

## TUNGSTEN SINGLE CRYSTAL AS HARDNESS STANDARD FOR NANOINDENTATION

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**Abstract** – Hardness standard of tungsten single crystal were utilized in order to check stability and reproducibility of nanoindentation testers. The pop-in load and maximum penetration depth were measured and monitored during several hundreds of repeated experiments. One of the tested instruments showed gradual increase of pop-in load and decrease of maximum depth, which indicate the indenter tip was damaged by repeated tests. The both parameters, the pop-in load and the maximum depth, can be used to detect the change of the area function; the pop-in force is more sensitive and should be easy for general users. Other applications of tungsten single crystal are also discussed.

**Keywords:** pop-in

### 1. INTRODUCTION

Nanoindentation technique can be a very powerful tool for various research and development fields. For nanoindentation measurement, roles of standard specimens are more important than those for conventional hardness test, since the calibration procedures of frame compliance of system and area functions of indenters are much more complicated in nanoindentation tests. For this purpose, fused silica is often used as a standard specimen in nanoindentation tests. It is uniform on a nanometer scale, and its large elastic recovery is suitable for calibration of indenter area functions [1]. However, macroscopic hardness measurement of fused silica is difficult due to cracking and this makes it hard to compare nanoscopic hardness with macroscopic hardness based on fused silica. On the other hand, tungsten single crystal (and other metallic materials)

are mainly plastic and can be easily measured by both conventional and nanoindentation hardness testers. In addition, electropolished tungsten has pop-in effect which is unique and of much interest and use. Considering the differences of the two specimens, tungsten single crystal can be a good candidate of hardness standard for some purposes.

In this paper, we investigate the possibility of tungsten single crystal as hardness standard in nanoindentation measurement.

### 2. SPECIMEN

Figure 1 shows a specimen made of tungsten single crystal used in this study. The tungsten is set in the central part of the specimen and it is surrounded by the outer ring for handling. The surface of tungsten is (100) plane and is mechanically and then electrically polished to obtain flat surface and uniform properties. UMV500 (standard specimen for micro Vickers hardness) that is made of steel was also tested for comparison.

### 3. EXPERIMENTAL PROCEDURE

#### 3.1. nanoindentation stability and pop-in

Since nanoindentation measurement is relatively sensitive to experimental conditions, it is always important to check stability, reproducibility and calibration of the system.

The pop-in effect of tungsten can be a good measure to evaluate the stability of measurement, since the pop-in happens at very low (at 20 nm for a new indenter) depth and is sensitive to the tip radius of indenter that is one of the most significant factor of measurement system. The pop-in should be clear to all users and this will be important, especially when nanoindentation test becomes common and is used by various kind of engineers. Therefore, we investigate pop-in effect of tungsten to check the stability of measurement through repeated nanoindentation tests.

#### 3.2. Repeated experiment

Nanoindentation tests were repeatedly carried out with the same experimental conditions in order to check the stability of nanoindentation measurement. Two specimens, tungsten and UMV500, and two commercial nanoindentation testers, No.1 and No.2, are used and all four combinations of specimens and testers are tested. Each specimen was tested for fifty times consecutively at

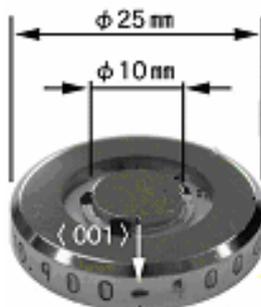


Fig. 1. Standard specimen made of tungsten single crystal.

different positions and then replaced by the other specimen. This process was repeated until 200-300 nanoindentation tests are counted for each specimen. The maximum force of nanoindentation test is 10 mN. The Berkovich indenters were used for each tester and were not replaced during the experiments.

#### 4. RESULT

##### 4.1. Stability of nanoindentation system

Figure 2 shows force-depth curves of tungsten single crystal obtained with No.1 tester. The pop-in effect is observed for both the first and the last (287th) tests, but pop-in force  $F_{pop}$  of the last test is much higher than that of the first test. This result and the decrease of maximum depth  $h_{max}$  imply that the area function of the indenter changed due to damage of repeated indentations.

Figure 3 shows detailed plots of pop-in force  $F_{pop}$  and maximum depth  $h_{max}$  against number of tests during repeated experiments. The maximum depth  $h_{max}$  gradually decreases from 285 nm to 260 nm, and the pop-in force  $F_{pop}$  gradually increases from 0.15 to 4.0 mN. The change of pop-in force is clearly more sensitive to maximum depth and should be easy for general users. As stated in the previous section, specimens were replaced after every 50 tests (the actual number of data of one process is not always 50 due to failure of the control program). Therefore gaps of data seen in Fig. 2 may come from exchanges to the other specimen occurred between a process and from slight difference of set-up of the specimen or environmental conditions, etc. Nevertheless, the trend of  $F_{pop}$  and  $h_{max}$  is clear. Note that the probability of pop-in is nearly 100% which means the dislocation density of the specimen is quite low.

Figure 4 shows the results of UMV500 obtained with No.1 tester. Only the maximum depth is plotted because

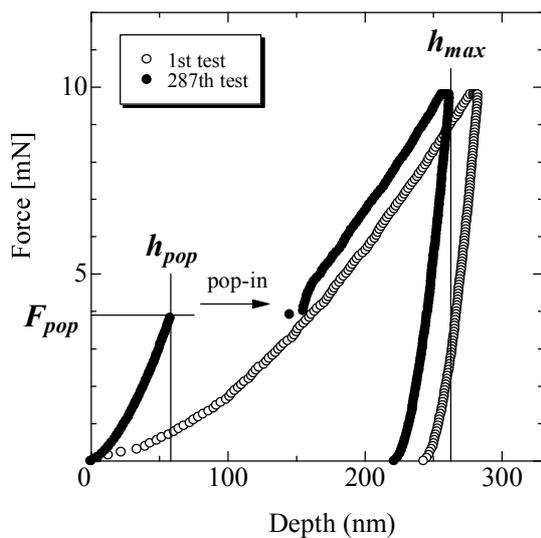


Fig. 2. The first and the last (287th) force-depth curves of tungsten obtained by No.1 tester.

UMV500 shows no pop-in. The trend is the same as tungsten, though the variation and scattering is quite high.

Figure 5 shows the results obtained with No.2 tester. In this case, the whole experiments are stable and no changes of  $F_{pop}$  and  $h_{max}$  were detected except several erratic data during the repeated experiments. This means the gradual change of pop-in force  $F_{pop}$  and maximum depth  $h_{max}$  shown in Fig. 3 is not caused by specimens, but by the measurement system (No.1 tester) itself.

These results indicate that tungsten is appropriate to check stability of nanoindentation measurement by two reasons: (1) It is uniform enough to detect changes of depth in several nanometers. (2) Pop-in force is quite sensitive to the tip radius of indenter. The second is an advantage of tungsten over fused silica.

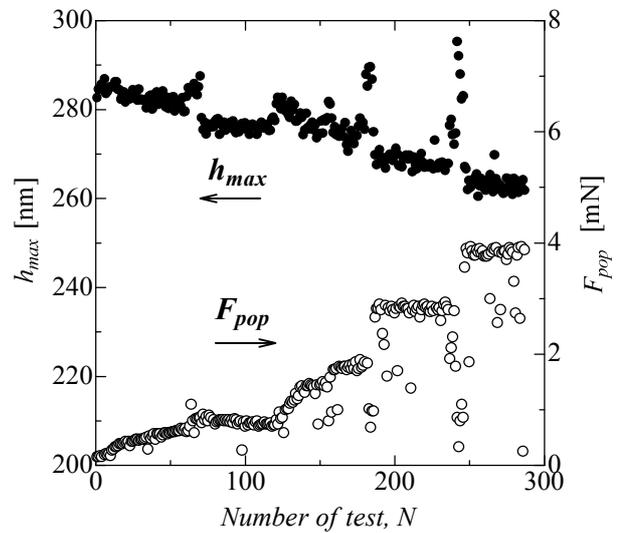


Fig. 3. Plots of pop-in force  $F_{pop}$  and maximum depth  $h_{max}$  against number of tests (tungsten, No.1 tester).

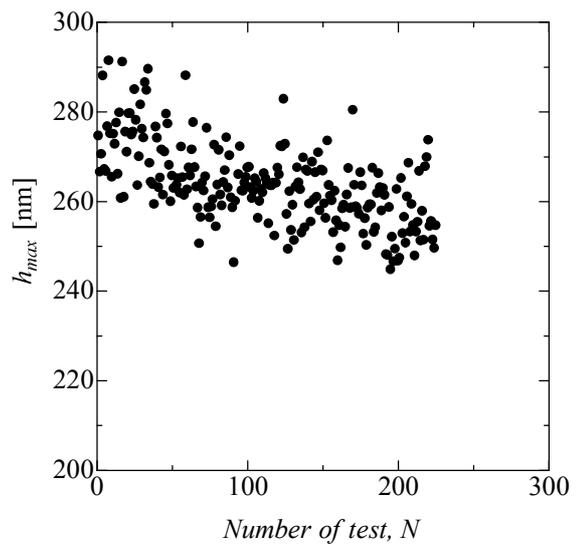


Fig. 4. Plot of maximum depth  $h_{max}$  against number of tests (UMV500, No.1 tester).

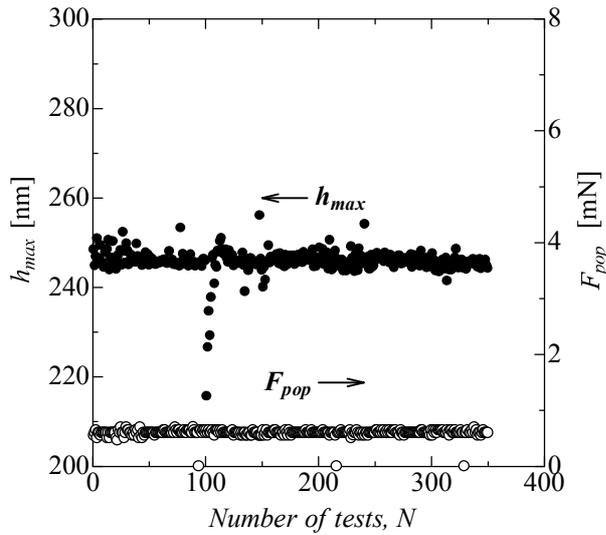


Fig. 5. Plots of pop-in force  $F_{pop}$  and maximum depth  $h_{max}$  against number of tests (tungsten, No.2 tester).

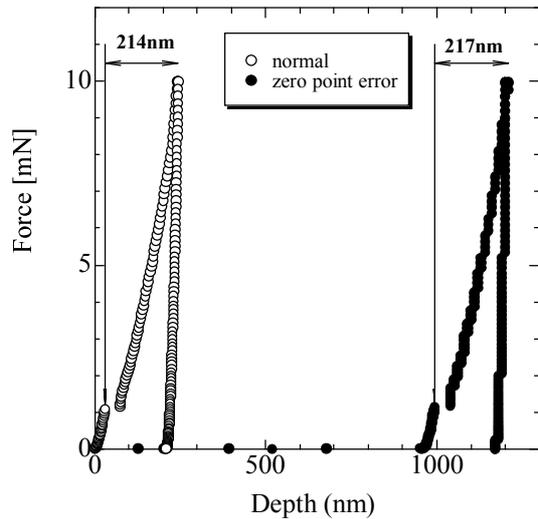


Fig. 6. Force-depth curves with and without zero point detection error (tungsten, No.2 tester).

#### 4.2. Discussion on other applications of tungsten

In some situations a nanoindentation tester fails to detect a zero point of force-depth curve. This error gives a force-depth curve an offset and therefore causes a wrong value of hardness. Figure 6 shows an obvious example obtained with No.2 tester. The curve with zero point error is apparently shifted by a large offset. Even in that case, the difference between  $h_{pop}$ , the pop-in depth just before pop-in occurs, and the maximum depth  $h_{max}$  is not much affected by this offset as shown in Fig. 6, since the offset is cancelled by subtraction. Monitoring this parameter,  $(h_{max} - h_{pop})$ , by tungsten or any pop-in material may help to check reliability of zero point detection or to calibrate doubtful data. For example, if the difference in depth is unchanged and maximum depth changes measurements by measurements, it is recommended to consider improving the zero point detection of the system.

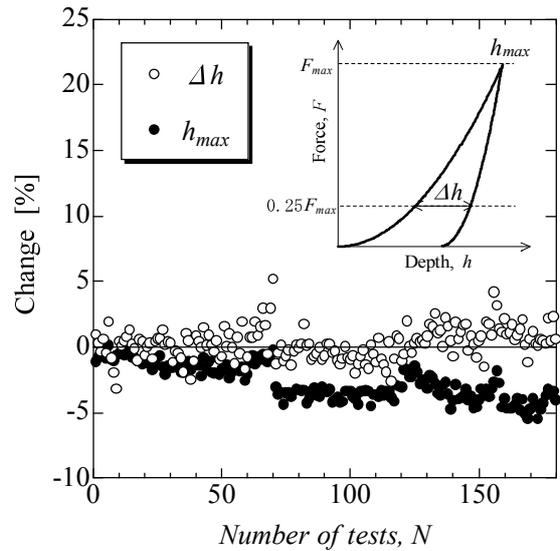


Fig. 7. Change in percentage of  $h_{max}$  and  $\Delta h$  (tungsten, No.1 tester).

This idea, i.e., use of the difference in depth, is one of the keys of a new hardness definition proposed by the authors [2]. The new hardness is based on  $\Delta h$ , the difference in depths between loading and unloading at the same force level (see the insert in Fig.7). Figure 7 shows the comparison between the maximum depth and the above-mentioned  $\Delta h$ . The maximum depth decreases by 5% at 150th tests as shown in Fig. 3, which means the area function varies and calibration process is necessary for each stage of experiments. On the other hand, the change of  $\Delta h$  is negligible and  $\Delta h$  can be regarded as 'constant' as a first approximation. It may seem strange that this simple difference method works to calibrate the change of area function, but the reason may lie in the fact that the area function is often approximated by an offset [3] and the difference method can cancel any offset error. The details of the new hardness are described elsewhere [2]. Note that the uniform properties of tungsten single crystal are also preferable in the new hardness method and therefore tungsten single crystal can be a standard specimen for the new hardness method, even if the pop-in becomes a disadvantage for calculating a difference,  $\Delta h$ .

## 5. CONCLUSION

The stability of nanoindentation system is inspected by tungsten single crystal and one of the two tested instruments showed the gradual increase of pop-in load and decrease of maximum depth. It is concluded that tungsten single crystal is appropriate to check stability of nanoindentation measurement by two reasons: (1) It is uniform enough to detect changes of depth in several nanometers. (2) Pop-in force is quite sensitive to the tip radius of indenter. Other applications of tungsten are also discussed and it is concluded that tungsten single crystal has potentials as hardness standard.

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