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A CONTACT POINT DETECTION FOR INDENTATION TEST OF LOW-K FILM

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Abstract − In the instrumented indentation test including nanoindentation, the mechanical parameters are calculated by test force and indenter contact/surface area. The area is determined as a function of depth, which is measured as the displacement from surface of sample. Therefore how to determine the surface is important, however, it is not easy to determine sample surface exactly during the test. Because nanoindentation machines detect the surface as a velocity decreasing point or stiffness increasing point with monitoring the indenter motion at the surface finding process. The detected surface point much deeper than the original sample surface and varies with depending on the mechanical properties of the sample.

In this report, we observed the surface contact behaviour of sapphire and low-k film using commercial indentation tester ENT-1100a (Elionix Co.). In the small penetration depth, observed results show the good agreement with Hertz's contact theory for both hard and very soft material. Then the first portion of the loading curve is fitted to the elastic theory, and surface point is determined as zero-force point of the fitted curve. We applied the proposed surface correction method for the elastic modulus measurement of low-k films, which have 150, 200, 300, 500 and 700 nm thickness on silicon substrate. The variation of estimated film modulus for each thickness sample was obtained as 6-11 GPa without correction and as 5-7 GPa after the proposed correction.

Keywords: nanoindentatin, contact point, zero-point estimation

1. INTRODUCTION

Nanoindentation test is widely used to evaluate mechanical properties of thin films [1], because of easy to use, such as sample preparation, size requirement and not long measurement time. On the other hand, measured value is depends on tester and system noise.

The system noise, including ground vibration effect, is directly couples to the surface finding sensitivity. In most popular tester, the preliminary contact point is determined and the machine control changes from surface finding process to test process. After (or during) test, recalculation will be done and final zero point is determined. Many nano-indentation testers may have such recalculation procedure, but the details are unclear.

The measurement requirement is getting difficult with film thickness decreasing. For instance, film thickness for semiconductor devices use less than 300 nm. The maximum indentation depth must be shallower than 100 nm. The accuracy of the surface contact point is much more important to evaluate mechanical properties of such thin films.

In this paper we use the commercial indentation tester, which is ranging micro-indentation and without re-calculation software for contact point. We applied Hertz's contact theory to calculating the contact point.

And we proposed the procedure for low dielectric films which has several GPa in indentation modulus and applied to the estimating film modulus.

2. EXPERIMENTAL CONDITION

2.1. Indentation tester

In this paper, we used ENT-1100a indentation tester (Elionix Co.). This is simple force control type machine. The displacement resolution is 0.3 nm and maximum force is 980 mN. There is no dynamic-stiffness monitoring system. Three different current-amplifiers were used for force generation, depending on its maximum applied load ranges, low, middle and high loads. So that the force resolution of surface finding process was varies depending on the maximum load range. To avoiding this problem, amplifier connections are changed from parallel to cascade connection.

For low-force range, low-force amplifier is only used. For middle and high load ranges, the low-force amplifier and additional amplifier is used. After this modification, the same force resolution is used for all test ranges at surface finding process.

The measurement software also modified. The sample surface was detected using indenter velocity change at the contact. In addition, the force-depth data before contact was deleted after once detected the surface in the original software. To investigating the force-depth behaviour at near the contact points, the software modified to keep 100 points force-depth data before detecting the sample surface. The surface detecting procedure is not modified. These two modifications are carried out through Elionix Co.

2.2. Sample and experimental conditions

Samples used in this experiment are low-dielectric (low-k) films deposited on the silicon single crystal. Thicknesses are 150, 200, 300, 500 and 700 nm. We do not know the details of chemical compositions of this sample, but that is a kind of SiOCH non-porous film and is prepared by the chemical vapour deposition technique. It is process ready quality, and little variation is expected in material property. The Young's modulus of this film is expected lower than 10 GPa [2]. These samples are prepared by Semiconductor Leading Edge Technologies Inc. (Selete).

Indenter used is Berkovich-type and its tip rounding (truncation length is about 14 nm) and that area function was evaluated by using fused silica as the reference. We use 0.5 μN for force increasing rate and 100 ms for single sampling time at the surface finding process. For the test process, we use 30 s for force application time, 1 s for force duration, 30 s for unloading time, respectively. The force application process is divided into 500 steps, and its sampling time is 60 ms.

Figure 1 shows the typical force-depth curve obtained by the preliminary test for 700 nm film. With increasing the test force, some pop-in events are observed (arrows), which may be by the cracking of the sample. We preliminary measured the first pop-in force, because the different mechanical response was expected before and after the pop-in event. Then we used much lower test force in the following experiments. Measured first pop-in forces are indicated in Table 1.

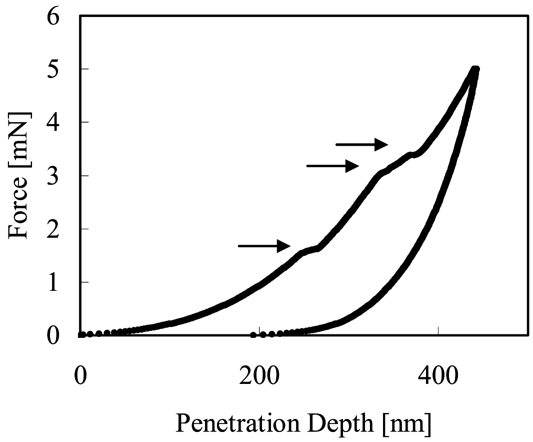

Fig. 1. Typical force-depth curve of low-k film.

Table 1. Measured pop-in force.

Film thickness	Pop-in force
150nm	0.5-0.6mN
200nm	0.7-0.8mN
300nm	1.1-1.2mN
500nm	1.1-1.2mN
700nm	1.5-1.6mN

2.3. Zero point detected by the Machine

Previously described, we modified the software to store force-depth data before machine detected the contact point. The machine re-set the force and the depth to zero when it found the contact in the surface finding process (hereafter, we called the point as "Machine Detected Zero Point"). Our new data points are 100 points before this point. However, through this software modification, the first data point (100 points before Machine detected zero point, is set to zero both of force and depth. Then we have to analyze that the difference in force steps between surface finding process and test process, and re-determined the Machine detected zero point, again. This re-determined point is corresponding to the requirement of software modification (100 points). We evaluated the force-depth data near Machine detected zero point, which is obtained as an ASCII file from ENT-1100a.

3. FORCE DEPTH BEHEVIOUR NEAR MACHINE DETECTED ZERO POINT

3.1. Indenter displacement near the surface contact

The indenter velocity is almost constant before contacting the surface. In the case of this experiment, the indenter displacement at single sampling is about 9-10 nm. The indenter displacement for single step is shown in fig. 2. Figure 2(a) is obtained by the sapphire sample, and (b) is obtained by a low-k sample, where the origin of figure (zero) is Machine Detected Zero Point. The indenter velocity is rapidly changed before and after the contact, and the indenter velocity start decreasing far before the Machine detected zero point. We indicate this region as affected region. The widths of these regions are about 9 nm for sapphire, and 15 nm for low-k film, respectively. The difference is about 5 nm depending on the sample. The maximum displacement is about 30nm for both samples, however this difference is non-negligibly large. The true contact point may be between in the affected region. And this difference sometimes affects the calibration results of area function. Tanaka et al. [3] noticed that and taken into account as surface damage length.

3.2. zero-point expected by the Hertz's contact theory contact

In accordance with the ISO standard [4], two methods are available to determine the contact point. The contact point is determined by the extrapolation by using a fitted function in Method 1. The other is contact point is determined by the first force or stiffness increasing point, in Method 2. At a first attempt, we applied the method 2. The contact point is chosen left-hand side of affected area in Fig. 2. In this case, the force is almost zero after contact point and the force starts increasing clearly apart from the contact-point even though the Sapphire sample. So that we decided this method is not applicable to this samples.

In the case of method 1, the question is what is appropriate function? In the case of nano-indentation, 10 to 30 nm region after the contact may be affected by the tip rounding. in addition, either of sapphire and low-k mainly shows elastic-like behaviour at this region. Normally, this method is used to the spherical indenter [5]. For Berkovich indenter, this analysis is used for very shallow indentation or fully elastic contact.

The force-depth curve following to the Hertz's contact theory, the force, F is described by the following relation $F=Ah^{3/2}$. where, the A is constant depending on the tip rounding and Young's modulus, and h is the displacement of indenter, respectively. We introduced the h_D to calculating appropriate correction regarding as a Machine Detected Zero Point. And re-plotted the force-depth curve using,

$$h = A' F^{2/3} - h_D . \qquad (1)$$

The re-plotted force-depth curve for sapphire is shown in Fig. 3. In this figure, the frame compliance and spring corrections were already done. This plot looks linear, and no difference found between loading and unloading curve. We found that the almost elastic behaviour is measured and we can treat the indenter used is almost spherical at this region. Figure 4 shows the re-plotted force-depth curve obtained by a low-k film. Small difference was found between loading and unloading curve, but the loading curve looks like a liner line. And we can consider that the elastic response dominate in this depth range.

To calculating h_D, we have examined the data-set using this fitting. The loading data will be dividing into two parts, the data from surface finding process and from the test process. The calculated gradient and h_D have some difference from the surface finding process and from test process. It may be comes from different force step, noise and unstable response near the contact. Finally, we decided to use the data only from test process. The zero points is determined as an intersection at F=0 of the linear regression for $F^{2/3}$of loading curve, fitted lines are shown in the figs. 3 and 4. The arrows in the figures are indicating Machine Detected Zero Point as "detected" and calculated point, as "corrected". We use the nearest data points from the calculated intersection only in the plots.

The procedure for the correction using in this study is as follows:

1. Correcting the spring effect by using the spring constant determent by the force-depth curve before affected region. And determine the force zero line, using the after spring corrected data points.
2. Remove the data from surface finding process.
3. Determine the h_D with titting to first 5 % portion of the test data, using eq.(1). In this fitting force is the variable.

Strictly speaking, the area function of the indenter using in this study should be re-calibrated by using the data after this process, but we use the same area function in this study. Because the absolute values is not required in this study.

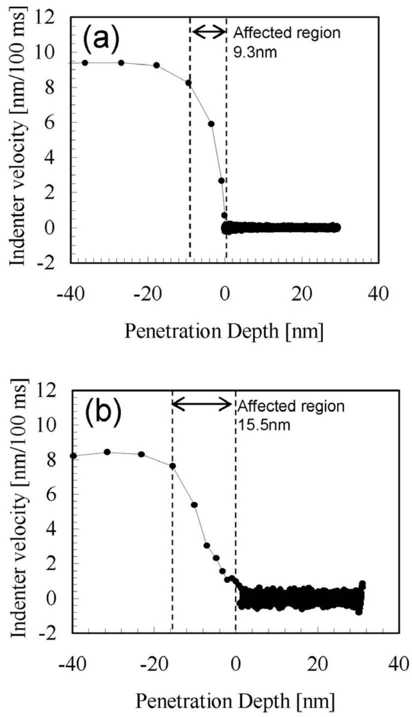

Fig. 2 Typical indenter velocity for Sapphire (a) and low-k film (b).

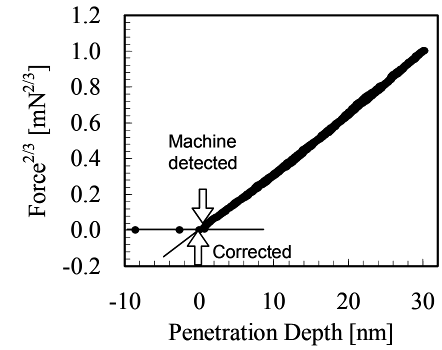

Fig. 3. Modified force-depth curve for sapphire.

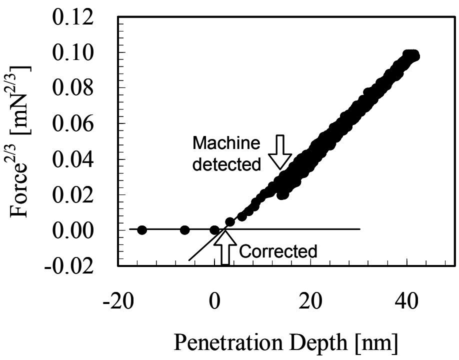

Fig. 4. Modified force-depth curve for low-k film.

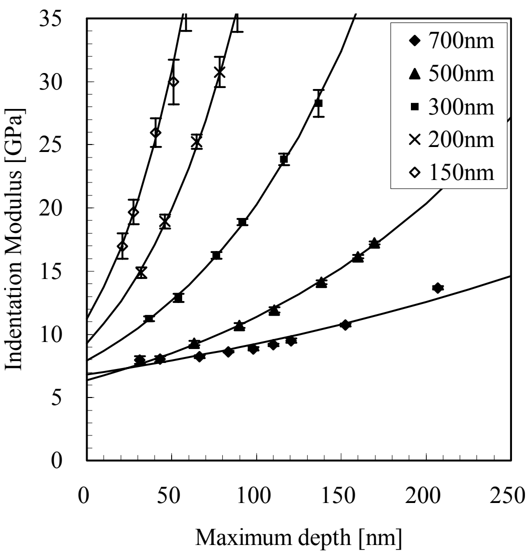

Fig. 5. Indentation modulus obtained by using Machine detected zero point.

4. ESTIMATION OF INDENTATION MODULUS OF LOW-K FILMS WITH VARIOUS THICKNESSES

We measured the indentation modulus of low-k films with various maximum indentation depths. The thicknesses are 150, 200, 300, 500 and 700 nm. 9 measurements have done for the same test force, and averaged. Figure 5 shows the indentation modulus variation against the maximum indentation depth using without correction. The standard deviation of measurements is indicated error bars. Figure 6(a) shows the indentation modulus variation with correction. The indentation modulus is increasing with increasing the maximum depth due to the high modulus of Silicon. This effect is much significant with decreasing the film thickness.

The semi-logarithmic plot of corrected indentation modulus is shown in fig. 6(b). There are some method to obtain the modulus of film [6], however, we use following simple way. We estimate the film modulus using this plot and linear (in logarithmic) fitting at a modulus at maximum depth equal to zero. The fitted curve is also shown in figs. 5 and 6(a). The estimated film modulus is indicated Table 2. The film modulus is from 6 to 11 GPa obtained by Machine detected zero point (uncorrected). From the result from 700 nm film the lowest maximum depth data looks high and line crossing to the result of 500 nm film. The standard deviation of measured modulus slightly larger than that of without correction (for instance about 40 nm maximum depth in 200 nm thickness sample).

The modulus obtained is increasing with decreasing film thickness in this case. For the corrected zero point was used, obtained modulus from 5 to 7 GPa. The variation of the expected film modulus is decreasing from 5 GPa to 2 GPa using this correction. This variation may be affected by the standard deviation of each point, that is about 2 GPa for 150 nm film.

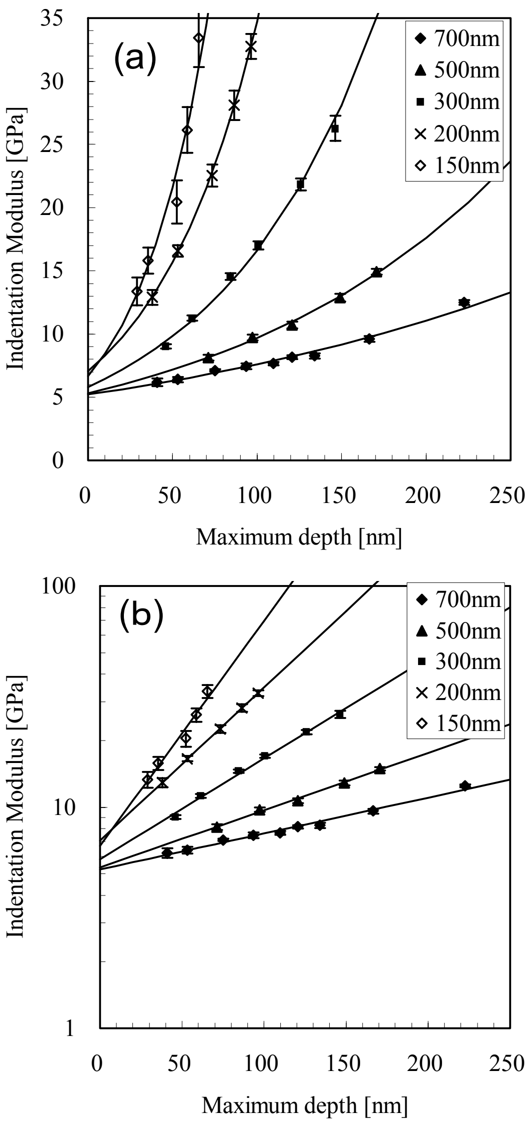

Fig. 6. Indentation modulus obtained with proposed contact point correction.

Table 2. Estimated Indentation modulus of low-k film extrapolated to penetration depth equal to zero.

Thickness	Indentation modulus [GPa]	
	uncorrected	corrected
150nm	11.2	6.7
200nm	9.3	7.1
300nm	7.9	5.8
500nm	6.4	5.3
700nm	6.8	5.2

5. SUMMARY

In this paper, we use the modified indentation tester, ENT-1100a for estimating the thin film properties of low-k films. We applied Hertz's contact theory to calculating the contact point.

Through the preliminary experiment, we found that the loading curve of low-k films approximately following to the

Hertz's contact theory near the contact region that is normally used to the fully elastic indentation. By using this result, we applied the Hertz's contact theory to determination of surface contact point for low-k films. The variation of estimated modulus of films is decreasing from 5 GPa to 2GPa. The variation of the results and thickness dependence is improved.

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