Materials Science and Engineering A 535 (2012) 197–201

Contents lists available at SciVerse ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Correlation between the plastic strain and the plastic pileup of the instrumented indentation by utilizing the interrupted tensile test

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ARTICLE INFO

Article history:
Received 18 November 2011
Received in revised form
29 November 2011
Accepted 13 December 2011
Available online 22 December 2011

Keywords:
Instrumented indentation
Pileup
Plastic strain
Vickers hardness
Interrupted tensile test

ABSTRACT

The experimental approach to analyze the direct correlation between the plastic pileup and the plastic strain of the Vickers indentation test was presented. The correlation between the yield strain and the plastic pileup was analyzed through the interrupted tensile test of SUS316L, the stainless steel in which the strain hardening phenomenon one of the two major factors affecting the plastic pileup is fixed. Finally, it was checked that the plastic strain is the parameter directly affecting the plastic pileup and that the relation through the linear function can be analyzed. Moreover, as for the depth of the indentation test, it was also checked that the plastic pileup can be corrected through the factor.

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1. Introduction

The instrumented indentation test applying the hardness test principle is the technology evaluating the mechanical characteristics of a material by minutely deforming the surface of a material from the penetration load and depth curve. The instrumented indentation test [1–15] has the characteristics different from the hardness tester measuring the contact area through the assistance of the optical equipment. Differently from the current hardening test that could not measure the indentation depth, the instrumented indentation test directly measuring the depth in the indentation process has the advantage of measuring the contact area from the load–depth curve without observing the indent. Hence, the specimen size is minute so that it can be also applicable to the material for which it is inconvenient to use the surface observation equipment or is impossible to use the optical equipment. The contact area is calculated from the contact depth and from the geometry of the indenter. However, because of the elasto-plastic deformation occurring around the indenter, the measured depth and the actual contact depth have different values. Therefore, it was the important research to analyze the elasto-plastic deformation for measuring the correct contact depth (h_c) and calculating the contact area (A_c).

The elastic deformation appears as the elastic deflection (h_d) that when the force is put on the point, the neighborhood around the force point not directly affected by the force is pulled down together due to the elastic bonding with the force point. As for the elastic deflection, the analytic formula using the Hertz contact formula assuming the contact of a spherical body exists. The Oliver–Pharr formula [2] for various indenters is being widely used:

$$h_d = \chi \frac{L_{\max}}{S}, \tag{1}$$

$L_{\max}$ is the maximum indentation load, S is initial unloading stiffness during unloading, and χ is the constant depending on the indenter shape (0.72 is used for Vickers and Berkovich indenters). When the plastic deformation occurs, the volume of the total system does not change so that it is accumulated around the indenter; it is called the plastic pileup (h_p). The analytical solution on the plastic pileup of the indentation test does not exist, so most of researches [4–14] were done by interpreting relation between the parameters representing the plastic deformation and the plastic pileup.

The plastic pileup is the phenomenon caused by the plastic deformation of a material and is known to be affected by the plastic strain and the strain hardening behavior [4–14]. And previous research [4–14] on the correlation between the plastic pileup, plastic strain and the strain hardening exponent (n) was done through

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doi:10.1016/j.msea.2011.12.063

the test on the 24 metal materials. An interesting fact is that differently from the matter known, the direct interrelationship did not appear between the strain hardening exponent and the plastic pileup. On the other hand, it was characterized that there is the linear relation between the yield strain inversely proportional to the plastic strain and the plastic pileup even there is scatterings for some materials. When the maximum load is engaged, the maximum depth from the reference surface becomes the maximum indentation depth (h_{max}). The actual contact occurring depth should add the plastic pileup to the maximum indentation depth. If the load is removed, the elastic part is recovered. The maximum depth of the indent remaining on the reference surface becomes the final indentation depth (h_f). The recovery occurs vertically so that the fixed diagonal length (d) of the indent is maintained.

The ratio of the contact depth reflecting the plastic pileup and the maximum indentation depth was expressed as the factor f_v. It was experimentally checked that f_v is linearly proportional to the yield strain [9,15]. The technology that can correct the plastic pileup only through the indentation test was proposed by finally expressing the yield strain as the depth of the indentation test:

$$f_v = 9.99 \times 10^{-3} \frac{h_{max}}{h_{max} - h_f} + 1.00, \tag{2}$$

$$h_c = f_v h_{max}. \tag{3}$$

The above result is useful to practically derive the contact area. However, it has the limitation that the relation between two factors involved in the plastic pileup was not clearly analyzed. In order to check the effect that each factor makes on the plastic pileup, it is important to check the effect of another variable in the situation that a variable is fixed. However, it is very difficult to keep the supply and demand of the material to check the effect of another factor by controlling one variable out of two variables (strain hardening and plastic strain). In order to resolve the problem, the research was done to analyze the independent relation between the plastic pileup and the plastic strain by manufacturing the specimen engaged the pre-strain through the interrupted tensile test in the austenitic stainless steel with the constant ratio of strain hardening.

2. Experiments

SUS316L, the stainless steel of the austenite series was selected as the material for the test. It is suspended after the engineering strain with 5%, 10%, 20%, and 30% is applied to the tensile specimen processed as the plate through the tensile test on the basis of ASTM standard E8-04 (2002). Afterwards, the middle surface of the tensile specimen is cut to prepare the indentation test. The tensile test is done through the Instron 5582 (Instron, Norwood, MA). The total 5 kinds of specimens including the specimen that did not go through the tensile test were prepared. The surface of the specimen was grounded with the 1 μm Al_2O_3 powder for the indentation test. Frontics AIS3000 (Frontics, Seoul, Korea) was used as the indentation tester. The Vickers indenter manufactured with diamond was used as the indenter. The indentation depth was controlled as 80 μm and the indenting rate was 0.3 mm/min. In order to calculate the contact depth, the surface observation using the optical microscope was made. The diagonal length (d) of the indent was measured. On the assumption of the ideal indenter shape, the contact depth (h_c) is calculated from the indenting angle (θ) and from the diagonal length of the indent:

$$h_c^V = \left(\frac{d}{2\sqrt{2}\tan\theta}\right) = \left(\frac{d}{2\sqrt{2}\tan 68^\circ}\right) \approx \frac{d}{7}. \tag{4}$$

The contact depth calculated from Eq. (4) was converted into f_v through the Eq. (3). As for the maximum indentation depth and the final indentation depth, the values measured from the indentation test were used.

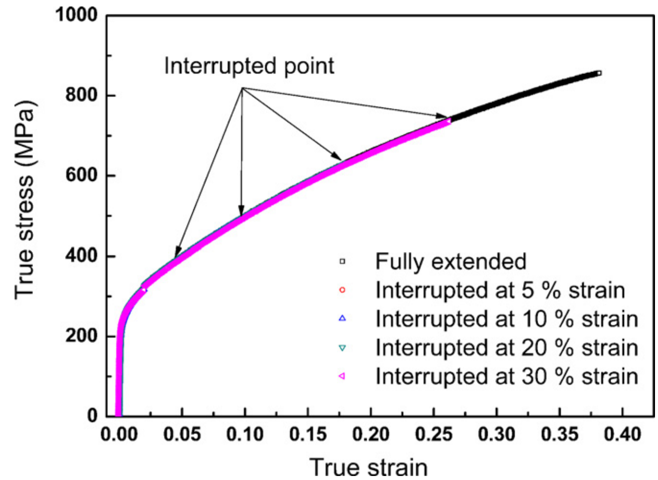

Fig. 1. The results of the tensile test to the interrupted point.

3. Results and discussion

The result of the tensile test on SUS316L is shown in Fig. 1. It represents the result of the tensile test being performed without interruption and the tensile curve interrupted in 5%, 10%, 20%, and 30% (the true strains with 4.9%, 9.5%, 18.2%, and 26.2%, respectively), respectively. The recurrence between tensile tests was very good. It could be checked that it is the same type of the material with the same tensile property. Therefore, it can be assumed that the specimen used in the test is the one of the same kind of the material having the difference of the pre-strain based on the interruption. It is known that as for the stainless steel of the austenite series, the shape of the plastic interval turned out to be straight. The result of the test showed that the plastic interval is straight. It was checked that the strain hardening is fixed regardless of the strain. If the pre-strain is given to the material, it becomes the specimen with the tensile property different from the material without pre-strain. Due to the phenomenon caused by the characteristics of the elasto-plastic deformation, the elastic deformation does not generate the permanent change of a material, but the plastic deformation generates the permanent deformation by changing the microstructure. The plasticity is also has directivity of deformation. If the load is removed from the specimen that went through the plastic deformation through the uniaxial tensile test, the elastic deformation part is recovered linearly. However, the permanent plastic deformation is maintained. If the load is reengaged, the elastic deformation is started again, but the plastic deformation appears after the permanent deformation due to the previous deformation. Therefore, the specimen with the pre-strain has the yield strength and strain that are different from those of the specimen with the deformation not achieved (Fig. 2). According to the same principle, the specimens with the 5%, 10%, 20%, and 30% pre-strains become the materials having the mutually different yield strength and strain with the different micro structure. The yield strength of the specimen without deformation and of the specimen with the 5%, 10%, 20%, and 30% pre-strains authorized is 252, 396, 490, 633, and 736 MPa, respectively. Their yield strain (σ_y/E) is 0.126%, 0.198%, 0.245%, 0.317%, and 0.368% respectively.

Here we needed to discuss about the strain hardening exponent which is used as a parameter of strain hardening of metal. Strain hardening exponent is defined from the Hollomon's equation which mathematically describes the stress–strain (σ–ε) relation during plastic deformation. Since it is generally related with strain

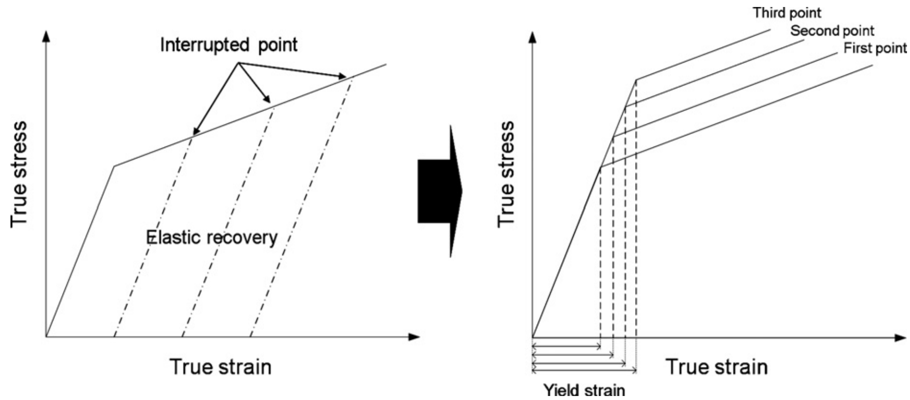

Fig. 2. The change of the yield strain using the interrupted tensile test.

hardening behavior (if strain hardening exponent is large, there is more strain hardening), many researcher tried to develop the empirical relation between strain hardening exponent and pileup in indentation [4–14]. In the case of austenitic steel which has linear plastic stress–strain relation, however, it is difficult to define strain hardening exponent. We illustrated logarithms of plastic stress–strain of interrupted tensile samples in Fig. 3. Each line is linear fit of each curves and its slope represent strain hardening exponent by Hollomon equation. Intuitively we know that Hollomon equation is not suitable for these materials and the definition of strain hardening exponent as a quantitative index is meaningless [16–19]. Even interrupted curves just translated in the stress–strain plane, strain hardening exponents of all interrupted specimens are different each others because it is the denoted as a logarithms plane. There are several well-known constitutive equations for describing the stress and strain curve of linearly hardened stainless steels, such as the Ludwigson and Voce equations [16,20,21]. However, these equations were devised on the basis of the mathematical description, and the parameters of constitutive equation are not enough to index strain-hardening effect independently [20]. For this reason we suggest the slope of stress–strain curve as a useful parameter for the strain-hardening index related to pileup in linear hardening materials. The affection of strain hardening on pileup is related with the hardness difference between the deformed zone which is hardening by plastic deformation and undeformed zone which is constraining the plastic deformation. In the case of linear hardening materials, the hardening ratio is constant for entire plastic deformation so we can assume that the strain hardening behavior of interrupted sample is almost coherent.

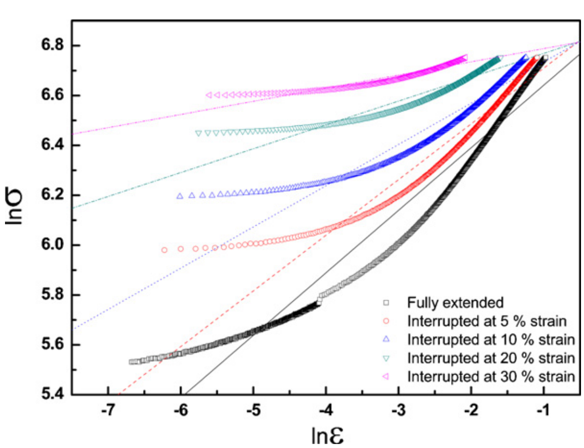

Fig. 3. The variation in strain hardening exponent of interrupted tensile test.

On the other hand, yield strain has been changed by interrupted tensile test and its effect on pileup can be demonstrated. The average strain in the lower part of the indenter by the indenter is the function of the indenter angle. Ahn and Kwon [22] defined the average strain by averaging the shear strain with the volume deformed by the indenter in the indenting direction and checked that the average strain is proportional to the contact angle. Moreover, the contact angle has the functional relation with the indenting angle so that it can be known that the average strain generated from the indentation test is inversely proportional to the indenting angle. In case of the Vickers indenter with the angle of the indenter fixed [23–25], the deformed volume is increased depending on the indentation depth, but the average strain is not changed.

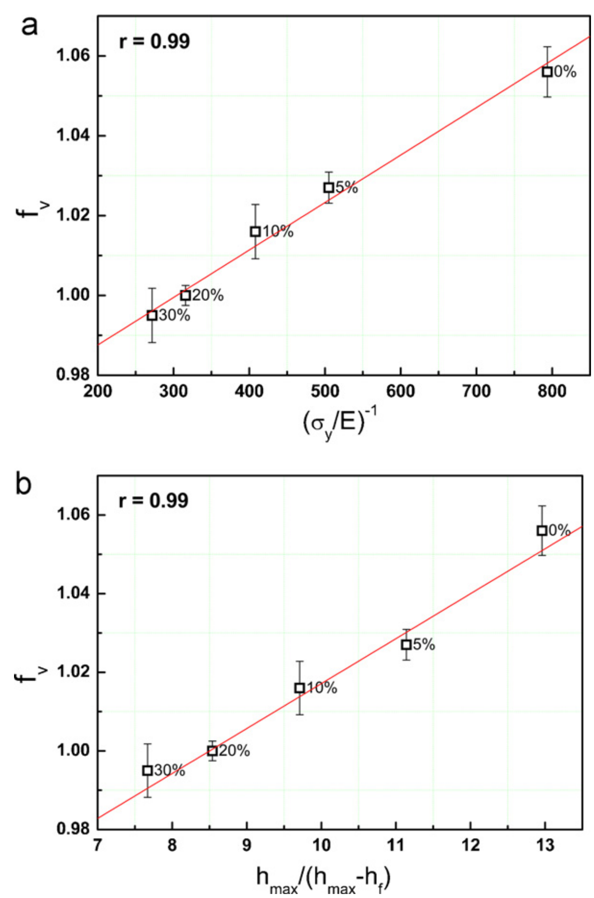

Fig. 4. The linear correlation between plastic pileup and (a) yield strain and (b) indentation depth.

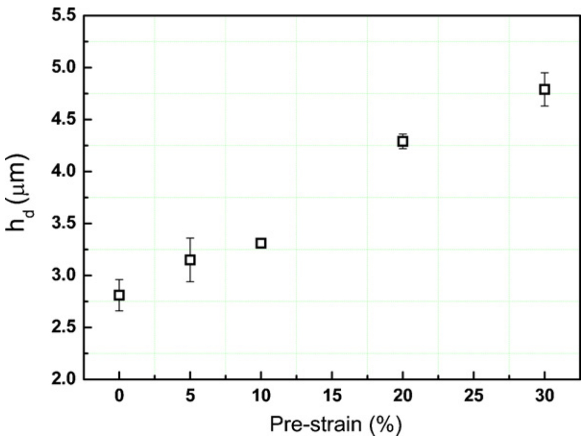

Fig. 5. The linear increase in elastic deflection as pre-strain increases.

It is defined as the geometrical self-similarity in the indentation test. Hence, the average deformation rate generated during the indentation of the Vickers indenter is fixed, and the quantity of the generation of the plastic strain is inversely proportional to the generation of the yield strain. Hence, if the plastic pileup of the specimen with the yield strain changed is compared, the relation between the yield strain (or plastic strain) being independent of the strain hardening and the plastic pileup can be verified experimentally. In order to directly check the inversely proportional relation with the elastic strain, the yield strain for four specimens and f_v are shown in Fig. 4. It can be checked in Fig. 4a that the reciprocal of the yield strain has the very highly related linear relation with f_v. The relation is as follows:

$$f_V = 1.1897 \times 10^{-4} \left(\frac{\sigma_y}{E}\right)^{-1} + 0.9638. \quad (5)$$

It shows the consistence with the result of the previous research. Especially, the test result is meaningful in a sense that it was checked that the plastic strain has direct correlation with the plastic pileup by changing only the yield strain in the same kind of materials of which strain hardening behavior is controlled.

It is noted that f_v does not directly represent pileup because it include elastic deflection as well; however, we can say that the variation in f_v in this discussion describes pileup. Because the elastic response is inherent regardless of pre-strain conditions, we expect that the variation of elastic deflection will constantly affect f_v. We found experimentally that elastic deflections constantly increase using Eq. (1) as 2.81 ± 0.15, 3.15 ± 0.04, 3.31 ± 0.21, 4.29 ± 0.07, and 4.79 ± 0.16 μm for specimens without deformation and for specimens with 5%, 10%, 20%, and 30% pre-strains, respectively. The linear increase of the elastic deflection is shown in Fig. 5.

On the basis of the above result, it can be concluded that the quantity of the generation of the plastic deformation directly induces the plastic pileup and that the plastic pileup can be estimated through the yield strain. However, the evaluation of the contact area only through the indentation test is still impossible only through the relation between the yield strain and the plastic pileup. The factor proposed to solve this problem is the ratio of the recovered indentation depth and the maximum indentation depth. The yield strain is understood as the ratio of the elastic strain (of the recovered deformation) to the maximum strain in the tensile test [6,9,15]. Application of the same principle to the indentation test can make the ratio that the recovered depth ($h_{max} - h_f$) takes from the total depth understood as the deformation quantity caused by elasticity. It was checked in Fig. 4b that f_v and $(h_{max} - h_f)/h_{max}$ are directly compared to express them as the following linear equation:

$$f_V = 1.1430 \times 10^{-2} \frac{h_{max}}{h_{max} - h_f} + 0.9028. \quad (6)$$

There are two occurring question from this relation: why constants are too much different from previous results and how the f_v can be below 1. One convincing answer is indenter bluntness. When we calculated the contact depth from the diagonal length, we assumed the perfect geometry of Vickers indenter. However, it is well known that there is an inevitable bluntness of sharp indenter and it misleading the geometrical calculation between contact depth and contact area [10,26–28]. Because the relation between contact depth and contact area depends on the tangential function of indenter angles so small difference in indenter angles cause large error in the calculation of contact depth. Miscalculated contact depth is directly related with each constants of Eq. (6) as f_v is including miscalculated angle. For this own indenter, however, this relation still valid to calculate the contact area (not real contact depth) from the indentation depths. Finally, we can conclude that Eq. (6) gives imaginary contact depth which evaluate contact depth using perfect indenter geometry from the indenter imperfect and yield strain of indentation.

4. Conclusion

The research presented the experimental approach to the analysis of the correlation of the plastic pileup generated in the corner of the Vickers indenter with the plastic strain. The interrupted tensile test was applied to stainless steel SUS316L material showing the linear strain hardening characteristics. The strain with four pre-strains authorized and the specimen without deformation were utilized. The plastic strain is inversely proportional to the yield strain in the Vickers indentation test with the average strain fixed so that the relation with the plastic pileup can be found through the yield strain. The yield strain was changed through the interrupted tensile test. The yield strain was directly compared with the plastic pileup. The reciprocal of the yield strain and the plastic pileup, f_v, have the linear relation of the linear equation each other. In order to correct the plastic pileup by utilizing only the indentation test, the elasticity strain was substituted for the yield strain. Finally, it was verified that the reciprocal of he yield strain of the indentation test $h_{max}/(h_{max} - h_f)$ and f_v have the linear relation of the linear equation each other. The previous research on the clarification of the relation of the plastic pileup with the yield strain without excluding the effect caused by the strain hardening was supplemented so that it was checked that the plastic strain is the direct factor affecting the plastic pileup and that the plastic pileup can be corrected through it.

Acknowledgment

This research was supported by the Converging Research Center Program through the Ministry of Education, Science and Technology (2011K000599).

References

[1] M.F. Doerner, W.D. Nix, J. Mater. Res. 1 (1986) 601–609.
[2] W.C. Oliver, G.M. Pharr, J. Mater. Res. 7 (1992) 1564–1583.
[3] Y.T. Cheng, C.M. Cheng, Mater. Sci. Eng. R 44 (2004) 91–149.
[4] J. Mencik, M.V. Swain, J. Mater. Res. 10 (1995) 1491–1501.
[5] K.W. McElhaney, J.J. Vlassak, W.D. Nix, J. Mater. Res. 13 (1998) 1300–1306.
[6] Y.T. Cheng, C.M. Cheng, Appl. Phys. Lett. 73 (1998) 614–616.
[7] Y.T. Cheng, C.M. Cheng, Surf. Coat. Tech. 133–134 (2000) 417–424.
[8] J. Alcala, A.C. Barone, M. Anglada, Acta Mater. 48 (2000) 3451–3464.
[9] J. Malzbendera, G. With, J. Mater. Res. 17 (2002) 502–511.
[10] Y. Choi, H.S. Lee, D. Kwon, J. Mater. Res. 19 (2004) 3307–3315.

[11] S.H. Kim, B.W. Lee, Y. Choi, D. Kwon, Mater. Sci. Eng. A 415 (2006) 59–65.
[12] Y.H. Lee, J.H. Hahn, S.H. Nahm, J.I. Jang, D. Kwon, J. Phys. D 41 (2008) 074027.
[13] O. Bartier, X. hernot, G. Mauvoisin, Mech. Mater. 42 (2010) 640–656.
[14] P. Zhang, S.X. Li, Z.F. Zhang, Mater. Sci. Eng. A 529 (2011) 62–73.
[15] S.K. Kang, J.Y. Kim, C.P. Park, H.U. Kim, D. Kwon, J. Mater. Res. 25 (2010) 337–343.
[16] D.C. Ludwigson, Metall. Mater. Trans. 2 (1971) 2825–2828.
[17] H.W. Swift, J. Mech. Phys. Solids 1 (1952) 1–18.
[18] K.G. Samuel, P. Rodriguez, J. Mater. Sci. 40 (2005) 5727–5731.
[19] J.D. Yoo, K.T. Park, Mater. Sci. Eng. A 496 (2008) 417–424.
[20] H.J. Kleemola, M.A. Nieminen, Metall. Mater. Trans. 5 (1974) 1863–1866.
[21] E. Voce, J. Inst. Metals 74 (1948) 537–562.
[22] J.H. Ahn, D. Kwon, J. Mater. Res. 16 (2001) 3170–3178.
[23] M. Dao, N. Chollacoop, K.J. Van Vliet, T.A. Venkatesh, S. Suresh, Acta Mater. 49 (2001) 3899–3918.
[24] X.L. Gao, X.N. Jing, G. Subhash, Int. J. Solids Struct. 43 (2006) 2193–2208.
[25] K.H. Kim, Y.C. Kim, E.c. Jeon, D. Kwon, Mater. Sci. Eng. A 528 (2011) 5259–5263.
[26] J.Y. Kim, B.W. Lee, D.T. Read, D. Kwon, Scr. Mater. 52 (2005) 353–358.
[27] S.K. Kang, J.Y. Kim, I.K. Kang, D. Kwon, J. Mater. Res. 24 (2009) 2965–2973.
[28] A.M. Korsunsky, A. Constantinescu, Thin Solid Films 517 (2009) 4835–4844.