HARDMEKO 2007
Recent Advancement of Theory and Practice in Hardness Measurement
19-21 November, 2007, Tsukuba, Japan

MEASUREMENT OF A RESIDUAL IMPRESSION BY THE LASER SCANNING MICROSCOPE WITH DIC UNIT

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Abstract – When the nanoindentation testing is carried out, it is very important to understand the residual impression and the surface around it. Generally Atomic Force Microscopy (AFM) is used to obtain them. However, it is necessary to understand correct shape in the point of cantilever in AFM procedure. And, measuring them by AFM procedure precisely, very long measurement time and very clean environment and so on are needed. So, there are many difficulties to use AFM procedure for measuring residual impression and the surface around it. On the other hand, there are some procedures to measure them. Scanning Electro Microscopy (SEM) is effective to the grasp of the shape of the surface and the ruggedness. But, this procedure needs the coating of the sample and the vacuum atmosphere. Therefore it is not a simple and quick procedure.

In this paper, authors used the laser scanning microscope with a differential interference contrast unit to obtain the data of a residual impression and the surface around it. This procedure is the very simple and quick procedure in no contact and measuring in various environmental conditions. The data of indentation depth from this procedure is obtained at the nano-meter order. The results from this procedure compare with the results from displacement of nanoindentation test.

Keywords: Nanoindentation, residual impression, laser scanning microscopy

1. INTRODUCTION

Nanoindentation test is a simple effective method for evaluating the mechanical properties of industrial materials. However it is very important to understand the residual impression and the surface around it. Error margin of depth influences the results immediately, because nanoindentation test is a kind of depth sensing indentation testing. To measure it more accurately, the geometry of the indenter tip and the residual impression is discussed very much. Generally Atomic Force Microscopy (AFM) is used to obtain them.[1] However, AFM procedure has some problems that should be solved. On the other hand, Scanning Electro Microscopy (SEM) is effective to the grasp of the shape of the surface and the ruggedness. But, this procedure needs the coating of the sample and the vacuum atmosphere. In this study, new measurement procedure using laser scanning microscope to obtain the residual impression and the surface around it.

2. MEASUREMENT BY LASER SCANNING MICROSCOPE

Generally laser scanning microscopy (LSM) is very effective technique to obtain high contrast images at short measurement time. In addition, LSM procedure is the very simple and quick procedure in no contact and measuring in various environmental conditions.

2.1. Measurement by confocal LSM

If confocal LSM system is used, it achieves out-of-focus rejection by two strategies a) by illuminating a single point of the specimen 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 specimen so that light emitted away from the point in the specimen being illuminated is blocked from reaching the detector. Therefore, confocal LSM system involves moving the focal plane of a microscope vertically through the sample while acquiring a series of images. The comparison of an image measured by normal microscope that is standard equipment of PICODENTER HM500 made by Helmut Fischer Gmbh + Co. KG. and an image measured by confocal LSM made by Lasertec Co. is shown in Fig.1. The lower image is especially clearer than upper image in respect of center part of residual impression. Also, contrast of lower image is much higher than upper image. Therefore, confocal LSM is very useful to obtain the X-Y image of residual impression. Especially for Vickers indentation testing at micro or nano range, the residual impression is much smaller than it at the macro range. The little error margin of the length of the diagonal of the residual impression make a very large error of the result of hardness value.

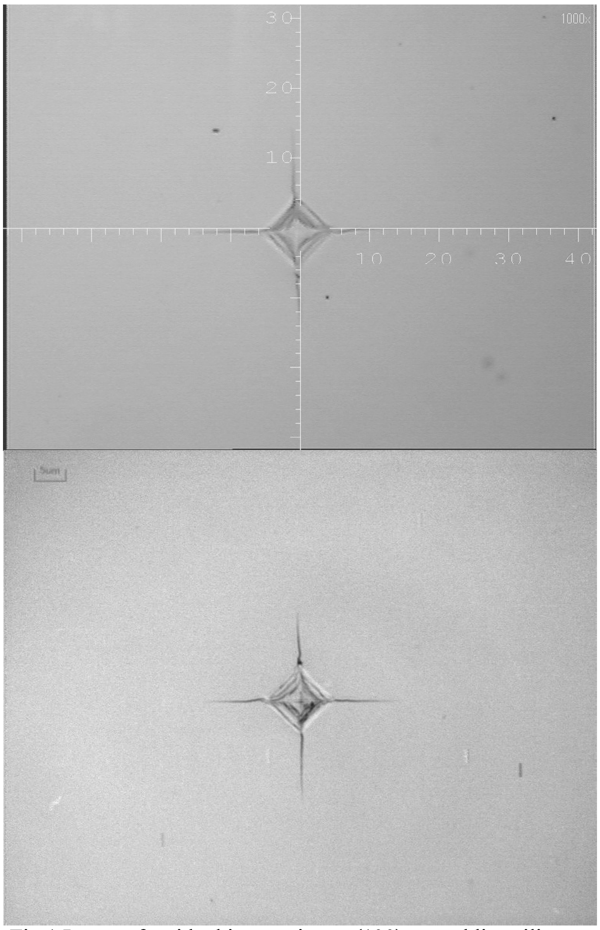

Fig.1.Image of residual impression at (100) crystal line silicon
Upper: measured by normal microscope
Lower: measured by confocal LSM

On the other hand, the images can later be assembled using software to form an apparent 3-D model. For depth sensing indentation testing, this 3-D observation function is very useful to obtain the images of the residual impression and surface around it. 3-D image is shown in Fig.2. Cross section of the residual impression is shown in Fig.3.

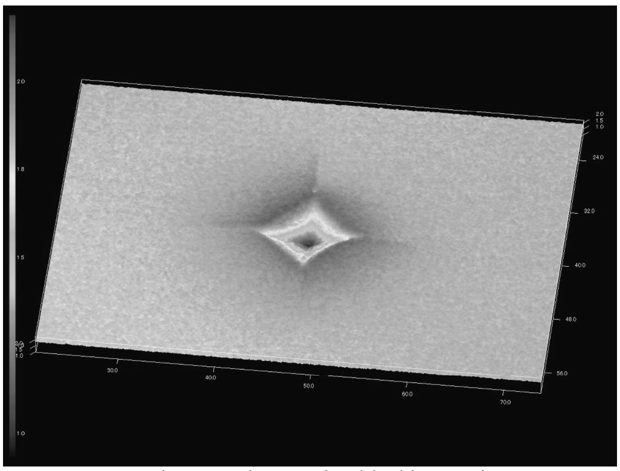

Fig.2. 3-D image of residual impression

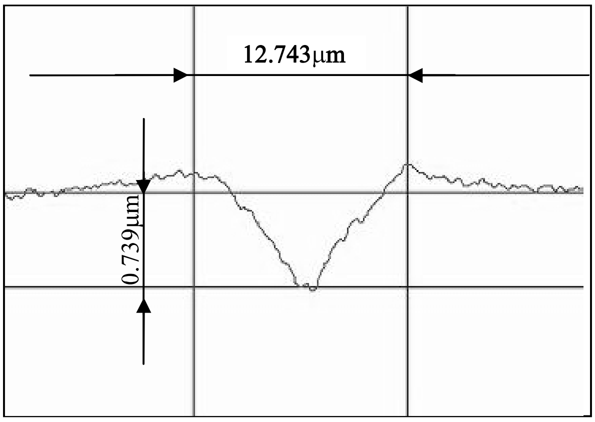

Fig.3 Cross section of the residual impression

The result of depth of the residual impression obtained from indentation test is 0.773μm. The agreement between the result from confocal LSM and the result from the indentation testing machine is nearly perfect. However the result from confocal LSM is average value at the center of the measurement point at the residual impression. Therefore, the results from confocal LSM is a little shallow compared with the results from the indentation testing machine. If residual impression is become smaller, the influence of this problem is not a little.
Generally, the resolution for depth measurement of AFM system is about roughly 0.5nm or less. The resolution for depth measurement of LSM is about 10nm or more. As for LSM and confocal LSM, 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, further device is necessary to use LSM for measurement of residual impression and around it.

2.2. Measurement by LSM with DIC unit

Differential Interference Contrast (DIC) is the technique of deriving contrast in an unstained specimen from differences in index of refraction of specimen components. DIC microscopy was introduced in the mid-1960s after the method was described by Nomarski in 1952. It is now widely used for various researches to obtain the micro or nano structures and surfaces. By this technique, 3-D image is obtained as the result from confocal LSM. The image from DIC is just like relief. The 3-D relief of DIC imaged specimens is an optical rather than a geometric relief. Now, DIC unit is installed in LSM system to obtain the residual impression and surface around it. The resolution for depth measurement of LSM with DIC unit is about 1.24nm. DIC image measured by LSM with DIC unit is shown in Fig.4. Specimen is the (100) crystal line silicon. Testing load is the 10mN. P-h curve is shown in Fig.5. In DIC measurement, there are many stripes at the image of surface. Stripe means the gradient of the surface. However this surface is almost flat, there are three or four stripes. The reason why there are stripes is to raise the magnification of the microscope. Though the lenses that have same magnification are used usually, the lenses that have different one are used this time.

In the close up image of the residual impression, it seems that the outline of it grows dim. But the data of depth measurement is obtained accuracy. Cross section of the residual impression is shown in Fig.6. Now red laser (wave length: 650nm) is used in LSM. If blue laser is used in it, the image of the residual impression is more clearly. Also, the vibration isolator is one of the important equipment to obtain the clear image of residual impression. In this measurement, simple passive type vibration isolator is used, active type is preferable. For nanoindentation testing, vertical direction of vibration isolation is needed, because it is the same direction as indentation testing. However, for measurement by using LSM with DIC unit, horizontal direction of vibration isolation is needed. If nanoindentation testing machine is built into LSM with DIC unit, performance of vibration isolation is needed for all X- Y- Z- direction.

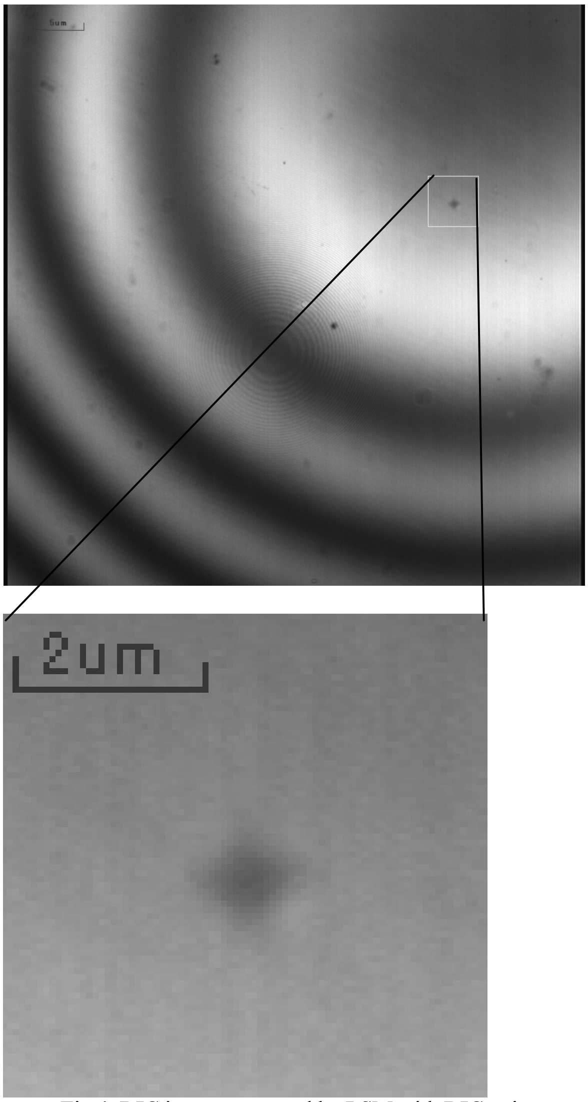

Fig.4. DIC image measured by LSM with DIC unit
Lower: Close up image of the residual impression

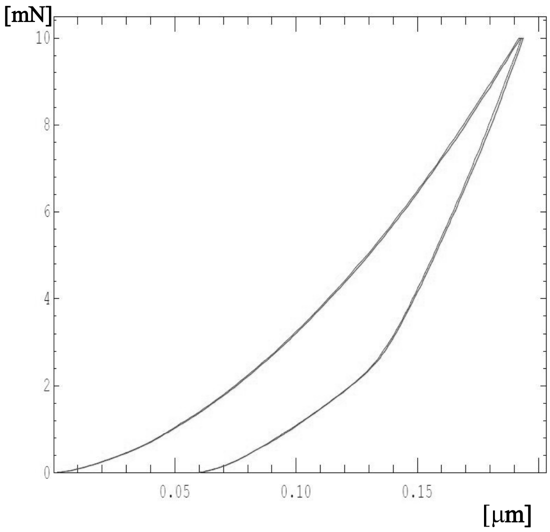

Fig.5 P-h curve of indentation test

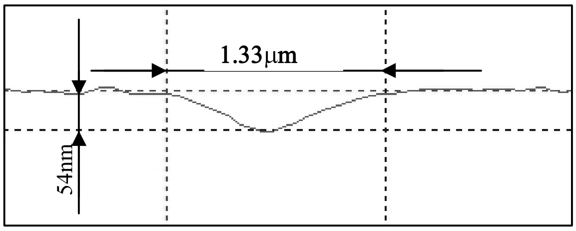

Fig.6. Cross section of the residual impression

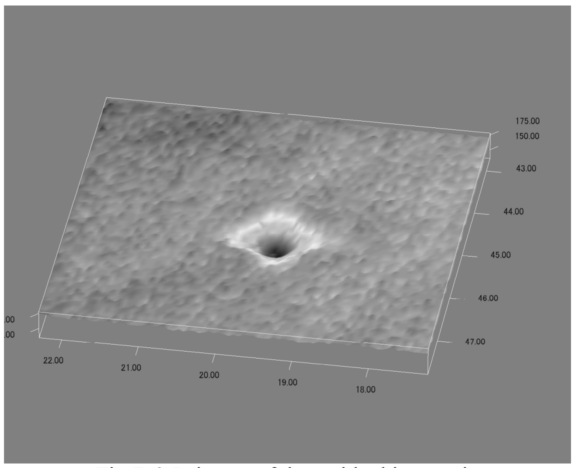

Fig.7. 3-D image of the residual impression

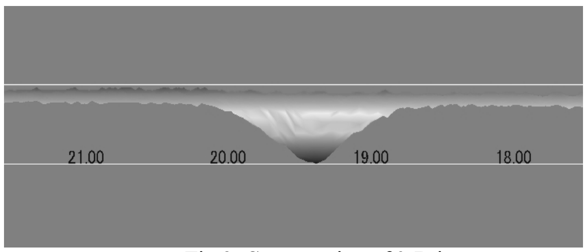

Fig.8. Cross section of 3-D image

3-D image and cross section of 3-D image of residual impression is shown in Fig.7 and Fig.8. Because it is difficult to know where there is an appropriate cross section, measuring the correct cross section data of the residual impression is very difficult. In using cross section of 3-D image depth data can be obtained more easily and simply. The depth of the residual impression from the measurement by LSM with DIC unit is 54nm. The result of depth of the residual impression obtained from indentation test is 60nm. The agreement between the result from LSM with DIC unit and the result from the indentation testing machine is considerably good. In the case of smaller indentation depth, the measurement result measured by LSM with DIC unit is shown in Fig.9 and Fig.10. There is roughness in vertical direction in Fig.10. This is the serious problem to measure the impression. The result of vertical measurement is the average value at the center part of measurement point mentioned above, for too small impression, the results from LSM with DIC unit is also shallow compared with the result from the indentation testing machine. Therefore, it is necessary to change the wavelength of the laser to obtain the vertical data of the small impression that the indentation depth is under 20nm. However, in many cases of measurement of the residual impression and surface around it, the utility of the LSM with DIC unit is can be confirmed. If the nanoindentation testing machine is built into LSM with DIC unit, there are a lot of very advantageous in limited indentation depth of residual impression.

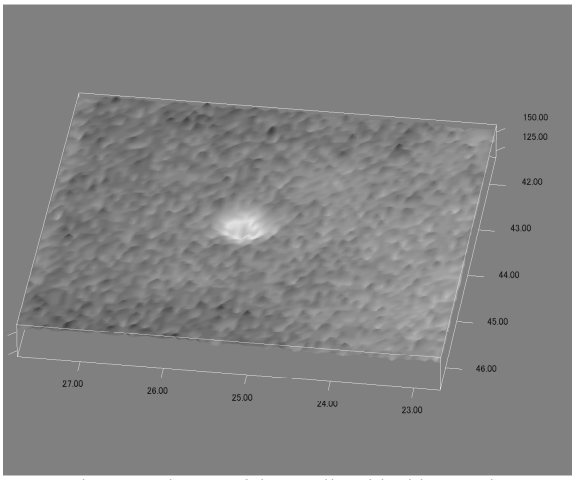

Fig.9. 3-D image of the small residual impression

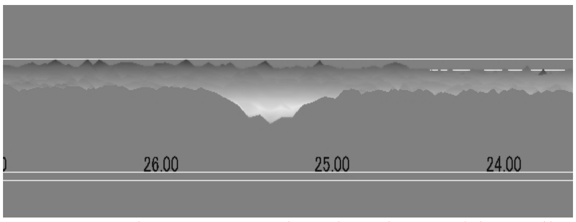

Fig.10. Cross section of 3-D image of the small residual impression

3. CONCLUSION

New measurement procedure using laser scanning microscope to obtain the residual impression and the surface around is proposed in this study. Each procedure that uses LSM or LSM with DIC unit is very simple, quick and useful procedure. Now the following conclusions are obtained.

1) Confocal laser scanning microscopy 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) Laser scanning microscopy with differential interference contrast unit is the useful instrument that can correspond to small residual impression under 100nm depth. Also, the further performance improvement can be expected by using blue laser diode and active vibration isolator.

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