Visual Abstract

The P-h^2 relationship as a function of (h_f/h_m) in indentation

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ABSTRACT. In the present study, a semi-empirical modeling of the mechanical response in pile-up mode is obtained by deriving the load-depth relationship during the indentation loading cycle. The advantage compared to the relations previously used is that this new expression is a function of the predictable criterion of the mode of deformation, (h_f/h_m), which makes it possible to distinguish the sink-in mode from the pile-up mode. A comparison between the proposed expression and the results of the instrumented indentation tests shows excellent agreement.

KEYWORDS. Indentation test; Mechanical response; Pile-up; Predictable criterion.

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INTRODUCTION

The study of the mechanical response by instrumented indentation has aroused the interest of many authors [1-4] although their experimental and numerical approaches are different who demonstrated that the load curve obtained from indentation tests can be described by the following power law:

$$P=Kh^2 \qquad (1)$$

where P is the indentation load applied to the indenter, h the indentation depth and K-factor a material-dependent constant. This relation was obtained experimentally as an alternative to classical approaches to determine the mechanical properties (Hainsworth et al., [1]), finite element approach (Zeng et Rowcliffe, [2]), and dimensional analysis (Cheng et Cheng, [3]). Then, relation (1) was used as a reference for a calibration of the indenter tip radius and load frame compliance (Sun et al., [4]). However, Hainsworth et al. [1] demonstrated that the value of the K-factor also depends on the geometry of the indenter tip and to the elastoplastic properties. These works [1-6] take as references the model proposed by [1] which is expressed as a function of the instrumented hardness, H, and the reduced modulus, Er. This expression of K as a function of reduced modulus of elasticity and instrumented hardness for sink-in mode has been refined by Malzbender et al. [5] and for pile-up mode has been refined by Habibi et al. [6] is formulated by Eqn. (2.a) and (2.b) respectively taking into account the geometry of the indenter and the tip defect as follows:

$$K = \begin{cases} E_r\left(\frac{1}{\sqrt{c}}\sqrt{\frac{E_r}{H}} + \varepsilon\sqrt{\frac{\pi}{4}}\sqrt{\frac{H}{E_r}}\right)^{-2} & (a) \\ E_r\left(\frac{1}{\alpha.\sqrt{c}}\sqrt{\frac{E_r}{H}} + \sqrt{\frac{\pi}{4}}\sqrt{\frac{H}{E_r}}\right)^{-2} & (b) \end{cases} \quad (2)$$

The deformation mode in the vicinity of the indenter depends on the elastoplastic response of the material under indentation. The two corresponding expressions of mechanical responses are different thus necessitating to previously considering one or the other model to determine the mechanical properties. However, to help the users to estimate the deformation mode, a simple criterion based on the ratio between the final depth and the corrected maximum depth reached by the indenter under the maximum load, (h_f/h_m), can be used [7-9]. The objective of this research is to refine the expression of K as a function of the criterion for identifying the deformation mode relating to the pile-up taking into account the geometry of the indenter and the tip defect. The advantage of the analytical expression proposed is its simplicity and the determination of (h_f/h_m) beforehand to determine the exact deformation mode of the material and to avoid the substitution by error of the sink-in mode by the pile-up mode or vice versa. The proposed model is then applied to bulk materials presenting pile-up deformation mode, i.e. copper, brass and bronze which have been the subject of previous investigations such as for example [10].

BACKGROUND

The methodology developed by Oliver and Pharr [11] to calculate hardness, H, and Young's modulus, E, in nanoindentation is currently applied in the majority of indentation work. On the other hand, this methodology is not justified for materials presenting pile-up as a mode of deformation [12-14]. From where, it is necessary to indicate the methods of calculation of the hardness and the modulus of Young in the two modes.
Oliver and Pharr [11] proposed to determine the reduced modulus Er, from the contact stiffness (the slope shown in Fig. 1.a) as follows:

$$E_r = \frac{S}{2}\sqrt{\frac{\pi}{A_c}} = \left(\frac{1-\nu^2}{E} + \frac{1-\nu_m^2}{E_m}\right)^{-1} \quad (3)$$

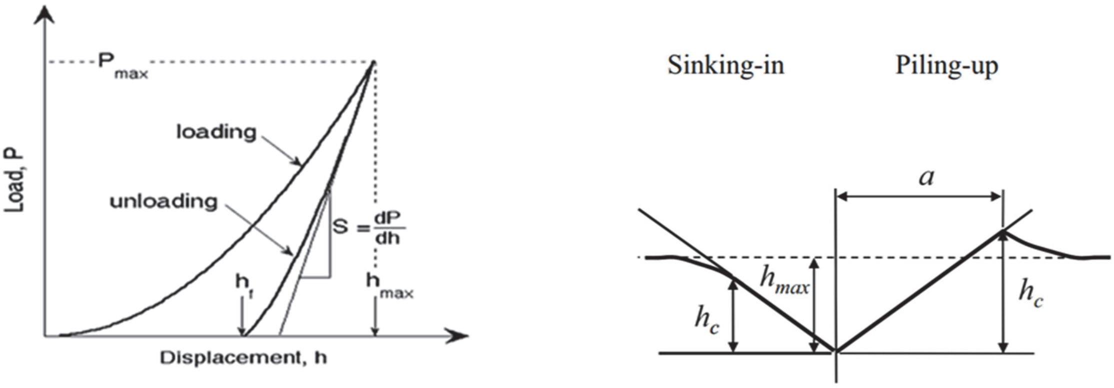
Indentation hardness is the ratio of the maximum applied, P, load to its projected contact, Ac, area between the indenter and the specimen as following:

$$H = \frac{P}{A_c} \quad (4)$$

Where E_m and ν_m are respectively the Young's modulus and the Poisson's ratio of the indented sample and E and ν are those of the indenter. Another relationship has been proposed previously [15] to express hardness as a function of indentation force and stiffness, concerning the two deformation modes by instrumented indentation.

$$A_c = \begin{cases} 24.56\left(h_m - \frac{P}{S} + h_d\right)^2 \; For\ sink-in[7](a) \\ 24.56\left(\alpha\left(h_m - \frac{P}{S}\right) + h_d\right)^2 \; For\ pile-up[8] \quad (b) \end{cases} \quad (5)$$

With the coefficient 24.56 resulting from consideration of the equivalent conical indenter associated with Vickers and Berkovich indenter tips have a semi-angle at the top of 70.3°.
Where h_m the maximum indentation depth, P the maximum applied load, S is the contact stiffness, ε a constant equals to 0.75 for Vickers and Berkovich indenters (for sink-in mode) and α is a constant equal to 1.2 (for pile-up mode) and h_d represents the length of the indenter tip defect.



(a) (b)

Figure 1: (a) Key parameters used in indentation characterization [7], (b) Schematic illustration of pile-up and sink-in around a pyramidal indenter [16].

The two semi-empirical relations (2.a) and (2.b) presented by the authors [5] and [6] respectively, are expressed as a function of the ratio of instrumented hardness on the reduced modulus, (H/Er). However, this characteristic and specific relationship of the material designated by (H/E_r) was expressed by the authors [8], according to the predictable criterion relating to the deformation mode (see Fig. (1.b)) designated by h_f/h_m, as follows:

$$\frac{H}{Er} = 0.2 \cdot \left(1 - \frac{h_f}{h_m}\right) \tag{6}$$

Note that h_f is the residual depth and h_m is the maximum depth as shown in Fig. (1.a). The calculated value of the h_f/h_m ratio designates the type of deformation mode under the indenter, namely the sink-in mode or the pile-up mode (see Fig. (1.b)). The critical value that separates the sink-in zone from that of the pile-up is estimated at 0.7 [7], 0.875 [8] and 0.83 [9]. For h_f/h_m lower than the critical value, the sink-in mode is predominant and for h_f/h_m higher than the critical value, the pile-up mode is predominant [7-9]. That value of 0.875 [8] is not appropriate for this case study because it does not take consider the corrections that have to be applied in order to take into account the bluntness of the indenter, the frame compliance or other factors which affect indentation response. From where, the analytical result when Γ=0.875 is used must be compared with data without correction while an analytical results with a Γ=0.83 [9] should be compared to data with correction of tip defect and compliance. Therefore, the critical value adopted during the present research, to identify the deformation mode by indentation is 0.83 [9].

MATERIALS AND EXPERIMENTAL METHOD

In this research, three distinct materials are studied: 99% pure commercial copper, bronze and 63/37 brass, hereafter designated by Cu99, SAE660 and C27200 respectively. The indentation test is applied to properly prepared specimens to limit surface roughness and the introduction of work hardening resulting from polishing followed by grinding with SiC papers of different grain sizes and finishing polishing using a series of diamond pastes up to a grain size of 1 μm. The tests are carried out using a CSM 2-107 instrumented microindenter (For a Vickers diamond indenter, Ei=1140 GPa and υi=0.07 [17]. The load range available on the indentation device varies from 0.1 to 30 N. The load resolution is given for 100 μN and the depth resolution for 0.3 nm.

Samples reference	Poisson's ratio ν	Forces range (N)
SAE660	0.30	0.2-10
Cu99	0.28	0.2-20
C27200	0.36	0.02-10

Table 1: Materials designation, Poisson's ratio, maximum force range, number of valid tests and tip defect lengths obtained by self-calibration.

The values of the loading and unloading rates (expressed in mN/min) were set at twice the value of the maximum applied load [18] and a dwell time of 15 s was imposed according to the standard indentation test procedure ASTM E92 and E384-10e2. Before analyzing the characteristic curves by indentation relating to the materials examined, the indentation device is systematically calibrated by identifying the conformity of the frame, C_f, by the self-calibration protocol.

RESULTS AND ANALYSES

Analysis of the criterion adopted by Giannakoupolos et al [8] concerning the relation between the ratio of hardness on the reduced modulus according to the indicator of the deformation mode applied in the present study on copper and its alloys is presented in the following figures:

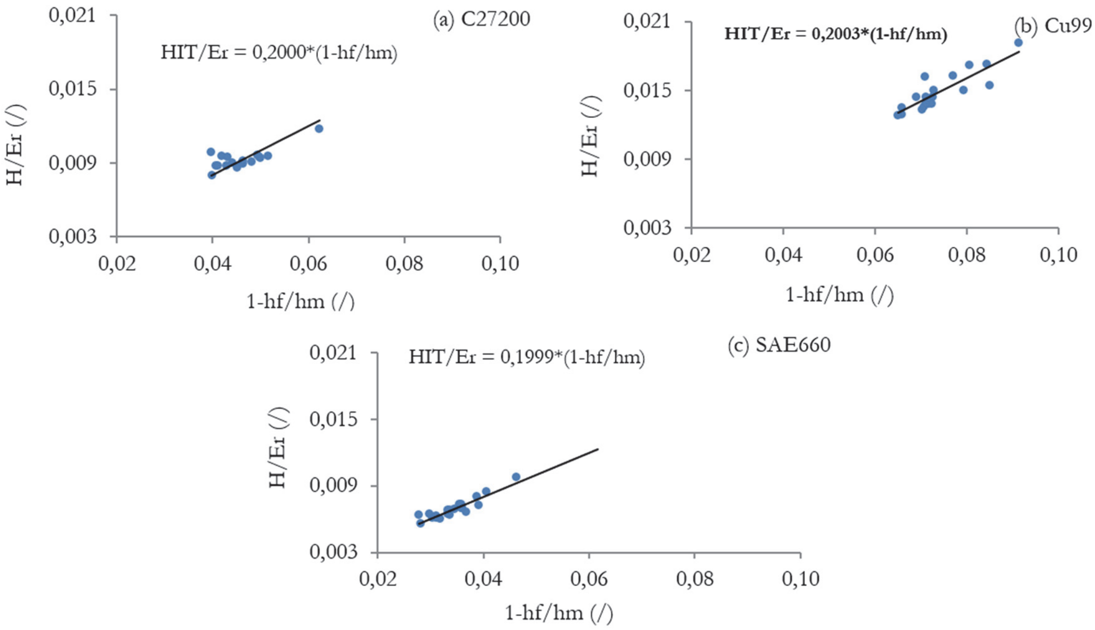


Figure 2: The (H/E_r)-$(1\text{-}h_f/h_m)$ relationship in indentation for C27200, Cu99 and SAE660.

The linear regressions from the graphical representations (see Figs. 3 (a,b,c)) show the following slopes: 0.2000, 0.2003 and 0.1999 for the materials C27200, Cu99, SAE660 respectively. These three slope values are equivalent to the value of the slope equal to 0.2 and which is proposed by Giannakopoulos et al. [8]. From where we confirm the value found by these authors [8] to express the relation (6).

Finally, we substitute the (H/E_r) ratio by the predictable deformation mode criterion, $\Gamma = h_f/h_m$ integrated in the relation $0.2*(1-h_f/h_m)$. The transformed analytic expressions (2.a) and (2.b) become as follows:

$$\begin{cases} Ksi = \dfrac{P}{\left(h_m + h_d\right)^2} = E_{rsi}\left[\sqrt{\dfrac{5}{c.(1-\Gamma)}} + \dfrac{\varepsilon}{2}\sqrt{\dfrac{\pi.(1-\Gamma)}{5}}\right]^{-2} & si \quad \Gamma < 0{,}83 \quad (a) \\ Kpu = \dfrac{P}{\left(h_m + h_d\right)^2} = E_{rpu}\left[\dfrac{1}{\alpha}\sqrt{\dfrac{5}{c.(1-\Gamma)}} + \dfrac{1}{2}\sqrt{\dfrac{\pi.(1-\Gamma)}{5}}\right]^{-2} & si \quad \Gamma > 0{,}83 \quad (b) \end{cases} \quad (7)$$

The Expressions (7.a) and (7.b) represent the new analytic expressions of mechanical responses corresponding to sink-in mode and pile-up mode respectively. These relations are expressed as a function of the reduced modulus specific to each deformation mode identified by the prediction mode criterion. We note that Γ separates the two zones of sink-in and pile-up for a critical value of 0.83 [9]. In the present work we focus on the validation of the expression (7.b) applied to copper and its alloys. Tab. 2 below shows that the three characterized materials admit a predominant pile-up deformation mode.

However the expression (7.a) will be used to compare the variations of results of calculation of mechanical responses in the event of substitution of a mode by another by error.

Samples reference	Γ	E_{rpu}(GPa)	E_{rsi} (GPa)	K_{anal} (GPa)	K_{exp} (GPa)	K_{anal}/E_{rpu}(/)
SAE660	0.96 ± 0.01	128 ± 19	150 ± 22	26.43 ± 0.05	26.30 ± 0.20	0,2065
Cu99	0.92 ± 0.01	91 ± 11	105 ± 12	37.00 ± 0.07	36,80± 0.80	0,4066
C27200	0.95 ± 0.01	91 ± 8	106 ± 8	23.79 ± 0.05	24.60 ± 0.20	0,2614

Table 2: Materials designation, reduced moduli E_{rsi} (sink-in mode) and E_{rpu} (pile-up mode), mechanical responses (Kanal, K_{exp}) and corresponding ratio (K_{anal}/E_{rpu}) for the tested materials.

We calculated E_r from Eqn. (3) as well as Eqns. (5.a) and (5.b) relating to sink-in, E_{rsi}, and pile-up, E_{rpu}, modes respectively. However, the experimental response, K_{exp}, is calculated directly from the experimental data ($P/(h_m+h_d)^2$) and the analytical response, K_{anal}, is calculated from Eqn. (7.b) proposed in this present research. The graphical representations of P-$(h_m+h_d)^2$ of the three materials are deliberately inverted to show their linear regressions without taking into account the tip defect (h_d=0) in continuous lines and taking into consideration the correction imposed by the tip defect equal to 205 nm, 221 nm, 245 nm in discontinuous lines for SAE660 (a), Cu99 (b) and C27200 (c) respectively (see Tab. 1).

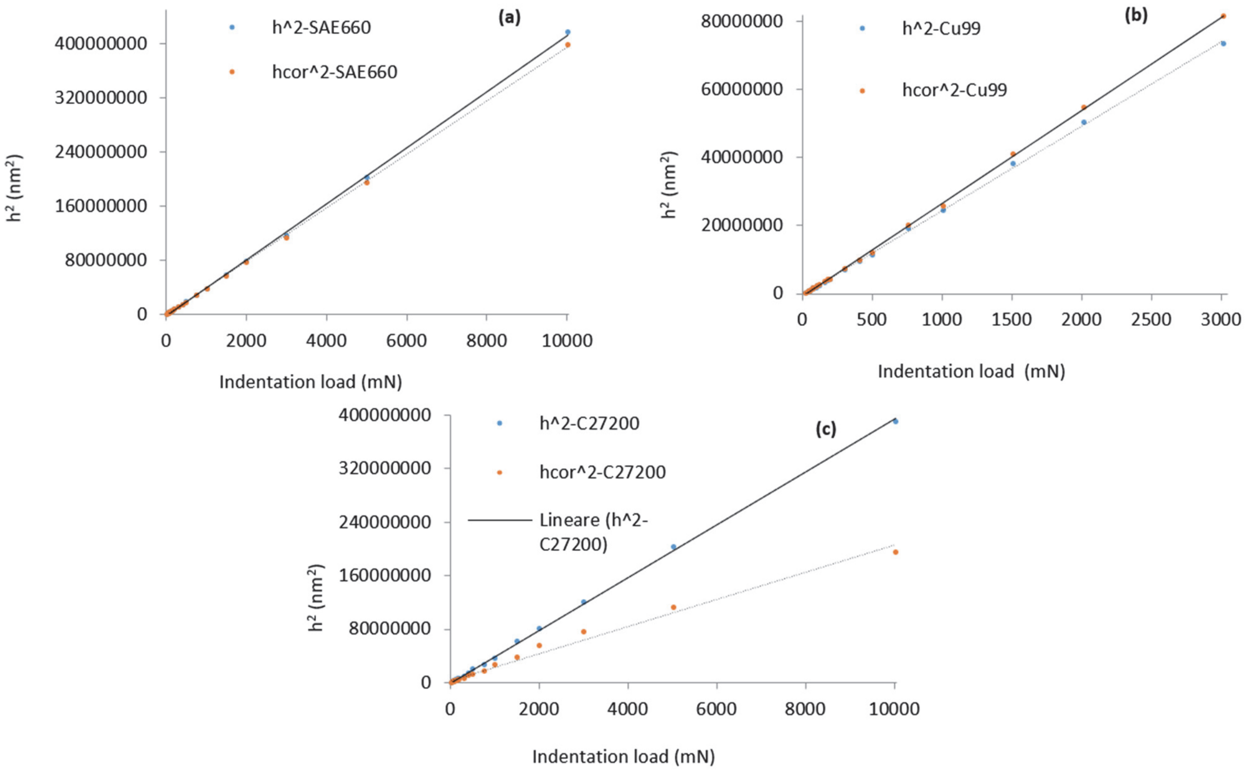


Figure 3: Linear regression of mechanical responses P-$(h_m+h_d)^2$ by indentation of bulk metallic materials.

The graphical representation (Fig. 4) consists in evaluating the difference between the linear regression taking into account h_d (with correction of h_m) and the regression without taking into consideration the truncation length, namely neglecting the value of h_d (without h_m correction) as shown in Tab. 3.

Tab. 3 shows an excellent factorial correlation of the linear regressions with very favourable reproducibility rates which tend towards 100% for the cases with or without the imposed corrections. However, the percentage differences between (h_d=0) and (h_d=205nm, h_d=221nm, h_d=245nm) for C27200, Cu99 and SAE660 register 48.70%, 9% and 4.3% respectively. Therefore, the differences are evident according to Tab. 3, which tend to affect the precision of the results of the mechanical responses and in particular when it comes to the scales of microindentation and eventually nanoindentation. The magnitudes of the estimated deviations vary from one material to another, for example for C27200 the difference is estimated at 48.7%, considered very substantial and is likely to generate significant characterization errors. For the other two materials, the values of 9% and 4.3% are significantly important in the reliability of the results of the expected mechanical responses. Hence the need to integrate the h_d in the corrections imposed by the tip defect to take into account the effect of the truncation length in the characterization calculations and in particular in the present work.

Samples reference	Regression without correction	Regression with correction	Percentage difference [%]
SAE660	$h^2 = 20265*P + 3.10^6$ with R^2=0.9868	$(h+h_d)^2$= 39505*P + 638539 with R^2=0,9995	48.7
Cu99	$h^2 = 24845*P – 452152$ with R^2=0.9995	$(h+h_d)^2$= 27288*P – 791652 with R^2=0,9996	9.0
C27200	$h^2 = 41414*P – 2.10^6$ with R^2=0.9994	$(h+h_d)^2$= 39622*P – 2.10^6 with R^2=0,9997	4.3

Table 3: Results of linear regressions with and without the introduction of correction imposed by the indenter tip defect.

The expressions proposed (Eqns. (7.a) and (7.b)) in this work and in particular Eqn. (7.b) relating to the pile-up is expressed as a function of the reduced modulus in pile-up mode. Figs. 5 show the variation of Er (pile-up) as a function of the maximum depth of indentation.

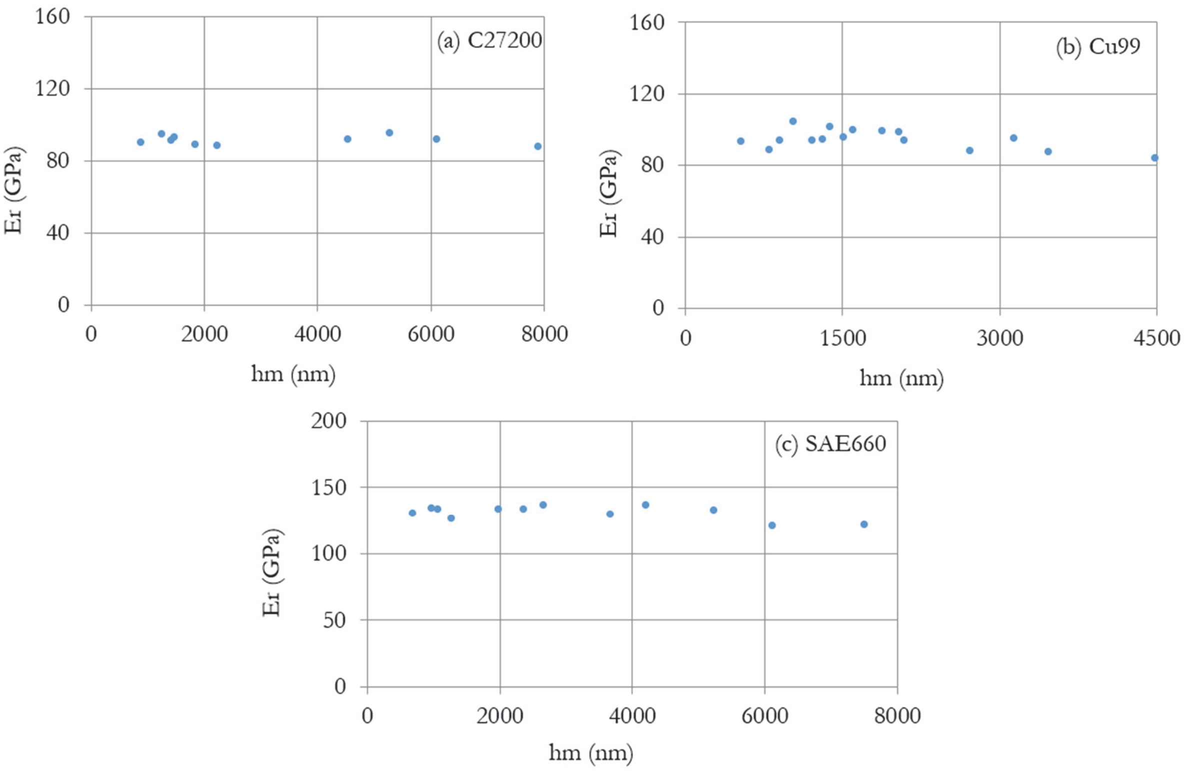


Figure 4: Evolution of reduced modulus in pile-up mode vs. the maximum indentation depth.

These Figs. 4 clearly show that the Er are substantially constant and the effect of the depths is almost insignificant for the three materials studied. Hence the confirmation of the intrinsic character of the module examined for copper and its alloys studied. For the three characterized materials, in pile-up mode, we express the relationship between the K_{exp} and the K_{anal} as shown in Figs. 5.

Linear regressions show very favourable factorial correlations with excellent experimental reproducibility rates. This shows that the expression specific to the pile-up mode (7.b) of semi-empirical analytical type is very compatible and coherent with the experimental data.

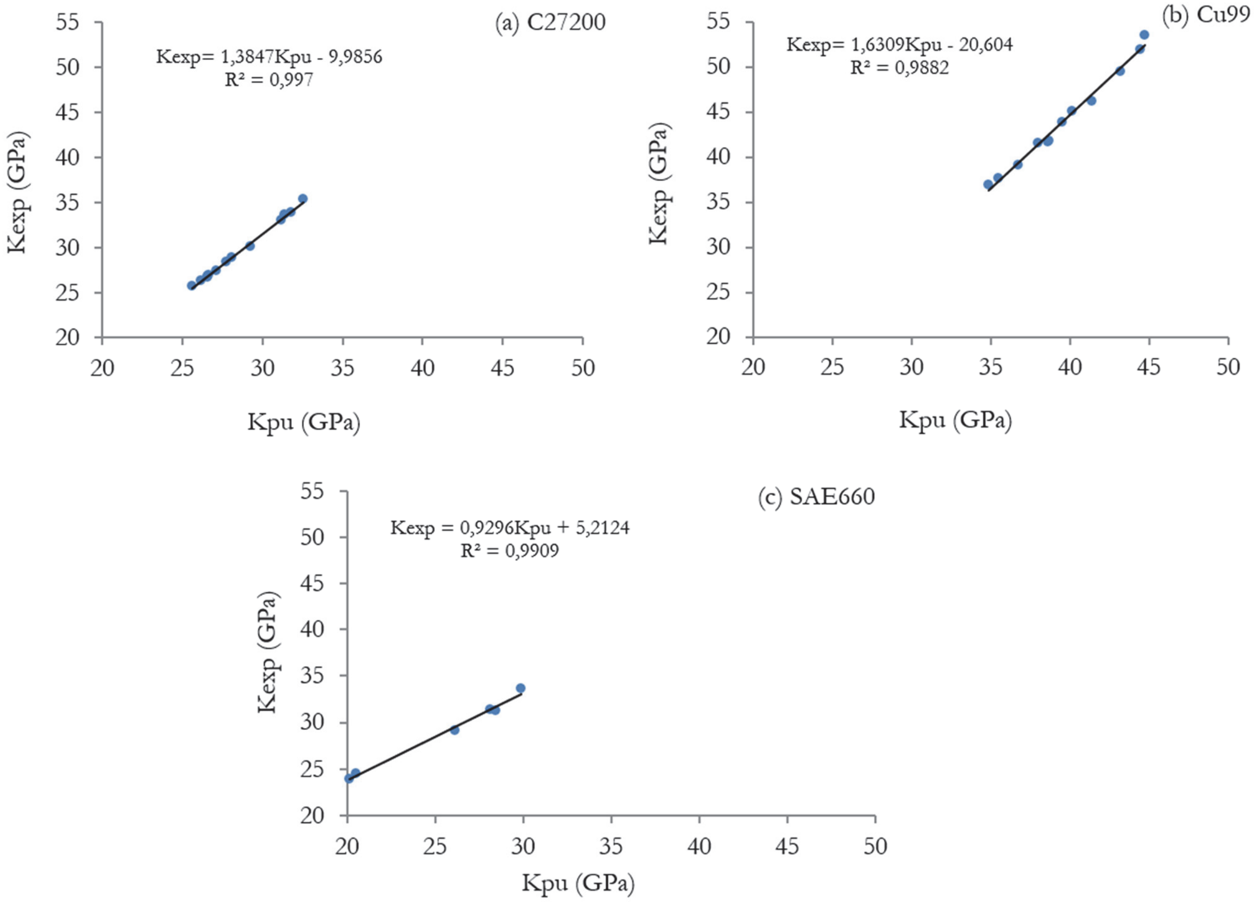


Figure 5: Correlation between K_{exp} and K_{anal} in the case of pile-up mode.

If we reformulate this same relation (7.b) by expressing K_{anal} on Er, we obtain a new relation which varies only as a function of Γ as is expressed in Eqn. 8:

$$\frac{K_{anal}}{E_{rpu}}=\frac{\mathrm{P}}{\left(\mathrm{h_m}+\mathrm{h_d}\right)^2.E_{rpu}}=\left[\frac{1}{\alpha}\sqrt{\frac{5}{\mathrm{c}.\left(1-\Gamma\right)}}+\frac{1}{2}\sqrt{\frac{\pi.\left(1-\Gamma\right)}{5}}\right]^{-2}\quad \text{if}\quad \Gamma>0.83 \tag{8}$$

The graphical representations 7 allow the understanding and interpretation of Eqn. (8). We have previously found that Figs. 5. (a,b,c) of the reduced pile-up moduli of copper and its alloys show a cloud of characteristic points which are substantially constant. When we study the K_{anal}/Er_{pu} variation we also find a substantially constant point trend as shown in Figs. 6. This implies logically and mathematically (by transitive relation) that the mechanical response K_{anal} is effectively constant. Hence the validation of the semi-empirical model proposed in the present investigations.

The histograms 8 represents a comparative study between the mechanical responses of the materials examined in pile-up mode, K_{pu}, calculated by Eqn. (7.b) and in sink-in mode, K_{si}, calculated by Eqn. (7.a) as well as the (direct) experimental expression assumed to be independent of the deformation modes, namely $P/(h_m+h_d)^2$ as follows:

For the three materials, $\Gamma=h_f/h_m$ is greater than the critical value of 0.83 [9] which shows the predominance of the pile-up mode. So we try to estimate the K_{SI}, K_{PU} and K_{EXP} responses in this interval defined by [7-9] as being the pile-up mode zone. We note that the three calculated responses K_{SI}, K_{PU} and K_{EXP} are different and we also notice that K_{PU} is closer to K_{EXP} unlike K_{SI}. Hence, the computational expression Eqn. 7b is more adequate for the pile-up than the inappropriate exploitation of Eqn. 7a relating to the sink-in if it is used in the case of a pile-up by error (not justified by the calculation of Γ less than 0.83). Therefore, the influence of the choice of the deformation mode expressed as a function of Γ on the reliability of the results of the mechanical responses. Finally we try to evaluate the differences between the different responses K_{SI}, K_{PU} and K_{EXP}.

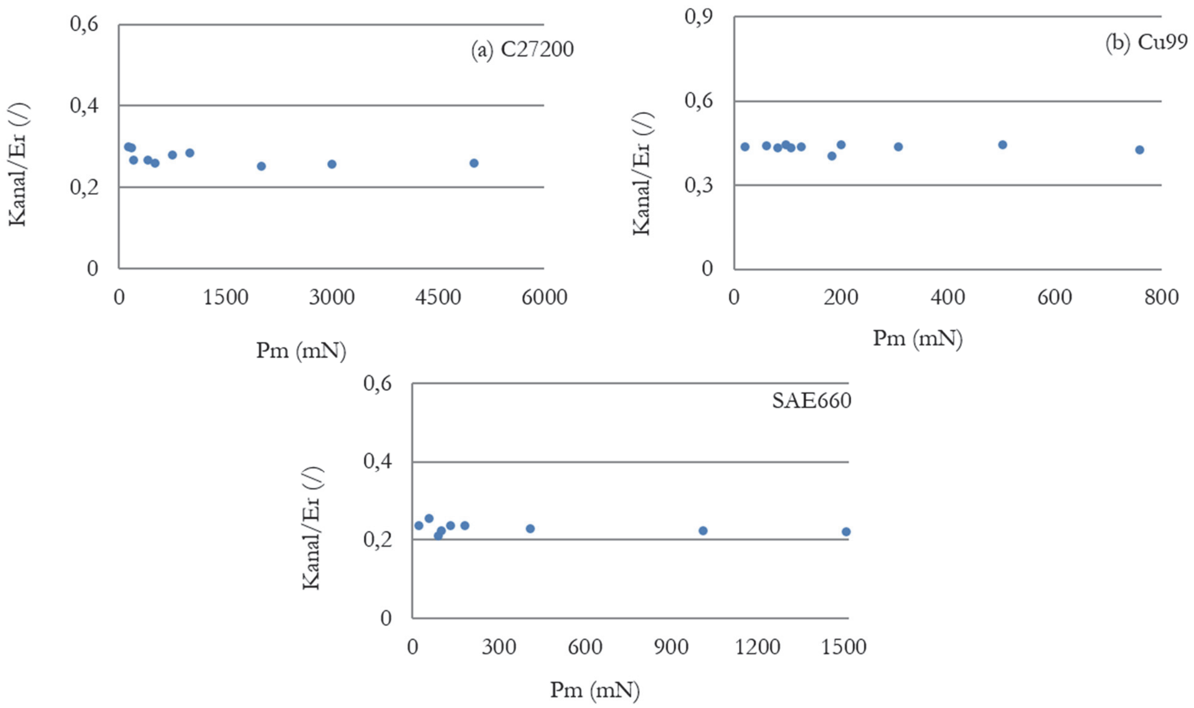


Figure 6: Appreciation of the characteristic point clouds of (K_{anal}/Er) according to the maximum indentation load.

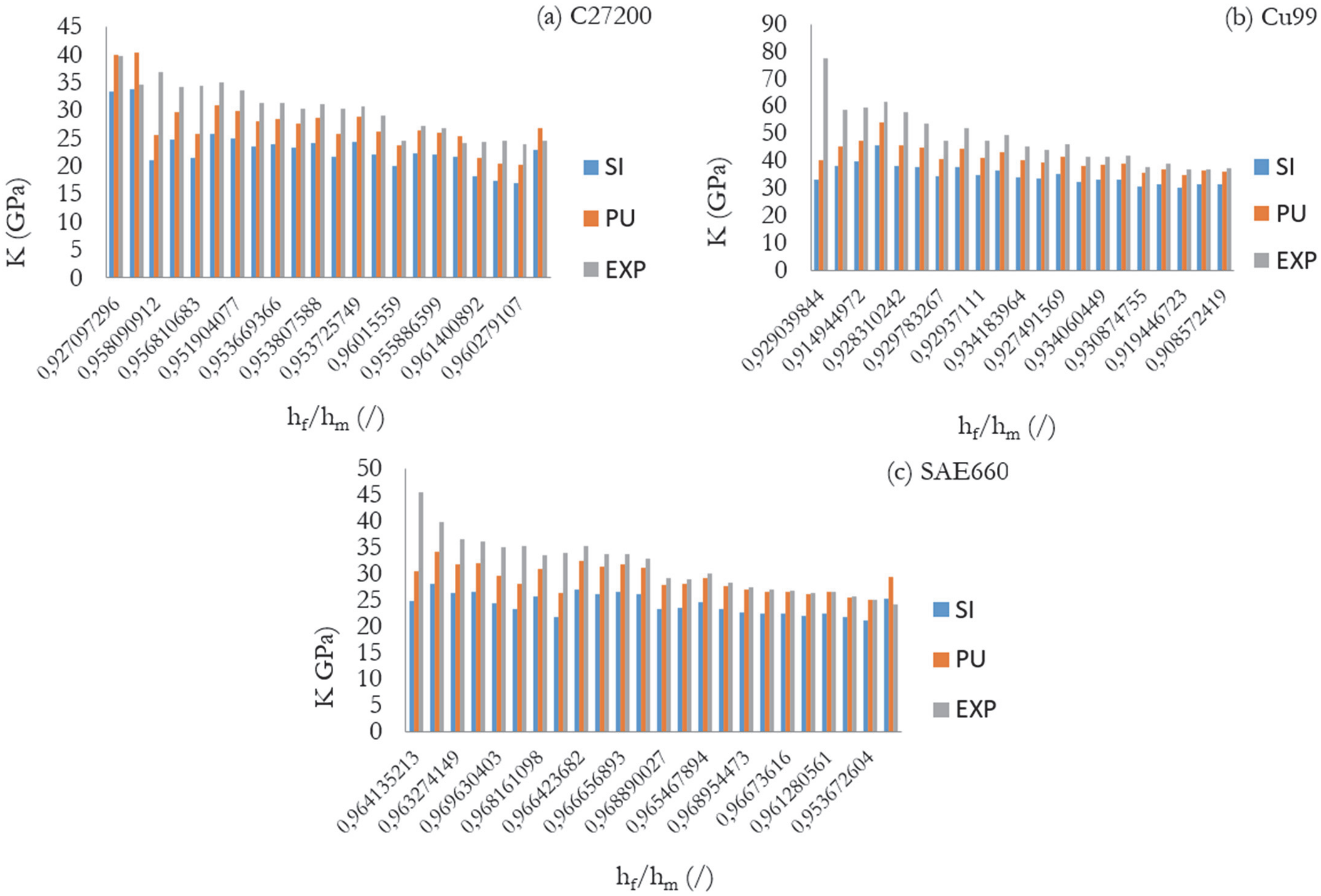


Figure 7: Assessment of K_{EXP}, K_{PU} and K_{SI} for copper and its alloys according to the deformation mode criterion.

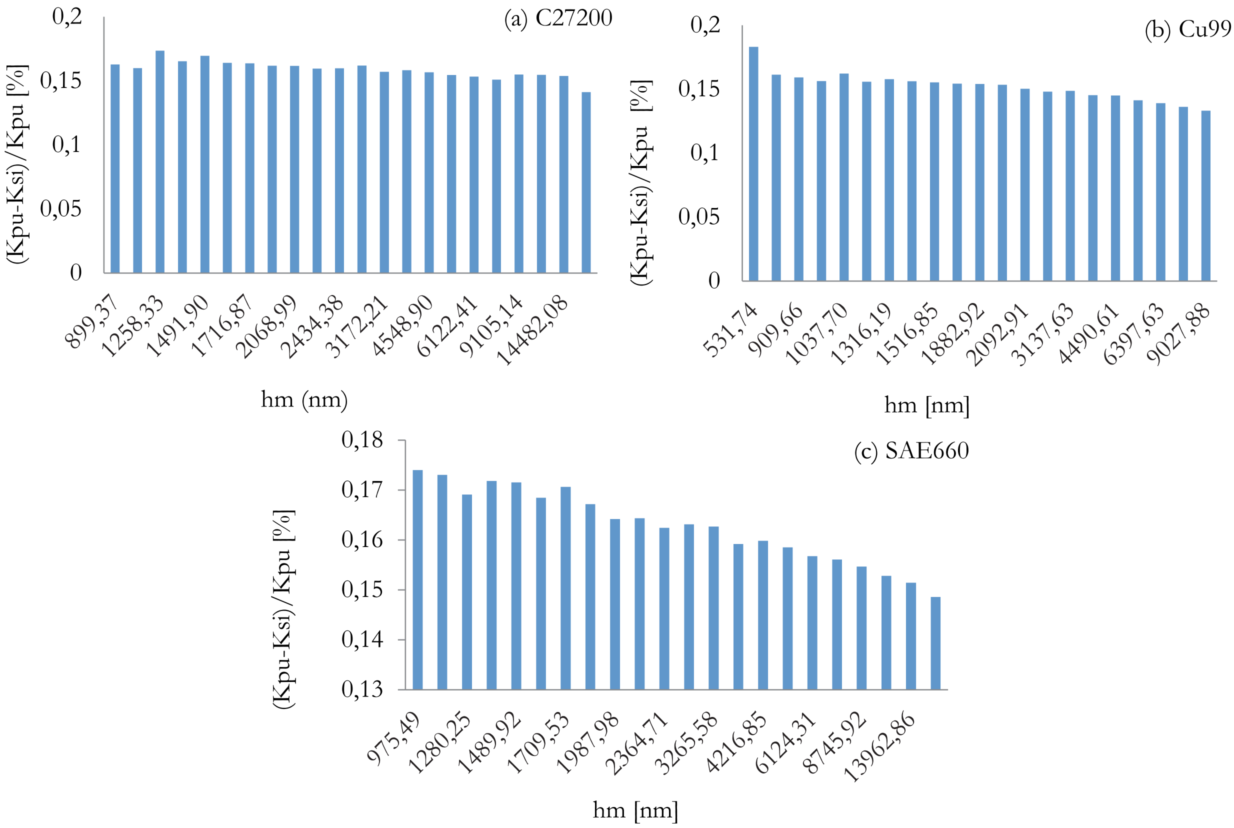


Figure 8: Histogram expressing the averages of the differences in the mechanical responses between the pile-up and the sink-in as a percentage.

Samples reference	(Kpu-Ksi)/Kpu [%]	(Esi-Epu)/Esi [%]
SAE660	16.28	14.67
Cu99	15.32	05.71
C27200	15.91	14.15

Table 4: Estimated mean differences in mechanical responses and reduced moduli between Sink-in mode and Pile-up mode.

The differences in the mechanical responses between the pile-up and sink-in deformation modes of the three materials studied are shown in Figs. 9 and are estimated on average at 16% as shown in Tab. 4 above. These mechanical responses are functions of the reduced moduli relating to the two deformation modes by indentation from where we note the influence of the reduced moduli differences on the results of the analytical expressions K_{pu} and K_{si}. The estimated differences of these modules are estimated at 14.67%, 5.71% and 14.15% for SAE660, Cu99 and C27200 respectively. As other authors have observed [8,19,20] who find an error that can reach 30% if the deformation mode of the projected area is not taken into account in the calculations. So, finally, the mechanical response is a function of the reduced modulus, the deformation prediction criterion expressed by the ratio of (h_f/h_m), thus reflecting the nature of the material. Obviously the chosen indenter geometry also has an influence on the mechanical response knowing that for the Vickers indenter the empirical constant integrated in the analytical expression is c=24.56 (see Eqns. (7.a) and (7.b)). However, in this work, we cannot estimate the exact influence of the geometry on the mechanical response because we will have to compare other indenters such as the Berkovich point and the spherical point. The taking into account of the deformation modes is dominating on the reliability and the precision of the mechanical responses results calculated by the Eqns. (2.a) and (2.b) on the one hand.

CONCLUSION

In conclusion, we have derived an analytical expression for the load-depth relationship during loading in an indentation experiment, namely Eqn. (7.b). The advantage over similar relationships derived previously is that the proposed equation is expressed as a function of the deformation mode prediction criterion and the reduced modulus which provides computational ease and caution in distinguishing between the sink -in and the pile-up. A comparison between the results achieved by the proposed analytical expression and the experimental expression shows an excellent correlation for the bulk metallic materials studied. The substitution of sink-in mode by pile-up mode and vice versa can generate a significant result error on the mechanical response which is estimated at around 16%. Taking into account the correction imposed by the tip defect is necessary because the length of truncation can considerably affect the results of mechanical responses. In perspective, investigations concerning the influence of work hardening on the mechanical response by indentation are provided.

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