Displacement measurement of an indentation tester using heterodyne interferometer

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Abstract

We have developed an heterodyne laser interferometer system to measure the indenter displacement of instrumented indentation at nano-/micro- ranges. The developed interferometer was applied to measure the displacement of an commercial type indentation tester. The interferometer system and the tester was set on the different anti-vibration systems. So the main difficulty of this measurement was how to got rid of the relative vibration effect. We designed the system as that the reference points of the system is set to the displacement measurement mirrors on the testers. The total stability of the displacement measurement system was about 10nm in spite of the larger amplitude of the relative vibration, which is larger than the 1 μm. We can successfully measure the indenter displacement by using newly designed interferometer system and is compared the displacement signal obtained from the tester.

Keywords: Nanoindentation; Displacement measurement; Heterodyne interferometer

Introduction

Nano-indentation is widely used to evaluate local elastic and plastic properties of thin film, coatings and semiconductor devices. There are many testing machines have developed and commercially used. However, the values obtained from these machines are different in a same sample that we cannot compare the values. The values are evaluated by the load-displacement curve, through the contact area calculated by the penetration depth. To measure the mechanical properties, the non-ideal tip shape should be taken into account.

All diamond indenters have each individual shape, although it were the brand-new indenters. These difference from the ideal indenter shape seriously affect the force-depth curve obtained from the indentation experiment especially at the nano- and micro- indentation ranges. This problem is main difficulty of the measurement in the range of the smaller depth. To avoid the problem, the shape was preliminary measured directly[1] or indirectly[2] and the depth-area function was determined and used to correct the non-ideal tip shape effect.

Another problem in a instrumented indentation test is the flame compliance correction. We can measure the hardness and Young's modulus when we finished the appropriate correction, on the depth-area and the flame compliance.

The direct measurement of the area function is diffecult to dairy use, because the specially designed scanning probe microscope were required. In addition, the probe tip shape effects is not negligible in the observation. Therefore, the tip-shape was evaluated by using indirect method originally Pharr and Oliver[2] in generally. Indirect measurement of the tip shape assuming 1)an uniform elastic properties of the sample, 2)a tip contact is well described by a elastic contact relation[3].

In this case the origin of the evaluation of the tip shape function, is how to get the force-displacement curves with high reliability.

We are studied the reliabillity of the depth measurement as a first step of our study of the standardization of the instrumented indentation test.

An interferometer designed to measure the small displacement on the other anti-vibration table

A commercial indentation tester was used

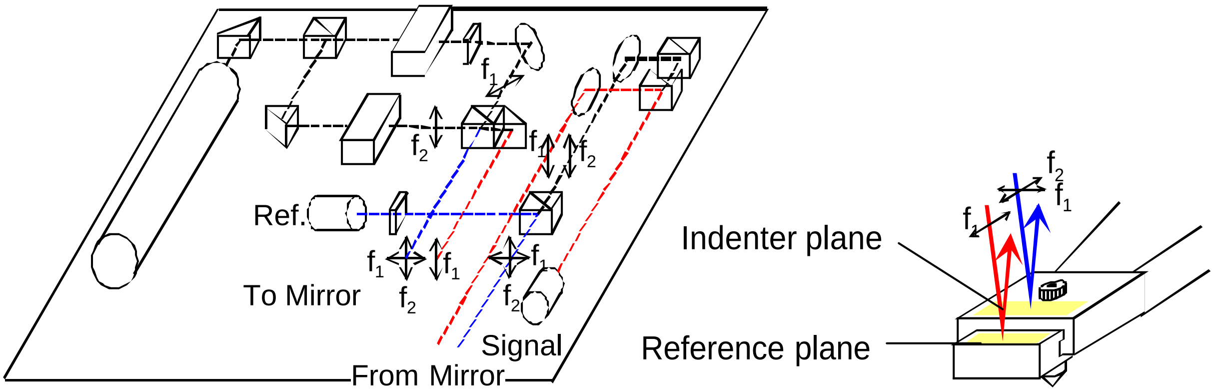

Fig 1 Schematic view of heterodyne interferometer system.
Right: Optical setup of the displacement measurement, reference plane (fixed) and the plane fixing the indenter.

in this experiment. This apparatus has following features: there are two balance-levers, the indenter is fixed the lever 1, and the indenter displacement was measured as a displacement between the levers. The lever 2, which is not fixed indenter, contacts to the sample surface and it uses as the origin of the depth measurement. Test force was applied by using force coil, fixed the other side of the indenter lever 1. Therefore, the levers are rotating around the fulcrum, however, the length of the arm is about 150 mm and the maximum measurable depth is about 20µm. Then the rotating angle is smaller enough and the rotating effect is negligible. The original position of the sample surface is set to almost same position by means of the focus plane of the microscope, which is attached on the flame.

The sample holder and the lever system compose the pendulum system mounted on the flame. Therefore the sample and the levers are protected from the vibration transmitted from the floor. However, this anti-vibration system makes us difficult to put the interferometer system near the indenter. This is the reason of why we cannot make the interferometer system on the testing machine to measure the indenter displacement.

One of the most serious problems is the relative vibration between the indentation tester and optical anti-vibration table. It can be predict that the amplitude of relative vibration is larger than a half of the light wavelength from our previous experiment. Previously constructed interferometer system set the reference plane at the same anti-vibration table of the interferometer. That has four optical paths and has very high resolution in a static experiment, however, is not work well in an actual condition due to the relative vibration. To avoid the problem, we have developed a new interferometer system.

Newly designed interferometer system is as following features: the reference plane is set on the tester. The light paths of the signal and reference have almost the same paths. Therefore the components, that are the relative displacement due to the relative vibration and the fluctuation of the refractive index, are shared between the signal and the reference.

Figure 1 shows schematic of the heterodyning interferometer system used in this experiment. The mirrors are glued near the indenter on both depth sensing devices. The optical source is a frequency stabilized He-Ne laser generated at 632.8 nm. The laser light was divided into two beams and these beams were guided to the acoust-optical modulators. We set the beat frequency as about 100 kHz to be able to detect the phase of the beat signals on the lock-in amplifier. The two frequency-shifted light was mixed at the polarized beam splitter.

One probe light (reference light) has two different frequencies, f1, f2, and these polarization-directions are set normal direction to each other. The reference light is detected after once reflect from the mirror on the tester. Detected light is used as a phase reference of the measurement of indenter displacement. The other light, whose frequency is f1, is used as signal light. The signal light is mixed with the f2 component of the probe light after once reflected on the indenter mirror and detected as the indenter displacement signal. The phase difference between these signals are detected by a lock-in amplifier. The phase readings of the lock-in amplifier is 0.5 degree. The calculated displacement resolution of the system is estimated about 0.5 nm, and the sampling rate is about 30~40 msec/sample, which is limited by the communication speed between computer and the instruments.

We observed the interference signal from the mirrors on the levers without indenter movement to evaluate the total stability of constructed system. Where the indenter was forced on the sample surface on the appropriate load. We start measurement 10 minute after to avoid the creep effect of the material. The relative vibration was simultaneously monitored using capacitance-type displacement sensor. Obtained displacement was shown in the fig. 2 and fig. 3. Figure 3 enlarges the ordinate of the interferometer signal. The noise amplitude of the interferometer was less than the 20nm, however, the amplitude of the relative vibration is larger than 1μm. The relative vibration was successfully decreased by the newly designed interferometer system. The residual signal of the interferometer system was analyzed by a correlation technique. The frequencies of the relative vibration and the interferometer are almost same, which is about 20Hz. Then it shows the relative vibration still affect the interferometer signal.

Simultaneous measurement of the indenter displacement

The indenter displacement of the actual indentation test was carried out. The Berkovich indenter was used to the experiment. The test piece was made of copper, which is commercially available as a Hardness standard sample for the micro Vickers and the nominal hardness was 200HV. The test force was chosen as 200mN, which gives about 1μm depth at the maximum force, that is predicted by the simple geometrical relation between the surface area and the depth of the Vickers indenter. The loading speeding is 10mN/sec and loading time, hold time, and unloading time are 20 sec, 10 sec and 10 sec, respectively. The time

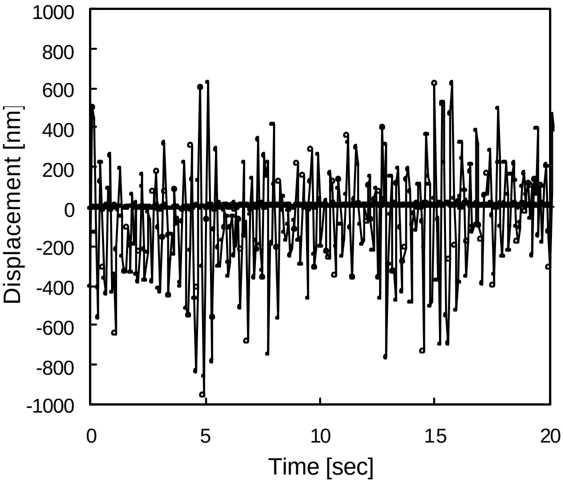

Fig. 2 The waveforms of the relative vibration and the interferometer. Stability of the interferometer signal. The interferometer signal from the fixed mirror was still stable under large relative vibration.

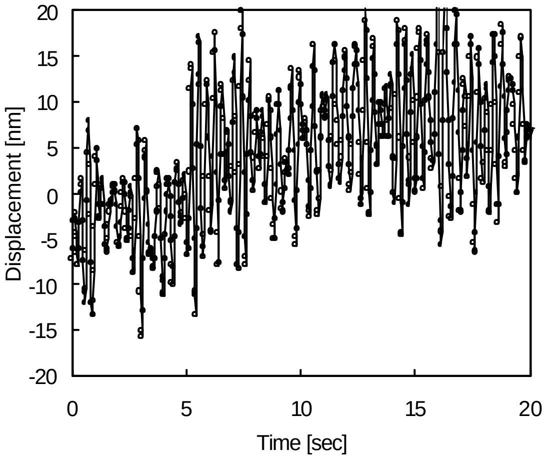

Fig. 3 Stability of the interferometer signal. The interferometer output signal has slightly noisy due to the relative vibration.

base of the sampling was determined by the internal timer of the PC. The error of the PC timer was estimated at about 1x10^{-4}. The phase of the heterodyne signal and the voltage signal obtained from the capacitance sensor of indentation tester were simultaneously observed and collected in the PC.

The raw output of the lock-in amplifier was not continuous, because of the observed displacement is much larger than the half of the wavelength, 632.8/2 nm. So the phase of the interferometer signal was increased/decreased when the phase difference between the nearest points was larger than the $\pi/3$. We automatically calculated the displacement following this procedure. The other side, the sensor output is not calibrated.

The interferometer starts working just before the test, because the reference mirrors came to the proper position at the time.

Figure 4 and 5 show the obtained indenter displacement from the interferometer system and the displacement signal of the sensor of the indentation tester, respectively. To compare the waveforms, we determined the voltage-displacement ratio as suitable to the displacement under hold segment.

We could not find remarkable discrepancy between the shape of these two curves. A thermal drift effect is slightly observed.

We successfully observed the indenter displacement in a actual testing condition.

Summary

We have developed an interferometer system that is designed to measure the small indenter displacement set at the other anti-vibration tables. The developed interferometer system can observe the indenter displacement in high stability. The noise reduction ratio is about 1/50. This interferometer system has been developed for the purpose of the study of standardization of the instrumented indentation test at nano-/micro- ranges. We are planning to make a more compact interferometer system applied to calibration of the nano-indentation tester.

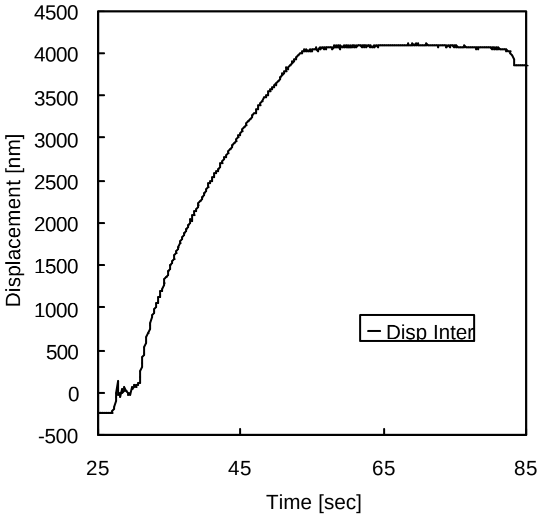

Fig. 4 Indenter displacement obtained from the Interferometer system.

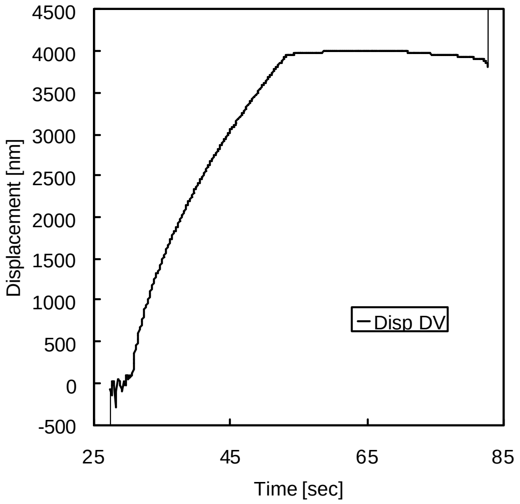

Fig. 5 Indenter displacement calculated from the Interferometer system.

References

[1] K.Herrmann, NM.Jennett, W.Wegener, J.Meneve, K.Hasche, and R.Seemann,"Progress in determination of the area function of indenters used for nanoindenatation" Thin Solid Films, 77(2000)394.

[2] W.C.Oliver and G.M.Pharr, “A improved technique for determinin hardness and elastic modulus using load and displacement sensidng indentaion experiments”, J.Mat.Res.7(1992)1568.

[3] M.F. Doerner and W.D.Nix, “A method for interpreting the data from depth-sensing indentation instruments, J.Mat.Res., 1(1986)601.

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