MILITARY TECHNICAL COURIER

Impact of Pile-up on Indentation Hardness of Neutron-Irradiated 15Ch2MFA Steel

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Abstract

Background/aim: The mechanical response of metallic materials during instrumented indentation is strongly influenced by the deformation mode beneath the indenter, which may involve sink-in or pile-up. This study aims to evaluate the impact of these modes on the prediction of the modified Meyer hardness of 15Ch2MFA steel and its associated mechanical properties in both non-irradiated and neutron-irradiated states. **Methods:** Analytical formulations were developed to determine the modified Meyer hardness value from the maximum indentation load, contact stiffness and reduced elastic modulus, while accounting for the deformation mode. The influence of substituting one deformation mode for the other was then assessed for both material states. **Results:** The results demonstrate that incorrectly identifying the deformation mode can introduce significant errors in hardness estimation. For non-irradiated 15Ch2MFA steel, the difference between the two modes of deformation was approximately 0.5%, corresponding to an overestimation of around 11 MPa. For neutron-irradiated steel, however, the effect was substantially greater: an error of around 8%, with an overestimation of approximately 186 MPa. **Conclusion:** Accurate identification of the indentation deformation mode is essential for reliable mechanical characterisation. This effect is particularly pronounced in the case of neutron-irradiated 15Ch2MFA steel, which indicates that irradiation-induced hardening increases the sensitivity of indentation-based hardness assessment to the assumed deformation mode.

1. Introduction

The continuous release of fresh research data is highly restricted by the use of neutron irradiation in experiments. This is due to the high cost and length of time required for an irradiation run, the restricted availability of space to irradiate samples, and the need for post-irradiation testing brought on by activation. One option for dealing with space and budgetary constraints is miniaturisation, a process in nuclear materials science that involves making samples smaller to enable more samples to be analysed at once. It is a strategy that can be employed to address these issues. However, another issue is the extent of the irradiation damage and the limited technical prospects, particularly in relation to the importance of the study. Due to the small sise of the test pieces, the indentation test is considered to be non-destructive in practice [1–3], which is an advantage of this mechanical characterisation test. The requirement to test components without compromising the performance of the system to which they belong (e.g. nuclear reactors) necessitates reducing the size of the samples to be characterised. This justifies using the instrumented indentation test to assess the mechanical properties of substances [4, 5]. Despite progress in predicting embrittlement and radioactive recovery [6–8], considerable material testing is still necessary to define the recovery process. This preliminary comparative study [1] focuses on the hardness of steel irradiated by neutrons and serves as a reference for the current work. This study evaluates the hardness in sink-in mode using the classical Oliver and Pharr methodology [9] and in pile-up mode using the finite element method approach developed by the authors [1]. It is widely recognised that the mode of deformation in the region where the indenter is applied depends on both the elastoplastic reaction of the indentable material and the indenter's shape. Traditionally, the contact surface is defined by relating it to the depth of

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contact and considering the specific deformation modes: sink-in and pile-up, which correspond to the models of Oliver and Pharr [9] and Loubet et al. [10], respectively. These two techniques and the associated contact depth expressions are substantially different, meaning that one or the other model must be used to derive the mechanical characteristics. Therefore, the deformation mode of the substance must be identified prior to testing. This enables the correct computation of the contacted surface and precise determination of instrumented hardness and Young's modulus values. Most researchers tend to use the simulation proposed by Oliver and Pharr [9], even though the actual deformation mode may differ. This may be because they are unaware of or do not use the development by Loubet et al. [10–12]. As many materials pile up around the impression, the mechanical characteristics derived from instrumented indentation tests are based on the Oliver and Pharr model. These models are not at all accurate. The objective of this work is to analyse hardness influenced by neutron irradiation from a different point of view. First, we will reconstruct the information implemented by the authors [1] in this study to extract the raw data from the direct numerical simulation method developed in publication [1]. These measurements are assumed to be independent of traditional indentation calculation methods [9, 10]. These results are then treated using the aforementioned two methods [5, 6] and compared with the direct method of the authors [1]. In particular, we demonstrate that the finite element method employed by the authors to estimate the pile-up effect is consistent with our Loubet et al.-based approach [10-12]. Finally, we propose two analytical expressions for Meyer hardening, one as a function of maximum indentation force and the other as a function of stiffness, for each deformation mode: sink-in and pile-up.

1.1. Background

The term 'hardness' signifies an increase in resistance to the elastoplastic transition. This phenomenon is the primary issue addressed in the study being presented. Controlled hardening is typically achieved through relevant thermo-mechanical treatments (e.g. quenching). This type of hardening can be exploited for beneficial purposes, such as strengthening the material to prevent plastic deformation. In contrast, radiation-induced hardening is a continuous process that accumulates over time. In severe cases, it can result in plastic deformation occurring before cracking. Ultimately, the material will shatter in a brittle manner, which is particularly hazardous as it would cause immediate damage to an element, as opposed to the safety margin provided by plasticity. Another type of embrittlement that a material can withstand is a reduction in plastic capacity, which leads to less plastic deformation. Both processes are linked to the build-up of radiation-induced defects, as previously mentioned. These flaws act as further barriers to dislocations, thereby increasing the stress required for them to move.

Oliver and Pharr [9] examined the approach for determining instrumented hardness (H) and Young's modulus (E) in nanoindentation, which is now widely used in research projects. However, this method does not apply to substrates that exhibit pile-up deformation [10]. Therefore, it is necessary to briefly describe how hardness and Young's modulus are determined in these cases.

In an instrumented indentation test, the hardness of a material is defined as its plastic resistance to a rigid indenter and quantified as the ratio between the maximum loading employed and the contact area between the indenter and the substance.

The instrumented hardness H takes into account the expected contact area:

$$H = \frac{P}{A_c} \tag{1}$$

where P denotes the applied load and A_c denotes the predicted contact area. Oliver and Pharr [9] proposed calculating the reduced modulus (E_r) using the contact stiffness (S) and the predicted contact area as follows:

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

where the reduced modulus is connected to the elastic-plastic characteristics of the indenter and the established substance as shown in Eq. (2). S is the contact stiffness corresponding to the slope of the unload curvature estimated at the maximum indentation depth, as shown below:

$$S = \frac{dP}{dh}\Big]_{h=h_m} \tag{3}$$

Usually, the unloading curve is fitted using a power law relating the indentation load P to the difference between the indentation depth h and the final indentation depth h_f, as follows:

$$P = B\left(h - h_f\right)^m \tag{4}$$

where B, m and h_f are values ascertained through sequential best-fit analysis. Typically, only the indentation data within the 40-98% range of the maximum load is utilised for this fitting procedure.

Equations (5) and (6) suggest that achieving reliable values for the two mechanical characteristics depends on the precision of the contact area between the indenter and the material being tested.

According to the subsequent relationship, most researchers consider ideal indenter geometry.

$$A_C = 24.56\, h_C^2 \tag{5}$$

The coefficient of 24.56 is derived by taking into account the corresponding conical indenter, which is equivalent to the Vickers and Berkovich indenter tips. These have a semi-angle at the apex of 70.3°. Furthermore, the contact depth (h_c) is affected by the deformation method. The most well-known model for sink-in materials is that proposed by Oliver and Pharr [5]. However, these investigators indicate that their methodology is inapplicable in the event of pile-up, even though it is widely and incorrectly used in deformation mode. Nonetheless, for pile-up materials, Loubet et al. [10-12] offered an estimate of h_c that can exceed the maximum indentation depth, h_m. This is in contrast to sink-

in materials, for which Oliver and Pharr [9] estimated a value of h_c that is always smaller than h_m.

Consequently, for a given indentation test, i.e. for a given requested stress and indenter depth, the contact depth defined by Loubet et al. is clearly greater than that defined by Oliver and Pharr. Consequently, the outcome varies based on the deformation method being examined. An underestimation or overestimation of the interaction depth is unavoidable, resulting in an overestimation or underestimation of the instrumented hardness and Young's modulus. Furthermore, it is evident that the two techniques cannot produce the same outcome for the same indentation test.

As a result, the deformation mode must first be classified. The two related interaction areas (see Figure 1) can be determined from the contact depth in equations (6) and (7) [13, 14], using the following relationships, where the indexes denote the sink-in and pile-up deformation modes respectively:

$$h_c = \begin{cases} h_m - \varepsilon \frac{P}{S} \text{For sink} - \text{in} & (6) \\ \alpha \left(h_m - \frac{P}{S} \right) \text{For pile} - \text{up} & (7) \end{cases}$$

h_m represents the ultimate indentation depth and P represents the ultimate load delivered. S is the contact stiffness and, for Vickers and Berkovich indenters, a value of 0.75 is used for ε. Additionally, α is a constant equal to 1.2.

2. Materials and Methods

This research work critically analyses previous research data [1], which mechanically characterised the neutron-irradiated 15Cr2MoV steel used in nuclear reactor vessel manufacture. The authors of this reference [1] characterised the pile-up phenomenon using the finite element method. This material has also been characterised by other authors, such as [13, 14]. Through this study, we aim to exploit and process the information in references [1, 13, 14] according to a second approach, applying semi-empirical expressions of the modified Meyer hardness specific to sink-in and pile-up.

2.1. Previous research

The reference research case study [1] examined quenched bainitic steel 15Cr2MoV, which is intended for use in manufacturing nuclear reactor vessels designated as VVER 440. The chemical composition of the steel can be found in previous research [13, 14]. The indentation test results for the steel in its two unirradiated and irradiated states can be found in reference [1]. The authors [1] determined the ultimate penetration depths, contact zones and ultimate pile-up heights for three types of examination and two material situations in this study [1], utilising finite element methodology (non-linear finite element simulations of the indentation test). This computational study sought to determine the impact of material pile-up on the consistency of the OP method's contact zone evaluation.

The contact areas were therefore calculated using simulated indentation load-displacement curves and compared with the 'actual' contact zones calculated using a finite element analysis, as shown in Figure 1. For all simulations, indentation was caused by a maximum force of 300 N, and the indenter was treated as rigid with no friction when interacting with the indented material. The Young's modulus E = 200 GPa and Poisson's ratio v = 0.3 were used to describe the elastic properties of 15Ch2MFA.

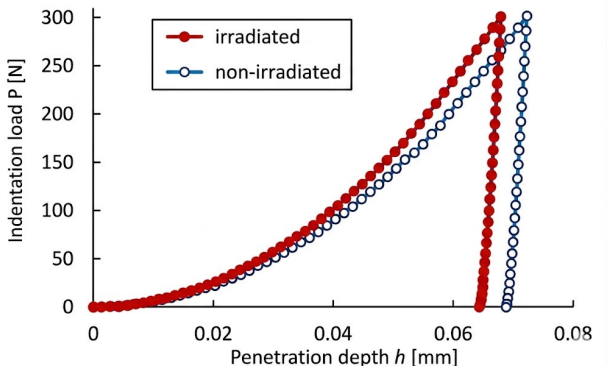


Figure 1. A comparison of the load-displacement curves for the Berkovich indenter and non- and neutron-irradiated indenters [1].

3. Meyer hardness models for pile-up and sink-in deformation modes

Meyer hardness can be calculated over the entire loading curve by taking the continuous values of the applied load (P) and the maximum indentation depth (h_m) into account. This hardening differs from Martens hardening in that it uses the projected contact area.

$$H_{Me} = \frac{P}{A} = \frac{P}{24.56\, h_m^2} \quad (8)$$

The two models expressed in equations (9) and (10) indicate the mechanical responses in terms of the ratio of the maximum indentation load to the ultimate depth reached by the indenter (P/h^2), according to authors [16] and [17], respectively. The two analytical expressions (10) and (11) are based on the contact depth descriptions calculated by Oliver and Pharr [9] and Loubet et al. [10] using equations (6) and (7) respectively (see Figure 2).

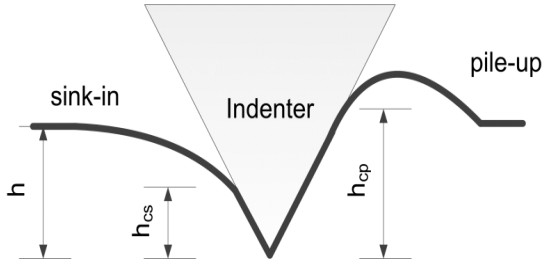


Figure 2. A graphic illustration showing an indentation pattern, and the sink-in and pile-up processes. It also shows the respective contact depths, h(cs) and h(cp), which are specified in equations (6) and (7) [16].

See references [15] and [16] for more information on determining equations (9) and (10). For the sink-in deformation type, the P/h^2 ratio is proportional to the model suggested by Malzbender et al. [15], as follows:

$$\frac{P}{h^2} = E_r \left(\frac{1}{\sqrt{c}} \sqrt{\frac{E_r}{H}} + \varepsilon \frac{\sqrt{\pi}}{2} \sqrt{\frac{H}{E_r}} \right)^{-2} \quad (9)$$

Finally, for the pile-up deformation type, the P/h² ratio is expressed according to a model proposed by Habibi et al. [16, 17] as follows:

$$\frac{P}{h^2} = E_r \left(\frac{1}{\alpha\sqrt{c}} \sqrt{\frac{E_r}{H}} + \frac{\sqrt{\pi}}{2} \sqrt{\frac{H}{E_r}} \right)^{-2} \quad (10)$$

The P/h² relation as a function of the critical distinction point of the indentation deformation type, estimated at 0.83, is decisive. See reference [18] for details.

According to Eq. (10), Martens' hardness is defined as the ratio of the maximum indentation load to the corresponding ultimate indentation depth (8). Unlike instrumented hardening, which is determined from contact depths calculated by equations (6) and (7), this hardness is independent of deformation type (7).

By substituting the H/E_r ratio for the P/S ratio, we obtain two new analytical expressions for calculating modified Meyer hardness according to the two deformation modes (see equation transformation steps (9) and (10) in previous research [19] to obtain equations (11) (sink-in) and (12) (pile-up), taking the Meyer hardness relation (8) into account as follows:

$$H_{Mesi} = \frac{1}{24.56} \left(\frac{\sqrt{\pi}}{2\sqrt{c}} \frac{1}{\left(\frac{\sqrt{P}}{S}\right) E_r} + \varepsilon \left(\frac{\sqrt{P}}{S}\right) \right)^{-2} \quad (11)$$

$$H_{Mepu} = \frac{1}{24.56} \left(\frac{\sqrt{\pi}}{2\alpha\sqrt{c}} \frac{1}{\left(\frac{\sqrt{P}}{S}\right) E_r} + \left(\frac{\sqrt{P}}{S}\right) \right)^{-2} \quad (12)$$

As can be seen above, the resulting system of equations describes Meyer hardness in sink-in (Eq. (11)) and pile-up (Eq. (12)) deformation types. Meyer hardness is closer to Oliver and Pharr hardness [9] because these two hardnesses use the same empirical constant, which is equal to 24.56. This is unlike Martens hardness, which uses a value of 26.43 for the largest projected contact area.

4. Results and discussion

4.1. Comparing the outcomes of the two methods

Suppose the type of strain mode is unknown. We calculate the properties of 15Cr2MoV using two models: one that treats the sink-in elaborated and developed by the authors [1], and one that treats the pile-up elaborated by the authors [6]. We also assume that the results obtained by numerical simulation (FEA) are neutral, i.e. that neither the methodology of Oliver and Pharr [9] nor that of Loubet et al. [10] is used for the calculation of the mechanical properties. Therefore, we can compare the results without the influence of the two known methods for estimating sink-in and pile-up. It is this neutrality of the data resulting from the direct method of numerical simulation that represents the strength and advantage of this study [1]. Often, the first or second method of calculation influences studies, making it impossible to compare them and provide an objective opinion on the reliability of the results. In the current study, we use the reverse methodology to extract data from the direct method (finite element analysis, FEA) employed by the authors to verify their approach and analyse and comment on an alternative approach. Referring to relations (6) and (7), the ratio (P/S) is calculated using equations (13) and (14) from values (h_m, A_c) extracted from Table 1 relating to the direct method by FEA [1], as follows:

$$\frac{P}{S} = \frac{1}{\varepsilon} \left[h_m - \left(\frac{A_{cOP}}{c} \right)^{1/2} \right] \quad (13)$$

$$\frac{P}{S} = h_m - \frac{1}{\alpha} \left(\frac{A_{cLA}}{c} \right)^{\frac{1}{2}} \quad (14)$$

The indentation force and contact stiffness are independent of the type of deformation of the indenter. The P and S variables are used directly when analysing the instrumented indentation P-h characteristic curve. According to the methods of Oliver and Pharr [9] and Loubet et al. [10, 11], the AcOP and AcLA are designated the contact areas, respectively. The resulting (P/S) values are then inserted into the conventional equations (13) and (14), which relate to the Oliver and Pharr [9] and Loubet et al. [10] approaches. The results are displayed in Table 1.

Table 1. Outcomes of the ratio (P_m/S) of 15Ch₂MFA using the two methods [9] and [10].

Indenter shape	Analysis dimension	Material state	Methods used for the calculation of (P/S) [mm]	
			Loubet et al. method [10]	Oliver and Pharr method [9]
			Pile-up mode	Sink-in mode
Berkovich	3D	N.I	0.005078718	-0.011535471
		I	0.003670209	-0.012771874
Equivalent cone	3D	N.I	0.00529083	-0.011181950
		I	0.003334562	-0.013053508
	2D	N.I	0.004717091	-0.011860404
		I	0.004334562	-0.011664619

The indentation force and contact stiffness are independent of the type of deformation of the indenter. The P and S variables are used directly when analysing the instrumented indentation P-h characteristic curve. According to the methods of Oliver and Pharr [9] and Loubet et al. [10, 11], the AcOP and AcLA are designated the contact areas, respectively. The resulting (P/S) values are then inserted into the conventional equations (13) and (14), which relate to the Oliver and Pharr [9] and Loubet et al. [10] approaches. The results are displayed in Table 1.

Table 2. Outcomes of the ratio (P_m/S) of 15Ch2MFA using the two methods [9] and [10].

Indenter shape	Material state (Analysis dimension)	Methods used to calculate A_c [mm²]		$\|A_c\|$ [%]
		Oliver and Pharr [9]	Loubet et al.[10]	
		Sink-in mode	Pile-up mode	
Berkvich	N.I(3D)	0.099396117	0.158	0.371
	I (3D)	0.084719528	0.146	0.420
Equivalent cone	N.I(3D)	0.100192115	0.157	0.362
	I (3D)	0.081289282	0.143	0.432
	N.I(2D)	0.095582222	0.155	0.383
	I (2D)	0.087032226	0.143	0.391

The difference rate of A_c between the two modes [%] is denoted by [%]. For reasons of clarity and readability of the results of the contact areas displayed in Table 2, it was preferred that they be presented separately in a different table (Table 2). Using the data and conditions in Table 1 will produce the following:

$$\left.\begin{aligned} H_{nop} &= 2.9486\ GPa \\ H_{iop} &= 1.15\ H_{nop} \end{aligned}\right\} \qquad (15)$$

To calculate H_{Dn} and H_{Di}. The following conditions are hence, $H_{iop} = 2.9486\ GPa$
used:

$$\left.\begin{aligned} \frac{H_{nop}}{H_{Dn}} &= 1.32 \\ \frac{H_{iop}}{H_{Di}} &= 1.40 \end{aligned}\right\} \text{hence,} \begin{cases} H_{Dn} = 1.9424\ GPa \\ H_{Di} = 2.1017\ GPa \end{cases} \qquad (16)$$

Therefore, Table 3 summarises the hardnesses relating to the conical type of indenter using two-dimensional numerical analysis. It will be as follows:

Table 3. Summary table of indentation hardnesses H_{nop}, H_{iop}, H_{Dn} and H_{Di} for the 2D conical indenter, calculated using the direct method (the numerical method assumed to be neutral) and the Oliver and Pharr method [9].

	Instrumented indentation hardness [GPa]		Rate/ Methods $H_{op}$and H_D
	Oliver and Pharr method [9]	Direct method by numerical simulation [10]	
Non-irradiated	2.564	1.942	24.26 %
Neutron-irradiated	2.9486	2.102	28.71 %
Rate compared to Material stateH_{NI} and H_I	13.04 %	7.61 %	/

Indices designation:
op: Oliver and Pharr method;
D: direct method;
i: irradiated;
n: non-irradiated.

J. Loubet et al., (1993) [10] assume that the maximum indentation load (P) is constant and equal to 300 N for all tests, in order to unify the conditions of these tests and draw objective conclusions.

4.2. Comparing the two Martens hardnesses for the two strain modes

The new analytic expressions (17) and (18), which are used to calculate the modified Martens hardnesses for the sink-in and pile-up types respectively, are given below:

$$\nabla H_{Mesi} = \frac{1}{2.43}\left(\frac{\sqrt{\pi}}{2\sqrt{24.5}}\frac{1}{\left(\frac{\sqrt{P_m}}{S}\right)E_r} + 0.72\left(\frac{\sqrt{P_m}}{S}\right)\right)^{-2} \qquad (17)$$

$$H_{Mepu} = \frac{1}{26.43}\left(\frac{\sqrt{\pi}}{2(1.2)\sqrt{24.5}}\frac{1}{\left(\frac{\sqrt{P_m}}{S}\right)E_r} + \left(\frac{\sqrt{P_m}}{S}\right)\right)^{-2} \qquad (18)$$

Table 4. Results of estimated Meyer hardnesses by the two analytical expressions.

State of the material examined	Meyer hardness, H_{Me} [GPa]		VR_1 [%]
	sink-in mode Eq. (17)	pile-up mode Eq. (18)	
Non irradiated	2.066	2.055	5.3‰
Neutron-irradiated	2.253	2.439	8 %
VR_2 [%]	8.03 %	15.44 %	/

Table 4 shows the results of the estimated Meyer hardnesses for the two modes, as proposed in this work, using the two analytical expressions (17) and (18).

VR1: denotes the hardness variation rate in percentage between the two expressions proposed (17) and (18) for the same material state. VR2: denotes the rate of variation of hardness in % between the two states of the material by the use of a single expression namely Eq. (17) for sink-in and Eq. (18) for pile-up, with:

$$VR\ [\%] = \left|\frac{H_1 - H_2}{(H_1;\ H_2)}\right| \qquad (19)$$

4.3. *Determination of the deformation mode by the method of Alcalá et al. [20].*

The coefficient Δ designates the relationship used by the authors Alcalá et al. [20] to characterize the sink-in type from the pile-up type [22,23]. When Δ is greater than 1, it is the predominance of the pile-up, and where applicable, when Δ is less than 1, it is the predominance of the sink-in. This ratio Δ is calculated as follows:

$$\text{Ratio } \Delta = \frac{A_{LA}}{24.56 \times h_{max}} \qquad (20)$$

We calculate the ratio Δ for 6 tests studied concerning the states of the material as well as the dimensions of its analysis (3D and 2D). Then, we draw the histogram (see Figure 3), illustrating the ratio of Alcalá et al. [20] for tests implemented directly. We calculate the ratio Δ for 6 tests studied concerning the states of the material as well as the dimensions of its analysis (3D and 2D). Then we draw the histogram (see Figure3), illustrating the ratio of Alcala et al. [20] for tests implemented directly.

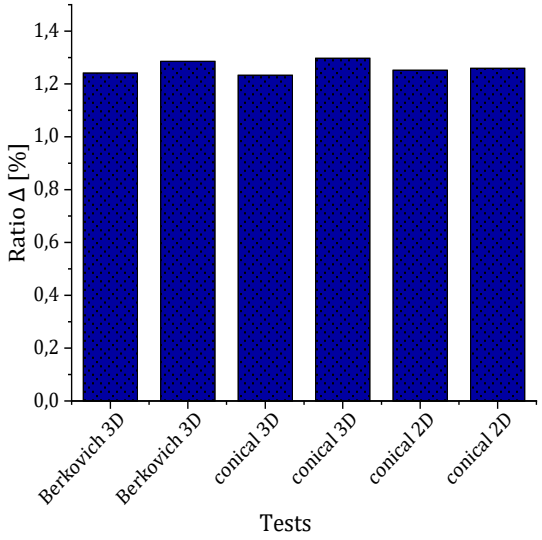


Figure 3. Histogram of Alcala et al. [20]to predict the deformation mode under the indenter.

The six characteristic columns of the tests that were made have amplitudes greater than 1, which is the average of the ratios, as shown by the relation:

$$\boldsymbol{Moy}_{\Delta} = 1.26 \pm 0.02 \qquad (21)$$

The results explain why 15Ch2MFA steel adopts a predominantly stacked deformation mode. The mode of deformation under the indenter is identified beforehand in order to choose the corresponding calculation equation along with the designated type (sink-in or pile-up). But in our case, we apprehended the reverse process to analyze and comment on the study without the prior influence of knowledge of the deformation mode to conduct the study objectively and assess the impact of pile-up on the appreciation of the hardness.

The graphical representation of Figure4 shows the characteristic points designating the shape of the indenter, the analysis dimension and the state of the material (see table IV) by the use of the Oliver and Pharr [9] methodology (blue circles), OP, and that of Loubet et al. [10], LA, (red circles). The characteristic points of the curve (P/S)-Ratio Δ designate the shape of the indenter, the examination dimension, and the state of the material. This curvature is subdivided into two-point clouds, red (LA) and blue (OP), respectively. It is noted that delta is greater than 1, so the steel examined adopts a pile-up mode deformation. However, we notice that the blue characteristic points (OP) estimated by the method [9] have negative signs, which have no physical meaning. Therefore, the conditions for applying this formulation are not justified.

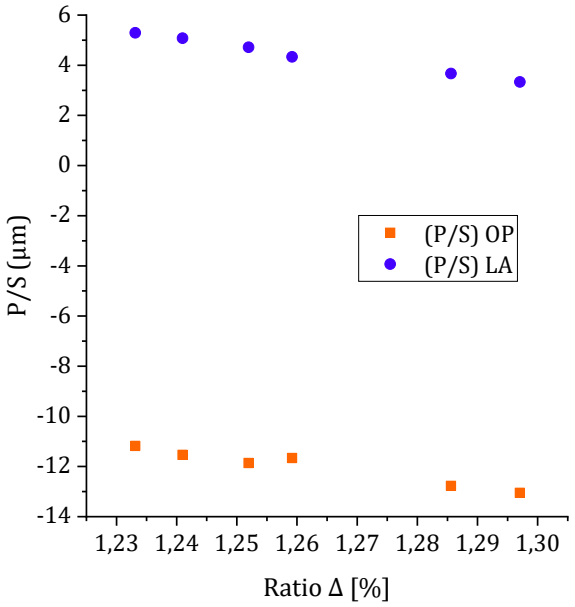


Figure4. The graphical representation of (P/S) vs. the criterion of Alcala et al [20].

4.4. *Analysis and commentary of the results*

Table 4 shows a perfect correlation between the author's study [1] results on contact areas and their related maximal depths (see Table 4) with the approach of Loubet et al. [10] for calculating AC in the situation of pile-up.

Contrary to the method of Oliver and Pharr [9], which shows its exploitation limit in the field of the pile-up by recording important variations.

The difference in A_c between the two modes of deformation calculated by the two corresponding preceding methods ([9] and [1,10]) is very significant and is estimated at an average of 39 %.

Table 4 displays correct values of the ratio (P/S) concerning the mode of deformation in pile-up since one finds conformal values of Ac between the direct method by FEA [1] and that of Loubet et al. [10].

However, the values of (P/S) relating to the sink-in are physically inadequate, bearing the negative sign. In the logic imposed by Oliver and Pharr [9] for the calculation of the ratio (P/S) by Eq. (7), the maximum height, hm, should be greater than the height $(A_{OP}/24.5)0.5$[20]. This means that the Oliver and Pharr method [9] can only be used to deal with sink-in deformation up to a certain point.

Table 3 gives the difference in hardness recorded between irradiated and unirradiated material by the Oliver and Pharr [9] methodology and is estimated at 13.04 %. While the direct method of the authors [10] by FEA records a more reasonable difference in hardness, estimated at 7.61% with an overestimation of 5.43 %.

However, the reliability of the two methods, namely the direct method and that of Oliver and Pharr [9], were recorded: 24.26 % and 28.71 %, respectively. With an estimated variation margin of around 4.5%. This can be explained by factors influencing the indentation results, such as the mode of plastic deformation beneath the indenter ora scale change relating to the instrumented indentation process, as has already been shown in previous research [20].

Table 4 records an upward trend in Meyer hardness from the initial state to the neutron irradiated state of the order of 8.03 % and 15.44 % for sink-in mode and pile-up type, respectively. This represents a difference of 7.41%, almost double.

The margin of difference between the use of the analytical expressions proposed (17) and (18) gives 0.53 % and 8 % respectively, for the characterization of the material in the initial state (unirradiated) and the final state (irradiated by neutrons). This is a difference of 11 MPa for the unirradiated state and 186 MPa for the irradiated state.

Figure 3 depicts the histogram shows the report from Alcala et al. [20]to predict the deformation mode under an indenter. The six characteristic columns of the tests developed have amplitudes greater than 1. This explains why 15Ch2MFA steel adopts a predominantly pile-up deformation mode.

5. Conclusions

Concerning the pile-up deformation mode, the results of mechanical characterization by indentation obtained by the direct numerical method of the authors [1] are in conformity with the results obtained by the method of Loubet et al. [10]. Unlike the Oliver and Pharr [9] methodology (see equation (6)), which shows its operating limit in the pile-up area by recording gross errors.

The difference in Ac between the two modes of deformation calculated by the two methods [9] and [1] is very significant and is estimated at an average of 39 %, which has a direct impact on the reasonable estimation of the increase in material hardness in irradiated and non-neutron irradiated states equal to 7.61%, with an overestimation of 5.43 % compared to the sink-in method [9].

The method adapted for this study is that of Loubet et al. [10], described as having excellent correlation with the direct method [1]. However, the direct method and that of Oliver and Pharr [9] both have a margin of error of about 4.5 percent.

The effect of the pile-up on Meyer hardness from the initial state to the neutron irradiated state, compared to the sink-in mode, is estimated at 7.41%, which is almost double. The margin of difference between the use of the proposed analytical expressions (17) and (18) gives: 5.3 percent and 8%, respectively, for the characterization of the material in the initial state and the final state. This represents an overestimation of 11 MPa and 186 MPa for the material in the unirradiated and neutron irradiated states, respectively.

The difference in modified Meyer hardnessH_{Me}generated by the ignorance of the corresponding studied mode or the substitution of one mode by the other by default, is estimated at (Equation (22)):

$$\Delta H_{Me} = \left| \frac{1}{24.56} \left[\left(\frac{\sqrt{\pi}}{2\sqrt{c}} \frac{1}{\left(\frac{\sqrt{P_m}}{S}\right)E_r} + \varepsilon \left(\frac{\sqrt{P_m}}{S}\right) \right)^{-2} - \left(\frac{\sqrt{\pi}}{2\alpha\sqrt{c}} \frac{1}{\left(\frac{\sqrt{P_m}}{S}\right)E_r} + \left(\frac{\sqrt{P_m}}{S}\right) \right)^{-2} \right] \right| \quad (22)$$

$$\begin{cases} H_{Me(Sink-in)} = \frac{1}{24.56} \left(\frac{\sqrt{\pi}}{2\sqrt{c}} \frac{1}{\left(\frac{\sqrt{P_m}}{S}\right)E_r} + \varepsilon \left(\frac{\sqrt{P_m}}{S}\right) \right)^{-2} \\ H_{Me(Pile-up)} = \frac{1}{24.56} \left(\frac{\sqrt{\pi}}{2\alpha\sqrt{c}} \frac{1}{\left(\frac{\sqrt{P_m}}{S}\right)E_r} + \left(\frac{\sqrt{P_m}}{S}\right) \right)^{-2} \end{cases} \quad (23)$$

For the steel studied, the empirical constants α and c are likely to vary with the variation in the geometry of the chosen tip and also with the designation of the real or projected contact areas used to calculate the hardness values. The coefficient $\alpha$is substituted by εin the case of the pile-up.

As presented in this system of equations, these analytical expressions make it possible to estimate the modified Meyer hardness $H_{Me} - \left(\frac{P}{S}\right)^{1/2}$ for the two modes of deformation under indenters, namely pile-up and sink-in, applied to a radioactive material (Equation (23)).

This resulting model not only provides a simple way to predict hardness as a function of P, S, and E_r for an elasto-plastic type of metallic material, but it also allows prediction by the reversible approach from the behavior of the loading curvature. In this perspective, it is interesting to study the effects of hardening and the passage of micro-nano scales to push the limits of this research, in particular in this strategic and sensitive field of nuclear reactors and radioactive materials.

References

[1] Materna A., Haušild P., Ondráček J., Kytka M., 2018. The Effect of Pile-Up on Indentation Hardness Evaluation of Neutron-Irradiated 15Ch2MFA Steel, Acta Physica Polonica A, 134(3). doi: http://doi.org/10.12693/APhysPolA.134.729

[2] Manahan M., Browning A., Argon A., Harling O., 1986. Miniaturized disk bend test technique development and application, in: The Use of Small-Scale Specimens for Testing Irradiated Material, ASTM International. doi: doi: https://doi.org/10.1520/STP32993S

[3] Fischer-Cripps A.C., 2011. Scaling relationships in nanoindentation, in: Nanoindentation, Springer, New York.

[4] Wang Z.-X., Shi H.-J., Lu J., Shi P., Ma X.-F., 2008. Small punch testing for assessing the fracture properties of reactor vessel steel with different thicknesses, Nuclear Engineering and Design, 238(12), pp. 3186–3193. https://doi.org/10.1016/j.nucengdes.2008.07.013

[5] Al Mazouzi A., Alamo A., Lidbury D., Moinereau D., Van Dyck S., 2011. PERFORM 60: Prediction of the effects of radiation for reactor pressure vessel and in-core materials using multi-scale modelling – 60 years foreseen plant lifetime, Nuclear Engineering and Design, 241(9), pp. 3403–3415. ***doi:*** https://doi: 10.1016/j.nucengdes.2011.07.023.

[6] Hasenhuetl E., Kasada R., Zhang Z., Yabuuchi K., Huang Y.-J., Kimura A., 2017. Evaluation of ion-irradiation hardening of tungsten single crystals by nanoindentation technique considering material pile-up effect, Materials Transactions, 58(5), pp. 749–756. doi: https://doi.org/10.2320/matertrans.M2016437

[7] Zhu L.-N., Xu B.-S., Wang H.-D., Wang C.-B., Yang D.-X., 2011. Measurement of mechanical properties of 1045 steel with significant pile-up by sharp indentation, Journal of Materials Science, 46(4), pp. 1083–1086. doi: https://doi.org/10.1007/s10853-010-4876-7

[8] Burik P., Pešek L., Voleský L., 2015. Pile-up correction of mechanical characteristics of individual phases in various steel by depth sensing indentation, Key Engineering Materials, 662, pp. 7–10. [Online]. Available at: www.scientific.net/KEM.662.7. [Accessed: 03 April 2026].

[9] Oliver W.C., Pharr G.M., 1992. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, Journal of Materials Research, 7(6), pp. 1564–1583. doi: https://doi.org/10.1557/JMR.1992.1564.

[10] Loubet J., Bauer M., Tonck A., Bec S., Gauthier-Manuel B., 1993. Nanoindentation with a surface force apparatus, in: Mechanical Properties and Deformation Behavior of Materials Having Ultra-Fine Microstructures, Springer, New York, pp. 429–447. doi: https://doi.org/10.1007/978-94-011-1765-4_28

[11] Guillonneau G., 2012. Nouvelles techniques de nano-indentation pour des conditions expérimentales difficiles: très faibles enfoncements, surfaces rugueuses, température, PhD Thesis, Ecole Centrale de Lyon, Lyon.

[12] Bec S., Tonck A., Georges J.-M., Georges E., Loubet J.-L., 1996. Improvements in the indentation method with a surface force apparatus, Philosophical Magazine A, 74(5), pp. 1061–1072. doi: https://doi.org/10.1080/01418619608239707

[13] Haušild P., Kytka M., Karlík M., Pešek P., 2005. Influence of irradiation on the ductile fracture of a reactor pressure vessel steel, Journal of Nuclear Materials, 341, pp. 184–188. doi: https://doi.org/10.1016/j.jnucmat.2005.01.019

[14] Haušild P., Materna A., Kytka M., 2015. Identification of neutron irradiation induced strain rate sensitivity change using inverse FEM analysis of Charpy test, Journal of Nuclear Materials, 359, pp. 122–126. doi: https://doi.org/10.1016/j.jnucmat.2015.01.024

[15] Malzbender J., Den Toonder J., 2000. The P–h² relationship in indentation, Journal of Materials Research, 15(5), pp. 1209–1212. doi: https://doi.org/10.1557/JMR.2000.0171

[16] Habibi S., Chicot D., Mejias A., Boutabout B., Zareb E., Semsoum D., Benaissa S., Mezough A., Merzouk H., 2021. The P–h² relationship on load–displacement curve considering pile-up deformation mode in instrumented indentation, Journal of Materials Research, 36, pp. 3074–3085. doi: https://doi.org/10.1557/s43578-021-00286-3

[17] Habibi S., Fares R., Batool K., Batool S., Mohamed Isa S.S.P., Abd-Elmonem A., Alhubieshi N., 2024. The methodology refinement through the predominant case of the pile-up to estimate the mechanical response by spherical indentation, Scientific Reports, 14, p. 29149. doi: https://doi.org/10.1038/s41598-024-80760-0

[18] Habibi S., 2022. The P–h² relationship as a function of (hf/hm) in indentation, Fracture and Structural Integrity, 16(62), pp. 613–623. doi: https://doi.org/10.3221/IGF-ESIS.62.42

[19] Djellal N., Mekki D.E., Navarro E., Marin P., 2022. Influence of Pr6O11 addition on structural and magnetic properties of mechanically alloyed Fe65Co35 nanoparticles, Fracture and Structural Integrity, 16(60), pp. 393–406. doi: https://doi.org/10.3221/IGF-ESIS.60.27

[20] Alcala J., Barone A., Anglada M., 2000. The influence of plastic hardening on surface deformation modes around Vickers and spherical indents, Acta Materialia, 48(13), pp. 3451–3464. doi: https://doi.org/10.1016/S1359-6454(00)00140-3

[21] Benaissa S., Habibi S., Semsoum D., Merzouk H., Mezough A., Boutabout B., Montagne A., 2021. Exploitation of static and dynamic methods for the analysis of the mechanical nanoproperties of polymethylmethacrylate by indentation, Fracture and Structural Integrity, 15(56), pp. 46–55. doi: https://doi.org/10.3221/IGF-ESIS.56.03

[22] Xu Z., Liu M., Gong J., 2025. Characterization of elastic modulus and hardness of brittle solids by instrumented indentation, Acta Mechanica Sinica, 41(424095), pp. 1–22. doi: https://doi.org/10.1007/s10409-024-24095-x

[23] Xu Z., Liu M., Yang F., 2024. Calculation of elastic constants of bulk metallic glasses from indentation tests, Journal of Materials Research and Technology, 28, pp. 4382–4391. doi: https://doi.org/10.1016/j.jmrt.2024.01.046

Uticaj naslaganja na oštrinu utiska na neutronski zračenom čeliku 15Ch2MFA

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Apstrakt

Uvod/Cilj: Mehanički odgovor metalnih materijala tokom instrumentalne indentacije u velikoj meri zavisi od režima deformacije ispod indentora, što može uključivati utonuće (sink-in) ili naslaganje (pile-up). Cilj ove studije je da proceni uticaj ovih režima na predviđanje modifikovane Mejerove tvrdoće čelika 15Ch2MFA i njegovih povezanih mehaničkih svojstava, kako u neradijacionom, tako i u neutronski zračenom stanju. **Metode:** Razvijene su analitičke formulacije za određivanje vrednosti modifikovane Mejerove tvrdoće na osnovu maksimalnog opterećenja pri utiskuivanju, kontaktne krutosti i smanjenog elastičnog modula, uz uzimanje u

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Ključne reči:
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obzir režima deformacije. Zatim je procenjivan uticaj zamenjivanja jednog režima deformacije drugim za oba stanja materijala. **Rezultati:** Rezultati pokazuju da nepravilno identifikovanje režima deformacije može dovesti do značajnih grešaka u procenjivanju tvrdoće. Za neradirajući čelik 15Ch2MFA, razlika između dva načina deformacije iznosila je približno 0,5%, što odgovara precenjivanju od oko 11 MPa. Međutim, kod neutronski radirajućeg čelika efekat je bio znatno veći: greška od oko 8%, sa precenjivanjem od približno 186 MPa. **Zaključak:** Precizna identifikacija režima deformacije pri utisku je od suštinskog značaja za pouzdanu mehaničku karakterizaciju. Ovaj efekat je naročito izražen kod čelika 15Ch2MFA izloženog neutronskoj radijaciji, što ukazuje da očvršćavanje izazvano radijacijom povećava osetljivost procene tvrdoće na osnovu utiska na pretpostavljeni režim deformacije.