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APPLICATION OF PSI/SCM MICROSCOPE FOR NANOINDENTATION TESTER

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Abstract – Instrumented indentation test is a simple and effective method for evaluating the mechanical properties such as elasticity/stiffness, hardness and adhesion. Generally it is the method that doesn't have to observe the residual impression and around the indentation area. However, it is necessary to observe the residual impression and surface of test piece to obtain the material behaviour such as pile-up/sink-in, crack. Recently atomic force microscope (AFM) and 3-D scanning electron microscope (3-D SEM) are used as the techniques for measuring the residual impression and surface of test piece in the three-dimensions.[1],[2] Especially, AFM is used mainly, however, it is necessary to correct shape in the point of cantilever in measuring procedure. And also, measuring them by AFM precisely, the vacuum atmosphere and clean environment and so on are needed. As for 3-D SEM, it is similar.

In this paper, authors focused the phase shifting interferometer (PSI)/ scanning confocal microscope (SCM) combined optical system for nanoindentation tester to observe the residual impression and the surface of the test piece. The utility of this optical system is confirmed, and the nanoindentation tester that is able to be the three-dimensional shape observation is developed. This tester is able to observe the three-dimensional shape easy and quick in complete noncontact and measuring in various environmental conditions. The observation data of residual impression and the surface of the test piece are obtained at the nano-meter order.

Keywords : Residual impresion, PSI/SCM microsocope, Surface observation

1. INTRODUCTION

Instrumented indentation test is generally known that the method should not to observe the residual impression and surface around it. However, it is important to observe the indentation area and to know the behaviour of material. After the indentation test carried out, researchers demand to know the indentation area in the microscope. In such a purpose, AFM and 3-D SEM are often used to observe them. When three-dimensional shape like residual impression is observed, contact point of cantilever is changes for AFM. And, cantilever undertakes a damage while observing, the reliability of the image decreases. The vacuum atmosphere is necessary for the observation by 3-D SEM. In the case of non electronic conductive materials, spatter is needed. They disturb an accurate surface observation. Any method has the advantage and the fault. Therefore, simple and quick observation system in ambient atmosphere for indentation area is needed. Recently scanning confocal microscope is used for measurement of rugged samples. There are advantages though this method is not suitable for the observation of the narrow field. The comparison of these observation techniques is shown in table.1.

On the other hand, nanoindentation test is able to measure the thin films and thin wirings that measurement in the past was very difficult. However, because cutting and buffing are difficult, they cannot be prepared for the test piece. In many cases, they are indented as they are. Tilt of the surface of test piece should be included in the uncertainty calculation. In the ISO14577-1, the tilt has been described as less than 1 degree. When it isn't possible to prepare the test piece, the demand of the ISO standard cannot be achieved. In such a case, it is necessary to correct the indentation test result by measuring the tilt. Therefore, for a high accurate indentation test, the system that observes three-dimensional shape on the surface of test piece is needed.

In this paper, the phase shifting interferometer (PSI)/ scanning confocal microscope (SCM) combined optical system for nanoindentation tester to observe the residual impression and the surface of the test piece is developed. From the observation data of the residual impression and the tilt of the surface of test piece, the utility of this optical system is confirmed. Finally, the indenter that is able to observe the three-dimensional shape is developed.

Table 1. Comparison of observation techniques

		AFM	SCM	3D-SEM
X-Y	wide field	×	◎	◎
	narrow field	◎	△	○
Z resolution		◎	○	○
easy observation		△	◎	×
ambient atmosphere		○	◎	×

2. PRINCEPAL OF PSI/SCM MICROSCOPE FOR NANOINDENTATION TESTER

There must be two features for optical system to observe the three-dimensional shape for nanoindentation tester. For tilt and curves of the test piece the wide observation area is needed. And narrow observation area is needed for the residual impression. Then, SCM optical system is used for wide observation area, and PSI optical system is used for narrow observation area.

2.1. Scanning confocal microscopy

When scanning confocal microscope (SCM) is used, it achieves out-of-focus rejection by two strategies a) by illuminating a single point of the test piece at any one time with a focussed beam, so that illumination intensity drops off rapidly above and below the plane of focus and b) by the use of blocking a pinhole aperture in a conjugate focal plane to the test piece so that light emitted away from the point in the test piece being illuminated is blocked from reaching the detector. Therefore, SCM involves moving the focal plane of a microscope vertically through the sample while acquiring a series of images. The principal scheme of SCM is shown in Fig.1

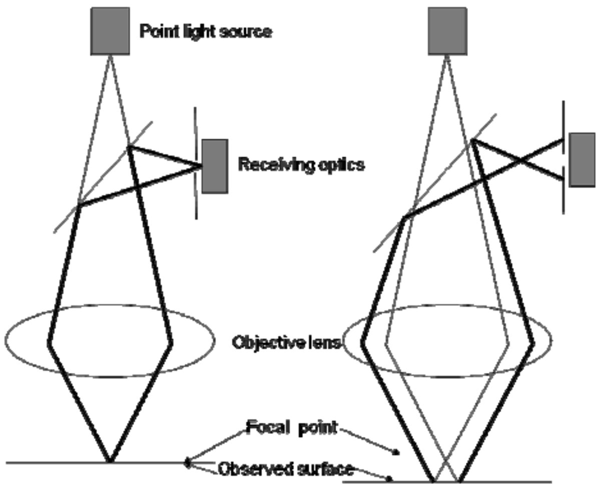

Fig.1 Scanning confocal microscope
Left: In focus situation
Right: Out of focus situation

In Fig.2, there are two images printed on paper with the laser printer. The upper image is taken by normal optical microscope, and the lower image is taken by SCM. High contrast and high resolution image is taken in deep focal depth by using SCM. And it takes only short time for the observation by SCM. SCM also take a three dimensional shape. The images can later be assembled using software to form an apparent three dimensional shape. The resolution for depth measurement of SCM is about 10nm. Generally, the resolution for depth measurement of AFM system is about roughly 0.5nm or less. As for SCM, the performance of the resolution at vertical direction is several digits worse than that of AFM system have disadvantages at resolution in vertical direction. In nano range, phase sifting interferometer is necessary to observe the residual impression.

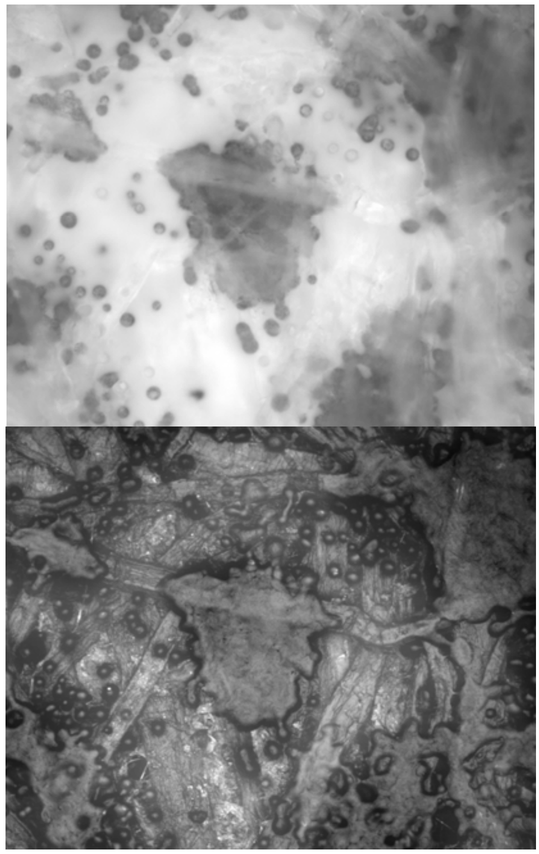

Fig.2.Image printed on paper with the laser printer
Upper: measured by normal optical microscope
Lower: measured by SCM

2.2. Phase sifting interferometry

Phase shifting interferometry (PSI) was proposed as a means to improve the measurement accuracy of a usual optical interference method rapidly in 1970's. Relative phase distribution on the entire side is calculated directly from the optical strength distribution obtained by using the interference-induced stripe as a probe and scanning the surface of the object, and the feature of this method is to convert the calculation result into the difference of three-dimensional height. Mirau interferometer and Linnik interferometer are well known as the PSI system. Linnik interferometer works only when the two path lengths are matched to within the coherence length of the light source. For a low-coherence source such as an LED, the coherence length is on the order of micrometers, so matching the path lengths requires precise adjustment of both the target and reference arms. In a Mirau interferometer, the interferometric system is customized for a single microscope lens, so the path lengths are matched during the manufacturing process. Consequently, taking three-dimensional interferometric images is as simple as changing focus on the microscope stage. In this study, Mirau interferometry is used as PSI optical system. The principal scheme of Mirau interferometry is shown in Fig.3 [3]

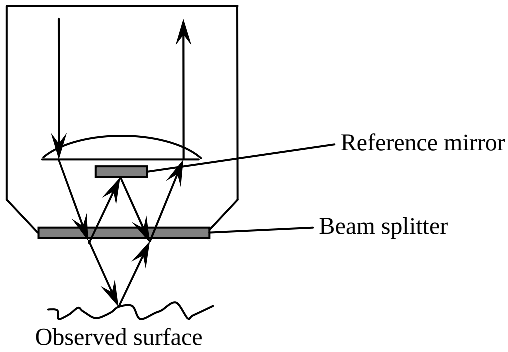

Fig.3 The principal scheme of Mirau interferometer lens

2. DEVELOPMENT THE NANOINDENTATION TESTER WITH PSI/SCM MICROSCOPE

The PSI/SCM microscope that has an excellent performance in the measurement of three-dimensional shape is selected, and it built into nanoindentation tester. In this system, PICODENTOR HM500 manufactured by Helmut Fischer Gmbh+ Co.KG is used as the nanoindentation testing machine, and 3CCD real color confocal microscope OPTELICS H1200 with interferometer option manufactured by Lasertec Corporation is used as the PSI/SCM microscope. [3] The specifications of this microscope as follows:

Light source: mercury vapour xenon lamp
Resolution 2048x2048 12bit each RGB
Wavelength selectivity: 436,488,546, 577,630 [nm]
Z-resolution: SCM:10nm / PSI:0.1nm

Fig.4 presents the nanoindentation tester with PSI/SCM microscope. Before and after the indentation test, three dimensional shapes observe the PSI/SCM microscope at the right side of this instrument. For nanoindentation test, vertical direction of vibration isolation is needed, because it is the same direction as indentation testing. However, for observation by PSI/SCM microscope, horizontal direction of vibration isolation is needed. Therefore, in this system equipped the vibration isolator for all X- Y- Z direction.

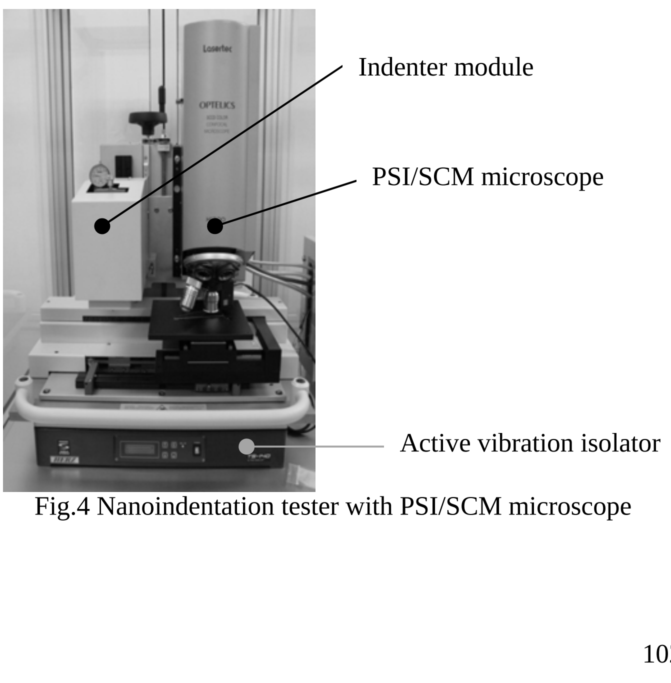

Fig.4 Nanoindentation tester with PSI/SCM microscope

For the large residual impression and the tilt of the surface of test piece, it is observed by SCM. The residual impression at the silicon wafer observed by SCM is shown in Fig.5, and the cross section of this residual impression is shown in Fig.6. And the observation example of the tilt and curves of test piece is shown in Fig.7. This sample is the case of the cylindrical pipe.

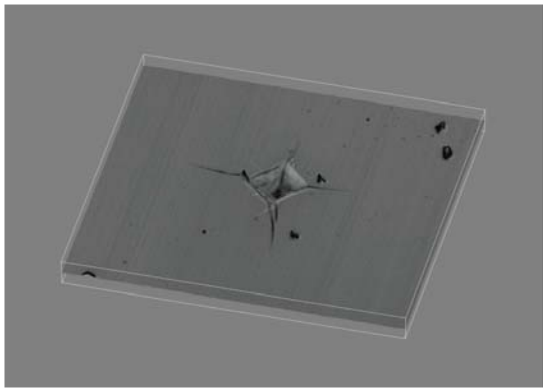

Fig.5 SCM image of the residual impression on silicon wafer

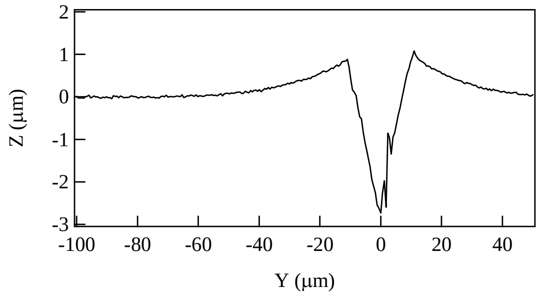

Fig.6 Cross section of the residual impression on silicon wafer

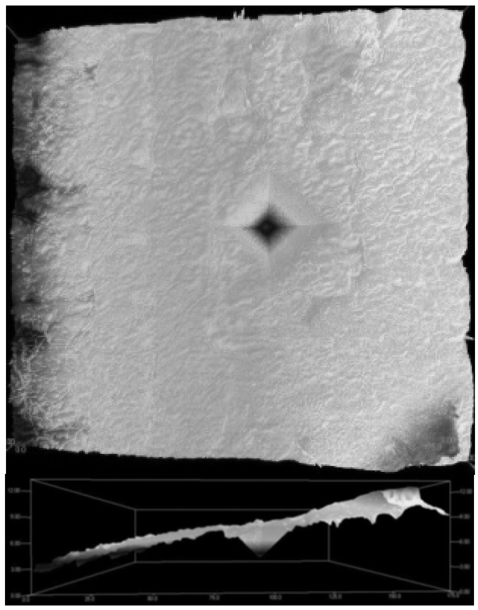

Fig.7 Observation example of the cylindrical pipe
Upper: Top view
Lower: Side view

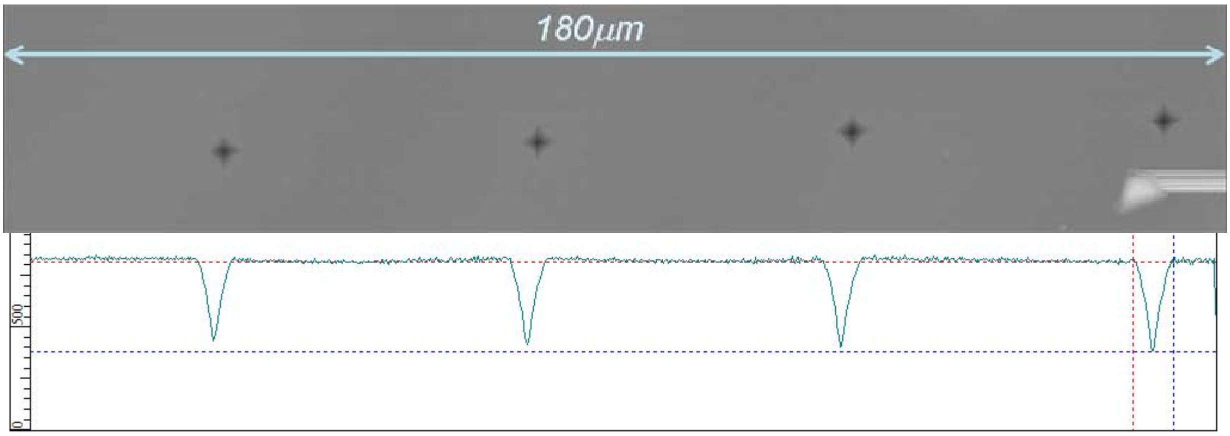

Fig.8 PSI image of the residual impression on silicon wafer

For the small residual impression, it is observed by PSI. The resolution of vertical measurement is 0.1 nm used by Mirau interferometer, and measurement time is about 2 or 3 seconds. This method is very easy and quick procedure in fully no contact and observation in various environmental conditions. The data of indentation depth from PSI procedure is obtained at the nano-meter order. This system also has advantage in observation area. When object lens that is 100 magnifications is used, PSI optical system is able to observe 180μm^2 at the same time. Observation result is shown in Fig.8. From the left to right, there are 4 residual impressions. Their indentation depth was observed at a time.

In many cases, this system is a performance enough to observation of the residual impression and the surface of the test piece. However, for much smaller residual impression under the sub nano-meter, this system can use the object type AFM system. General AFM system has the range of the observation of several microns. It is difficult to search the small residual impression by only AFM system. The area should be observed can be selected by using PSI/SCM system together before the observation of AFM. AFM is a useful tool, however, there are difficulties in narrowness in observational area and the damage of the cantilever and so on. In this study, utility in nanoindentation tester of PSI/SCM microscope is confirmed. The purposes in the future are the expansion of the range of the observation of this PSI/SCM microscope and the relation of the three dimensional shape and the mechanical properties.

4. CONCLUSIONS

In this work, authors focused the PSI / SCM combined optical system for nanoindentation tester to observe the residual impression and the surface of the test piece. The nanoindentation tester with PSI / SCM microscope was developed, and the following conclusions were obtained.

1) Confocal scanning microscope is very useful technique to obtain the indentation depth of residual impression at submicron order. However there are some problems should be solved in small impression under submicron order.
2) Phase Sifting Interferometer microscope is the useful instrument that can correspond to small residual impression in depth of submicron order.In the case of smaller indentation depth under the tens of nanometers, There is necessity for using other techniques like AFM together.
3) The expansion of the range of the observation by the PSI/SCM microscope, and the relation of the three dimensional shape and the mechanical properties are necessary for confirming further utility of this three dimensional shape observation system.

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