High-accuracy compliance correction for nonlinear mechanical testing: Improving Small Punch Test characterization

David Sánchez-Ávila a,b,*, Alberto Orozco-Caballero b,d, Elkin Martínez c,e, Luis Portolés c, Rafael Barea a, Fernando Carreño b

a Department of Industrial Engineering, Nebrija University, C/Pirineos 55, 28040 Madrid, Spain
b Department of Physical Metallurgy, CENIM-CSIC, Avda. Gregorio del Amo 8, Madrid 28040, Spain
c AIDIMME-Metal Processing Research Institute, Parque Tecnológico, Avda. Leonardo Da Vinci, 38, Valencia, Spain
d Department of Mechanical Engineering, Chemistry and Industrial Design, ETS Ingeniería y Diseño Industrial, Polytechnic University of Madrid, Ronda de Valencia 3, 28012 Madrid, Spain
e * Corresponding author at: Department of Industrial Engineering, Nebrija University, C/Pirineos 55, 28040 Madrid, Spain.
E-mail address: d.sanchezav@gmail.com (D. Sánchez-Ávila).

A R T I C L E   I N F O
Keywords:
316L stainless steel
SPT (Small Punch Test)
FEM (Finite Element Modelling)
SLM (Selective Laser Melting)
Plastic deformation
Non-linear Compliance Correction

A B S T R A C T
Small punch test, SPT, of metallic materials is a very convenient mechanical test since it requires a small volume of material. However, SPT data from different laboratories suffer from large scatter, mainly due to the different experimental set-ups, especially when displacement is measured without an LVDT. Such uncertainty is attributed predominantly to the large effect of elastic displacement associated to the low stiffness of the device set-up, which has not been taken properly into account so far. Additionally, we show that large stiffness variations during SPT are taking place. Thus, in this study, we present a methodology for performing the elastic correction in SPT load vs. displacement curves through a load-unload cycles test, allowing the attainment of the elastic displacement and associated stiffness for each unload step as a function of increasing SPT displacement.

The proposed methodology provides SPT load vs. plastic displacement curves, which offer more accurate mechanical data and more reliable comparisons among different laboratories and materials, independently on the employed SPT set-up.

1. Introduction

Additive manufacturing (AM) techniques such as selective laser melting (SLM) are nowadays being integrated in the manufacturing industry for performing rapid prototyping and obtaining complex geometries. These techniques offer several advantages in terms of freedom of design and production flexibility so they are being implemented for numerous applications in the nuclear, aerospace, automotive and biomedical industries [1–7]. SLM and other AM technologies are nowadays expensive due to costly raw materials, usually metallic powder, the long production time and the heating source (electron beam or laser). Good quality and appropriated metallic powder is usually obtained by complex and low production yield methods such as powder atomization [8–10]. As usual occurs when new structural materials are developed, a complete mechanical characterization has to be performed in order to optimize processing parameters and final mechanical properties [11,12]. The most universal mechanical test is tensile testing since it provides reliable stress and ductility values. Nevertheless, it requires for a relatively large volume of material. Due to the high associated cost of AM processes and the heterogeneous and porous microstructure of the manufactured pieces, the conventional tensile testing is not always suitable, evidencing the need for an alternative miniature testing method, such as SPT.

Small Punch Test (SPT) was suggested by Manahan et al. [13] and then developed by Mao et al. [14] to determine the mechanical properties mainly on irradiated and other materials [14–19]. SPT is used in the nuclear industry to characterize and evaluate the neutron irradiation damage and for evaluating the life in service of pipes and pressure vessels in petrochemical and energy industries. This test is considered a quasi-non-destructive test used as a part of the safety plan of the remaining life of nuclear plants [20–23]. Since it is not possible to perform tensile tests on service parts, the aim of using alternative mechanical tests, such as punch, bending and nanoindentation tests, is to obtain the same mechanical parameters, i.e. yield stress, UTS and ductility [6,24–28]. Therefore, data handling should follow similar
For instance, the load can be applied using a ball pushed by a puncher. SPT data from different laboratories is the use of different testing rigs. From the total displacement. Correspondingly, when measuring hard material-rig system as a function of the material deflection. Different transducer) measurement [46] . These different set-ups produce differences in the stiffness of each experimental -elastic displacements of the device depend on each set-up. Additionally, displacement is measured using the puncher displacement, the elastic load is applied and that displacement is added to the readings [50] .

It is our searchers are getting involved in the SPT development and standardization [29–36] . The European Committee for Standardization established a standard document where some key considerations are taken into account [37] . Nevertheless, the elastic displacement (compliance) correction has still not been given sufficient attention probably due to its intrinsic difficult estimation, especially in the case of SPT. Moreover, an additional source of data scatter when comparing SPT data from different laboratories is the use of different testing rigs. For instance, the load can be applied using a ball pushed by a puncher [35,38–40] , or using a rounded puncher (hemispherical ended punch) [41–45] . Additionally, there are mainly two different displacement measurement methods: top measurement and LVDT (linear variable differential transducer) measurement [46] . These different set-ups combinations produce differences in the stiffness of each experimental SPT device and thus, induce differences in the values of the measured properties, like, for example, the values of the deflection. It is our contention that using the plastic strain instead of the total strain is advantageous since it is an intrinsic response of the material and makes the SPT curves rig-independent. However, the elastic component of the total displacement in the raw load-displacement curves comprises both, the material elastic response and the machine elastic response. Moreover, this elastic response is not linear throughout the SPT test and thus, it is important to perform proper elastic displacement calculations for each material-rig system as a function of the material deflection.

The compliance correction is a common practice carried out for tensile and compression tests [47] . Due to the design of these tests, it is an easy standard operation to eliminate the compliance of both the material and the machine by subtracting the linear elastic displacement from the total displacement. Correspondingly, when measuring hardness and modulus by nanoindentation, the compliance is usually measured and the indenter contribution decoupled/corrected [48,49] . This is because the supporting frame is elastically deformed when the load is applied and that displacement is added to the readings [50] . Similarly, the same approach holds for SPT when position is not measured with an LVDT positioned below the sample: when the displacement is measured using the puncher displacement, the elastic displacement of the device and sample are added to the readings. The elastic displacements of the device depend on each set-up. Additionally, such elastic displacement is a function of the total displacement. In fact, the SPT stiffness increases considerably during the test, as high as an order of magnitude, in contrast to tensile and compression tests.

In this research, we selected 316L austenitic steel to perform such analysis. This material is widely used for structural applications and can be found in pipes and many other structural components close to nuclear power reactor cores [51–54] . Additionally, 316L it is also used in marine, energy, aerospace, petrochemical and medical environments due to its properties such as good ductility, fracture toughness, high strength and corrosion resistance. The 316L stainless steel is widely available, on its properties such as good ductility, fracture toughness, high strength and corrosion resistance. The 316L stainless steel is widely available, on many applications including marine environments, oil and gas industries, and various other areas such as the petrochemical industry due to its excellent corrosion resistance.

In this paper, we propose a method to perform the subtraction of the elastic displacement in SPT measurements, using a 316L steel that has been processed by Selective Laser Melting (SLM). We calculate the elastic displacements for each unloading step by means of several cyclic loading–unloading steps. Additionally, Finite Element Modelling (FEM) has been carried out to discern the relative influence of the device vs. material stiffness during SPT. The objective of this article is to propose a new method to obtain more accurate SPT load vs. plastic displacement curves, which would improve the correlations and reduce the scatter of the calculated mechanical property values among laboratories using different set-ups. The proposed methodology will surely help in future standardizations for obtaining more reliable SPT measurements.

2. Material and methods

2.1. Material and processing: Selective Laser Melting parameters

316L stainless steel was used in the form of a powder which composition is presented in Table 1. The powder was obtained by gas-atomization and presents particle diameters in the range 25–52 μm. The samples were manufactured using a SLM-AM Concept Laser M3 machine, equipped with a Diode-pumped solid state Nd:YAG laser which provides a wavelength of 1064 nm, operating at a maximum power of 100 W in a nitrogen atmosphere to avoid oxidation. The manufacturing conditions are presented in Table 2. AM blocks and cylindrical bars were manufactured with dimensions of 26 × 26 × 100 mm3 and ∅ = 10 mm L = 100 mm, respectively. In order to know the mechanical parameters and later compare with SPT, dog-bone tensile test samples were extracted from AM blocks having the tensile axis aligned with the building direction (vertical axis). The tensile tests were performed at room temperature and at an initial strain rate of 10−4 s−1, obtaining the values of yield strength (YS), ultimate tensile strength (UTS), uniform elongation (εu) and elongation to failure (εf) shown in Table 3. The properties of materials used in the simulation with FEM are also present in Table 3.

2.2. Small punch test

Samples of 10 mm in diameter and 0.6 mm in thickness, were machined from the cross-section of the AM cylinders. Both sample faces were ground up to a 2000-grit paper to reach a final thickness of 0.500 ± 0.005 mm.

A schematic of the SPT device is presented in Fig. 1a. The sample is clamped on its external edge and deformed by a steel ball of 2.4 mm in diameter pushed by a punch. The SPT device is attached to a universal 10 kN load cell Servosis ME-405 testing machine. The SPT was performed at room temperature using loading/unloading cycles of 0.2 mm loading displacement (forward) and 0.1 mm unloading displacement (backward). The complex geometry and the non-linear deformation of the SPT test make it necessary to perform several cycles to identify the elastic and/or plastic displacement at different displacements. The purpose of these cycles is to obtain information from each unloading step, giving stiffness/compliance data.

2.3. Finite element modelling

The SPT finite element model has been implemented using Comsol™ software. The model consists in a 2D-axisymmetric geometry formed by a clamped specimen at its external part with a spherical punch that moves downwards in small increments. The model is comprised by two entities: the sample, formed by 600 quadratic elements distributed in a quadratic mesh, and the ball formed by 548 triangular elements, as shown in Fig. 1a. The model allows large deformations and it does not implement damage.

The vertical displacement, d, is applied by the ball which transmits the load to the disk. This model does not consider time dependent properties. The contact between the ball and disk used a penalty method with a constant friction coefficient, μ=0.4. We selected the same type of
Table 1
Chemical composition of the gas-atomized 316L powder.

| Steel grade | Cr  | Ni  | Mo  | Si  | Mn  | V   | Co  | Cu  | C   | S   | Fe  |
|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 316L        | 16.53 | 11.55 | 2   | 0.7 | 0.54 | 0.06 | 0.062 | 0.029 | 0.015 | 0.007 | Bal |

Table 2
SLM manufacturing parameters.

| Laser parameters | Power (W) | Line offset (mm) | Speed (mm/s) | Focus diameter (mm) |
|-------------------|-----------|------------------|--------------|--------------------|
| 316L              | 98.86     | 0.14             | 200          | 0.2                |

Table 3
Tensile test data of experimental and FEM limit materials. Yield stress (YS), ultimate tensile stress (UTS), uniform elongation (\(\varepsilon_u\)), elongation to fracture (\(\varepsilon_f\)) and the ultimate tensile stress to yield stress ratio.

| Material                  | YS (MPa) | UTS (MPa) | \(\varepsilon_u\) (MPa) | \(\varepsilon_f\) (MPa) | UTS/YS |
|---------------------------|----------|-----------|--------------------------|--------------------------|--------|
| 316L SLM (Experimental)   | 600      | 894       | 0.2                      | 0.217                    | 1.49   |
| 316L Annealed (FEM)       | 172      | 480       | 0.34                     | 2                        | 2.790  |
| 316L Hardened (FEM)       | 965      | 1280      | 0.01                     | 2                        | 1.362  |

contact as other authors [31,55,56]. The nodes are distributed with a density of the mesh governed by physics, so that there is a high density of elements in the zone of high stress gradient.

In this FEM analysis two “extreme” materials, in terms of strength, have been used to feed the model. It is reasonable to assume that any conventional 316L steel will have a mechanical performance between these two limit cases. The simulation was performed in two 316L stainless steel tempers: 316L annealed, A, and hardened, H, which properties were implemented parametrically and are shown in Table 3. A linear hardening rate has been employed for the FEM materials up to \(\varepsilon_u\), since it is the common behaviour for ductile 316L austenitic stainless steels.

In order to avoid premature failure during deformation on the FEM model, the true strain to failure, \(\varepsilon_f\), has been set to a maximum of 2, so that once the material reaches the UTS, such stress is maintained up to \(\varepsilon_f\). Additionally, 10 different load/unload cycles have been simulated for both materials, as shown in Fig. 6. The very first FEM unloading cycle is performed at just 10 \(\mu\)m from the beginning. Although this first unloading/loading cycle it is not evident in the figure, it was properly taken into account and quantified.

3. Results and discussion

The tensile test is designed to test a simple uniform geometry applying a simple uniaxial stress field. The result is a nonlinear F–d curve showing the elastic and the plastic behaviour of a practically constant “effective” volume of material, which corresponds to, in principle, the gauge length. “Effective” refers to the fact that some additional deforming volume adjacent to gauge volume could be incorporated if the material presents a remarkable strain hardening. Usually, the elastic displacement is calculated as the displacement for which the yield stress is reached and then, subtracted from the total displacement for presenting, after a simple calculation, a true stress–true plastic strain (\(\sigma–\varepsilon\)) curve.

However, in the case of the SPT, the effective tested volume, the applied stress and the obtained strain fields are neither constant nor uniform, and vary continuously throughout the test. During the SPT test (see Fig. 1.a), the ball progressively deforms non uniformly the sample which suffers progressively increasing elastic and plastic strains as the test progresses. In fact, the sample stiffness increases progressively as the ball deforms an increasing effective volume of material with increasing ball displacement [57,58].

Additionally, for both tensile and SPT tests, the whole testing machine and set-up rig are not perfectly rigid so there is a compliance component that needs to be considered. While in the tensile test such compliance is constant and considered in the elastic correction, in the SPT set-up it increases with the test and this fact has not been adequately considered. It will be shown in this paper that if the machine is not very rigid, the elastic displacement affects considerably the total displacement of the SPT curve, reaching deviations higher than 10%.

3.1. Cycling loading Small Punch Tests

The calculation of the instantaneous elastic and plastic components require several loading–unloading cycles along the test. Fig. 2 shows the experimental SPT F–d curve of a 316L SLM stainless steel tested under 14 loading–unloading cycles. The calculation of the elastic component is performed at each unloading step, following a similar approach to the one proposed by Oliver and Pharr for nanoindentation curves [59]. The slope of the unloading curve, in the region close to the maximum load reached at a given position, \(d\), provides the global stiffness of both, the sample and SPT set-up, at such position. As it is observed in Fig. 2, the slopes of each unloading step increase as \(d\) and \(F\) increase. This behaviour is a consequence of the evolution of the SPT test: initially, the ball is just interacting in a very small area at the very centre of the sample, while as the test progresses the ball-sample contact area increases. In fact, the contact radius between the ball and the sample is continuously increasing during the SPT test. In terms of stiffness, the increase on the ball-sample contact area during the test is related to an increase of the
effective gauge volume size. Additionally, it is expected that the larger the applied loads, the larger the set-up stiffness. The contribution of these two additive effects on the measured stiffness can be estimated using finite elements modelling and its analysis will be performed below. Experimentally, we can measure the global stiffness/compliance (sample and SPT set-up) and use its value to obtain the elastic and plastic displacements from the total displacement. At this point it is convenient to remind to the reader that, in our SPT experiments, the total displacement is measured from the top of the sample and not through a LVDT gauge situated below the sample.

### 3.2. Methodology for obtaining force vs. plastic displacement SPT curve

A convenient way for calculating elastic and plastic displacements is to switch axis of Fig. 2, as shown in Fig. 3a, and thus, plotting the total displacement vs. force (d-F). In this representation, the slopes of each unloading cycle are the values of the compliance (C) at each step. The compliance is the inverse of the stiffness (k), C = 1/k, being i the number of each unloading step, which takes values 1 to 14. The compliances values are high at the beginning of the test and decrease continuously with increasing load, contrary to what occurs to the stiffness values.

Based on Hooke’s law, the elastic displacement, d_{ei}, for each unloading step i, is given by the expression d_{ei} = C_{i} \cdot F_{i}, where F_{i} is the reached load when the unloading step starts. Therefore, the total displacement, d_{t}, will be given by the expression:

\[ d_{t} = C_{i} \cdot F_{i} + d_{pi} \]  

where \( d_{pi} \) is the corresponding plastic displacement for a total displacement \( d_{t} \).

The discrete values of the different \( C_{i} = 1/k_{i} \) and \( d_{pi} \) as a function of \( d_{i} \) are plotted in Fig. 3b. Additionally, the values of the global stiffness, \( k_{i} \), are plotted in Fig. 4. In order to obtain continuous functions of the three variables \( C, k \) and \( d_{pi} \), the discrete values were fit using polynomial functions. The resulting continuous fitting curves have been plotted in Fig. 3b and Fig. 4 (solid lines). Sixth-order polynomial fits have been chosen for the compliance (Eq. (2)) and the plastic displacement (Eq. (3)), and a fifth-order polynomial for the stiffness (Eq. (4)). The values of the coefficients are given in Table 4. These polynomials are valid from \( d = 0.2 \) to 1.5 mm for the tested 316L SLM stainless steel using our SPT set-up. For \( d \) values below 0.2 mm it would be necessary to assume an initial value of \( C \) when \( d \) tends to 0, which introduce some uncertainty in the calculations. Therefore, Eqs. (2) and (4) cannot be used at \( d < 0.2 \) mm, i.e. under the first unloading step. Nevertheless, the plastic displacement is \( d_{pi} = 0 \) when \( d = 0 \) and thus, this variable can be extrapolated towards \( d = 0 \).

\[
C = \frac{1}{k} = b_{0}d^{6} + b_{1}d^{5} + b_{2}d^{4} + b_{3}d^{3} + b_{4}d^{2} + b_{5}d + b_{6}
\]  

\[
d_{pi} = a_{0}d^{6} + a_{1}d^{5} + a_{2}d^{4} + a_{3}d^{3} + a_{4}d^{2} + a_{5}d^{1} + a_{6}
\]  

\[
k = g_{0}d^{6} + g_{1}d^{5} + g_{2}d^{4} + g_{3}d^{3} + g_{4}d^{2} + g_{5}d^{1} + g_{6}
\]  

By using Eq. (3) we can now represent the values of \( F \) vs. \( d_{pi} \). The original SPT F-d curve (uncorrected, black line) is compared with the corrected F- \( d_{pi} \) (blue line) curve in Fig. 5. The difference between both curves corresponds to the elastic displacement. It is worth mentioning that the correction is successful since all the unloading slopes in the corrected curve become completely vertical (slope → ∞). Note that if only the initial stiffness was used to correct the original curve, a large overcorrection should be obtained for high displacement values. At the highest total displacement values, the difference between measured \( d \) and \( d_{pi} \) values is more than 10%. This difference is more evident when comparing the beginning of the corrected and uncorrected curves. For
Table 4

Coefficients of polynomial fits of C (Eq. (2)), \( d_p \) (Eq. (3)) and \( k \) (Eq. (4)) for SPT 316L SLM steel.

| Eq (2) | \( b_0 \) | \( b_1 \) | \( b_2 \) | \( b_3 \) | \( b_4 \) | \( b_5 \) |
|--------|-----------|-----------|-----------|-----------|-----------|-----------|
| C      | \(-7.399E-07\) | \(3.596E-06\) | \(-6.707E-06\) | \(5.940E-06\) | \(-2.293E-06\) | \(-2.600E-08\) | \(3.310E-07\) |
| Eq (3) | \( a_0 \) | \( a_1 \) | \( a_2 \) | \( a_3 \) | \( a_4 \) | \( a_5 \) | \( a_6 \) |
| \( d_p \) | \(2.842E-01\) | \(-1.540E+00\) | \(3.597E+00\) | \(-4.407E+00\) | \(2.832E+00\) | \(9.420E+02\) | \(-3.000E-04\) |
| Eq (4) | \( g_0 \) | \( g_1 \) | \( g_2 \) | \( g_3 \) | \( g_4 \) | \( g_5 \) |
| \( k \) | \(0\) | \(1.355E-07\) | \(-5.963E+07\) | \(8.882E+07\) | \(-5.696E+07\) | \(2.334E+07\) | \(6.150E+05\) |

Fig. 5. Load-unload vs. total displacement (\( d \)) and plastic displacement (\( d_p \)) SPT curves obtained for 316L SLM.

instance, as shown in Fig. 5, when total displacement is 0.2 mm, the corresponding plastic displacement is only 0.1 mm, which is a 50% difference at this displacement. Additionally, the form of the curve is also modified, so now, more reliable predictions of mechanical parameters, such as yield stress, can be performed.

3.3. FEM analysis

The proposed experimental procedure allows to extract a \( F-d_p \) curve, which will be enough for obtaining the corresponding mechanical parameters in most of the scenarios. Nevertheless, just by using the experimental data it is not possible to discern the relative contribution of the sample and the SPT set-up in terms of elastic displacement. Such analysis can be performed by FEM. In this study, we used the FEM mesh shown in Fig. 1b and imposed an infinite stiffness to the ball. Since the FEM mesh does not consider any other grip or part of the set-up and the ball behaves as a perfect rigid body, the stiffness/compliance will be associated exclusively to the 316L sample. We simulated the plastic behaviour of two extreme materials, namely 316L annealed (low strength) and 316L hardened (high strength), both having the same elastic modulus. This will help to elucidate if the different plastic behaviour has an influence on the overall SPT stiffness values. Additionally, these two extreme behaviours are reasonable upper and lower limits for the behaviour of any intermediate 316L steel, as the one used in the experiments.

The simulated force vs. ball displacement SPT curves for both materials (H, hardened, and A, annealed) are shown in Fig. 6. The FEM simulation was set to perform 10 loading-unloading cycles every 0.2 mm, starting at 10 \( \mu \)m. Additionally, in the same graph, the corrected curves, where \( F \) is plotted against \( d_p \), for each simulated test are included. A small window in Fig. 6 shows a detail of the second unloading step (at \( d = 0.2 \) mm) of the uncorrected and corrected curves of the 316L hardened (H) steel. First, it is worth mentioning that the unloading slopes of the \( F \) vs. \( d \) loading/unloading cycles present much higher values than the experimental ones (Figs. 2, 3 and 5) for both annealed and hardened model 316L steels. Additionally, it must be pointed out that the load values sustained by the experimental 316L SLM are approximately in the middle of the model annealed and hardened 316L steel values of Fig. 6. Following the same procedure with the FEM results as with the experimental 316L SLM, the different \( k \) and \( d_p \) values were obtained as a function of ball position \( d \). In Fig. 7.a the values of \( k \) vs. \( d \) for the three steels (316L annealed, in red, 316L hardened, in blue, and SLM in black) are plotted in a logarithmic scale, while Fig. 7.b presents the values of \( d_p \) vs. \( d \) for the model annealed and hardened steels. The latter figure clearly shows that \( d_p \) values are practically the same for both model materials, except for a mere 40 \( \mu \)m difference at the beginning of the test for the hardened steel. Additionally, the slope of \( d_p \) vs. \( d \) is almost 1 in the whole range, which points to a low elastic contribution of the sample since the ball does not contribute. The huge difference in the yield stress values between the model hardened and annealed steels is reflected into different initial elastoplastic behaviours: while the annealed material deforms plastically early at the very beginning of the test, the hardened one presents a larger elastic response. This is the origin of the initial difference in the \( d_p \) between both materials. Such small difference in \( d_p \) for both model materials and the slope 1 in the linear relation between \( d_p \) and \( d \) (Fig. 7b) proves that during SPT the elastic contribution of the material is almost negligible compared to the elastic contribution of the SPT set up. The high elastic displacement values observed in the experimental conditions (Fig. 3) and the remarkable modification of the \( F-d_p \) curve compared to the experimental \( F-d \) (Fig. 5) corroborate this claim. In fact, Fig. 7.a shows relatively similar total stiffness values for the modelled 316L annealed and hardened steels, and both much higher than for the experimental 316L SLM steel. Equations (5) and (6) and Table 5, present the polynomial fitting equations and fitting, respectively, for both model materials:

\[
\begin{align*}
  k_a &= c_9 d^6 + c_8 d^5 + c_7 d^4 + c_6 d^3 + c_5 d^2 + c_4 d + c_3 \\
  k_h &= f_9 d^6 + f_8 d^5 + f_7 d^4 + f_6 d^3 + f_5 d^2 + f_4 d + f_3
\end{align*}
\]  

(5)  

(6)

where \( k_a \) and \( k_h \) are the stiffness of the annealed and hardened 316L steels, respectively.

Comparing the stiffness values obtained experimentally and by FEM we can infer some key insights. The \( k \) values for the model hardened 316L range from 1.38 10^7 N/m at 10 \( \mu \)m to 36.3 10^7 N/m at 1.8 mm, i.e.,
performed at each laboratory [60]. The coefficients for such SPT set-up, similarly to nanoindentation corrections, are lower than the modelled steels at the beginning of the SPT test and this difference increases to up to about 25 times lower at the end of the test. By polynomial fitting the stiffness values, a proper separation of elastic and plastic components can be performed as a function of ball displacement so that from the load–displacement curve (\(F - d\)) it can be properly obtained the load-plastic displacement curve (\(F - d_p\)).

2. The total elastic displacement is given by two contributions: the first one is the elastic behaviour of the material, and the second one is the compliance of the SPT set-up. FEM analysis shows that the elastic contribution of the material is very low compared to the one from the SPT set-up. This fact highlights the importance of performing such correction, especially when comparing data sets from different laboratories.

3. The analysis of the correction of the experimental curve (\(F - d\)) shows that the elastic displacement measured using the SPT set-up employed in this research is, at least, 10% of the total displacement curve. This difference is more evident when comparing the beginning of the corrected and uncorrected curves (about 50%). Additionally, there is a clear change on the shape of the curve, which has an influence on the estimation of the corresponding mechanical parameters of the sample such as the yield strength.

4. Once the employed SPT set-up compliance is calibrated, it is possible to correct previous tests by performing various loading/unloading cycles to obtain the elastic and plastic, displacements as a function of ball position so that from the load–displacement curve (\(F - d\)) it can be properly obtained the load-plastic displacement curve (\(F - d_p\)). The method succeeds in its purpose and proves that comparisons between data sets from different materials and SPT set-ups can only be properly done using \(F - d_p\) curves.

4. Conclusions

The measurement of the displacement values, \(d\), during Small Punch Tests, SPT, depends noticeably on the compliance of the SPT set-up, which is different for each laboratory. Therefore, improved comparisions and analyses can be made considering only the \(F\) vs. \(d\) (plastic displacement) curve. In this study, we present a method for separating elastic and plastic displacements by performing loading-unloading cycles during SPT. This is not trivial because the elastic displacement depends on the amount and geometry of the deformed material at each ball position. The experimental results were compared with FEM analyses allowing us to obtain the following conclusions:

1. A method for stiffness/compliance measurement during SPT by using load-unload cycles has been proposed. Each unloading slope provides the stiffness value at each displacement value. We found large differences in the stiffness values from the beginning until the end of the test. By polynomial fitting the stiffness values, a proper separation of elastic and plastic components can be performed as a function of ball displacement so that from the load–displacement curve (\(F - d\)) it can be properly obtained the load-plastic displacement curve (\(F - d_p\)).

2. The method for stiffness/compliance measurement during SPT by using load-unload cycles has been proposed. Each unloading slope provides the stiffness value at each displacement value. We found large differences in the stiffness values from the beginning until the end of the test. By polynomial fitting the stiffness values, a proper separation of elastic and plastic components can be performed as a function of ball displacement so that from the load–displacement curve (\(F - d\)) it can be properly obtained the load-plastic displacement curve (\(F - d_p\)).

26 times increase from beginning to the end of the test. In contrast, the experimental stiffness values increase about three times from the beginning of the unloading/loading cycles, at \(d = 0.2\) mm, to the end, at \(d = 1.2\) mm. Additionally, the experimental \(k\) values are about 8 times lower than the modelled steels at the beginning of the SPT test and this difference increases up to about 25 times lower at the end of the test.

Finally, all the stiffness values increase as the test proceeds, indicating that the volume of material undergoing deformation is increasing continuously, proving that simulations capture properly such behaviour.

Table 5

| Eq. (4) | \(a_0\) | \(a_1\) | \(a_2\) | \(a_3\) | \(a_4\) | \(a_5\) | \(a_6\) |
|-------|-------|-------|-------|-------|-------|-------|-------|
| \(k_a\) | 1.895E07 | 1.271E08 | -3.784E08 | 5.441E08 | -2.867E08 | 9.833E07 | 1.407E07 |
| \(k_b\) | 6.892E07 | -3.380E08 | 4.533E08 | -2.773E07 | -1.137E08 | 9.134E07 | 1.293E07 |


4. Conclusions

The measurement of the displacement values, \(d\), during Small Punch Tests, SPT, depends noticeably on the compliance of the SPT set-up, which is different for each laboratory. Therefore, improved comparisions and analyses can be made considering only the \(F\) vs. \(d\) (plastic displacement) curve. In this study, we present a method for separating elastic and plastic displacements by performing loading-unloading cycles during SPT. This is not trivial because the elastic displacement depends on the amount and geometry of the deformed material at each ball position. The experimental results were compared with FEM analyses allowing us to obtain the following conclusions:

1. A method for stiffness/compliance measurement during SPT by using load-unload cycles has been proposed. Each unloading slope provides the stiffness value at each displacement value. We found large differences in the stiffness values from the beginning until the end of the test. By polynomial fitting the stiffness values, a proper separation of elastic and plastic components can be performed as a function of ball displacement so that from the load–displacement curve (\(F - d\)) it can be properly obtained the load-plastic displacement curve (\(F - d_p\)).

2. The total elastic displacement is given by two contributions: the first one is the elastic behaviour of the material, and the second one is the compliance of the SPT set-up. FEM analysis shows that the elastic contribution of the material is very low compared to the one from the SPT set-up. This fact highlights the importance of performing such correction, especially when comparing data sets from different laboratories.

3. The analysis of the correction of the experimental curve (\(F - d\)) shows that the elastic displacement measured using the SPT set-up employed in this research is, at least, 10% of the total displacement curve. This difference is more evident when comparing the beginning of the corrected and uncorrected curves (about 50%). Additionally, there is a clear change on the shape of the curve, which has an influence on the estimation of the corresponding mechanical parameters of the sample such as the yield strength.

4. Once the employed SPT set-up compliance is calibrated, it is possible to correct previous tests. Therefore, the load–plastic displacement curve (\(F - d_p\)) should be similar for the same material tested at any laboratory with similar device geometry, and thus, more adequate than the \(F - d\) curve for evaluations and mechanical parameters analysis.

CRediT authorship contribution statement

David Sánchez-Ávila: Conceptualization, Methodology, Formal analysis, Investigation, Visualization, Writing - original draft, Writing - review & editing. Alberto Orozco-Caballero: Investigation, Methodology, Software, Visualization, Writing - review & editing. Elkin Martínez: Resources, Writing - review & editing. Luis Portoles: Resources, Writing - review & editing. Rafael Barea: Software, Supervision, Writing - original draft, Writing - review & editing. Fernando Carreno: Conceptualization, Methodology, Formal analysis, Investigation, Resources, Visualization, Writing - original draft, Writing - review &
Dealing of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

Financial support from MINECO (Spain), Project MAT2015-68919-C3-1-R (MINECO/FEDER) is gratefully acknowledged. D. Sánchez-Avila thanks Nebrija University for a PhD scholarship, and a research contract at CENIM, CSIC financed by the aforementioned project. A. Orozco-Caballero thanks also a research contract at CENIM, CSIC financed by same project.

References

[1] Y. Zhai, B.o. Huang, X. Mao, M. Zheng, Effect of hot isostatic pressing on microstructure and mechanical properties of ClAM steel produced by selective laser melting, J. Nucl. Mater. 512 (2019) 111–121, https://doi.org/10.1016/j. jnucmat.2018.12.028.

[2] M. Song, M.i. Wang, X. Lou, R.B. Rebak, G.S. Was, Radiation damage and irradiation-assisted stress corrosion cracking of additively manufactured 316L stainless steels, J. Nucl. Mater. 513 (2019) 33–44, https://doi.org/10.1016/j. jnucmat.2018.10.044.

[3] H. Zhou, M. Zhao, Z. Ma, D.Z. Zhang, G. Fu, Sheet and network based functionally graded lattice structures manufactured by selective laser melting: Design, microstructure and mechanical properties, Mater. Sci. Eng., A 728 (2018) 259–272, https://doi.org/10.1016/j.msea.2017.03.006.

[4] N. Soro, H. Attar, E. Brodie, M. Veidt, A. Molotnikov, M.S. Dargusch, Evaluation of the mechanical compatibility of additively manufactured porous Ti-25Ta alloy for load-bearing implant applications, J. Mech. Behav. Biomater. Mater. 97 (2019) 149–158, https://doi.org/10.1016/j.jmbbm.2019.05.019.

[5] –. Ríos-Márquez, E. Altstadt, M. Houska, I. Simonovski, D. Baraldi, S. Holmström, Determining the ultimate tensile strength of fuel cladding tubes by small punch testing, J. Nucl. Mater. 509 (2018) 620–630, https://doi.org/10.1016/j. jnucmat.2018.07.041.

[6] A. Kareev, A. Prasithipiyayong, D. Kruemel, D.M. Collins, P. Hooseman, S. Roberts, An analytical method to extract irradiation hardening from nanoindentation hardness-depth curves, J. Nucl. Mater. 498 (2018) 274–281, https://doi.org/10.1016/j. jnucmat.2017.10.049.

[7] O. Grydin, A. Andreiev, M.J. Holzweissig, I. Simonovski, D. Baraldi, S. Holmström, E. Altstadt, M. Houska, I. Simonovski, D. Baraldi, S. Holmström, Determining the ultimate tensile strength of fuel cladding tubes by small punch testing, J. Nucl. Mater. 509 (2018) 620–630, https://doi.org/10.1016/j. jnucmat.2018.07.041.

[8] –. Soro, H. Attar, E. Brodie, M. Veidt, A. Molotnikov, M.S. Dargusch, Evaluation of the mechanical compatibility of additively manufactured porous Ti-25Ta alloy for load-bearing implant applications, J. Mech. Behav. Biomater. Mater. 97 (2019) 149–158, https://doi.org/10.1016/j.jmbbm.2019.05.019.

[9] –. Ríos-Márquez, E. Altstadt, M. Houska, I. Simonovski, D. Baraldi, S. Holmström, Determining the ultimate tensile strength of fuel cladding tubes by small punch testing, J. Nucl. Mater. 509 (2018) 620–630, https://doi.org/10.1016/j. jnucmat.2018.07.041.

[10] A. Kareev, A. Prasithipiyayong, D. Kruemel, D.M. Collins, P. Hooseman, S. Roberts, An analytical method to extract irradiation hardening from nanoindentation hardness-depth curves, J. Nucl. Mater. 498 (2018) 274–281, https://doi.org/10.1016/j. jnucmat.2017.10.049.

[11] O. Grydin, A. Andreiev, M.J. Holzweissig, I. Simonovski, D. Baraldi, S. Holmström, E. Altstadt, M. Houska, I. Simonovski, D. Baraldi, S. Holmström, Determining the ultimate tensile strength of fuel cladding tubes by small punch testing, J. Nucl. Mater. 509 (2018) 620–630, https://doi.org/10.1016/j. jnucmat.2018.07.041.

[12] A. Kareev, A. Prasithipiyayong, D. Kruemel, D.M. Collins, P. Hooseman, S. Roberts, An analytical method to extract irradiation hardening from nanoindentation hardness-depth curves, J. Nucl. Mater. 498 (2018) 274–281, https://doi.org/10.1016/j. jnucmat.2017.10.049.

[13] O. Grydin, A. Andreiev, M.J. Holzweissig, I. Simonovski, D. Baraldi, S. Holmström, E. Altstadt, M. Houska, I. Simonovski, D. Baraldi, S. Holmström, Determining the ultimate tensile strength of fuel cladding tubes by small punch testing, J. Nucl. Mater. 509 (2018) 620–630, https://doi.org/10.1016/j. jnucmat.2018.07.041.

[14] A. Kareev, A. Prasithipiyayong, D. Kruemel, D.M. Collins, P. Hooseman, S. Roberts, An analytical method to extract irradiation hardening from nanoindentation hardness-depth curves, J. Nucl. Mater. 498 (2018) 274–281, https://doi.org/10.1016/j. jnucmat.2017.10.049.

[15] O. Grydin, A. Andreiev, M.J. Holzweissig, I. Simonovski, D. Baraldi, S. Holmström, E. Altstadt, M. Houska, I. Simonovski, D. Baraldi, S. Holmström, Determining the ultimate tensile strength of fuel cladding tubes by small punch testing, J. Nucl. Mater. 509 (2018) 620–630, https://doi.org/10.1016/j. jnucmat.2018.07.041.

[16] A. Kareev, A. Prasithipiyayong, D. Kruemel, D.M. Collins, P. Hooseman, S. Roberts, An analytical method to extract irradiation hardening from nanoindentation hardness-depth curves, J. Nucl. Mater. 498 (2018) 274–281, https://doi.org/10.1016/j. jnucmat.2017.10.049.
