

Derivation of tensile flow characteristics for austenitic materials from instrumented indentation technique

K-W Lee, K-H Kim, J-Y Kim and D Kwon

Department of Materials Science Engineering, Seoul National University, Seoul, Korea

E-mail: case77@snu.ac.kr

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Abstract

In this study, a method for deriving the tensile flow characteristics of austenitic materials from an instrumented indentation technique is presented along with its experimental verification. We proposed a modified algorithm for austenitic materials that takes their hardening behaviour into account. First, the true strain based on sine function instead of tangent function was adapted. It was proved that the sine function shows constant degrees of hardening which is a main characteristic of the hardening of austenitic materials. Second, a simple and linear constitutive equation was newly suggested to optimize indentation flow curves. The modified approach was experimentally verified by comparing tensile properties of five austenitic materials from uniaxial tensile test and instrumented indentation tests.

1. Introduction

Instrumented indentation techniques (IIT) have been studied as an effective nondestructive method to evaluate the mechanical properties of metallic materials [1–10]. These techniques are applicable in situations where traditional fracture tests cannot be conducted, for example, on in-service structures such as power, petroleum and chemical plants. Since the safety and reliability assessment of these systems have become key issues, the need for nondestructive testing such as IIT has also increased.

Safety assessment depends on the precise estimation of mechanical properties. Tensile properties such as yield strength, tensile strength and work-hardening exponent are the most fundamental mechanical properties in safety assessment of industrial facilities. Much work on evaluating tensile properties using IIT has been reported [3–6], among which the representative strain and stress approach proposed by Ahn and Kwon [3] is one of the most systematic. In general, their approach is applicable to power-law hardening materials. However, it is difficult to use for linear hardening materials such as austenitic stainless steels due to its basic assumption of obedience to the Hollomon equation [11], since they show totally different plastic deformation characteristics, i.e. hardening characteristics.

In this study we proposed a modified algorithm for austenitic stainless steels that takes their hardening behaviour into account. In our work the definition of the true strain differs from that in previous research: a sine function instead of tangent function was used for linear hardening materials by variation of differential values of the true stress. We also suggested that the new equation optimizes the linear hardening behaviour by representative strain–stress. Using the modified representative strain and proposed constitutive equation, tensile properties obtained from uniaxial tensile tests and instrumented indentation tests were compared for six stainless steels.

2. Algorithm for evaluation of tensile properties

The algorithm for evaluating tensile properties has four steps: step 0—determine real contact area; step 1—define representative stress and strain; step 2—fit to constitutive equation and step 3—evaluate tensile properties. Details are as follows.

Figure 1 shows a typical indentation load–depth curve obtainable during instrumented spherical indentations on steel. Unlike curves from sharp indentation using a pyramidal indenter, loading curves are quite linear due to the counterbalance of spherical geometry and work hardening in

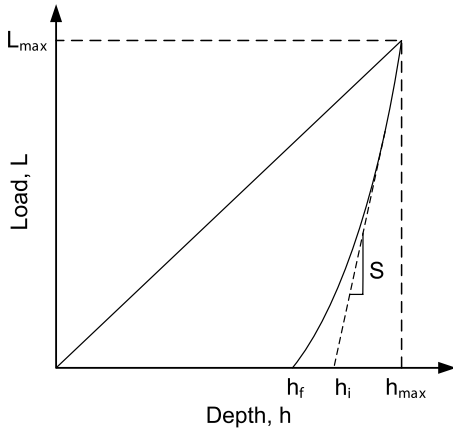


Figure 1. Schematic diagram of typical load–depth curve obtainable during instrumented spherical indentations on steel.

tested steel. Several depths are defined from this curve. The maximum indentation depth h_{\max} is the total displacement of the material and the indenter at maximum load L_{\max} , including elastic and plastic deformation. In unloading, elastic deformation is the indentation stiffness (S) of the specimen and the indenter. Thus, the final depth h_f is the plastic deformation of the material.

A contact depth h_c^* at maximum indentation load can be evaluated by analysing the unloading curve using the concepts of indenter geometry and elastic deflection [2]:

$$h_c^* = h_{\max} - \omega(h_{\max} - h_i), \quad (1)$$

where h_i is the intercept indentation depth and the indenter shape parameter ω is 0.75 for a spherical indenter. The material pile-up around the indentation enlarges the contact radius (from the analysis of elastic deflection) by an extent that is determined by the work-hardening exponent n and the ratio of maximum indentation depth and indenter radius h_{\max}/R [12]:

$$h_{\text{pile}}^* = h_c^* \cdot f(n, h_{\max}/R), \quad (2)$$

where h_{pile}^* is the plastic pile-up depth.

The mean pressure P_m obtained by dividing the maximum load L_{\max} by the contact area πa_c^2 is well known to be about three times the representative stress σ_R for fully plastic deformation of steels [13]. In other words, the representative stress can be expressed as

$$\sigma_R = \left(\frac{1}{\Psi}\right) P_m = \left(\frac{1}{\Psi}\right) \left(\frac{L_{\max}}{\pi a_c^2}\right), \quad (3)$$

where Ψ is a plastic constraint factor, here taken as 3, and a_c is the contact area. On the basis of the deformation shape and strain distribution under a spherical indenter, Ahn and Kwon [3] proposed a new definition using the tangent function and a strain proportional constant, α :

$$\varepsilon_R = \left(\frac{\alpha}{\sqrt{1 - (a_c/R)^2}}\right) \left(\frac{a_c}{R}\right) = \alpha \tan \gamma, \quad (4)$$

where α was determined as 0.14 by finite element analysis for various materials [14], R is the indenter radius and γ is the half-angle between the indenter and the material. The true stress

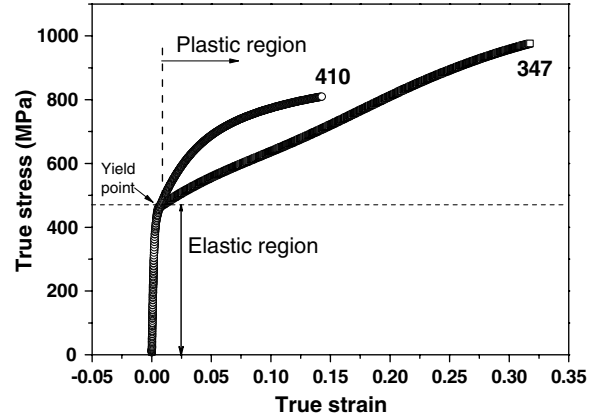


Figure 2. Comparison of uniaxial tensile curves of stainless steels 410 and 347.

and strain points obtained from the indentation test are then fitted to a constitutive equation by a simple power-law-type Hollmon equation:

$$\sigma = K \cdot \varepsilon^n, \quad (5)$$

where n is the work-hardening exponent and the gradient of the curve, K is the strength coefficient and σ and ε are, respectively, the representative stress and strain values. This approach assumes that the flow curve of many metals in the uniform plastic deformation region can be expressed by (5). With most materials there is a gradual transition from elastic to plastic behaviour, and the point at which plastic deformation begins is hard to define with precision. Although there are various criteria for the initiation of yielding, the yield strength obtained by an offset method is commonly used for design and specification purpose because it avoids the practical difficulties of measuring the elastic limit or proportional limit [11]. Thus, the yield strain can be determined as the intersection point of an elastic line whose slope is the elastic modulus 0.2% offset from the origin and a plastic curve of the constitutive equation. The uniform tensile strain should be same as the work-hardening exponent, by the theory of instability in tension [11], and from this the indentation tensile strength can be determined. The indentation yield strengths and the indentation tensile strengths lie within 10% and 5% errors of those from uniaxial tensile tests [15]. This is currently limited to power-law hardening materials, i.e. those that obey (5).

3. Analysis of work-hardening behaviours

Figure 2 shows two stainless steels, stainless steels 347 and 410, having different work-hardening behaviours. Since the two steels have similar elastic moduli and yield strength, they have similar tensile curves before yielding. However, stainless steel 410 shows continuously decreasing hardening ($d\sigma/d\varepsilon$) after yielding, while stainless steel 347 shows constant and greater hardening than stainless steel 410 until the uniform elongation point. Overall, stainless steel 347 has higher ultimate tensile strength than stainless steel 410.

The main reason that the two steels show entirely different hardening phenomena has been reported as the difference in stacking fault energy (SFE) [16, 17]. Low values of SFE

Table 1. Typical values of stacking fault energy (SFE) [11].

Metal	SFE (10^{-3} J m^{-2})
Stainless steel 303	8
Stainless steel 304	20
Stainless steel 310	45
Silver	25
Gold	50
Copper	80
Nickel	150
Aluminium	200

increase the width of stacking faults. This effectively increases the work-hardening rate, since it is harder for dislocations to cross slip; the degree of work hardening is therefore increased [17]. The two stainless steels 347 and 410 have different crystal structures: 347 with austenite stainless steel is face-centred cubic (FCC) and 410 with martensite stainless steel are body-centred cubic (BCC); at times body centred tetragonal (BCT) in the harden condition. BCC materials present generally higher values of SFE because their many slip systems make free cross slip possible. The SFE of FCC materials is comparatively low due to their fewer slip systems. Also, it has been reported that chemical composition is an important variable for SFE values [18–21], and that high concentrations of manganese and nitrogen as well-known austenite stabilizers typically reduce the SFE [18, 20]. These effects are well revealed particularly in austenite stainless steel, which is high in both constituents. The typical SFE values in table 1 indicate that the SFE values of austenite stainless steel are particularly low.

This difference in work-hardening behaviour raises new issues for evaluating the tensile properties of austenite stainless steels, since (5) does not apply. Ludwigson [16] suggested a new constitutive equation to describe their unique hardening behaviour:

$$\sigma = K_1 \varepsilon^{n_1} + \exp(K_2 + n_2 \varepsilon), \quad (6)$$

where K_1 , n_1 , K_2 and n_2 are all materials constants with no physical meaning. This equation contains so many constants that they cannot easily be determined. In order to obtain the four constants accurately, many true stress–strain points are needed, which is possible in uniaxial tensile test. However, it is difficult to obtain them in instrumented indentation tests because of the limited representative stress–strain points (about 10–15 points). Thus, this equation is inadequate for representing the behaviour of austenitic steels using IIT. Other expressions have also been discussed in the literature [22, 23], but it is hard to apply for similar reasons.

It is also important to consider whether (4), the definition of representative strain proposed by Ahn and Kwon [3], could be reflected to the deformation behaviour of austenite stainless steel. To optimize this algorithm, the constitutive equation and representative strain need significant reappraisal.

4. Optimization of constitutive equation and representative strain

As described above, the austenite stainless steels show constant values of $d\sigma/d\varepsilon$, that is, linear hardening behaviour in the

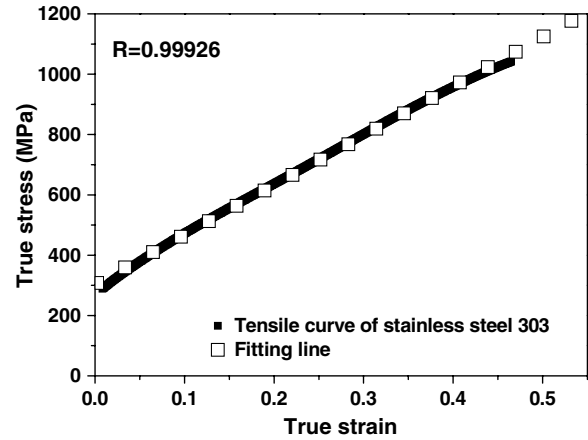


Figure 3. Comparison of uniaxial tensile curve of stainless steel 303 and a fitting line of equation (3).

plastic deformation range, a behaviour also observed in some FCC materials [24–27]. To express their hardening behaviour with a new equation, the tensile curve for stainless steel 303 was fitted as a linear line, as illustrated in figure 3. Generally, the degree of linearity is expressed by a correlation coefficient, R . As linearity becomes higher, the absolute value of the coefficient approaches 1. Most austenitic steels had coefficients above 0.995; it means that their tensile curves can be expressed by the linear function. In our research their hardening behaviour is expressed by a simple linear equation:

$$\sigma = A + E_T \varepsilon, \quad (7)$$

where A and E_T are material constants. Since in general metals have a yield strain of about 0.005, the constant A has almost the same value as the yield strength. The tangent modulus, E_T , is an index on the degree of hardening. Since the linear equation has just two constants, they can be easily calculated using IIT. Thus the linear constitutive equation is able to describe the best equation for expressing the hardening behaviour of austenitic steels using IIT.

In general, two kinds of strain definition are used; tangent function suggested by Ahn and Kwon [3] and sine function by Tabor [13], as in (8). From a study of indentation deformation, Tabor concluded that the true strain was proportional to the ratio d/D and could be expressed by the sine function:

$$\varepsilon = \beta \frac{d}{D} = \beta \sin \gamma, \quad (8)$$

where β is generally taken as 0.2, D is the diameter of the spherical indenter, d is the diameter of the residual imprint and γ is the half-angle between the indenter and the material. Although this definition has been widely used in similar indentation works, it has the indispensable limitation of the sine function: the maximum strain must be less than β (generally 0.2), and this makes it difficult to evaluate the indentation flow curve of ductile materials.

The main difference between (4) and (8) is the plastic deformation behaviour, due to the characteristics of trigonometric functions. Of the two strain definitions, the suitability in evaluating tensile properties, Jeon *et al* [28],

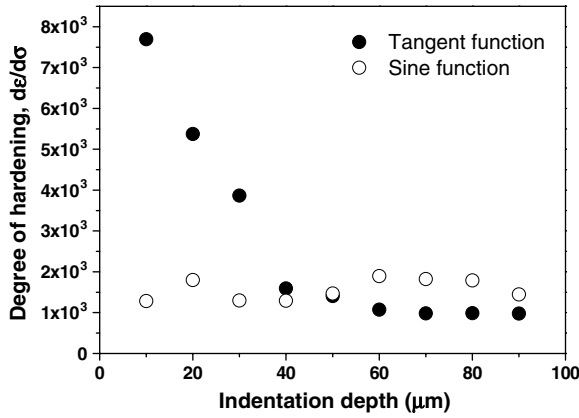


Figure 4. Differential values of stress indentation flow curves derived by tangent function strain and sine function strain for stainless steel 304L.

was analysed based on derived behaviours of the work-hardening exponent. By studying the increase in true strain with penetration depth, they demonstrated that the true strain based on the sine function yielded much larger work-hardening behaviour with increasing plastic deformation. The tangent function was proved to be optimal for deriving the indentation flow curve of power-law hardening materials.

A degree of hardening $d\sigma/d\varepsilon$ was introduced to determine the proper definition to represent linear hardening behaviour. Two indentation flow curves were derived from the indentation load–depth curve of stainless steel 304L using (4) and (8), and then the values of $d\sigma/d\varepsilon$ on each flow curve were calculated, in figure 4. The only difference between two flow curves was how to define true strain. The values from the tangent function decreased continuously with indentation depth, as shown in the tensile curves of the power-law-hardening materials in the plastic deformation range. On the other hand, the values from the sine function were relatively constant regardless of indentation depth, which corresponds to linear hardening behaviour. Therefore, the sine function should be used in representing the indentation flow curve of linear hardening materials instead of the tangent function used for the power-law hardening materials.

5. Experimental verification

Spherical indentation testing was used to verify the modified algorithm for tensile properties. The stainless steels prepared here are classified into two groups from uniaxial tensile testing: martensite stainless steel 410, material showing power-law hardening behaviour, and five austenitic stainless steels 303, 304L, 316, 321 and 347, materials showing linear hardening behaviour. Their chemical compositions are given in table 2. Surfaces were polished with $1\ \mu\text{m}\ \text{Al}_2\text{O}_3$ powder and indentation tests were carried out with the AIS 2100 portable indentation system (Frontics Inc., South Korea). The indenter was a tungsten carbide ball of $250\ \mu\text{m}$ radius, and indentation speed was $0.3\ \text{mm}\ \text{min}^{-1}$. The final maximum depth was $150\ \mu\text{m}$ and 15 multiple unloadings down to 50% of maximum load at each point were applied. All tests were

performed at room temperature. Uniaxial tensile tests were performed at a cross-head speed of $1\ \text{mm}\ \text{min}^{-1}$ for specimens with gauge length $25\ \text{mm}$ and diameter $6\ \text{mm}$ according to ASTM standard [29].

If a yield point and necking point can be determined from the IIT results, the indentation yield strength and the indentation tensile strength can be evaluated. The yield point is the intersection of the plastic curve and the elastic line offset to 0.2% strain, as mentioned above. The plastic curve can be regarded as the line obtained from (7). The slope of the elastic line is the elastic modulus which can be easily evaluated by the IIT [2]. Therefore, the elastic line can be also obtained, and the yield point, as well:

$$E(\varepsilon_y - 0.002) = A + E_T\varepsilon_Y, \tag{9}$$

where E is the elastic modulus and ε_y is the yield strain.

The necking point is known to have the same uniform tensile strain as the work-hardening exponent (from the concept of instability in tension [11]), and this equivalence is used to determine the indentation tensile strength of power-law hardening materials. In linear hardening materials, however, this concept is difficult to apply directly because their work-hardening exponent is hard to define (that of general steels is defined as the increase in hardness and strength caused by plastic deformation [30], a definition limited to materials whose plastic-flow behaviour obeys the power curves as in (5)). Nevertheless, we calculated the work-hardening exponent using the same concept because it is the only method available. To determine the necking point in linear hardening material, we modified the concept of instability in tension as follows:

$$\varepsilon_u = C \cdot n, \tag{10}$$

where ε_u is the uniform tensile strain and n is the work-hardening exponent calculated using (5). C is an empirical value taken as about 1.5 here. This relation is the experimental result, showing a linear relationship of the uniform tensile strain value with the work-hardening exponent. However, this relationship has no physical meaning yet, and the matter requires further study.

Using the concepts described above, the indentation tensile properties were evaluated and are compared in table 3 and figure 5 which results from uniaxial tensile tests. The error ranges of the indentation yield strengths are within 10%, and of the tensile strengths within 5%. We thus see that the IIT can be applied to evaluating the tensile properties not only of power-law-hardening materials but also of linear hardening materials.

6. Conclusion

1. Materials having low SFE such as austenitic stainless steels have a linear hardening characteristic which is more severe hardening than power-law hardening since the stacking fault blocks the dislocation movement. Their linear hardening characteristic can be described in the form of the linear function

$$\sigma = A + E_T\varepsilon,$$

Table 2. Chemical compositions of stainless steels used.

Stainless steel (AISI)	Chemical composition (%)								
	C	Mn	Si	S	P	Cr	Ni	Mo	N
303	0.14	1.82	0.56	0.141	0.18	18.22	9.31	0.21	—
304L	0.022	1.65	0.251	0.026	0.037	18.19	9.12	0.24	0.086
316	0.059	1.69	0.38	0.021	0.036	16.96	12.42	2.11	—
321	0.054	1.76	0.4	0.027	0.022	17.36	9.22	0.21	0.025
347	0.052	1.74	0.48	0.026	0.032	18.43	9.87	—	—
410	0.103	0.82	0.77	0.024	0.03	12.09	0.26	—	—

Table 3. Tensile properties obtained from tensile tests and instrumented indentation tests.

Stainless steel (AISI)	Yield strength (MPa)			Tensile strength (MPa)		
	Tensile	IIT	Error (%)	Tensile	IIT	Error (%)
303	264.4	284.6	-7.6	1051.4	1067.1	-1.5
304L	361.7	332.7	8.0	1120.8	1082.4	3.4
316	357.2	336.5	5.7	908.6	941	-3.5
321	356.6	402.9	-10.2	866.8	872.8	-0.7
347	455.27	482.5	-5.9	967.6	934.3	3.4
410	431.47	471.9	-8.5	792.1	836.5	-5.3

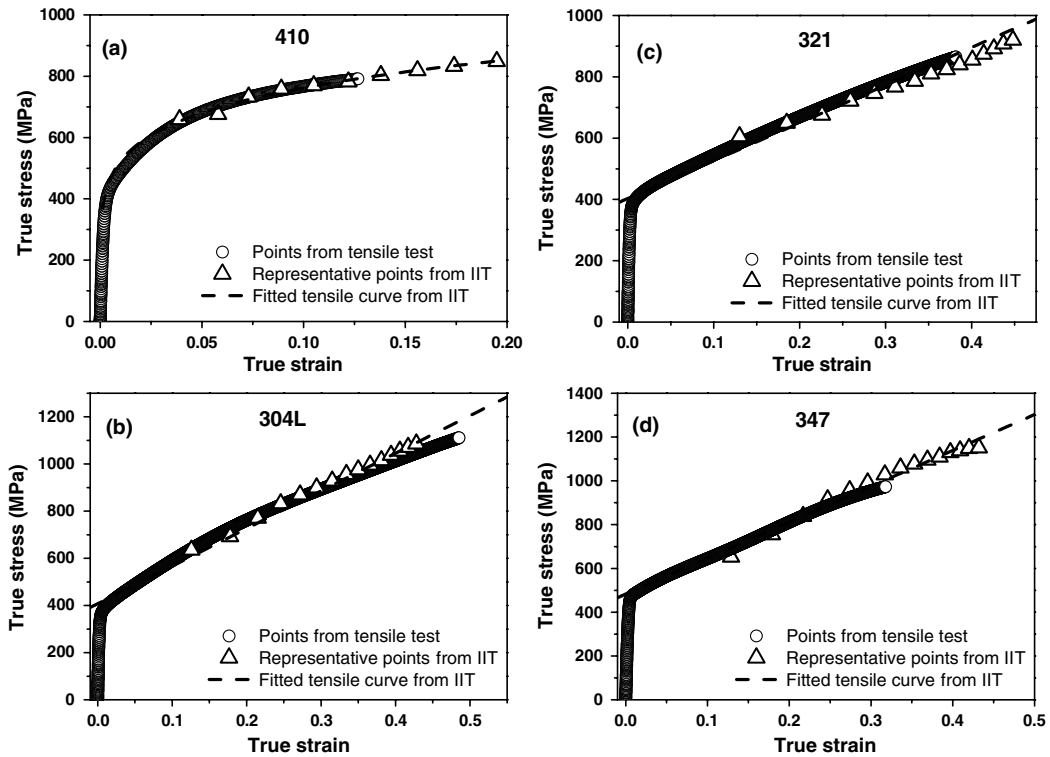


Figure 5. Comparison between the tensile curves from the tensile tests and the instrumented indentation tests for the stainless steels (a) 410 (power-law hardening behaviour), (b) 304L, (c) 321 and (d) 347 (linear hardening behaviour).

where A has a similar value with the yield strength and E_T means a degree of hardening during plastic deformation.

- There are two kinds of true strain definitions in the IIT: tangent function and sine function. The tangent function corresponds to decreasing degrees of hardening and the sine function is suitable for a constant degree of hardening. Since the constant degree of hardening is a main characteristic of linear hardening, the sine function

could be used for evaluating the tensile flow characteristic of the linear hardening materials.

- It is experimentally verified that the tensile properties can be evaluated accurately through the true strain definition using sine function and the constitutive equation in the form of linear function which are described above. The yield strength and the tensile strength have small error ranges within 10% and 5%, respectively.

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