EFFECT OF PILE-UP ON THE MECHANICAL CHARACTERISTICS OF STEEL WITH DIFFERENT STRAIN HISTORY BY DEPTH SENSING INDENTATION

Peter BURIK[a], Ladislav PEŠEK[b], Lukáš VOLESKÝ[a]

[a]Technical University of Liberec, Faculty of Mechanical Engineering, Department of Material Science, Liberec, Czech Republic, EU, peter.burik@tul.cz, lukas.volesky@tul.cz
[b]Technical University of Kosice, Faculty of Metallurgy, Department of Materials Science, Kosice, Slovakia, EU, ladislav.pesek@tuke.sk

Abstract

Depth sensing indentation allows deriving mechanical properties from the indentation load-displacement data using a micromechanical model developed by Oliver & Pharr (O&P). However, O&P analysis on the indentation unloading curve is developed from a purely elastic contact mechanics (sink-in). The applicability of O&P analysis is limited by the materials pile-up. However, when it does, the contact area is larger than that predicted by elastic contact theory (material sinks-in during purely elastic contact), and both hardness, H, and Young's modulus, E, are overestimated, because their evaluation depends on the contact area deduced from the load-displacement data. H may be thus overestimated by up to 60 % and E by up to 30 % depending on the extent of pile-up [1,2]. It is therefore important to determine the effect of pile-up on obtained mechanical characteristics of the material by depth sensing indentation.

The work experimentally analyses the effect of pile-up on the mechanical characteristics H and E of steel sample with different strain hardening history by Depth Sensing Indentation. Mechanical characteristics are determined by O&P analysis. Pile-up height was measured by atomic force microscopy (AFM).

Keywords: Berkovich diamond indenter, pile-up, hardness, Young's modulus, thin steel sheets

1. INTRODUCTION

In the O&P analysis, the H and E are determined from the load-displacement curve: The hardness is defined as:

$$H_{IT}^{O\&P} = \frac{P_{max}}{A_p} \tag{1}$$

projected contact area at P_{max} (HV=0.0925 H_{IT}). The reduced Young's modulus E_r is derived from the relation where P_{max} is the maximum indentation load and A_p is the:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A_p}} \tag{2}$$

where S is the contact stiffness computed from the initial slope of the unloading curve at the P_{max}, β is a constant that depends on the geometry of the indenter (β = 1.034 for a Berkovich indenter) and E_r is the reduced modulus given by:

$$\frac{1}{E_r} = \left(\frac{1-\nu_s^2}{E_{IT}^{O\&P}} + \frac{1-\nu_i^2}{E_i}\right)^{-1} \tag{3}$$

where ν_s and ν_i are the Poisson's ratios of the samples and the indenter, respectively, and $E_{IT}^{O\&P}$ and E_i are the corresponding Young's modulus (for a diamond indenter, E_i = 1141 GPa and ν_i = 0.07). From Eq. (2) and Eq. (3) can be the indentation modulus $E_{IT}^{O\&P}$ determined.

The projected contact area A_p is calculated by evaluating an empirically determined indenter shape function A = f(h_c). For an ideal Berkovich indenter, it is given by:

$$A_p^{O\&P} = 24.56 h_c^{2\,O\&P} \tag{4}$$

where $h_c^{O\&P}$ is the contact depth between material and indenter at the P_{max}, which is also deduced from the load–displacement curve using:

$$h_c^{O\&P} = h_{max} - \varepsilon \frac{P_{max}}{S} \tag{5}$$

where ε is a constant related to the geometry of the indenter (for a Berkovich indenter, ε = 0.75) and h_{max} is the maximum indentation depth [3]. O&P analysis on the nanoindentation unloading curve is developed from a purely elastic contact mechanics and that $h_c^{O\&P}$ is always smaller than h_{max}. If plastically deformed material piles-up around the indentation and corresponding contact boundary rises above the original sample surface, $h_c^{O\&P}$ (or $A_C^{O\&P}$) cannot be consistent with the real contact depth h_C (or real contact area A_C) anymore (Fig. 1) and the O&P analysis can strongly overestimate the H (up to 60%) and E (up to 30%) [4]. E is less affected because it is proportional to $1/\sqrt{A}$ (Eq. 2), whereas the H depends on 1/A (Eq. 1) [2]. The fundamental material properties affecting pile-up are the ratio of the yield stress $R_{p0.2}$ to Young's modulus ($R_{p0.2}/E$) and the work-hardening behaviour. In general, pile-up is greatest in materials with low $R_{p0.2}/E$ and little or no capacity for work hardening, that is, "soft" metals that have been cold worked prior to indentation. The ability to work harden inhibits pile-up because as material adjacent to the indenter at the surface hardens during deformation, it constrains the upward flow of material to the surface. The pile-up is not significant, irrespective of the work-hardening behaviour, when $R_{p0.2}/E > 0.03$ [2].

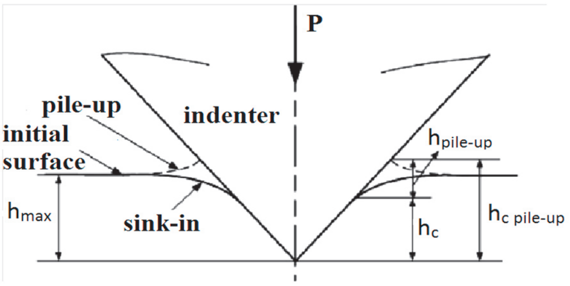


Fig. 1 Definition of real contact depth for material with pile-up [5]

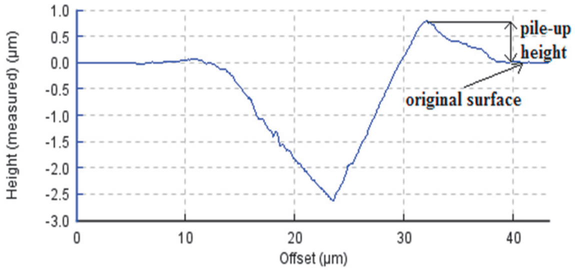


Fig. 2 Schema of pile-up height measuring

The true contact depth $h_{c\,pile\text{-}up}$ when pile-up occurs, **Fig. 1**, is equal to:

$$h_{c\,pile-up} = h_c + h_{pile-up} \tag{6}$$

The contact depth error calculated by Eq. (5) is:

$$\Delta h_c = h_c - h_{c\,pile-up} = -h_{pile-up} \tag{7}$$

Assuming the apparent values of $H_{IT}^{O\&P}$ and $E_{IT}^{O\&P}$ calculated by Eqs. (1) and (2), while the true values are H_0 and E_0, respectively. Thus, if we take only account for the influences of pile-up errors on the calculations of H and E, the following relationships can be obtained by analyzing Eqs. (1), (2) and (4) [5]:

$$\frac{H_0}{H_{IT}^{O\&P}} = \left(\frac{h_{c\,pile-up}}{h_c}\right)^{-2} \tag{8}$$

$$\frac{E_0}{E_{IT}^{O\&P}} = \left(\frac{h_{c\,pile-up}}{h_c}\right)^{-1} \tag{9}$$

Combining Eqs. (8) and (9), then [5]:

$$H_0 = H_{IT}^{O\&P}\left(1 + \frac{h_{pile-up}}{h_c}\right)^{-2}; \left(1 + \frac{h_{pile-up}}{h_c}\right)^{-2} = r_H \;\; => H_0 = H_{IT}^{O\&P} r_H \tag{10}$$

$$E_0 = E_{IT}^{O\&P}\left(1 + \frac{h_{pile-up}}{h_c}\right)^{-1}; \left(1 + \frac{h_{pile-up}}{h_c}\right)^{-1} = r_E \;\; => E_0 = E_{IT}^{O\&P} r_E \tag{11}$$

2. MATERIAL AND METHODS

The materials used in this study are thin steel sheets: (i) deep drawing IF steel XSG (ferrite), (ii) microalloyed steel HR 45 (ferrite-pearlite), and (iii) dual phase steel DP 600 (ferrite-martensite) with different deformation-strengthening characteristics. Thickness t and mechanical properties of the steels used are in Table 1. The measurements were carried out on loaded tensile specimens with polished surface in order to obtain hardness gradient. Longitudinal true deformation ψ after the tensile test was calculated by Eq. (12):

$$\psi = \frac{S_0 - S_u}{S_0} \tag{12}$$

where S_0 and S_u are the original cross-section and final specimen cross section area, respectively. Indentations, 5 for each load, were carried out with a CSM instrument equipped with a Berkovich diamond indenter at a constant loading rate of 400 mN/min from 0 to the maximum force (200 and 400 mN), with 10 s hold period and constant unloading, the distance between the indentations was 100 µm. The force-displacement curves were analyzed using the O&P analysis. After indentations, pile-up height was measured by atomic force microscopy (**Fig. 2**).

Table 1 Mechanical properties of investigated steels

Steel	$R_p0.2$ [MPa]	R_m [MPa]	A_{80} [%]	HV 1	n	t [mm]
DP 600	388	581	26.1	202	0.16	1.60
HR 45	360 R_e	449	27	179	0.139	1.80
XSG	177	286	47.2	120	0.211	1.95

3. RESULTS AND DISCUSSION

The measured data E and H determined by O&P analysis at different longitudinal position of a broken tensile specimen, thus with different deformation, are in **Fig. 3, 4**. XSG steel has greatest longitudinal true deformation ψ (0.81 - 0.23) and DP 600 steel has lowest ψ value (0.43 – 0.13), at the same measured length of the specimen. The measured HV value increases with increasing longitudinal specimen deformation. HV exhibit also a load effect. In general, HV tend to decline with increased load. Longitudinal deformation does not affect the E. Theoretical value of E was assumed E_0 = 200 GP a for all steels, using the DSI technique measured value E was around 210 GPa.

The mechanical characteristics of steels are affected beside the longitudinal true deformation also by pile-up. The maximum height of pile-up, different for each indent edge, is in the middle of the indent edge, in the majority of cases.

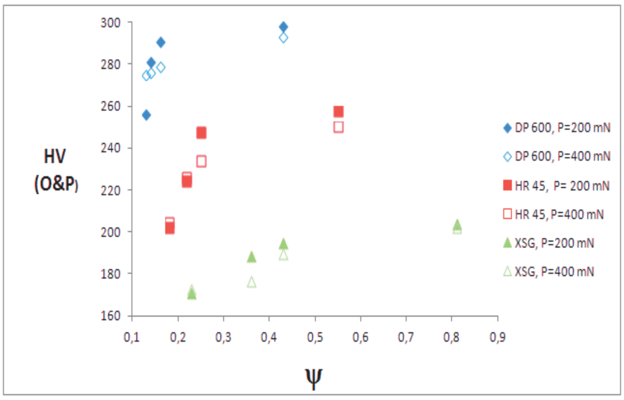


Fig. 3 Dependence of Vickers hardness HV on true deformation ψ

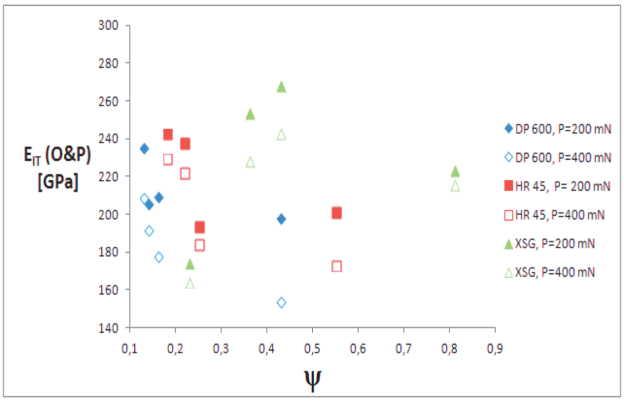


Fig. 4 Dependence of indentation Young's longitudinal modulus E on longitudinal true deformation ψ

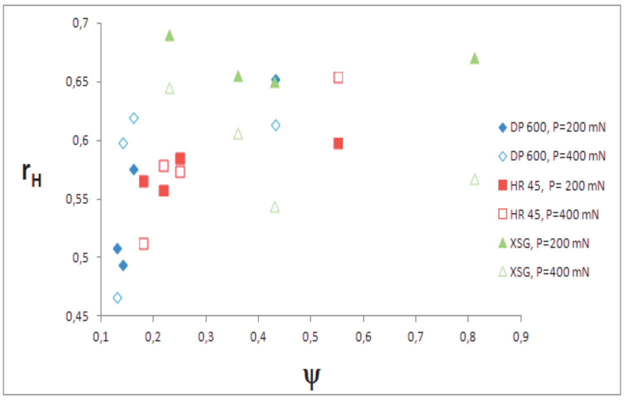


Fig. 5 Dependence of coefficient r_H on longitudinal deformation ψ

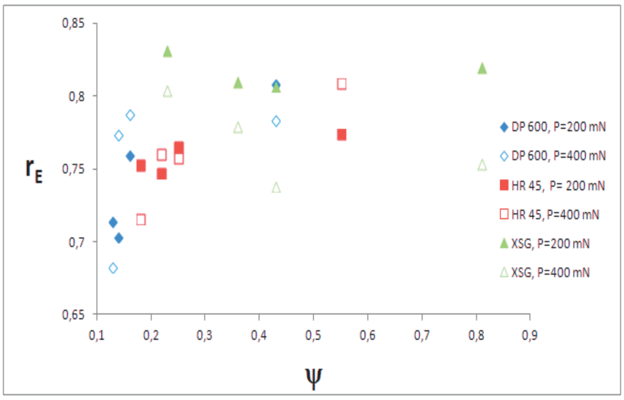
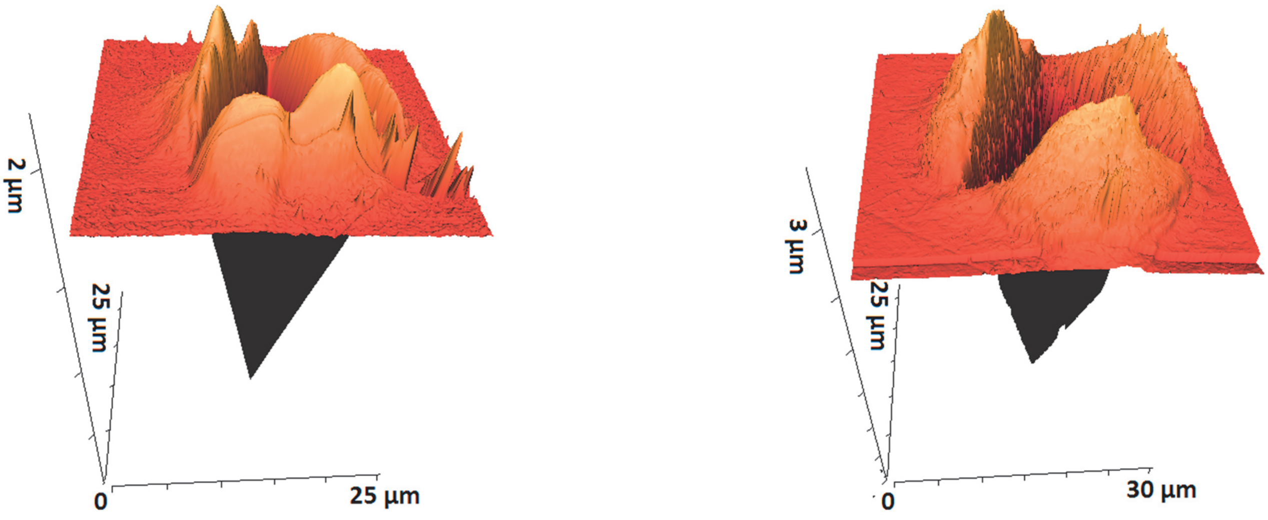


Fig. 6 Dependence of coefficient r_E on true longitudinal true deformation ψ

Pile-up height effect is expressed by a coefficient r_H for hardness and a coefficient r_E for Young's modulus (Eqs. 10, 11), **Fig. 5, 6**. Coefficients r_H and r_E increase with increasing ψ value deformation. The maximum force does not affect significantly the coefficients. Pile-up height has greater effect on the H than on the E. Values of H are underestimated with a maximum shift of 53 % and values of E are underestimated with a maximum shift of 32 %. DP 600 steel has lower both coefficients r_H and r_E. H is overestimated from 38 % to 53 %, and E is overstated from 19 to 32%. HR 45 steel has a similar effect of the pile-up height on mechanical characteristics as DP 600 steel. H is overestimated from 35% to 49 %, and E is overestimated from 19% to 29%. XSG steel has the greater coefficients r_H and r_E. H is overestimated from 31 % to 46 %, and E is overestimated from 17 to 26%. Coefficients r_H and r_E varies due to great variance of pile-up height at the indent. The coefficient respect an average height value measured on all three lobes at the edge of indent, **Fig. 7**.



a) b)

Fig. 7 Indentation impressions of HR 45 steel, magnification in z-direction = 15000 x, longitudinal true deformation ψ: a) 0.55, b) 0.18

4. CONCLUSIONS

Values of the mechanical characteristics of the steel are significantly affected by the longitudinal true deformation, indentation size effect and pile-up when using the depth sensing indentation technique. The effect of pile-up is more expressive than both indentation size effect and longitudinal true deformation. The pile-up effect decreases with an increasing of longitudinal true deformation for reasons of strain hardening. Strain hardening increasing constrains the upward flow of material to the surface. The pile-up has a greater effect on hardness than on Young's modulus. The Young's modulus is less affected because it is proportional to $1/\sqrt{A}$, whereas the hardness depends on $1/A$.

The pile-up effect increases with the strain-hardening exponent decreasing. Minimum effect of pile-up is for the XSG steel, n = 0.211, where Young's modulus is underestimated at maximum by 26 % and hardness is overestimated at maximum by 46 %. Maximum effect of pile-up is for the DP 600 steel, n = 0.16, where Young's modulus is overestimated at maximum by 32 % and hardness is overestimated at maximum by 53 %. Results are consistent well with the results of finite element simulation investigated by Bolshakov and Pharr [6].

O&P analysis is suitable for measuring the mechanical characteristics of steel by depth sensing indentation technique only after correction of measured values respecting the pile-up effect. Coefficients r_H and r_E are given for loads (200, 400 mN) and for longitudinal deformation of the steels.

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