The nano-indentation test has been developed as a substitute for the micro-Vickers hardness test, because indentation profile is difficult to measure by means of optical microscopy. In case of the micro-Vickers hardness test, the surface area of indentation is calculated from two diagonals of a regular tetragon by observing a surface profile of the indentation under the assumption of an ideal pyramid applied at the maximum force. In case of Martens hardness test specified in ISO 14577, the surface area of the indentation is calculated from the indentation depth at the maximum force under the assumption of an ideal pyramid with a truncation. To obtain the true hardness value, evaluating the true contact surface area is very important. However, from the viewpoint of industrial utility, agreement must be obtained between micro-Vickers hardness and Martens (nano-Vickers) hardness. We propose a nano-Vickers hardness test employing indentation profile observation using scanning probe microscopy (SPM).

A reliable hardness value cannot be obtained without evaluating the shape of the indenter tip with a truncation and observing the profile of the indentation in the nano-indentation test [1][2]. We evaluated the shape of the indenter tip and the vertical profile of the indentation by SPM. Vertical section profiles can be obtained only by SPM observation and give important information about indentation profile. However, few studies have evaluated the vertical profile of the indentation. Horizontal section profile of the indenter tip and the indentation at an arbitrary distance from the top of the indenter and the bottom of the indentation were obtained by a particle analysis method, and give important information about the indenter tip profile and the indentation bottom profile. In this study, we discuss the validity of the SPM observation method in the nano-Vickers hardness test.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

Indentation testing was performed using a Fischer Scope H100. A pyramidal (Vickers) indenter and a trigonal pyramidal (Berkovich) indenter were used. Maximum testing forces were 50 mN and 100 mN, respectively. The tips of indenters and indentation profiles were observed using a scanning probe microscope (Nanopics 1000, Seiko Instruments). Scanning was performed in non-contact mode and the scanning speed was 130 sec per frame. The measurement accuracy for length and depth were checked by reference to silicon gratings (TGT01 and TGG01, MikroMasch). A horizontal cross-sectional area was measured using a particle analysis program included in the system software of SPM.

The specimen used was PVD TiN thin film coated on SUS304 steel. The TiN film had a thickness of approximately 2000 nm.

3. RESULTS AND DISCUSSION

3.1 Evaluation of indenter shape

Figure 1 shows typical SPM images of the Berkovich indenter and the Vickers indenter used in this work. Generally, the truncation derived from the vertical cross-sectional profile is defined as the height between the point of intersection between tangent lines and the top of the indenter tip. The truncations of Berkovich indenter and Vickers indenter are approximately 105 nm and 58 nm, respectively.

Figure 2 shows horizontal cross-sectional profiles of the Berkovich indenter at the indicated height from the tip. The profile at 10 nm from the tip forms a triangle, and the vertexes of the triangle are slightly rounded. The area of each triangle is calculated automatically by software based

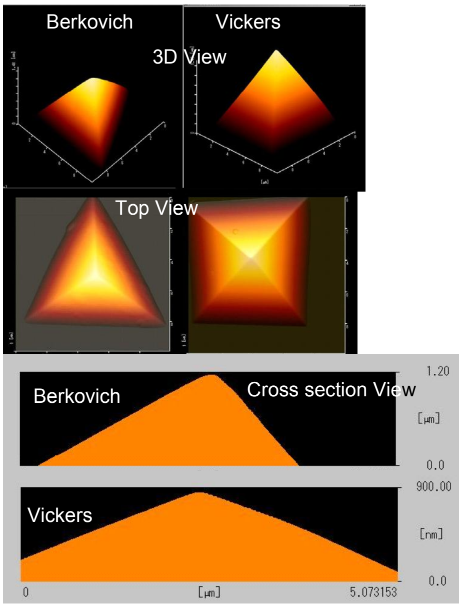

Fig. 1 Typical SPM images of a Berkovich indenter and a Vickers indenter

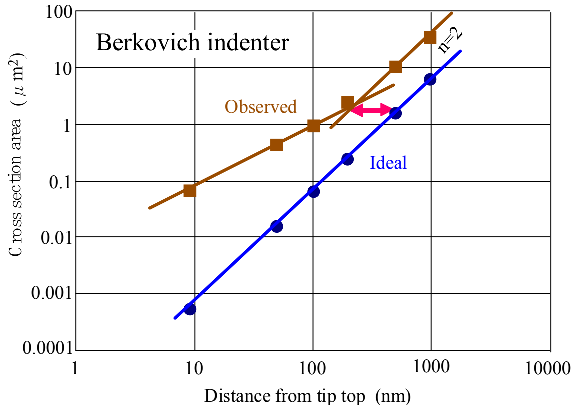

Fig. 3 Relationship between distance from the tip top and horizontal section area

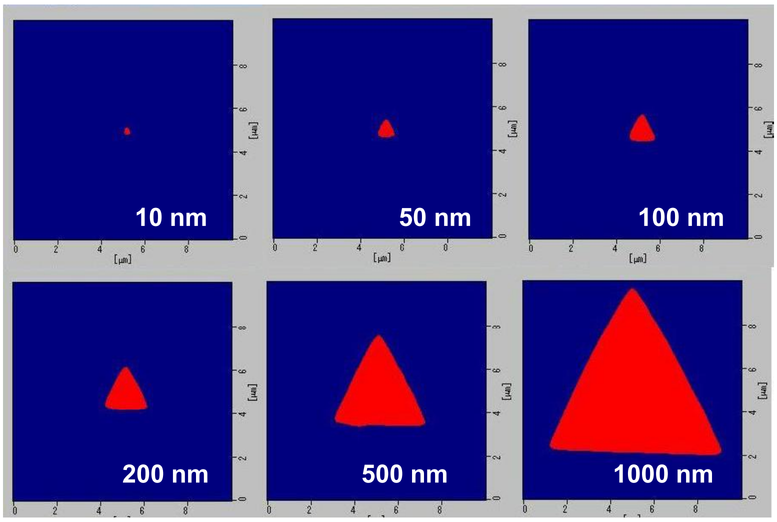

Fig. 2 Horizontal cross section profiles of the Berkovich indenter at the indicated heights from the tip top

on a particle analysis method.

Figure 3 shows the relationship between the distance from the tip and the horizontal section area. In case of an ideal indenter that assumes the shape of a perfect trigonal pyramid, a straightline with a slope 2 (n=2) is obtained. In the case of the real indenter that has the rounded tip, two straight lines with the slope 2 (n=2) and 1 (n=1) are obtained. The straight line with slope n=1 represents a relation between the distance from the tip and the horizontal section area for a spherical indenter. The difference between the ideal relation and the observed relation at the intersection of two straight lines in Fig. 3 is defined as the truncation height of the real indenter. The truncation height derived from Fig. 3 is approximately 300 nm; this is very large compared with values reported in other studies [3].

Figure 4 shows horizontal cross-sectional profiles of the Vickers indenter at the indicated heights from the tip top. The profiles are not square, since the indenter does not stand vertically. The profiles at a height of 10 nm and a distance of 50 nm from the tip form rectangles, due to the conjunction of the tip. The area of each horizontal cross section is calculated automatically using the software based on a particle analysis method.

Figure 5 shows the relationship between distance from the tip and the horizontal section area. In the case of an ideal indenter that assumes the form of a perfect pyramid, a

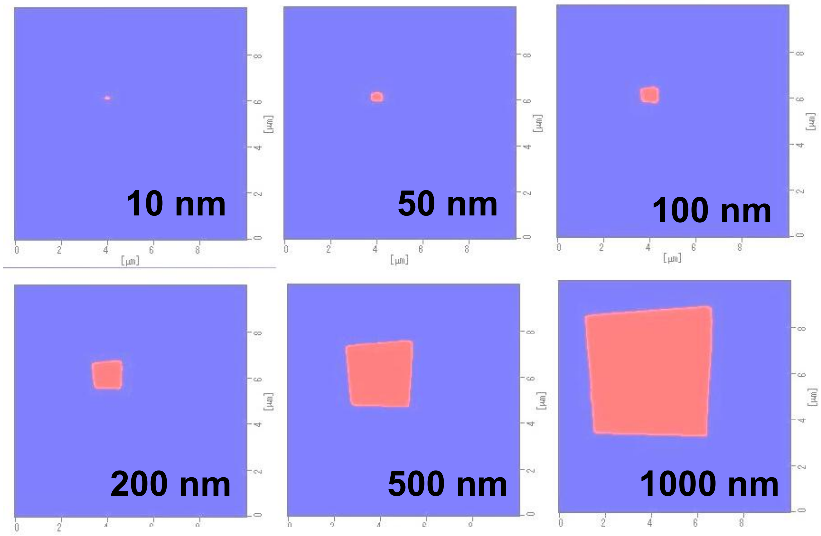

Fig. 4 Horizontal cross section profiles of the Vickers indenter at the indicated heights from the tip top

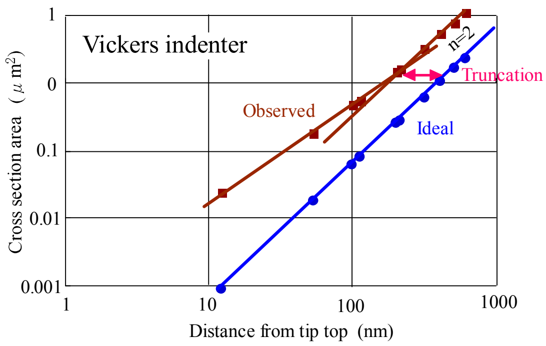

Fig. 5 Relationship between distance from the tip and the horizontal section area

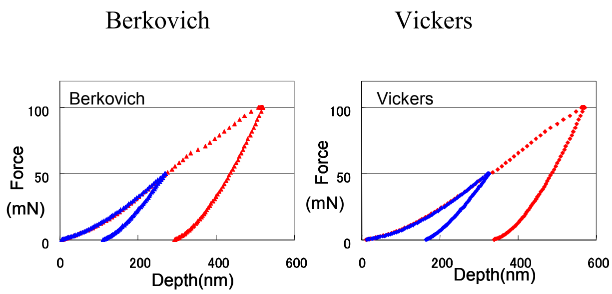

Fig.6 Typical force/indentation depth curves for TiN film coated on SUS 304 stainless steel by use of the Berkovich indenter and the Vickers indenter

straight line with the slope 2 (n=2) is obtained. In the case of a real indenter that has a rounded tip, two straight lines having slopes 2 and 1.5 appear. Consequently, the tip of this Vickers indenter is not as rounded as that of the Berkovich indenter. The truncation height derived from Fig. 5 is approximately 200 nm, much larger than that of Fig. 1.

3.2 Indentation test

Figure 6 shows typical force/indentation depth curves for TiN film coated on SUS304 stainless steel by using the Berkovich indenter and the Vickers indenter. In the case of using the Berkovich indenter at a maximum force of 100 mN, sinking is observed over an indentation depth of approximately 320 nm under a force of 70 mN. We select as high a testing force as possible to find the indentation profile easily, because we need to move the test piece on the SPM apparatus after indentation testing. The indentation test is performed under a maximum force of 50 mN. The average value of 14 measurements of maximum indentation depth and the average off-load depth are approximately 266 nm and 108 nm, respectively.

In case of using the Vickers indenter at a maximum force of 100 mN, sinking is observed over an indentation depth of approximately 450 nm and a force of 80 mN. The indentation test is performed under a maximum force of 50 mN. The average value of 11 measurements for maximum indentation depth and the average off-load depth are approximately 320 nm and 160 nm, respectively.

Comparing the Berkovich indenter and the Vickers indenter, the indentation depth of the Vickers indenter is larger than that of the Berkovich indenter, because of the truncation height of indenter. The hardness obtained by use of the correction value of the indenter truncation is much lower than the known value reported [3][4]

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3.3 Observation of indentation and evaluation of hardness

Basically, Berkovich hardness is calculated from side lengths of a triangle by measuring a surface profile of the

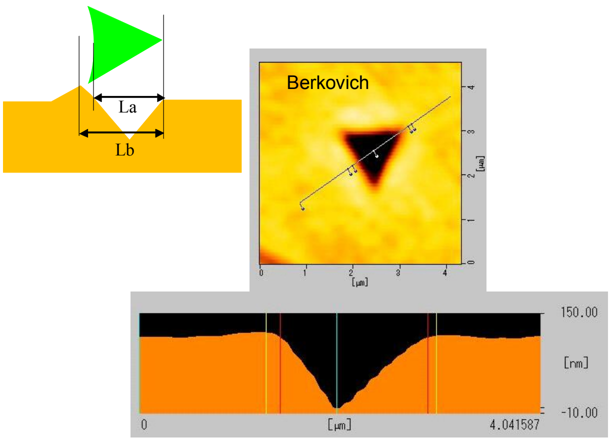

Fig. 7 Typical top view and cross section view of a indentation of the Berkovich indenter

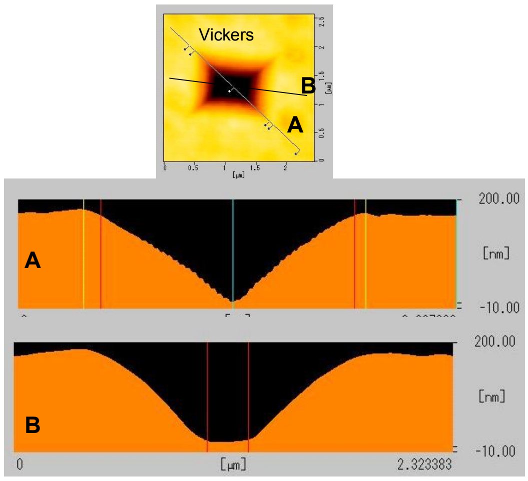

Fig. 8 Typical top view and cross section view of a indentation of the Vickers indenter

Table 1 Berkovich hardness calculated from vertical cross section profile of indentation

	Depth of indentation				Perpendicular length from the vertex to the base									
	h (nm)				La (nm)				Hardness	Lb (nm)				Hardness
No.	1	2	3	Average	1	2	3	Average		1	2	3	Average	
1	132	131	131	131	1533	1368	1778	1560	3289	1859	1647	1889	1799	2473
2	120	114	126	120	1891	1576	2018	1828	2394	2155	1814	2386	2118	1783
3	126	123	124	124	1741	1543	2063	1782	2519	2028	1831	2315	2058	1889
4	129	111	110	116	1681	1431	1832	1648	2945	1736	1671	1994	1800	2468
5	128	133	123	128	1582	1383	1620	1528	3426	1728	1730	1879	1779	2528
6	131	111	131	124	1420	1534	1841	1598	3133	1761	1614	1995	1790	2496
7	129	123	117	123	1587	1547	1823	1652	2930	1879	1565	2185	1876	2272
8	69	107	55	77	1456	1501	1746	1568	3255	1703	1656	1841	1733	2664
9	122	140	129	130	1448	1463	1620	1510	3508	1704	1728	1815	1749	2616
10	128	127	120	125	1440	1345	1713	1499	3560	1716	1647	1825	1729	2675
11	135	128	119	127	1470	1393	1710	1524	3443	1657	1595	1851	1701	2766
12	110	125	107	114	1366	1237	1472	1358	4336	1593	1442	1713	1583	3194
Average	122	123	116	120	1551	1443	1770	1588	3172	1793	1662	1974	1810	2443

Table 2 Vickers hardness calculated from vertical cross section profile of indentation

	Depth of indentation h (nm)			Diagonal length (nm)			Hardness
No.	1	2	Average	1	2	Average	
1	177	154	165	2094	2047	2071	2206
2	167	166	167	1974	1978	1976	2421
3	165	171	168	1951	2027	1989	2389
4	155	167	161	2003	1949	1976	2422
5	171	166	169	2046	2049	2048	2256
6	162	166	164	2061	2054	2058	2234
7	163	167	165	2051	2044	2048	2255
8	160	168	164	1956	1922	1939	2516
9	170	169	169	1945	2084	2015	2330
Average	166	166	166	2009	2017	2013	2337

indentation. Detecting an edge position of the triangle from the top view, as shown in Fig. 7, is difficult. However, detecting an edge position from a vertical cross section view, as shown in Fig. 7, is easy. Generally, a side length of a triangle is obtained by measuring a perpendicular length from the vertex on the base. As the periphery part of indentation rise, known as piling-up, the perpendicular length is a distance measured between the vertex edge and the point equal to surface level. This length is defined as La. The distance between the vertex edge and the top point of periphery part is defined as Lb. Some Berkovich hardness values calculated from La and Lb are shown in Table 1. Essentially, hardness should be calculated from La. Hardness value obtained from La is larger than the known hardness value of TiN, which is about 2000. The average hardness value from Lb is approximately 2400, which is close to the known hardness value.

Figure 8 shows the top view and the cross section profiles of Vickers indentation. In the profile of vertical cross section along A direction line, the edge position of the diagonal, is determined more easily than in the case of the top view profile.

Some Vickers hardness values calculated from the diagonal are shown in Table 2. The average hardness value is about 2300, which is as large as the Berkovich hardness shown above.

A conjunction of the tip is observed in the direction B line of Fig. 8. The conjunction line length is approximately 150 nm. As shown in Fig. 8, Vickers indentation assumes a rectangular shape due to the conjunction line of indenter tip; the hardness should be calculated from the two side lengths. Therefore, the two side lengths should be measured from the lengthwise and crosswise section images. When the positions of sides are decided, piling-up should be considered. Essentially, the side length is a distance measured between the edge and the point equal to surface level. However, more discussions of this aspect are required.

4. CONCLUSION

(1) The relation between height and the horizontal section area from the top of a Berkovich indenter was obtained from the particle analysis result of an SPM image. The relation can be represented by two straight lines. In a part near the top, a straight line of slope 1 corresponds to a spherical indenter, and a straight line of slope 2 corresponds to a pyramidal indenter from the top in the part of 200 nm or more.

(2) In the above-mentioned relation, the truncation height in the top can be estimated as the difference between the observed straight line and the ideal straight line with perfect pyramidal shape. The truncation height estimated from this result becomes about 300 nm, which is large compared with previously reported values.

(3) The method of estimating truncation for Berkovich indenter could not be applied because there is a conjunction line in the top of Vickers indenter.

(4) The relation with two straight lines from the relation between height and the horizontal section area from the top of a Vickers indenter as well as the case of Berkovich indenter is observed. The slope of the straight line in a part near the top becomes about 1.5, and a slope of 2 of the straight line is observed from the top in the part of 200 nm or more.

(5) The vertex and the base positions of triangle are difficult to determine accurately from the top view of an SPM image. When Berkovich hardness is calculated from the indentation, the method of measuring the length of the perpendicular from the vertex to the base in the vertical section image is suitable.

(6) Because Vickers indentation assumes a rectangular shape due to the conjunction line of indenter tip, the method of calculating the indentation area from the diagonal length is unsuitable. Then, two side lengths should be measured from the lengthwise and crosswise section images. When the positions of the sides are determined, piling-up should be considered. Essentially, the side length is a distance measured between the edge and the point equal to surface level. However such parameters in the cross section profile must be defined as the principle of method to obtain a reliable nano-hardness value.

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