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APPLICATION OF CONTINUOUS MULTIPLE LOADING WITH SPHERICAL-TIP NANOIDENTATION TO MECHANICAL PROPERTY EVALUATIONS OF INDUSTRIAL MATERIALS

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Abstract – There are increasing demands for measuring mechanical properties of small volumes of metal or small-sized materials used for Micro-Electro-Mechanical-Systems (MEMS). This is basically because such mechanical properties are closely related to the strength and reliability of the metal or systems. In this work, a nanoindentation technique with a spherical-tip is applied to the evaluation of elastic-plastic properties such as stress-strain relationship of industrial materials such as steel and aluminium. A continuous multiple loading method is employed to determine the stress-strain curve. A set of 21 times of loading/unloading sequences with increasing terminal load are made and load-displacement curves with the different terminal loads from 0.1 mN to 100 mN are then continuously obtained and converted to a stress-strain curve. The influence of measurement condition on the obtained stress-strain curve is examined and an appropriate condition is determined. Based on the examinations, stress-strain curves have been estimated for steels and aluminium. It has been found that the estimated stress-strain curves correlate closely with those measured by tensile or compression tests when the influence of the residual stress of the specimen surface is taken into account. In addition, some issues affecting the results are discussed.

Keywords: spherical indentation, stress-strain curve, continuous multiple loading

1. INTRODUCTION

In the fields of science and engineering, there are growing demands for measuring materials properties of small-sized materials and thin films used for Micro-Electro-Mechanical-Systems (MEMS) as well as any other industrial constructions. In particular, mechanical properties which are directly related to the elastic and plastic behaviours are strongly required to be measured because accurate values of such properties are indispensable for better machine design and CAE applications with higher reliability. Unfortunately, there is little knowledge about such microscopic mechanical properties even for widely used industrial materials such as steels, aluminium, and other metals. This is basically because of difficulty in measuring mechanical properties of materials in submicron or submillimeter volumes.

Nanoindentation has been known as a sophisticated technique providing effective measurements of both hardness and elastic modulus at a small volume on a material surface [1][2]. In fact, the hardness and Young's modulus obtained by nanoindentation can be used as representative values for plastic and elastic properties, respectively, in machine designs, CAE analyses, and materials science researches. However, those values are not always good enough for practical applications. It is still very attractive to know a stress-strain relationship which is directly related to material deformation behaviour in elastic and plastic regions, and therefore strongly required to measure it.

Due to the importance of determining tensile materials properties from the experimentally measured indentation load-depth curve, in the past decade, numerous researches on the application of nanoindentation to the estimations of mechanical properties have been made extensively [1]-[16]. In these investigations, spherical indenters are often used and attempts have been made to determine stress-strain curves. Among them, a continuous multiple indentation method is being tried to determine tensile properties of industrial materials [3][4][7][9][11]. The method called "multiple partial unloading" or "continuous multiple loading" seems to be one of the promising candidates to determine tensile properties. It is noted that the standardization of the method is being considered. The optimum values for experimental parameters used in the method were examined to determine indentation tensile properties quantitatively [9]. Although the method is attractive and has the potential for the tensile properties determination, there remains unclear whether the method is appropriate for practical uses or not. In this work, the continuous multiple loading method is applied to stress-strain curve estimation of a steel and the influence of measurement condition on the obtained stress-strain is examined. Based on the examinations, stress-strain curves of steels and aluminium are estimated.

2. DETERMINING STRESS AND STRAIN

When a spherical indenter penetrates into a material as shown in Fig. 1, the deformation behaviour is classified into three stages of elastic, elastic-plastic, and fully plastic. When the stress field due to the indentation satisfies the

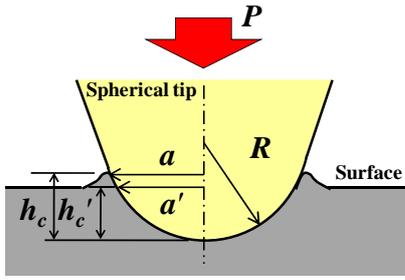


Fig. 1. Schematic of spherical indentation.

yield condition, local plastic deformation starts to occur beneath the spherical indenter and expands to free surface. The mean contact pressure beneath the indenter then increases rapidly. The expanded semi-spherical plastic zone grows into the surrounding elastic zone as the indenter penetration depth increases. The mean contact pressure gradually increases in the fully plastic zone. This deformation behaviour is similar to the work-hardening behaviour during uniaxial tensile test. When the indenter penetration proceeds, so-called “piling-up” or “sinking-in” phenomenon occurs at the edge of indenter and leads to consistent change in contact area for a given depth. It is noted that Fig. 1 represents a situation with piling-up, where, R is the radius of spherical tip, a and h_c are the contact radius and contact depth with considering piling-up, respectively, and a' and h_c' are those without considering piling-up.

Since the stress and strain fields beneath the indenter during loading are not homogeneous, it is reasonable to use an averaged value beneath the indenter rather than a certain value at a specific position, as suggested by Ahn and Kwon [7]. The terms of representative stress σ_R and representative strain ϵ_R are used here as an averaged stress and strain beneath the indenter, respectively.

Representative stress can be obtained from the mean contact pressure P_m to be,

$$\sigma_R = \frac{P_m}{c} = \frac{P}{c \pi a^2} \quad (1)$$

Where, P is a maximum applied load, c is related to a parameter called the constraint factor depending on deformation regime [5]. We use $c=2.8$ which corresponds to the fully plastic deformation regime [18], for simplicity. The contact radius a is determined from

$$a = \sqrt{\frac{5(2-n)}{2(4+n)}} a' \quad (2)$$

Where, n is work-hardening exponent. The value of n is calculated from the relation $P \propto K a^{(2+n)}$ which was derived from Meyer’s law, where K is a material constant. Taking logarithms of the relation, a linear regression of $\log(P)$ on $\log(a')$ gives $2+n$ as the gradient. It should be noted that the validity of this procedure is not fully verified.

Tabor showed that $\epsilon=0.2 \tan \beta$ is an appropriate expression for representative strain in conical indentation, where β is the angle between the indenter flank and the

material surface [17]. In spherical indentation, an appropriate equivalent expression is

$$\epsilon_R = 0.2 \tan \beta \approx 0.2 \sin \beta \approx 0.2(a/R) \quad (3)$$

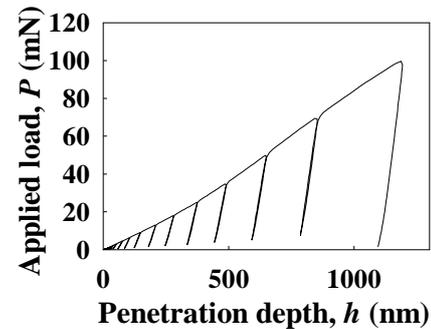
Maximum strain occurs just beneath the center of the region of contact and reduces with radial distance from the symmetry axis until at the periphery of the indentation. It is reduced to about 20 % of the maximum value [4]. Although there are other strain models proposed for the spherical indentation, the strain shown in (3) is employed in this work, for simplicity. It is considered from the empirical Tabor relation that $P_m/2.8$ and $0.2(a/R)$ approximately represent the uniaxial stress and strain, respectively.

3. EXPERIMENTAL PROCEDURES

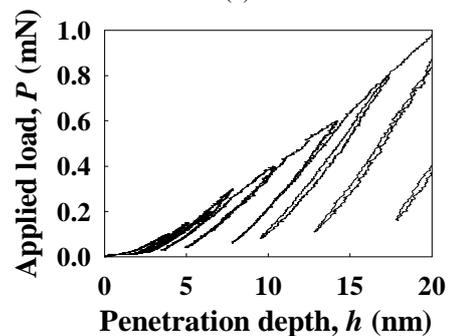
Specimens used are cold rolled carbon steel sheets (JIS G3141) and aluminium alloy sheet (JIS A2017-T351, so-called duralumin). The specimen dimension for indentation tests is about 5 x 5 x 1 mm. The specimen surface was ground and polished with 1 μm Al_2O_3 powders.

A load-controlled indentation tester ENT-2100 from Elionix Inc., Japan, is employed. The tester provides continuous multiple loading/partial-unloading indentations with load and displacement resolutions of 4 nN and 0.06 nm, respectively. A diamond spherical indenter with 9.7 μm tip radius is used for indentation.

Fig. 2 shows a typical result of continuous multiple indentations measured for a steel specimen. In the method, the load is removed not totally but partially, and then reloading and unloading are repeated until the final maximum load up to 100 mN as shown in Fig. 2(a). In this work, a terminal load at each partial loading step is



(a)



(b)

Fig. 2. Load-depth curve for continuous multiple loading, (a) the whole, (b) the detail in the beginning of loading.

increased from 0.1 mN to 100 mN (maximum), and the number of the partial unloading is set to be 21. Since both representative stress and strain can be determined from a loading/unloading curve at each step, stress-strain curve is then given as a set of 21 data points of the stress and strain.

4. RESULTS AND DISCUSSION

4.1. Reproducibility of load-depth curves

Since there are some experimental parameters which may affect the behaviour of the load-depth curves obtained by the multiple loading indentations, it is important to examine the influence of such parameters. In this work, the following parameters are examined; (i) indentation speed during loading, (ii) holding time at terminal load of each partial loading, (iii) unloading ratio to each terminal load, and (iv) value of the initial load at the first loading.

Fig. 3 shows the stress-strain curves measured for a steel specimen with different conditions of the unloading ratio. We can see that there is no influence of unloading ratio on the curves. The similar tendency was found in the influence of the holding time and the initial load. On the other hand, a measurable influence is found in the stress-strain curves measured with different indentation speeds as shown in Fig. 4. We can see that fast speed makes higher stress in the region shown by a circle in Fig. 4. This may correspond to the result that a material appears to be “harder” at higher loading rates [15].

It is also important to examine the reproducibility of the stress-strain curve. To confirm it, the same measurements

under the condition of indentation speed 0.02 mm/min, holding time 0 s, unloading ratio 90%, and initial load 0.1 mN were repeated twenty times. The averaged stress-strain curve and its 68% confidence interval are shown in Fig. 5.

4.2. Evaluations of steels and aluminum

Stress-strain curves of steels and aluminum are evaluated by the multiple loading/unloading indentations under the condition mentioned above. The stress-strain curve measured for a steel specimen is shown in Fig. 6. It is found that the nanoindentation result depicted by black circles shows extremely higher values than those obtained by uniaxial tensile and compression tests. It is observed from an x-ray diffraction measurement that compressive residual stress of -180 MPa exists on the specimen surface even after fine polishing. Since the depth of the existing region of the residual stress is considered to be a few micron at least, the residual stress must affect the nanoindentation result. Based on this consideration, 180 MPa is simply subtracted from the measured stress values and such corrected result is depicted by white circles in Fig. 6.

Three kinds of steels (JIS G3141 2B, 4B, SB) with different heat treatments have been evaluated and the obtained stress-strain curves are shown in Fig. 7. We can see a remarkable difference among the curves, depending on the heat treatment. It is highly probable that stresses measured here include the contribution from the residual stress on the specimen surface. Nevertheless, we can see an interesting fact that the stress level corresponds to that of the tensile

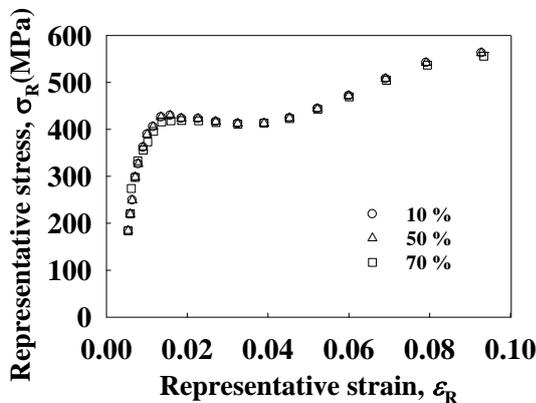


Fig. 3. Influence of unloading rate on the stress-strain curve.

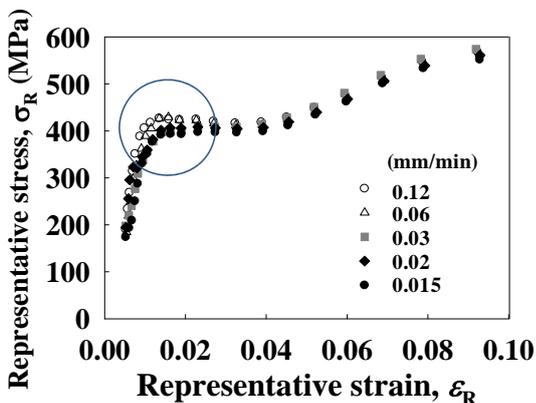


Fig. 4. Influence of indentation speed on the stress-strain curve.

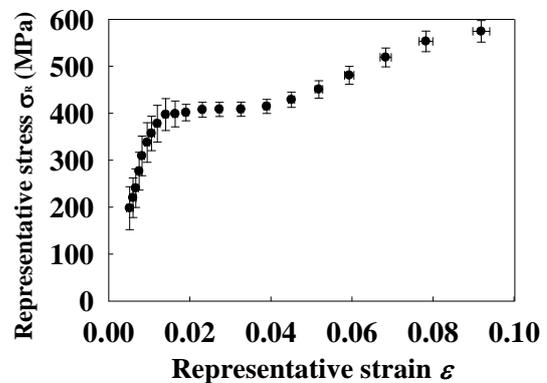


Fig. 5. Reproducibility in measuring stress-strain curve.

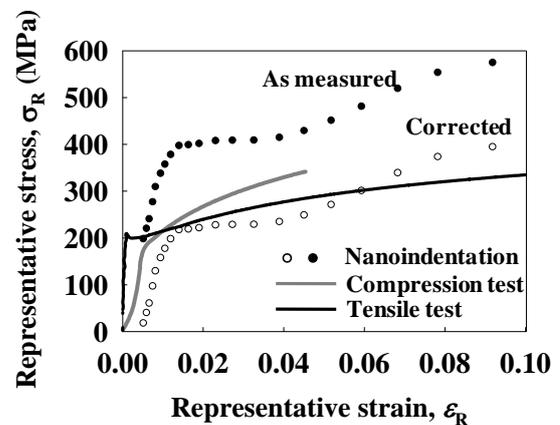


Fig. 6. Comparison between stress-strain curves measured by nanoindentation and uniaxial tests.

strength of each material. The same indentation test is applied to the aluminium alloy and the similar result to the steel is obtained as shown in Fig. 8. It is noted that there remain other factors affecting the stress-strain curve, such as not only the size but also the irregularity and geometry of spherical tip, morphology of material surface before and after indentation, and environmental effect in a laboratory. Clarifying the influence due to such factors will be the future works.

4. CONCLUSIONS

Multiple loading/unloading indentations with spherical indenter have been applied to evaluations of steels and aluminium, to determine stress-strain curves. It has been found that the method is useful to obtain unique results reflecting mechanical properties at a small volume if appropriate conditions are employed in the measurements.

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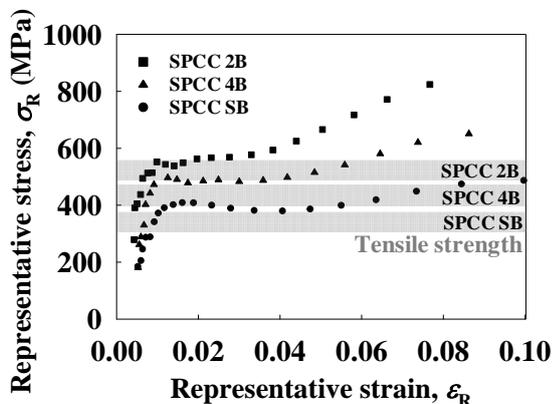


Fig. 7. Stress-strain curves measured for three kinds of steels.

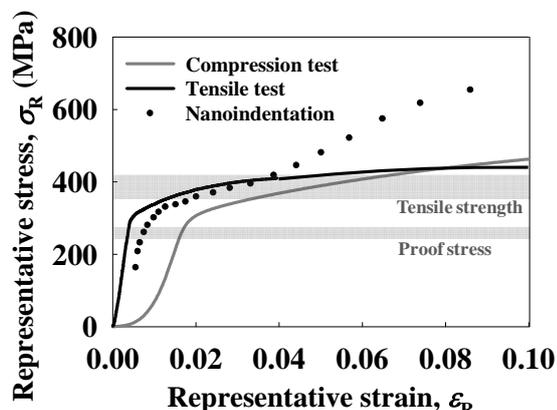


Fig. 8. Stress-strain curves measured for the aluminium alloy.

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