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COMPARISON OF METHODS FOR ASSESSING MATERIAL PROPERTIES FROM SPHERICAL INDENTATION TEST ON THREE TYPES OF STEEL

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Abstract. *To reduce the costs associated with the conventional tensile test, the Instrumented Spherical Indentation Test (ISIT) can be adopted. The ISIT consists of applying a controlled force through a spherical probe on the material's surface while measuring the resulting penetration depth. From these data, different analysis methods for characterizing mechanical properties of materials were developed in the past and, more recently, optimization based approaches were also proposed. In this context, the main objective of the present paper is to compare the results obtained with classical analysis methods (specifically Haggag's method and ISO/TR29381) with a recent proposal (Yu's method) for three common types of steel (4340 28HRC, 4340 40HRC and 4130M 25 HRC). In particular, the accuracy in determining the yield stress σ_y, the ultimate tensile strength σ_{UTS} and the strain hardening parameter n is analysed. The results showed that, for the considered materials, Yu's method can produce more accurate values for σ_y with an error between 2.5% and 4.3%. However, for the determination of n, the classical Haggag's method was more consistent, with an error of 3.1%. Lastly, all investigated methods resulted on adequate estimates for σ_{UTS} meanwhile, with a maximum error of 6.5%, the ISO method proved to be the most consistent among the others. These results can help the choice of an appropriate method for analysing ISIT results for steels depending on the property of interest.*

Keywords: *Mechanical Properties, Spherical indentation, Strain Hardening, Strength Coefficient, Yield stress.*

1. INTRODUCTION

One of the steps during a mechanical project is the material specification, that is, selecting the materials which fulfill the performance requirements (Chung, 2006). In this step, knowing in advance the mechanical properties of the available materials is crucial for the designer. These properties can be obtained from conventional tests such as tensile, tenacity and hardness (Fredel *et al.*, 2015).

In particular, the tensile test can be employed to evaluate various mechanical properties of materials that are of great importance to the project, such as Yield Stress σ_{y}, Young's Modulus E, Ultimate Tensile Strength σ_{UTS}, Strength Coefficient K and Strain Hardening Exponent n. This test consists of deforming a specimen by applying a uniaxial and incremental tensile load until it fractures. During this procedure, the sample is fixed to the testing machine which is designed to lengthen the specimen at a constant rate in addition to measure the applied load (typically with a load cell) and the elongation of the sample (with a strain gauge). Thus, the material properties can be estimated from the stress-strain curve (Callister, 2000).

Another widely used approach to characterize the mechanical properties of materials is the Standart Test Method for Linear-Elastic Plane Strain Fracture Toughness K_{ic} of Metallic Materials (ASTM International, 2013). According to Anderson (2017), a material fractures occurs when some sufficient stress applied at the atomic level breaks the bonds that hold atoms together. The fracture energy can also be influenced by crack meandering and branching, which increase the surface area. As a result, the microstructure and mechanical properties of materials are greatly influenced by the orientation of grains caused by the crack. This sensitivity can be noticed by performing tenacity tests, which consists of a specimen with lateral notch with a pre-crack through a cyclic load applied. The specimen is fastened by means of fixing claws that will pull the specimen parallel to the crack orientation until it fractures. The displacement generated will be obtained by the opening of the crack and can be measured through a strain gauge (Melo, 2019). Besides from the aforementioned parameters, the experimental data generate from this test can be used to evaluate the Fracture Toughness $K_{J_{IC}}$.

The methods described so far are classified as destructive tests, since they result in the loss of the tested specimen. As alternative, the non-destructive test called indentation can be adopted Tabor (2000). This consists of applying a load P through a spherical (or other shape such as conical or pyramidal) indenter on the material producing a deformation with depth h on its the surface. The indenter is pressed against the sample until a chosen strength or depth is achieved. After that, it is removed at a constant rate proportional to the pressing rate until the force or displacement reaches a small percentage of the maximum applied load. The total indentation depth h_t is usually defined as 24% of the indenter radius R (Tairui *et al.*, 2019). This loading/unloading cycle is repeated for a chosen number of cycles. Thus, a $P \times h$ curve can be constructed from the data acquired during the experiment (see Fig. 1 for a single loading/unloading cycle), where the contact stiffness S is defined by the slope of the initial unloading curve, when the material response is totally elastic. Moreover, by extrapolating the tangent line of unloading curve until zero load, the residual indentation depth h_p is defined. From this curve, various methods were purposed for the determination of material properties. For instance, Haggag and Murty (1997), ISO (2008), Li *et al.* (2016), Yu *et al.* (2023).

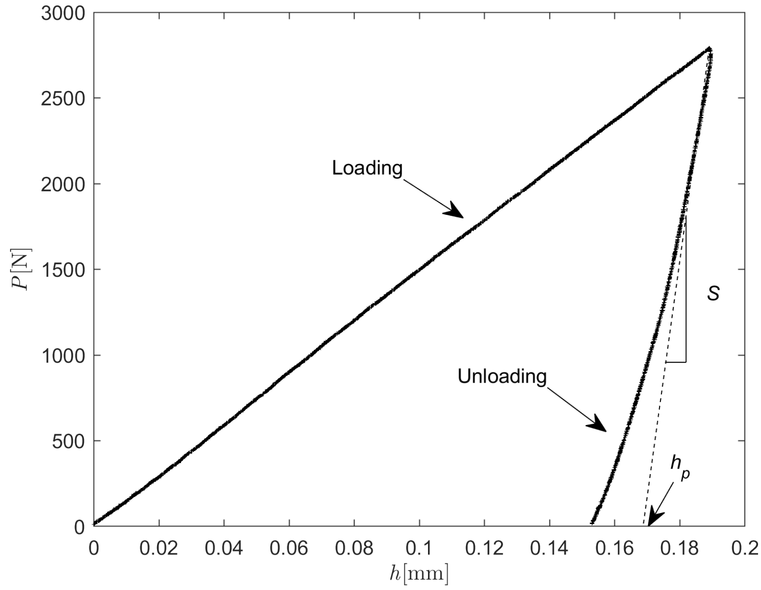


Figure 1: Illustration of a load-depth curve typical of Instrumented Spherical Indentation Test (ISIT).

In this context, the main objective of the present paper is to compare different methods that use indentation data to estimate mechanical properties of materials. The well established methods from Haggag and Murty (1997) and ISO (2008), and the recent proposal of Yu *et al.* (2023) will be employed and their results will be compared to the ones obtained with the conventional tensile test. With this objective, these methods were implemented in *Matlab*® and employed for estimating material properties from spherical indentation tests for three commonly adopted steels with known properties. Specifically, 4340 28HRC, 4340 40HRC and 4130M 25HRC. Such comparison can be helpful for the choosing of a analysis method for these type of materials. Moreover, the impact of the total penetration ratio (i.e. the final h_t/R value) on the accuracy of estimated parameters is also investigated.

The remainder of this paper is organized as follows: The considered analysis methods are described in Section 2.The obtained results are then presented and discussed in Section 3. Concluding remarks and suggestions for future work are given in Section 4.

2. DESCRIPTION OF ALGORITHMS FOR ESTIMATING MECHANICAL PROPERTIES FROM INDENTATION TEST

2.1 Haggag's Method

Haggag and Murty (1997) proposed a portable stress-strain microprobe system which can be employed to evaluate the true-stress/true-plastic-strain behavior and determine the mechanical properties of different metallic materials. The system consists of a software that automates the indentation test controlled by a computer providing visually available graphs in real time and digital display of load-depth test data. Initially the sample is indented with a spherical tungsten indenter while monitoring load and depth by means of load cell and linear variable differential transformer (LVDT), respectively. From these data, the mechanical properties (σ_{y}, σ_{UTS}, K, n) are calculated following the procedure shown in Fig. 2.

In the flowchart, the input variables are the indenter diameter D and Young's modulus of the indenter E_i and the specimen E, as well as the maximum load applied during the i-th loading cycle $P_{max,i}$ and its associated $h_{p,i}$. From these inputs, the plastic indentation diameter $d_{p,i}$ can be calculated and used to obtain the true plastic strain $\epsilon_{p,i}$ and the flow stress σ_i for $i = 1,\ 2, \dots,\ N_c$, being N_c the chosen number of loading/unloading cycles. The constant δ depends on the stage of deformation of the plastic zone under the indenter and varies between 1.12 and $2.87\alpha_m$ being $\alpha_m = 1.2$. Thus, the Hollomon power law $\sigma = K\epsilon^n$ is fitted to the generated data resulting on the parameters K and n enabling the σ_{UTS} calculus. As proposed in Nicolosi (2015), herein the Yield Stress is calculated from $\sigma_y = \beta_m\eta + b_m$, where η is obtained from the regression analysis of d_t/D versus P/d_t^2, $\beta_m = 0.4324$ and $b_m = -329$. Note that this differs from the original proposal of Haggag and Murty (1997) where $\sigma_y = \beta_m\eta$, with $\beta_m = 0.2285$. However, as shown in Nicolosi et al. (2015), the adopted approximation generate better results for the considered steels.

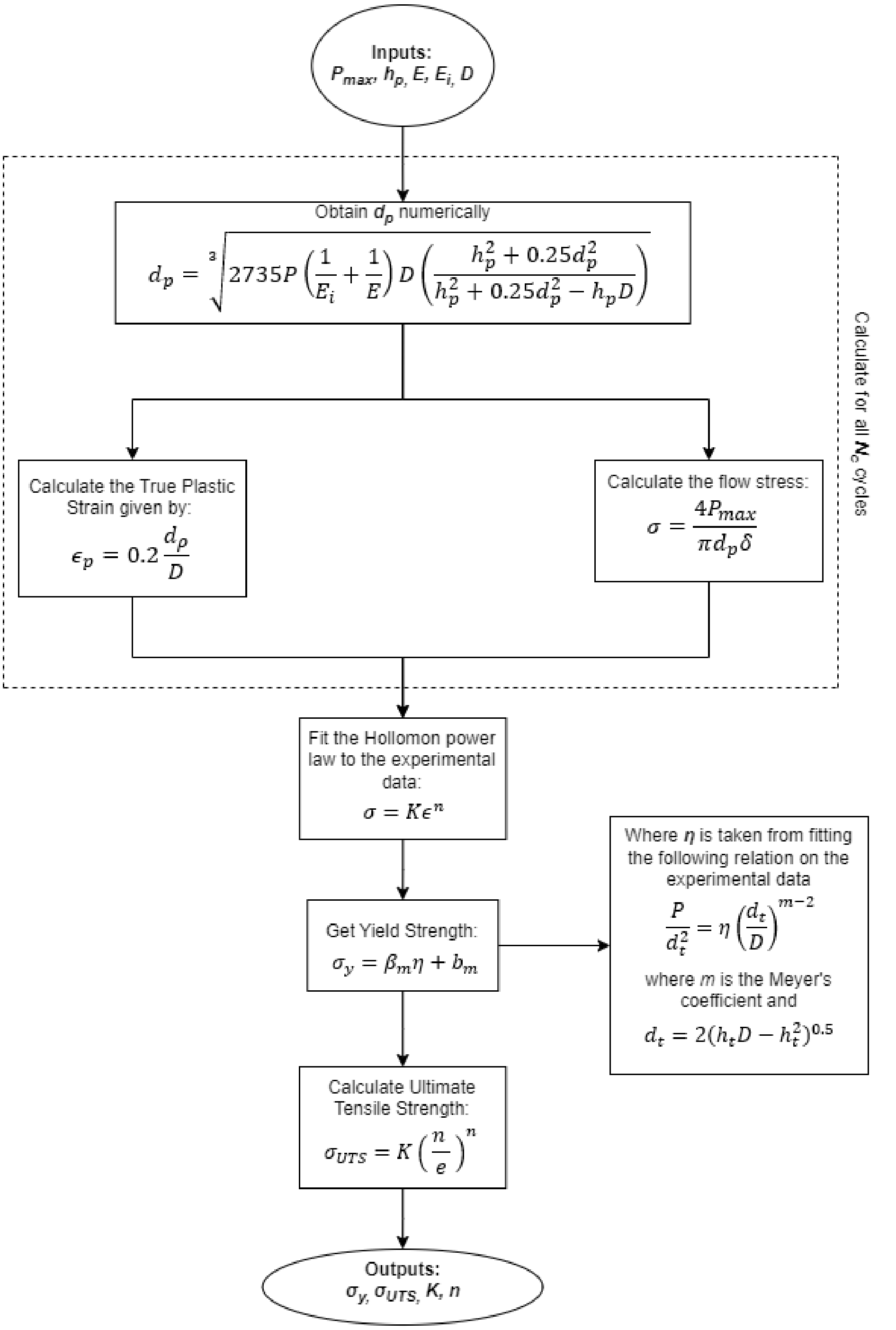


Figure 2: Flowchart of Haggag's method for calculating material properties from ISIT.

2.2 Dutch guideline's Method (ISO 29381/2008)

Another method to estimate the material properties from ISIT data can be found in ISO/TR29381 (ISO, 2008). The key idea is to optimize the Strain Hardening Exponent n in order to fit the Hollomon power law to the experimental data. This method can be implemented following the flowchart of Fig. 3 as presented in Li *et al.* (2016).

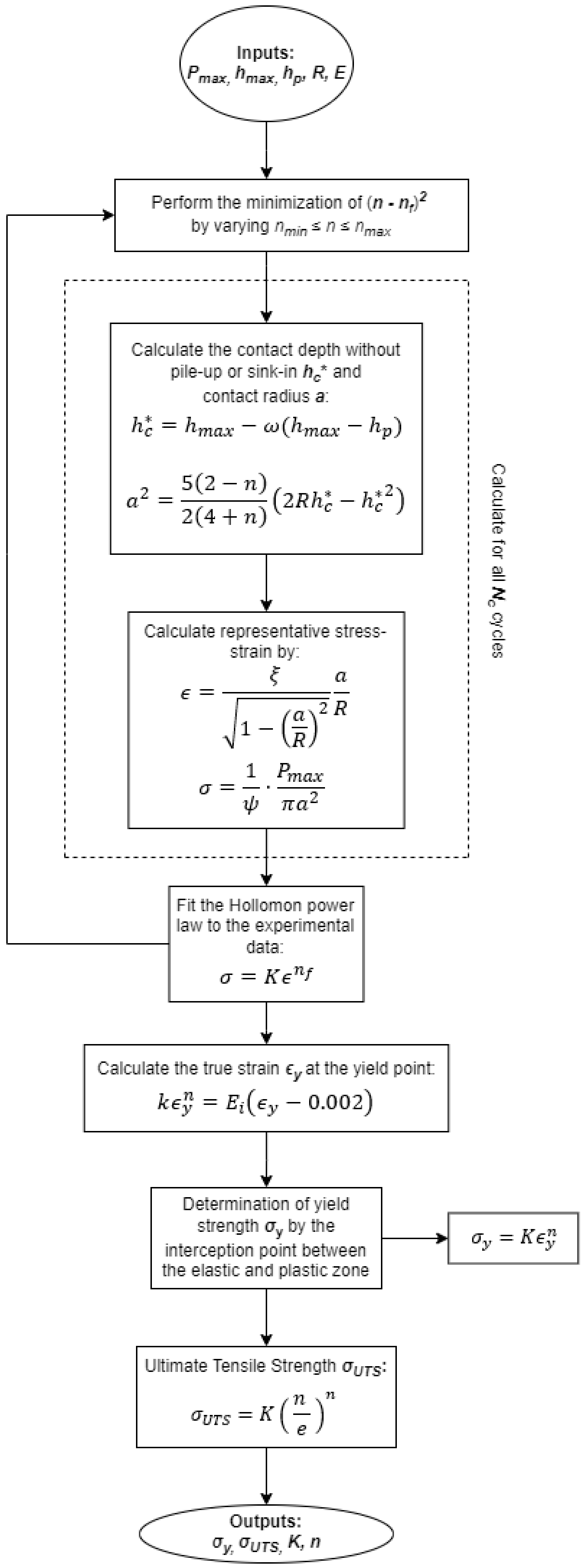


Figure 3: Flowchart of Dutch guideline method for calculating material properties from ISIT.

As described by the flowchart, the inputs for this method are h_p, E and the indenter radius R, as well as the maximum load and depth of each loading cycle (denoted respectively by P_{max} and h_{max}) taken from the indentation test. An initial value of n is defined in order to calculate the contact depth between the indenter and the specimen without considering pile-up or sink-in assuming $\omega = 0.75$ value and the contact radius a. After, the representative stress-strain can be obtained where $\xi = 0.14$ is a material independent constant and $\psi = 3.0$ (Francis, 1976). Thus, by solving a nonlinear least squares problem the objective is to minimize the disparity between observed stress values (σ) and computed values determined by the equation for various n values. This optimization is performed by varying n iterative until finding the value that minimizes the difference, the fitted n_f. Finally, from n and K it is possible to obtain ϵ_y which is the strain deformation at the yield point, also allows the calculation of σ_y. As can be seen, σ_{UTS} was obtained as in the Haggag's method.

2.3 Yu's Method

The recent paper of Yu *et al.* (2023) presents a new methodology to extracting tensile properties from ISIT. A procedure is proposed for calculating the Young's modulus (E) of the material. However, in this context, we will employ the theoretical value for consistency in comparison with other methods. The tensile properties are estimated by using an improved representative stress-strain approach with the Meyer power law correction. Fig. 4 shows its flowchart. The main difference from the method described in Section 2.2 is that the nonlinear least squares problem is solved to determine K and n. From these values, the contact radius a is calculated as before and E can be found by the respective equation assuming $\beta = 1$ for spherical indenters. Thus, the coefficients m and η of the Meyer power law are obtained by fitting such law on the experimental data. In the sequence, the Yield Stress is obtained from $\sigma_y = \beta_m \eta + b_m$. Even though Yu *et al.* (2023) suggest values for β_m and b_m, herein the values presented in Section 2.1 will be adopted since they were determined for the materials tested in our work. Lastly, one calculates σ_{UTS} using the conventional expression. The paper describes the testing using two different types of portable indentation machine set as 15 number of cycles with three indented loading–unloading curves each to verify the accuracy and repeatability of the proposed ISIT methodology.

3. RESULTS AND DISCUSSION

The methods described in Section 2 have been implemented in *MATLAB*® version R2019a. In particular, such methods were employed to calculate the mechanical properties of 4340 28HRC, 4340 40HRC and 4130M 25HRC steels whose chemical composition can be found in the Tab.1.

Table 1: Chemical composition (%) of the different materials studied.

	AISI 4130M	**AISI 4340 28/40HRC**
C	0.29	0.47
Mn	0.769	0.723
Cr	1.48	0.791
Ni	0.126	1.72
Mo	0.691	0.311
Si	0.3150	0.33
P	0.0139	0.0118
S	0.0032	0.0011
Al	0.0383	0.0268
Cu	0.216	0.0978
Nb	0.0239	0.0069
Ti	0.0042	0.0167
W	-	0.0301
V	0.0097	0.006
B	0.0013	0.0015

These three steels were selected due to their isotropic nature, meaning they exhibit consistent physical properties regardless of direction. Moreover, they possess well-defined characteristics which allows verifying the properties obtained from each analysis method. The specimens adopted for indentation were generated by the following procedure: A specimen is removed from each material (block of approximately 25.10 x 65 x 10 mm) and grinding so that surface imperfections are removed from the material. Thus, sanding is performed to remove scratches of the previous step. This process was done initially with a sandpaper of less granulation degree which was gradually increased so that the surface becomes increasingly smooth and with less porosity. The grit sandpapers used during preparation were 240, 360, 600 and 1200. The specimen is then polished with cloths and diamond lubricant of $9\,\mu\mathrm{m}$, $6\,\mu\mathrm{m}$ and $3\,\mu\mathrm{m}$ to remove any imperfection left in the specimen. Finally, the specimen is cleaned with alcohol to avoid oxidation.

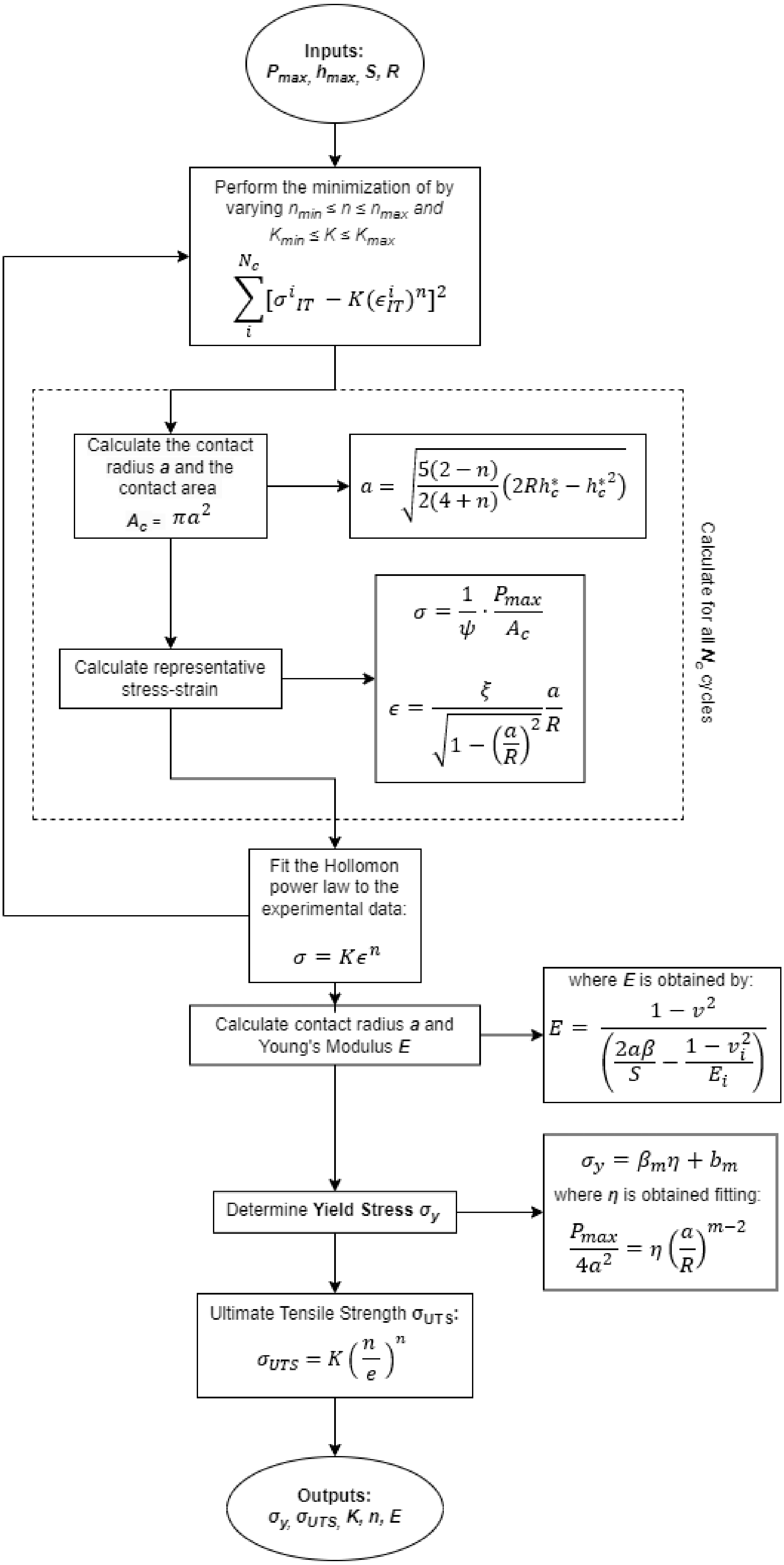
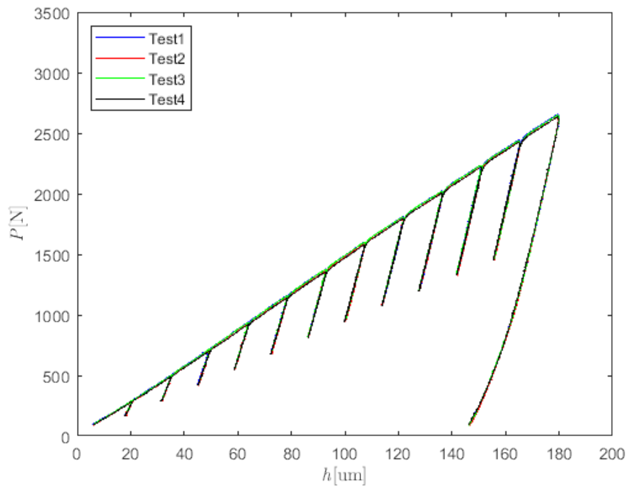


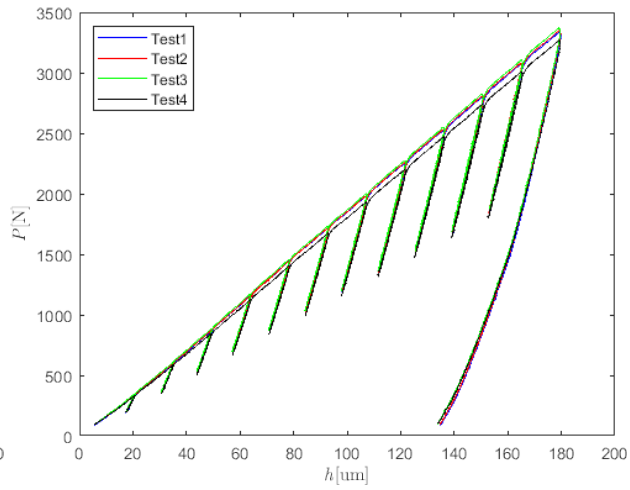
Figure 4: Flowchart of Yu's method for calculating material properties from ISIT.

Once the material preparation is finished, the specimens can be tested. This was accomplished by using the Haggag's equipment (Haggag and Murty, 1997) using a tungsten carbide spherical indenter with a $D = 1.5\,\mathrm{mm}$. In the present work 12 loading/unloading cycles were done during the tests. The obtained $P \times h$ curves for each material are shown in Fig. 5. Note that four indentations were performed on each material. As can be seen, the results from these indentations are similar, as expected.

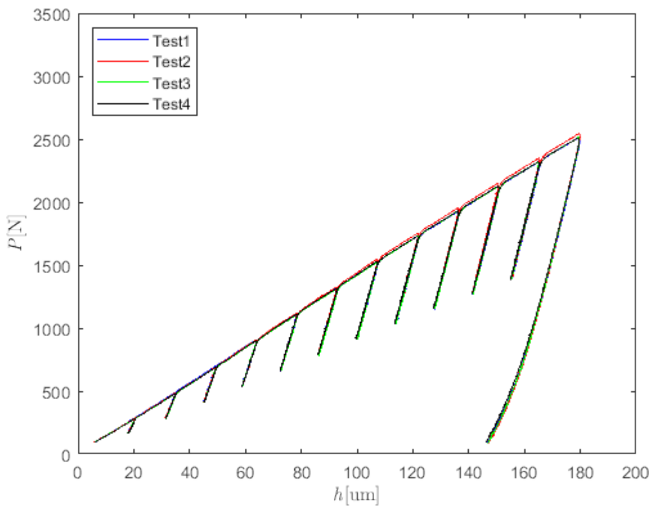
The average values of load and depth were adopted for employing the methods described in Section 2. The indentation results shown on Tables 2, 3 and 4 are compared with the results from a tensile test carried out within the laboratory following the standard test method for tension testing of metallic materials (ASTM International, 2013). The optimization procedures were performed by considering $n_{min} = 0.05$, $n_{max} = 0.3$, $K_{min} = 1500$ and $K_{max} = 3000$.



(a) Load/Depth curve for 4340 28HRC material.



(b) Load/Depth curve for 4340 40HRC material.



(c) Load/Depth curve for 4130M 25HRC material.

Figure 5: Results of indentation tests on the adopted metals.

Table 2: Mechanical properties calculated from indentation methods and convetional tensile test for the material 4340 28HRC.

Material Properties (MPa)	**Conventional Tensile Test**	**Haggag**	**ISO**	**Yu**	**Error (%)**		
					Haggag	**ISO**	**Yu**
Yield Stress σ_y	776.0	976.6	885.0	798.2	25.8	14.0	2.9
Ultimate Tensile Strength σ_{UTS}	911.9	944.3	971.1	982.9	3.6	6.5	7.8
Strain Hardening parameter n	0.11	0.11	0.05	0.05	3.1	54.5	54.5

For 4340 28HRC steel is observed that the Yu's method presented better results for the calculation of σ_y while Haggag's show certain consistency for σ_{UTS} and n as can be seen in Tab. 2. For 4340 40 HRC shown in Tab. 3, the ISO and Yu's method resulted on best estimates for σ_y and σ_{UTS} (absolute errors of 1.3 % and 0.9 %, respectively). For the working hardness exponent, Yu's method and the ISO approach showed equal results (an absolute error of 17 %), where the obtained $n = 0.05$ was the defined lower bound for n. The results presented in Tab. 4 demonstrate that Yu's method had a smaller error for σ_y (about 4.3 %) while ISO show good results for σ_{UTS} with an absolute error of 4.9 %. The hardening exponent n was best represented by Haggag's method.

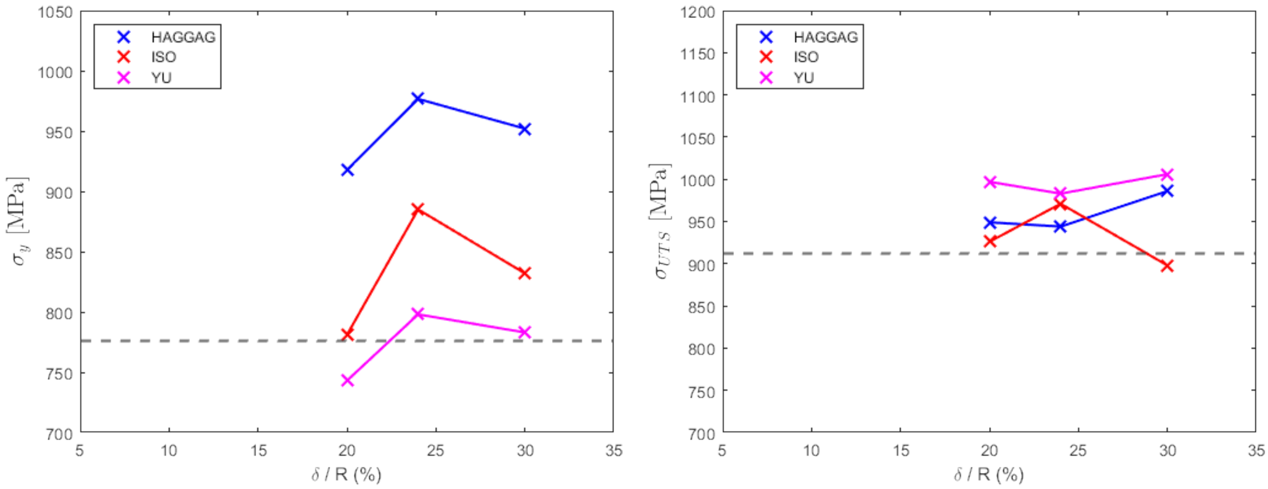
Table 3: Mechanical properties calculated from indentation methods and convetional tensile test for the material 4340 40HRC.

Material Properties (MPa)	Conventional Tensile Test	Haggag	ISO	Yu	Error (%)		
					Haggag	ISO	Yu
Yield Stress σ_y	1152.0	1314.0	1137.0	1123.0	14.1	1.3	2.5
Ultimate Tensile Strength σ_{UTS}	1248.0	1216.0	1206.0	1260.0	2.6	3.4	0.9
Strain Hardening parameter n	0.06	0.09	0.05	0.05	63.3	16.6	16.6

Table 4: Mechanical properties calculated from indentation methods and convetional tensile test for the material 4130M 25HRC.

Material Properties (MPa)	Conventional Tensile Test	Haggag	ISO	Yu	Error (%)		
					Haggag	ISO	Yu
Yield Stress σ_y	775.8	898.9	823.7	742.8	15.9	6.2	4.3
Ultimate Tensile Strength σ_{UTS}	848.4	914.5	890.0	946.8	7.8	4.9	11.6
Strain Hardening parameter n	0.124	0.09	0.05	0.05	24.2	59.7	59.7

As mentioned in Campbell *et al.* (2018), if the plastic deformation beneath the indenter is too low, the resulting data may not be adequated to calculate the material properties. Therefore, in order to verify the influence of penetration ratio ($\delta = h_t/R$) on the estimated properties, indentations were performed for three different penetration ratios: 20 %, 24 % and 30 %. The results are shown in Figures 6, 7 and 8. The gray dashed line points the material parameter from the tensile test.



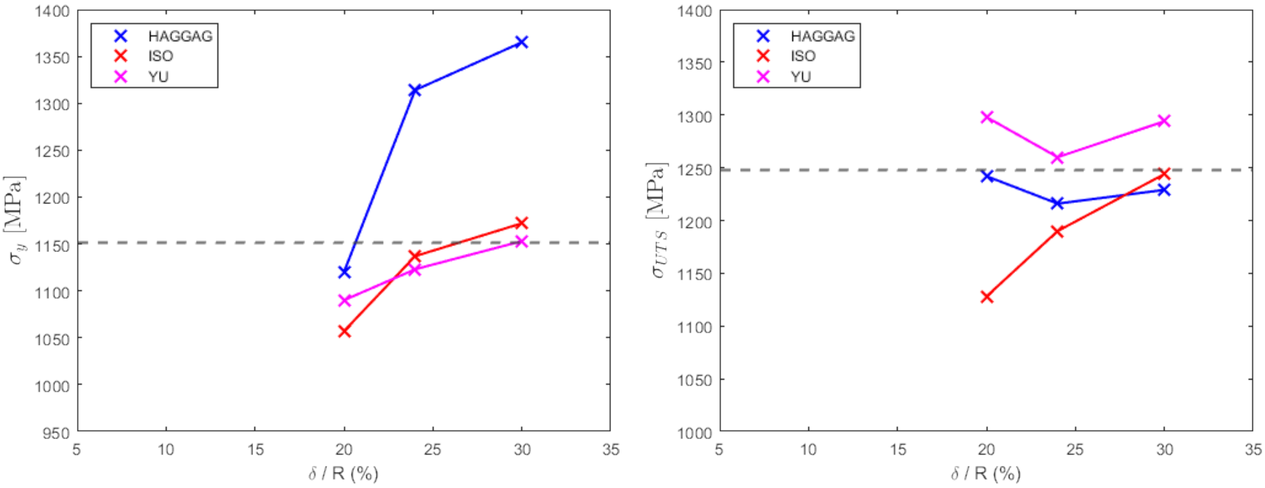
(a) σ_y results for the three presented methods.

(b) σ_{UTS} results for the three presented methods.

Figure 6: Graph of the stresses in function of the (%) penetration ratio for 4340 28HRC steel.

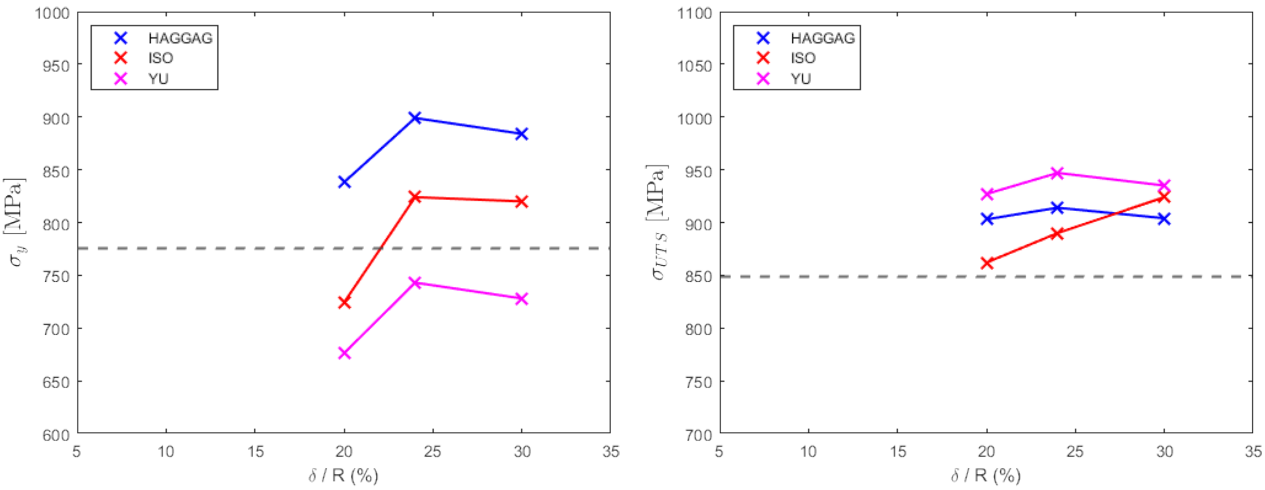
From the test carried out in the 4340 28 HRC steel, it is possible to observe that for a penetration of 20% give better results for all ISIT methods.

In contrast, for 4340 40 HRC, increasing the penetration ratio resulted on better estimates for the ISO 2008 and Yu's methods, while for Haggag's method the results shows some variations. Lastly, for 4130M 25HRC, larger values of δ were associated with larger estimates of σ_y. While for values of σ_{UTS} the methods presented very consistent results regardless of δ value.



(a) σ_y results for the three presented methods. (b) σ_{UTS} results for the three presented methods.

Figure 7: Graph of the stresses in function of the (%) penetration ratio for 4340 40HRC steel.



(a) σ_y results for the three presented methods. (b) σ_{UTS} results for the three presented methods.

Figure 8: Graph of the stresses in function of the (%) penetration ratio for 4130M 25HRC steel.

Despite some variations between the methods in Fig.8 the yield stress predictions at a 20% penetration ratio were notably more accurate using both the ISO and Haggag methods, along with consistent outcomes for σ_{UTS}. Conversely, when employing a 30% penetration ratio, the Haggag, ISO and Yu's method generated closely values for σ_{UTS}.

It can be observed that for low hardness steels such as 4340 28HRC and 4130M, the methods presented closer values when using a δ of 20%. On the other hand, for harder materials like 4340 40HRC, a larger δ leads to better results. Therefore, the penetration ratio (δ/R) can be chosen according to each material to be analyzed, using a smaller δ for materials with lower hardness and a larger δ for materials with higher hardness.

It must be pointed out that the penetration ratio analysis was not performed for the strain hardening exponent n due to the large errors of Yu's and ISO's methods. These errors can be attributed to several sources, including uncertainties in the experimental data, the sensitivity of the methods to variations in input data, and the inherent complexity of the material under investigation. Accurately assessing the strain hardening exponent n is critical for understanding the behavior of materials subjected to plastic deformation. Given the importance of this constant, it is imperative to conduct a more thorough and precise assessment in future studies. Such an investigation may involve developing new analysis techniques or adapting existing methods to mitigate the observed errors.

4. CONCLUSION

This study involved the comparison of different methods for calculating material parameters from spherical indentation data for three different steels. Specifically, the results of ISIT for 4340 28HRC, 4340 40HRC and 4130 25 HRC were analyzed with the methods of Haggag and Murty (1997), ISO (2008) and Yu *et al.* (2023) and compared with the ones of conventional tensile test. Through this comparison, we identified strengths and limitations of each method. The ISO 29381 method has consistently demonstrated its reliability in estimating ultimate tensile strength (σ_{UTS}) values, showcasing an error ranging from 3.37% to a maximum of 6.5%. This makes it a strong contender for evaluating this parameter. On the other hand, Yu's method excelled in predicting yield stress values, demonstrating exceptional robustness with a maximum error rate of 4.25%. However, the results for n fell short of expectations, exhibiting significant disparities. It was observed that the resulting was equal to the minimum n allowed value during the optimization. By following the Haggag's method a good estimate for n could be calculated for the 4340 28 HRC. However, the results for 4340 40 HRC and 4130 25 HRC were not adequated.

An analysis of influence of the penetration ratio on the results were also performed. It showed that tests with 24% penetration ratio, results on good agreement among all methods and all materials, with some notable exceptions, such as the yield stress analysis for 4340 28HRC steel. It's worth noting that materials with a high n value tend to exhibit significant deformations over a broader area, while simultaneously constraining the maximum levels in close proximity to the indenter. This suggests that, for materials demonstrating substantial strain hardening, deeper penetration is advisable to explore higher strain regions along the stress-strain curve more effectively.

The findings of this study contribute to the field of material characterization by providing an investigation of the advantages and limitations of instrumented indentation techniques for a specific class of materials. Future work could extend our analysis to a greater class of materials. Alternatively, comparisons with artificial intelligence based methods would be interesting.

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6. RESPONSIBILITY NOTICE

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