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INSTRUMENTED INDENTATION TEST FOR HIGH-TEMPERATURE SPECIMEN

Takeshi Sawa [1], *Masaharu Tsujii* [2], *Masaru Kawazoe* [3]

Mitutoyo Corporation, 1-20-1 Sakado, Takatsu-ku, Kawasaki-shi, Kanagawa-ken, 213-8533, Japan
[1] takeshi_sawa@mitutoyo.co.jp, [2] masaharu_tsujii@mitutoyo.co.jp, [3] masaru_kawazoe@mitutoyo.co.jp

Abstract: Concerning the specimen heating table attached to an instrumented indentation testing machine, one of the hardness testing machines, for realizing the hardness test of heated specimen, thermal influences to the instrumented indentation testing machine by attaching the specimen heating table have been examined. Consequently, it has been clarified that test result errors are mainly induced by the fact that the thermal expansion by the increase in temperature of the indenter (the metal part fixing the diamond tip) is added to the measured indentation depth, so that the accurate indentation depth cannot be measured. Furthermore, it has been considered that the increase in temperature of the indenter is mainly caused by the thermal transfer (thermal conduction) while the indenter contacts with the high-temperature specimen, and it has been clarified that the behavior of temperature change of the indenter depends on specimen materials and testing conditions. In this paper, newly invented measures for obtaining correct indentation depth in high-temperature tests are described.

Keywords: Instrumented indentation test, High-temperature environment, Thermal expansion, Hardness

1. INTRODUCTION

Nowadays plastics and thin-film materials are essential materials for various machines and constructions. Recently these materials are often used in high-temperature environments, and therefore demands for evaluating the material characteristics in high temperature are increasing to help the development and evaluation of these materials. To satisfy these demands, we have struggled to develop a specimen heating table to be attached to hardness testing machines for realizing the hardness test of heated specimens.

On the other hand, the instrumented indentation test is optimum for evaluating plastics and thin-film materials, and to standardize its testing method, various researches have been performed [1], [2], [3] and the instrumented indentation test is now recognized in the markets. The instrumented indentation test is one of the indentation hardness tests such as the Vickers hardness test. In the instrumented indentation test, the test force, F and the indentation depth, h are measured continuously during the indentation process to evaluate various mechanical properties of specimens by analyzing the obtained F-h curve. Especially, the instrumented indentation test where the indentation depth is very small is called “Nanoindentation test” or “Ultra-micro hardness test”, and attracts attention as the evaluation method of ultra-thin films [4], [5]. Accordingly, we have developed a specimen heating table that can be mounted on the instrumented indentation testing machine. In this paper, influences to test results by mounting the specimen heating table, and the test results of heated specimens are examined.

2. INSTRUMENT

We mounted the specimen heating table on the Mitutoyo-made micro zone testing machines “MZT-500 series” that are instrumented indentation testing machines.

2.1. Specimen Heating Table

The specimen heating table consists of a specimen heating unit and a temperature control unit. The specimen heating unit includes a specimen table for heating the specimen, a specimen fixing mechanism, and a stage attaching part for attaching the heating unit to the stage of the testing machine. The specimen heating unit is connected to the temperature control unit via a heater power supply cord and a temperature sensor cord. The temperature of the specimen table is controlled by the PID at an accuracy of ±2°C related to the set temperature. The maximum set temperature of this instrument is 250°C, and the time required to increase the temperature from 20°C to 250°C is about five minutes.

Figure 1 shows the schematic internal structure of the specimen heating table. A ceramic thin heater is adopted as the heater and is arranged in such a manner that the heater directly contacts with the specimen table. In the instrumented indentation test, the thermal expansion of the specimen table and stage directly influences the indentation depth measurement. Accordingly, we use a material with low CTE (Coefficient of Thermal Expansion) and high coefficient of thermal conductivity* for the specimen table, and use a heat insulated structure between the specimen table and the stage attaching part. In addition, in the specimen fixing mechanism, we use a mechanism of fixing the specimen by spring force* to prevent the specimen pressing force from changing by the thermal expansion.

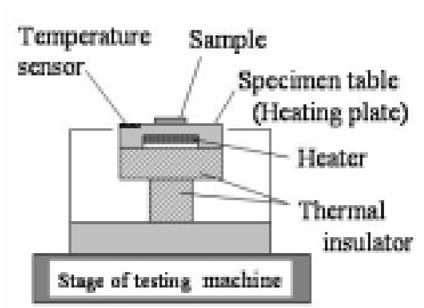


Fig. 1 Schematic structure of specimen heating table

2.2. Instrumented Indentation Testing Machine

As shown in Fig. 2, for the loading mechanism of the micro zone testing machines "MZT-500 series," a balance lever mechanism is adopted. The test force is generated by the force coil, and the indentation depth is measured by a capacitance type displacement sensor arranged in the upper side of the indenter and aligned with the indenter. In the present system, to reduce the heat applied to the loading lever and the displacement sensor, we provide a thermal shield* arranged in the lower side of the loading lever and exposing only the indenter tip. In addition, the objective lens for observing the specimen surface is a long working distance type.

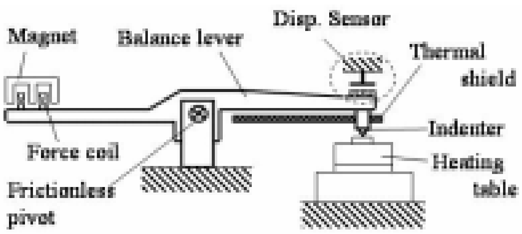


Fig. 2 Test force loading mechanism of MZT-500 series

3. EXPERIMENTS

3.1. Experimental Conditions

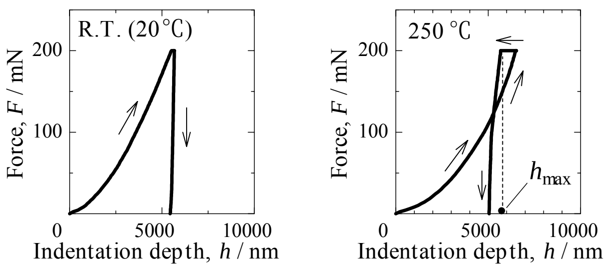
To verify high-temperature test results, we prepare two types of specimens, namely pure aluminum plate (by roll process) and fused silica plate. The dimension of the both specimens is 20 × 20 mm, and the specimen thickness is 2 mm. In the instrumented indentation test, the aluminum hardly presents elastic recovery when the test force is unloaded. Therefore, for the aluminum specimen, the indentation depth estimated from the indentation image based on the indenter's geometrical shape in the traditional Vickers hardness test substantially coincides with the maximum indentation depth of the *F*-*h* curve. Accordingly, the aluminum is optimum to verify the reliability of indentation depth measurement. On the other hand, the fused silica has a very high reproducibility at the instrumented indentation test and has appropriate elastic recovery in the *F*-*h* curve, so that the modulus of elasticity can be calculated accurately. Accordingly, the fused silica is frequently used as the reference specimen for the instrumented indentation test. In addition, the fused silica is well knows as a material with an ultra-low CTE (Coefficient of Thermal Expansion).

We perform tests for these two types of specimens at the room temperature and at a high temperature. In the high-temperature test, the specimen is heated to 250°C that is the highest set temperature of this instrument. The room-temperature test is performed in the 20°C environment. In all the tests, a triangular pyramid-shaped diamond indenter (Berkovich indenter) is used and the test force is 200 mN. In the test sequence, the loading period is 10 seconds, the test force holding period is 10 seconds, and the unloading period is 10 seconds. In addition, before experiments, we have confirmed that the test force and the observation image magnification by the microscope do not change according to the specimen temperature, by using an electronic force balance and a reference scale made of quarts.

3.2. Experimental Results

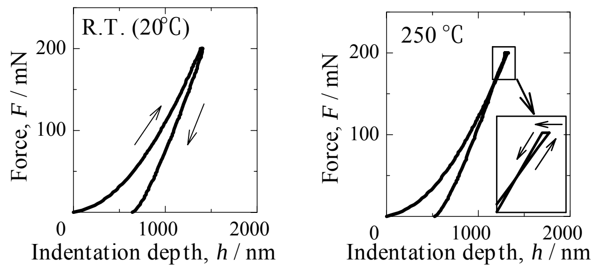
Figure 3 (a) and (b) shows the test results for the aluminum at the room temperature and at the high temperature, respectively. As shown in Fig. 3, in the high-temperature test, the displacement is largely decreased during the test force holding period.

Figure 4 shows the test results for the fused silica. As shown in Fig. 4, in the high-temperature test, the displacement is slightly decreased during the test force holding period similarly to the test results for the aluminum, but the decrease amount for the fused silica is smaller than that for the aluminum. The phenomenon that the indentation depth is decreased while the test force is kept constant is not considered correct from the material mechanics, and therefore this phenomenon suggests that the test results are influenced by attaching the specimen heating table. Furthermore, it has been clarified that the decrease amount of the indentation depth depends on the specimen material and the indentation depth.



(a) Room temperature **(b) High temperature**

Fig. 3 *F-h* curve for aluminum specimen



(a) Room temperature **(b) High temperature**

Fig. 4 *F-h* curve for fused silica specimen

4. DISCUSSION

4.1. Factors of Decrease in Displacement

One of the reasons of the phenomenon shown in Fig. 3 (b) and Fig. 4 (b) is that since the stage and the specimen heating table are thermally expanded during the hardness test, the indenter is lifted so that a decrease in the indentation depth is output virtually. In addition, if the indenter (the metal part fixing the diamond tip) thermally expands, a similar result is obtained. However, if the thermal expansion of the stage and specimen heating table is the cause, as the time elapses after turning on the power of the specimen heating table, the temperature becomes stable so that the decrease amount of the indentation depth will decrease. Actually, as the time elapses, the decrease amount of the indentation depth during holding the test force is decreased, but the decrease amount does not become zero and is saturated in two or three hours. Accordingly, to examine the cause of the decrease in indentation depth, we measured temperatures of every portions of the hardness testing machine during the actual testing. Consequently, it was found that the temperature of the indenter becomes extremely high during the hardness testing.

Figure 5 shows the measurement results of the side-surface temperature of the indenter at the position spaced from the indenter tip by 3 mm, during the instrumented indentation test for the high-temperature specimen. The temperature is measured by a radiation thermometer. As the indenter is penetrated, the temperature is increased. The temperature increase amount for the aluminum specimen is greatly larger than that for the fused silica specimen. The indenter temperature at the beginning of the indentation is about 42°C for the both materials, and this temperature hardly changes when the indenter is held so that the indenter slightly contacts with the specimen. Accordingly, the degree of increase in temperature depends on the specimen material and the contact area between the indenter and the specimen, which area is related to the indentation depth.

For the high-temperature test, a method where the test force is held in the latter half of the unloading period and the changing ratio of the displacement during the holding interval is assumed as the thermal drift amount so that the whole test result is corrected, and an indenter pre-heating method are frequently used.[6], [7] The former method is introduced as the general thermal drift correcting method in the room-temperature tests by the ISO standard[3]. However, the both methods assume that the indenter is not expanded during the indentation test, namely assume that the indenter temperature does not change. Accordingly, these methods cannot be used in the high-temperature tests for materials such as aluminum where the indenter temperature largely changes during the indentation test.

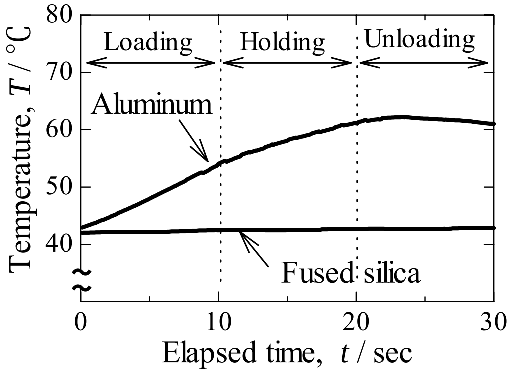


Fig. 5 Temperature change of indenter during high-temperature test

4.2. Measures for Solving Issues

As measures for solving the thermal expansion of the indenter, we have invented two measures, namely 1) Method of continuously measuring the temperature of the indenter during the indentation test to correct the thermal expansion amount based on the measured indenter temperature *, and 2) Method of selecting an appropriate indenter material * to reduce the thermal expansion amount.

1) Assuming the increase in indenter temperature as ΔT, the corrected indention depth, h_{cor} is expressed in the following equation using a coefficient, α.

$$h_{cor} = h + \alpha \Delta T \quad (1)$$

One method of obtaining the coefficient α is to use the dimension of the indentation. Assuming that the length, L of one side of the indentation is proportional to the indentation depth, determine the α in such a manner that the ratio between the maximum indentation depths in the F-h curve at the room temperature and the high temperature is equal to the ratio between indentation lengths. Note that in the Eq. (1), it is assumed that the temperature distribution of the indenter is uniform through the inside of the indenter at the temperature of the measuring point.

2) Use a low CTE material such as Invar, or a low thermal conductivity material such as ceramics as the indenter material. As the tip material of the indenter, the conventional diamond having high stiffness is effective, but since the diamond has a very high coefficient of thermal conductivity, other materials such as sapphire having lower coefficients of thermal conductivity than diamond are effective for soft materials.

4.3. *Execution of Measures*

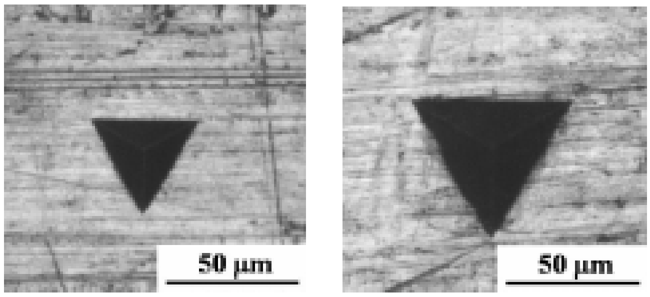
1) The measures using the Eq. (1) is verified. Figure 6 (a) and (b) shows the indentation images for the aluminum at the room-temperature test and at the high-temperature test, respectively. The average length, L of the sides of the indentation read from Fig. 6 (a) is 41 μm. By assuming that the indentation form is similar to the indenter form, the indentation depth, h can be estimated according to the Eq. (2) that is induced from the geometrical form of the Berkovich indenter.

$$h = \frac{L}{2\sqrt{3}\ \tan\ 65\ °} \qquad (2)$$

According to the Eq. (2), h is calculated to be 5520 nm that well coincides with the maximum indentation depth (h_{max} = 5650 nm) in Fig. 3 (a) by an error of 2.3 %. Thus, for aluminum specimens, the indentation depth estimated from the indentation image based on the geometrical indenter form substantially coincides with the maximum indentation depth, h_{max} in the F-h curve. On the other hand, L read from Fig. 6 (b) is 60 μm. According to the Eq. (2), h is calculated to be 8080 nm that largely differs from h_{max} in Fig. 3 (b). Therefore, to perform the correction indicated by the Eq. (1), the coefficient, α is obtained in such a manner that h_{max} in Fig. 3 (b) becomes 8080 nm using the result shown in Fig. 5. Consequently, α is 140. By using this value of α and by measuring the indenter temperature during the indentation process shown in Fig. 5, high-temperature test results shown in Fig. 3 (b) and Fig. 4 (b) can be corrected. Note that since the α value changes when the temperature measuring position changes, it is necessary to perform indentation tests while measuring the indenter temperature at the same measuring position as the measuring position used for obtaining the α value.

2) The material of the indenter (the metal part fixing the diamond tip) is changed from stainless steel to Super Invar, and experiments are performed.

Figures 7 and 8 show the results, where the above two types of measures are applied, for aluminum specimen and fused silica specimen, respectively. The thick dashed line shows the corrected result of Fig. 3 (b) and Fig. 4 (b) by using the Eq. (1), and the thick solid line shows the test result by changing the indenter material. In addition, the thin dashed line shows the room-temperature result, namely Fig. 3 (a) and Fig. 4 (a), to make a comparison. The experiment results by the two types of measures substantially coincide to each other. Accordingly, the invented two types of measures are appropriate.



(a) Room temperature **(b) High temperature**

Fig. 6 Indentation image of aluminum specimen by indentation test

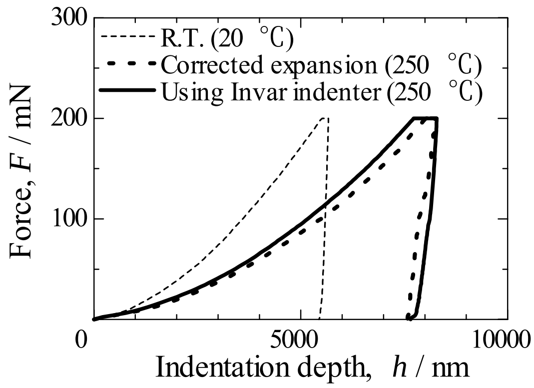


Fig. 7 *F-h* curve for aluminum specimen where measures for solving thermal expansion of indenter are applied, and data at room temperature

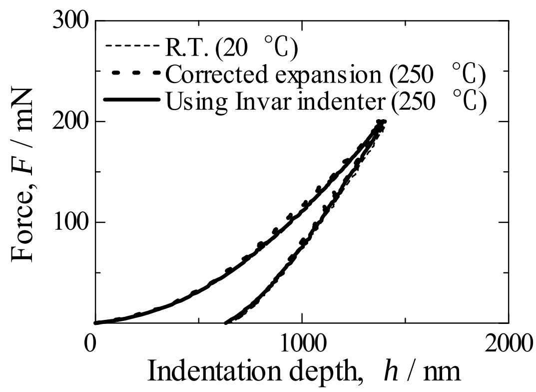


Fig. 8 *F-h* curve for fused silica specimen where measures for solving thermal expansion of indenter are applied, and data at room temperature

The test results for aluminum specimen and fused silica specimen at high temperature shown in Figs. 7 and 8 are summarized. When the aluminum specimen temperature is increased from 20°C to 250°C, h_{max} in the *F-h* curve is increased by about 43 %, which corresponds to a decrease of about 86 % in hardness. On the other hand, the *F-h* curve for fused silica specimen hardly changes between at 20°C and at 250°C, so that the hardness hardly changes. According to Beake and Smith [7], the temperature coefficient for the fused silica hardness is induced to be -1.1MPa/°C by experiments. Therefore, according to their theory, when the specimen temperature is increased from 20°C to 250°C, the hardness is decreased by 253 MPa. By assuming that the hardness of fused silica at the room temperature is 8930 MPa [7], the decrease ratio of hardness is 2.8 %, which corresponds to a slight increase of 1.4 % in the h_{max} value.

5. CONCLUSION

We have developed a specimen heating table attached to the instrumented indentation testing machine, one of the hardness testing machines, for performing the hardness test of heated specimens. In the high-temperature hardness tests, the test result is largely influenced by the increase in indenter temperature caused by the thermal conduction from the high-temperature specimen, and therefore the correct indentation depth cannot be measured due to the thermal expansion. As the measures for solving this issue, we have invented the following two methods.

1) Measuring the indenter temperature continuously during the indentation test to correct the thermal expansion amount based on the measured indenter temperature

2) Selecting an appropriate indenter material to reduce the thermal expansion amount

We have performed instrumented indentation tests for pure aluminum specimen and fused silica specimen at the room temperature and high temperature, and by analyzing the obtained *F-h* curves and the indentation dimensions, the above two measures have been verified to be appropriate. It has been found that when the specimen temperature is increased from 20°C to 250°C, the hardness of the aluminum is reduced by about 50 %, but the hardness of the fused silica hardly changes. We plan to accumulate data by performing high-temperature tests for plastic materials and thin-film materials, for which the instrumented indentation test is effective.

*: Patent pending

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