Eun-chae Jeon
Division of Nano-Mechanical Systems,
Korea Institute of Machinery & Materials,
Daejeon, 305-343, Korea
e-mail: jeonec@kimm.re.kr

Joo-Seung Park
Technology & Standards Policy Division,
Korea Agency for Technology and Standards,
Gwacheon, 427-723, Korea
e-mail: joospark@ats.go.kr

Doo-Sun Choi
Division of Nano-Mechanical Systems,
Korea Institute of Machinery & Materials,
Daejeon, 305-343, Korea
e-mail: choids@kimm.re.kr

Kug-Hwan Kim
e-mail: samadhi0@snu.ac.kr

Dongil Kwon
e-mail: dongilk@snu.ac.kr

School of Materials Science and Engineering,
Seoul National University,
Seoul, 151-742, Korea

A Method for Estimating Uncertainty of Indentation Tensile Properties in Instrumented Indentation Test

The instrumented indentation test, which measures indentation tensile properties, has attracted interest recently because this test can replace uniaxial tensile test. An international standard for instrumented indentation test has been recently legislated. However, the uncertainty of the indentation tensile properties has never been estimated. The indentation tensile properties cannot be obtained directly from experimental raw data as can the Brinell hardness, which makes estimation of the uncertainty difficult. The simplifying uncertainty estimation model for the indentation tensile properties proposed here overcomes this problem. Though the influence quantities are generally defined by experimental variances when estimating uncertainty, here they are obtained by calculation from indentation load-depth curves. This model was verified by round-robin test with several institutions. The average uncertainties were estimated as 18.9% and 9.8% for the indentation yield strength and indentation tensile strength, respectively. The values were independent of the materials' mechanical properties but varied with environmental conditions such as experimental instruments and operators. The uncertainties for the indentation yield and tensile strengths were greater than those for the uniaxial tensile test. These larger uncertainties were caused by measuring local properties in the instrumented indentation test. The two tests had the same tendency to have smaller uncertainties for tensile strength than yield strength. These results suggest that the simplified model can be used to estimate the uncertainty in indentation tensile properties.

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Keywords: instrumented indentation test, uncertainty, indentation tensile property

1 Introduction

The instrumented indentation test (IIT) can evaluate several kinds of mechanical properties by recording variation in indentation load and indentation depth. The IIT covers a wide range in indentation load, from hundreds of newtons to a few millinewtons, covering the range from bulk scale to nanoscale. Its nondestructive nature and extremely few limitations on specimen condition enable the IIT to replace many conventional tests, among them are the tensile test and fracture test. Because of the IIT's many advantages, it has been investigated as a method of evaluating hardness [1,2], elastic modulus [3,4], tensile properties [5–7], fracture toughness [8,9], and residual stress [10,11]. Recent research has focused on using IIT to evaluate tensile properties, defined as indentation tensile properties, using spherical indenters. A national standard [12] has already been developed, and the ISO technical report [13] was legislated.

Although the international standard is currently under development, research on uncertainty estimation has not yet been presented. Uncertainty estimation is essential for standardization of material testing methods because it is an index of reliability. However, not only is uncertainty estimation very difficult, but the method for evaluating indentation tensile properties has several calibration and calculation stages that influence the uncertainty. Therefore, a simplifying uncertainty estimation model for the indentation tensile properties is developed here and verified by round-robin test (RRT) with several institutions. Some researchers have used round-robin tests in the standardization [14,15], but this work focused on hardness and elastic modulus, not on the indentation tensile properties and uses pointed indenters, not spherical indenters.

2 Theoretical Analysis

2.1 Evaluation of Indentation Tensile Properties.

Indentation tensile properties are, according to the ISO technical report [13], defined as "mechanical properties of materials such as indentation yield strength, indentation tensile strength, and indentation work-hardening exponent obtained by analyzing the true stress-strain curve determined by instrumented indentation test." The indentation tensile properties can be matched one-to-one with the conventional tensile properties measured by uniaxial tensile tests. If the uniaxial tensile test cannot be applied on a certain specimen, the indentation tensile properties can be reported as the general tensile properties of the specimen.

The indentation tensile properties can be evaluated by previous research [5,6] and the national standard [12,13]; here the method for doing so is concisely explained. Tensile properties are a relationship between true strain and true stress. The true strain in IIT is defined as a tangent function of a half angle of the spherical indenter in Fig. 1

$$\varepsilon = \alpha \tan \gamma = \frac{\alpha}{\sqrt{1-(a_c/R)^2}}\frac{a_c}{R} \qquad (1)$$

where α is a proportional constant and has a value of 0.12 [16]. The tangent function is a maximum shear strain derived by differentiating the displacement in the depth direction (u_z) [5]. A sine function proposed by Tabor [17] is widely used for the true strain.

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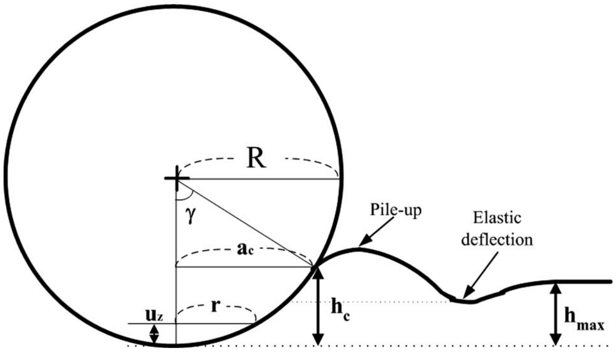

Fig. 1 Schematic of material deformation around a spherical indenter

However, it has been shown in the author's previous research [6] that the tangent function describes Hollomon-type work-hardening characteristics better than the sine function. The ISO final draft accepts the tangent function as the definition of true strain [13], and hence the tangent function was adopted in this research. It is well known that the true stress is proportional to mean contact pressure in a fully plastic region [17]

$$\sigma = \frac{1}{\Psi}\frac{L}{\pi a_c^2} \tag{2}$$

L is the indentation load, and Ψ is a constraint factor with value 3.0 [5,17,18].

The true strain and the true stress are inserted into the Hollomon equation

$$\sigma = K\varepsilon^n \tag{3}$$

where K is a strength coefficient and n is a work-hardening exponent. They can be determined by log-scale linear fitting of a few pairs of true strain and true stress from an indentation load-depth curve. Finally, the indentation yield strength (σ_y) and the indentation tensile strength (σ_{UTS}) are calculated by defining a yield strain (ε_y) and a uniform strain (ε_u), respectively. With the 0.2% offset concept, the indentation yield strength is defined as

$$\sigma_y = K\varepsilon_y^n = E(\varepsilon_y - 0.002) \tag{4}$$

where E is an elastic modulus, which can be evaluated by the Oliver–Pharr method [4]. The uniform strain is assumed to be same with the work-hardening exponent based on instability in tension [19]. The indentation tensile strength can be obtained by

$$\sigma_{\mathrm{UTS}} = K\varepsilon_u^n = Kn^n \tag{5}$$

2.2 How to Estimate Uncertainty in Indentation Tensile Properties.

"ISO guide to the Expression of Uncertainty in Measurement" [20] is widely used to estimate the uncertainty of testing methods. Based on "ISO guide," American Association for Laboratory Accreditation (A2LA) published a similar guidance [21]. The guidance of A2LA defines the final property, generally the goal of the testing method as a *measurand*. If the measurand can be expressed as $y=f(x_1,x_2,\ldots)$, the parameters $(x_1,x_2,\ldots)$ are defined as *influence quantities*. The sum of the uncertainties of all influence quantities is the uncertainty of the measurand. The measurands of this study are the indentation yield strength and the indentation tensile strength. Their mathematical models are Eqs. (4) and (5), respectively.

Generally, the influence quantities are experimental data, which can be obtained directly from experiments, and there is an equation consisted of the measurand and the influence quantities. However, the mathematical model of the indentation yield strength consists of the strength coefficient (K), the yield strain (ε_y), and the work-hardening exponent (n), which are not experimental data but calculated from experimental data. It is assumed in this study that strength coefficient (K), the yield strain (ε_y), and the work-hardening exponent (n) can be regarded as experimental data, and they contain all uncertainty of experimental procedures and calculation procedures. Therefore, they were defined as the influence quantities. Each A-type uncertainty (u_K, u_n, and $u_{\varepsilon y}$) was calculated by

$$\bar{x} = \frac{1}{j}\sum_{i=1}^{j} x_i \tag{6a}$$

$$u_x = \sqrt{\frac{\sum_{i=1}^{j}(x_i - \bar{x})^2}{j-1}} \tag{6b}$$

where x_i is experimental data and j is a number of experiments. Next, the sensitive coefficients (c_K, c_n, and $c_{\varepsilon y}$) were obtained by

$$c_K = \frac{\partial \sigma_y}{\partial K}, \quad c_n = \frac{\partial \sigma_y}{\partial n}, \quad \text{and} \quad c_{\varepsilon_y} = \frac{\partial \sigma_y}{\partial \varepsilon_y} \tag{7}$$

The sensitive coefficient is a kind of weighting value when summing all uncertainties of the influence quantities. Using Eq. (6) and (7), the combined standard uncertainty (u_c) was defined as

$$u_c^2 = \sum_i c_i^2 u_i^2 = c_K^2 u_K^2 + c_n^2 u_n^2 + c_{\varepsilon_y}^2 u_{\varepsilon_y}^2 \tag{8}$$

This equation is valid when the parameters are not correlated [20]. However, physical parameters (not chemical parameters) can be regarded independent. Sometimes the combined standard uncertainty is used as the final uncertainty; however, the expanded uncertainty (U) is more reliable in estimating the uncertainty of a measurand

$$U_{\sigma_y} = ku_c = \sqrt{c_K^2 u_K^2 + c_n^2 u_n^2 + c_{\varepsilon_y}^2 u_{\varepsilon_y}^2} \tag{9}$$

The value of k is a coverage factor and appears in Student's t-table. Generally, k is from 2 to 3 in case of 95% confidence interval; for example, k is 2.87 when the degree of freedom is 4. The expanded uncertainty in Eq. (9), $U_{\sigma y}$, is the uncertainty of the indentation yield strength, the target of this study. Finally, the uncertainty is reported either as an absolute value or a relative percentage of an average value of the indentation yield strength in Eq. (10)

$$\text{uncertainty}(\%) = \frac{U_{\sigma_y}}{\overline{\sigma_y}} \times 100 \tag{10}$$

The relative percentage is better when comparing the uncertainties of several testing methods. The uncertainty of the indentation tensile strength can be estimated by a similar procedure from Eq. (5). The influence quantities are the strength coefficient and the work-hardening exponent.

3 Experimental Procedures

RRT was performed with five Korean institutions in order to verify the proposed model and to improve the reliability of the experiments. The five institutions were Seoul National University (SNU), Korea Electric Power Research Institute (KEPRI), Posco, Doosan Heavy Industries and Construction Co. Ltd. (DHIC), and Korea Gas Corporation (KOGAS). The commercial metals Al70XX, API X65, SCM400, STD11, and S45C were used for RRT. All materials followed power law hardening in Eq. (3), which was confirmed by analysis of uniaxial tensile curves. They were machined to $25\times25\times20$ mm^3, carefully polished with 1 μm alumina carefully and distributed to each institution. The same IIT experiments were performed under the conditions of a newly prepared standard of procedure (SOP) according to Korean Standard B0950 for minimizing experimental errors. The maxi-

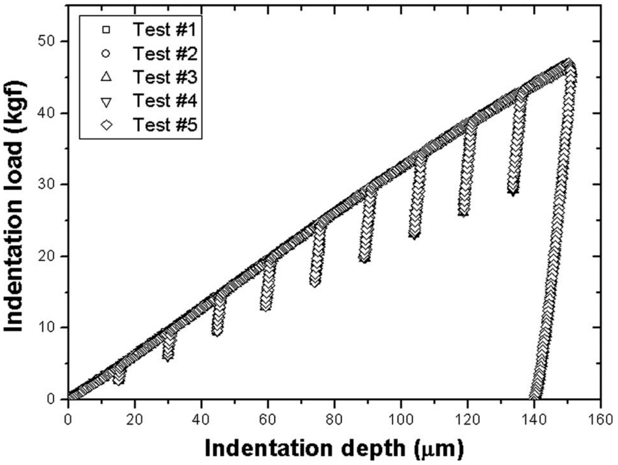

Fig. 2 Five indentation load-depth curves for S45C steel from KEPRI

mum indentation depth was 150 μm. The spherical indenter diameter was 500 μm and made of WC (tungsten carbide). Since the maximum indentation depth was fixed for all materials, the maximum force varied from 350 N to 700 N depending on the materials' mechanical properties. The instruments were provided by the same company and have 0.98N and 0.2 μm resolution.

4 Results and Discussion

The five indentation load-depth curves for S45C steel obtained by KEPRI are displayed in Fig. 2. Their reproducibility is great enough that they seem to be just one curve. Most of other curves from the other institutions showed similar reproducibility. The five experimental data sets for each material from each institution were analyzed by the method in Sec. 2.1 to yield the indentation tensile properties, and the uncertainties of IIT were estimated on the basis of these properties (a few data were not analyzed because they contained uncontrolled experimental errors). Then the uncertainties of the indentation tensile properties were estimated. The results are presented in Table 1 (indentation yield strength) and Table 2 (indentation tensile strength); the institutions are identified only by numbers to prevent unnecessary misunderstanding. The uncertainties of the indentation yield strength range from 6.2% to 30.9%, and the average value is 18.2%. The uncertainties of the indentation tensile strength are from 3.2% to 22.8%, and the average value is 9.8%. Generally, the uncertainties of the indentation yield strength are about double those of the indentation tensile strength because of the rapid variation in the true stress at the yield point, as discussed in Sec. 4.2.

Table 1 Estimated uncertainties of indentation yield strength

Institute	Al70	STD11	S45C	SCM400	API X65
1	10.3	15.2	21.5	27.5	18.8
2	6.2	9.2	14.2	12.6	25.8
3	11.1	-	21.6	27.8	25.6
4	30.2	8.1	30.6	30.9	-
5	20.9	15.2	15.3	12.1	13.1

Table 2 Estimated uncertainties of indentation tensile strength

Institute	Al70	STD11	S45C	SCM400	API X65
1	3.7	7.2	11.1	17.6	6.7
2	3.2	3.5	6.6	6.2	10.6
3	5.1	-	10.9	12.6	11.9
4	22.8	5.2	14.8	16.8	-
5	18.2	8.8	7.6	9.5	7.9

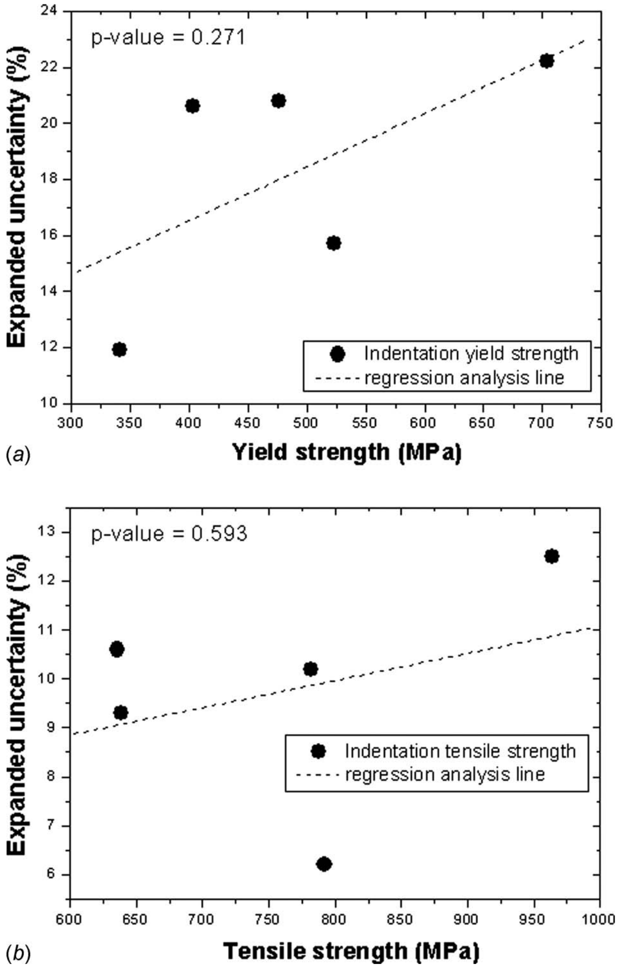

Fig. 3 Regression analysis of uncertainties with (*a*) indentation yield strength and (*b*) indentation tensile strength

4.1 Effects on Uncertainty of Material Properties and Environmental Conditions. As described in Eq. (10), the uncertainty includes an average value of the mechanical properties. Therefore, the uncertainty in this study (a relative value) could be influenced by the material's mechanical properties. For example, the uncertainty seems to be small if the mechanical properties of the material are large. It should be verified whether the relative values are dependent on the mechanical properties or not. A regression analysis, widely used to analyze whether or not parameters depend on each other, was done to verify the relationship of the uncertainty and the mechanical properties, the indentation tensile properties. If the p-value obtained from the regression analysis is greater than 0.05, the parameters has no linear correlation with a 95% confidence interval [22]. The regression tests were applied on the average values of the indentation tensile properties and the uncertainty. As shown in Fig. 3, the p-values are 0.271 and 0.593 for the indentation yield strength and indentation tensile strength, respectively. These values are much larger than 0.05, and thus the mechanical properties and the uncertainty expressed by the relative value have no linear correlation absolutely.

Generally, the uncertainty is affected by experimental instruments and operators [21]. The RRT of this study was performed under the same experimental conditions; however, conditions of experimental instruments and operators differed. The graph in Fig. 4 indicates that the uncertainty of institution 4 is almost double

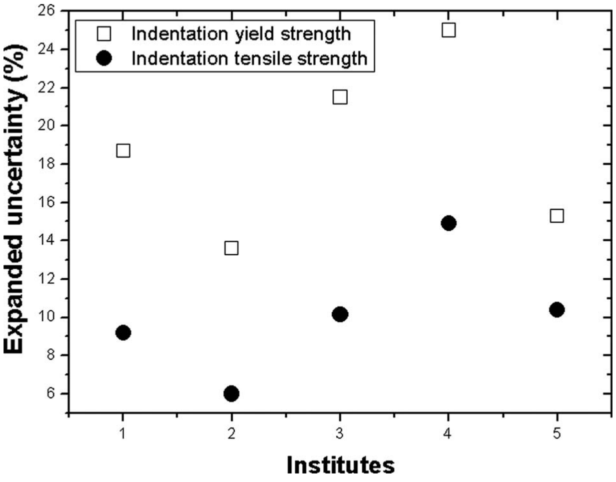

Fig. 4 Variation in uncertainties from the five institutions

the uncertainty of institution 2. Moreover, if the uncertainty of the indentation yield strength from a certain institution is small, the uncertainty of its indentation tensile strength is small, too, and vice versa. Though the two uncertainties are based on different governing equations, Eqs. (4) and (5), they show similar variation at all five institutions. This interesting point indicates a very important fact. The uncertainty estimated in this study is affected by environment conditions such as experimental instruments, operators, laboratory conditions, and the like, which are different among the institutions in this study. The uncertainty thus contains experimental uncertainties even though the influence quantities (strength coefficient, yield strain, and work-hardening exponent) in this study are not obtained directly from experiments. The assumption of this study was that the final equation contains all uncertainty including experimental procedures though the parameters of the final equation are not experimental data. This assumption is verified and confirmed in Fig. 4.

4.2 Effects of Influence Quantities on Uncertainty. The expanded uncertainty can be expressed as the sum of the uncertainty of each influence quantity. Sometimes a certain influence quantity governs the expanded uncertainty. It helps to decrease the expanded uncertainty to control the dominant influence quantity's uncertainty. The degree of contribution (DOC) of each influence quantity is defined as

$$\mathrm{DOC}(\%) = \frac{c_i^2 u_i^2}{u_c^2} \times 100 \tag{11}$$

It is clear that the sum of DOCs is 100. The DOC values of influence quantities are displayed in Tables 3 and 4 for the indentation yield strength and indentation tensile strength, respectively. The work-hardening exponent whose DOC value is 67.4% is a dominant influence quantity in the uncertainty for the indentation yield strength. On the other hand, DOC values of the work-hardening exponent are less than 10% and much smaller than DOC values of the strength coefficient for the indentation tensile strength. The work-hardening characteristic of metals explains why the exponent has totally different effects on the two mechanical properties.

Table 3 Contribution of influence quantities on uncertainties of indentation yield strength

Institute	Strength coefficient (%)	Yield strain (%)	Work-hardening exponent (%)
1	15.7	12.2	72.0
2	14.2	23.9	61.9
3	15.6	15.4	69.0
4	23.5	13.6	63.0
5	26.2	2.8	71.0
Total average	19.0	13.6	67.4

Table 4 Contribution of influence quantities on uncertainties of indentation tensile strength

Institute	Strength coefficient (%)	Work-hardening exponent (%)
1	91.8	8.2
2	94.0	6.0
3	97.2	2.8
4	93.7	6.3
5	92.1	7.9
Total average	93.8	6.2

A general uniaxial tensile curve is displayed in Fig. 5. During the uniaxial tensile test, a metal experiences linear elastic deformation at the beginning stage and then exponential plastic deformation governed by Eq. (3) after a yield point. The true stress increases very rapidly near the yield point and then increases more and more slowly as necking proceeds; finally, at the necking point, no work-hardening occurs. The true stress corresponding to the yield point and the initial necking point are the yield strength and the tensile strength, respectively. The increase in the true stress can be expressed quantitatively by differential values ($d\sigma/d\varepsilon$) of the true stress in Fig. 6. The differential value is more than 15,000 MPa near the yield point, but goes down to 1,000 MPa at the necking point. Since the variation in the true stress is very severe at the yield point, even a small change in the yield point makes a very large difference in the indentation yield strength. Therefore, the uncertainty of the indentation yield strength is larger than the uncertainty of the indentation tensile strength. Such severe variation at the yield point is affected primarily by the work-hardening exponent that is a power in the governing equation (Eq. (3)). According to Fig. 6, the differential value goes down from 20,000 MPa to 15,000 MPa, when the work-hardening exponent decreases from 0.267 to 0.132. On the other hand, the differential value near the necking point is almost independent of the work-hardening exponent. Since the differential value at the necking point is same as the tensile strength theoretically [19], the differential values at the necking point have small gaps, less than several tens or a few hundreds of MPa. The metals tested in this study have about 350 MPa differences in tensile strength. Therefore, the work-hardening exponent is not a dominant influence quantity for

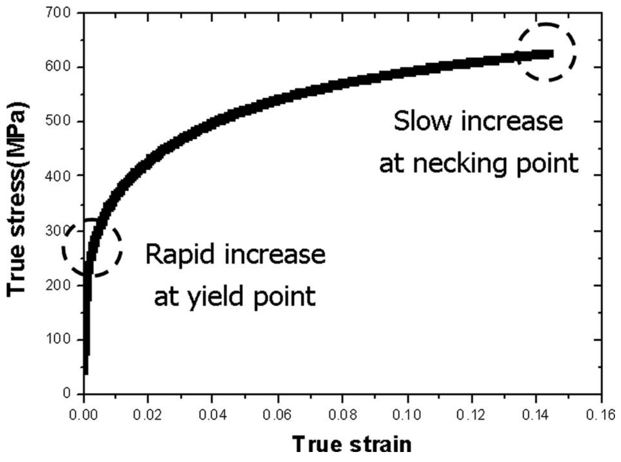

Fig. 5 General example of uniaxial tensile curves

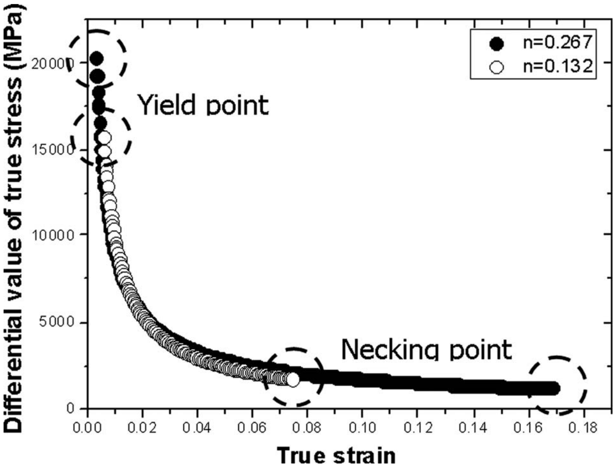

Fig. 6 Differential values of true stress of tensile curve

the uncertainty of the indentation tensile strength, but it is a dominant influence quantity for the uncertainty of the indentation yield strength.

4.3 Comparison to Uncertainty of Uniaxial Tensile Test. Since the IIT is intended to replace the uniaxial tensile test, it is meaningful to compare their uncertainties. The uncertainty of the uniaxial tensile tests has generally been less than 5% in previous research [23,24]. Loveday reported that the uncertainties of uniaxial tensile tests were 2.3–4.6% for the yield strength, based on the tests of three kinds of steels and one nickel-based alloy. Loveday applied a fixed coverage factor $k=2.0$ regardless of the degrees of freedom. However, the coverage factor of this study varied with the degree of freedom according to Student's *t*-table, and generally $k=2.4$ was used. Considering the difference in the coverage factors, Loveday's results can be modified to be about 2.8–5.5%. Silva reported uncertainties of 3.2% and 2.15% for the yield strength and the tensile strength, respectively. Again taking the difference in coverage factor into account, his results can be changed to about 3.9% and 2.8%.

The results of the two researches are similar for the yield strength: as in this study, the uncertainty of the tensile strength is smaller than that of the yield strength. However, the two results in previous researches for uniaxial tensile tests are less than the results for the IIT in this study. The reason is the size of the tested materials. The uniaxial tensile test uses specimens several tens of mm in size and measures the average value over the specimen volume. Even if the specimen is not uniform, raw data show little variation and the inhomogeneity is not revealed. However, the IIT measures an area of less than 1 mm in diameter: the residual indentation diameter in this study was about 450 μm. Local properties and inhomogeneity are thus much more reflected in the indentation load-depth curves, and the raw data inevitably have larger bias than in the uniaxial tensile test. Therefore, the IIT is expected to have a greater uncertainty than the uniaxial tensile test.

Other less important reasons for the greater uncertainty are the accuracy of the test instruments and the optimization of experimental procedures. Since uniaxial tensile test has gone on now for more than 100 years, very accurate test instruments have already been developed and optimal experimental procedures appear in international standards. The IIT, on the other hand, has a short history, and its optimization is not yet complete. Though the Vickers and Brinell hardness tests have very long histories and testers are provided by many vendors, instruments for the IIT at the macroscale, let alone at the nanoscale, have been provided by just few companies only for two or so decades. Improvements in test instruments and experimental procedures will reduce the uncertainty of the IIT.

5 Conclusions

A new method for estimating the uncertainty of indentation tensile properties in instrumented indentation test was suggested. Round-robin tests to verify the new method by five institutions showed the following.

(1) The uncertainties had average values of 18.2% and 9.8% for the indentation yield strength and the indentation tensile strength, respectively. The uncertainty in the indentation yield strength is higher because work-hardening is much more severe near a yield point.
(2) The uncertainty does not depend on the materials' mechanical properties. It does, however, depend on environmental conditions such as experimental instruments, operators, laboratory conditions, and the like, although the new model does not start from experimental parameters. This verifies the reliability of the new model.
(3) The work-hardening exponent is important in the uncertainty in the indentation yield strength but not in that of the indentation tensile properties. The difference in work-hardening at a yield point and a necking point is again the reason.
(4) The uncertainty in the instrumented indentation test is greater than the uncertainty in the uniaxial tensile test. Though the uncertainty in the IIT can be reduced by optimizing the instrumented indentation experiments, it will always remain greater because of the size of the IIT specimen test regions.

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Nomenclature

a_c = contact radius
c_i = sensitive coefficient
E = elastic modulus
h_c = contact depth
$h_{\max}$ = indentation depth
j = number of data
K = strength coefficient
k = coverage factor
L = indentation load
n = work-hardening exponent
R = indenter radius
u_c = combined standard uncertainty
U_i = expanded uncertainty
u_i = A-type uncertainty
x_i = influence quantity
y = measurand
α = constant related with true strain
ε = true strain
ε_u = uniform strain
ε_y = yield strain
γ = half angle between indenter and material
σ = true stress
σ_{UTS} = (indentation) tensile strength
σ_y = (indentation) yield strength
Ψ = constraint factor

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