Identification of Fe-Zn coating behaviors by a new reverse approach using artificial intelligence

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Abstract
Nanoindentation is a technique commonly used to measure the mechanical properties of thin films even at depths less than 1 μm. In fact, the characterization is based on the study of the load/displacement curves resulting from the nanoindentation test. We aim to use the backtracking search optimization algorithm (BSA) to improve the extraction of $P(h)$ curves performed by nanoindentation on galvannealed Fe-Zn coating in terms of precision and dispersion. Indeed, the originality of this study is not limited only to the $P(h)$ curve extraction methods, but also to its application in modeling the reverse approach for the case of Fe-Zn coating deposited on a Dual-Phase DP600 steel substrate. Indeed, the BSA approach showed more precision (with respect to the determined mean value) and less dispersion (magnitude of error around the identified mean value) compared to the Least-squares approach. The average error with the BSA and LS methods is respectively 0.89% and 3.16% for the yield stress ($\sigma_y$) and 3.17% and 7.93% for the strain hardening exponent ($n$). This reduced the error variability in the prediction of the constitutive law to 72% and 60% for $\sigma_y$ and $n$, respectively. Thus, we solved the problems of accessibility, uniqueness of the solution, precision (+10%) and dispersion (−85%) of the required prediction models for the Fe-Zn coating behavior.

1. Introduction

The nanoindentation test has been widely used in various reverse approaches to determine the elastoplastic, isotropic, or anisotropic properties of indented surfaces. In this context, some approaches have been established for different indenters (Vickers [1, 2], spherical [3, 4], Berkovich or conical [5, 6]) based on the indenter/specimen contact theories and on resulting plastic deformation. The $P(h)$ curve parameters were processed using dimensionless functions [7–9], nanoindentation fingerprints [10–13] or Finite Element (FE) simulations [14]. The main objective of these studies is to ensure the uniqueness of the solution in determining the mechanical properties by the reverse identification approach. Moreover, reverse methods allow extracting the mechanical behavior of coated surfaces from instrumented indentation [7–19]. In addition, Panich et al [16] discussed the impact of the indenter geometry and the gradient of the mechanical characteristics in the coating/substrate system on the evolution of the load-displacement $P(h)$ curves. They found that the critical indentation depth depends on the roughness, indenter tip radius and resistance ratio of the coating/substrate system. These approaches have allowed considerable progress in the study of $P(h)$ curves retracing the elastoplastic behavior of indented surfaces. However, the various challenges to achieve a universal methodology have not been successful. Indeed, they do not guarantee an explicit and unique solution for the identification of the coating behavior, especially for the case of the inverse methods.
Any direct or reverse approach for the mechanical behavior identification requires the physical quantity extraction governing the required $P(h)$ curves evolution. In practice, the mathematical modeling of the various experimental or simulation results is carried out by classical approaches (Newton-Raphson, gradients, least-squares, etc) based on the minimum potential of local solutions [20, 21]. These modeling approaches are better suited to linear problems which are based on the choice of the initial value and the number of iterations. However, in the case of non-linear problems, reverse optimization techniques based on artificial intelligence tools, such as genetic algorithms, fuzzy logic, and neural networks, are often used [22]. In this regard, the meta-heuristic BSA approach presents one of these reverse optimization techniques based on fast converging stochastic algorithms [23–25]. This approach is based on three basic operators which are selection, mutation and crossing starting from an initial base population.

For identification based on experimentation, the BSA algorithms invented in 2013 by Civicioglu [23] have attracted attention in the electrical systems regulation, computing, industry and energy [26]. However, these algorithms have not been evaluated in extracting the $P(h)$ curves of the nanoindentation test to identify the coating behavior. In nanoindentation, the experiment is established on smooth surfaces or on a FE sufficiently homogeneous and stable. Thus, the extraction of the $P(h)$ curves evolution does not include any significant error. Meanwhile, in the case of the galvannealed Fe-Zn coating, the extraction of these parameters is delicate because the indentation is performed on finished surfaces without prior polishing due to their low thickness and scratch sensitivity. Moreover, the hardness ratio is 340/320HV for the coating/substrate system, unlike the cases of PVD/CVD (Physical/Chemical Vapor Deposition) coatings for example. Indeed, these industrial coatings are sufficiently hard (1200–3000HV) compared to their substrates [10–13, 27–29].

Regarding the thin layers induced by industrial zinc-based coatings (Zn–O, Zn–Ni, Zn–Mg, etc), several scientific studies [30–33] have examined their behavior, their microhardness and their corrosion and wear resistance. In the case of Zinc coatings alloyed with Iron, some work has also aimed to understand the kinetics of their required formation [34–36], their microstructural textures, and their corrosion resistance [37, 38]. However, few authors studied the galvannealed Fe-Zn coatings deposited on High-Limit-Elastic (HLE) steels, especially for the case of the dual-phase DP600 substrate [39, 40].

The use of CSM (Continuous Stiffness Measurement) mode in nanoindentation, coupled with BSA algorithms and dimensionless functions allowed to identify the elastoplastic Fe-Zn coatings behavior. On the other hand, the methodology approved by Dao et al [7–9] was used in the reverse identification of the elastoplastic constitutive laws of industrial Fe-Zn coatings. This improves the precision and reduces the dispersion when extracting the $P(h)$ curves from the nanoindentation. In our study, we need to explore a new approach (BSA) as an alternative to classical Least-Squares approaches. To do this, we proceeded as follows:

i. From the reference data of the $P(h)$ curves with known constructive parameters, we validated the BSA approach in the extraction of the physical quantities governing the evolution of the $P(h)$ curves. The goal of this step is to evaluate the precision and dispersion of the extraction approach.

ii. We used the FE method to simulate nanoindentation coupled with known constitutive laws, then extracted the resulting $P(h)$ curves and found the predefined behavior model by inverse approach. Thus, we evaluated the precision and dispersion of the reverse identification of the predefined constitutive laws.

iii. We used the experimental nanoindentation data applied to Fe–Zn coated surfaces, to extract the parameters of the experimental $P(h)$ curves. Then, we applied the reverse approach to identify the mechanical constitutive laws of the Fe-Zn coating.

2. Methodology and approaches

2.1. Materials substrate and Fe-Zn coating

The base substrate is a cold-rolled HLE-DP600 Dual-Phase steel sheet with a Ferrite-Martensite microstructure. The martensite, finely dispersed in a ferrite matrix, plays an essential role in the mechanical behavior by controlling the local damage and failure mechanisms [41–43]. The various mechanical characterization tests permitted to obtain the DP600 properties confirming the Hollomon relation modeled by the expression (equation (1)):

$$
\sigma = E \varepsilon \ (\varepsilon \leq \varepsilon_y) \\
\sigma_{p} = K \varepsilon_{p} \Gamma (\varepsilon > \varepsilon_y)
$$

(1)

With $\sigma_p$ is the plastic stress, $\varepsilon_p$ the plastic strain, $\varepsilon_y$ the yield strain, $E$ the Young’s modulus, $K$ the hardening modulus, and $n$ the hardening exponent. In fact, the mechanical characteristics of the DP600 substrate used in
the nanoindentation test simulation are given in table 1. The DP600 substrate should be considered isotropic \((R = 0.97)\) and can be modeled by the Hollomon’s relation with \(K = 1070\) and \(n = 0.186\).

The continuous dip galvanizing process for thin sheets provides industrial coatings that can be completed by various forming processes (Stamping, welding, painting, etc). In this study, the Fe-Zn coating deposited on the DP600 Dual-Phase steel was delivered by the ArcelorMittal Company. The typical coating process consists of continuous galvanization in a liquid zinc bath at 480 °C for 8 min followed by a post-galvanization treatment (520 °C for 16 s). The post-galvanization treatment induces iron diffusion from the substrate into the zinc layer, essentially revealing the formation of a bilayer coating: the δ phase \((FeZn_{10})\) and the Γ phase \((Fe,Zn_{10})\) [34, 40, 44–52]. The mechanical behavior of the Zinc coatings mechanical behavior has been modeled by some authors using the modified Hollomon’s relation [53] which considers a restriction of the elastic/elastoplastic behavior on the stress-strain curve (equation (2)):

\[
\sigma = E\varepsilon (\varepsilon \leq \varepsilon_y) \\
\sigma = \sigma_y \left[ 1 + \frac{E}{\sigma_y} (\varepsilon - \varepsilon_y) \right]^n (\varepsilon > \varepsilon_y) \quad (2)
\]

The identification of the different parameters of the above model is the subject of our study. They are useful in the case of modeling the behavior of the Fe-Zn coating, in order to validate the efficiency of the BSA algorithms. In this regard, this identification is also essential to analyze the local kinetic mechanisms of damage/failure during shaping by plastic deformation when stamping or bending thin sheets for example [47–50, 52].

2.2. Experimental details
2.2.1. Experimental procedure: nanoindentation test
The Fe-Zn coated DP600 sheet has been cut by laser process with a size of about 20 × 20 × 1 mm³. Surface impurities were removed by ethylene and acetone solution using the electrolysis techniques for a period of 10 min before cleaning with distilled water. Subsequently, the samples were fixed on a suitable support for mounting on the instrumented indentation device. The experimental tests were conducted in CSM mode using an MTS Nano-Indenter XP® device equipped with a Berkovich type diamond indenter with a tip radius of 50 nm. The CSM mode was used for continuous measurement of harmonic contact stiffness as a function of indenter displacement. This makes it possible to calculate the mechanical properties (Hardness and Young’s modulus) as a function of the displacement of the indenter. The indentation measurements were performed in a controlled manner at a maximum depth of 1000 nm where the logarithmic indentation strain rate was maintained at 0.05 s⁻¹ and the superimposed harmonic oscillating force frequency was 45 Hz. Thus, the Young’s modulus, hardness and standard deviations of displacement were so continuously registered. The instrument resolution is about 50 nN for the load and 0.01 nm for the displacement indenter. Ten nanoindentation tests were realized on the Fe-Zn coating surface and the instrument was calibrated using a standard of fused silica before the series of tests. For modeling purposes, deformation during loading is assumed to be both elastic and plastic in nanoindentation. The Young’s modulus \(E\) and the Poisson’s ratio \(\nu\) of the pyramidal diamond indenters are respectively 1100 GPa and 0.07. These quantities will be exploited in the calculations of the Fe-Zn coatings behavior laws parameters in the reverse identification established by the coupling between the BSA algorithms [23] and the Dao approach [7].

2.2.2. Fitting the \(P(h)\) curve
Different models governing the \(P(h)\) evolution with dependent variables have been proposed by some authors [7–13, 16–19]. A typical evolution of the \(P(h)\) curves is given by figure 1 revealing two distinct domains: an elastoplastic loading followed by an elastic unloading. The model’s reliability is strongly correlated with the parameter extraction from the \(P(h)\) curve resulting from the experimental or numerical approach. The precision for this extraction depends directly on the preparation of the surface for indentation as well as the polishing test conditions [18, 19]. Table 2 presents some models from the literature adopted in this work to extract the indentation curve parameters used to calculate the mechanical behavior of Fe-Zn thin films [7–9, 18, 19].

| Table 1. Typical mechanical characteristics of Fe-Zn coated DP600 steel. |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| DP600           | \(E\) [GPa]     | \(\sigma_y\) [MPa] | \(R_m\) [MPa]  | \(\nu\) [−]    | \(A\) [%]       | \(K\) [MPa]   | \(n\) [−]       |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 201.4           | 366.67          | 768.67          | 0.293           | 24.33           | 1069.67         | 0.186          | 0.97            |

Note: \(E\): Young’s Modulus; \(\sigma_y\): Yield Stress; \(R_m\): Tensile Strength; \(\nu\): Poisson’s coefficient; \(A\): Relative elongation at rupture; \(K\): Hardening modulus; \(n\): Hardening exponent and \(R\): Anisotropy coefficient.
Table 2. Models and parameters of the P(h) curve [7–10, 18, 19].

| Loading parameter’s                        | Unloading parameter’s                                |
|--------------------------------------------|------------------------------------------------------|
| $P_l(h) = Ch^2$: Evolution model of the Loading curve | $P_u(h) = \alpha.(h−hr)^{m}$: Evolution model of the Unloading curve |
| $C$ [N/nm²]: Constant curvature of the loading curve | $W_p/W_e$: Plastic/Elastic Energy loading/unloading. |
| $a_m$: The contact radius measured at $h_m$ | $\alpha$ [mN/nm $^{-m}$]: Constant of the model of the unloading phase |
| $A_m = \pi\sqrt{a_m}$: The true projected contact area | $m$ [–]: Exhibitor of the law power of the phase of discharge |
| $P_{in}$ [mN]: Maximum load in indentation | $S$ [mN/nm] $= \alpha.m.(h_m-h_r)^{m-1}$: Elastic unloading stiffness |
| $h_m$ [nm]: Maximum penetration in nanoindentation | $hr$ [nm]: Residual’s depths of the imprint |

The parameter extraction from the indentation curve is generally achieved by the classical LS approaches [7, 21]. Indeed, the extracted parameters make it possible to reconstruct the $P(h)$ curves evolution, and therefore to model the mechanical behavior of the indented surfaces in various reverse approaches [7–9, 16–19]. As an alternative to the Olivier-Pharr method [28, 29], a new reverse approach was proposed by Kossman et al [28]. This approach uses LS simulation and introduces a random Gaussian noise in the displacement data to improve robustness identification. This allows a more precise determination of the stiffness and the elastic modulus in nanoindentation. In this regard, the progress made by these authors has considerably improved the parameterization of the $P_l(h)$ unloading curves but is typically based on conventional extraction techniques and for more homogeneous structures than the Fe–Zn case. The method of nanoindentation is more adaptable to materials that generate experimental curves near parabolic shape or the curves follow Kick’s law [7].

To validate the contribution of the $P(h)$ curve extraction with the BSA approach, an implicit axisymmetric simulation using the standard ABAQUS® was retained. In fact, an axisymmetric model with semi-finished geometry compared to the maximum indentation is established. The elastoplastic model of Song et al [35] was adopted for the case of the Fe-Zn coatings. In addition, the limits variability $P(h)$, $E(h)$ and hardness $H(h)$ evolutions resulting from the nanoindentation tests were used to define the reference combinations of the Fe-Zn coating behavior laws. Furthermore, eight reference combinations were selected using the same extreme boundaries of the mechanical quantities ($E$, $\sigma_y$ and $n$) which were affected in the Fe-Zn film behavior during the FE simulations.

For more precision and rapid convergence of FE simulations, the mesh is refined in the indentation zone. The equivalent Berkovich indenter is a conical shape angle of 70.3° with the same projected area-depth function. For this purpose, we will obtain the reconstruction of the eight $P(h)$ curves resulting from the FE simulations whose pre-established reference characteristics will thus be known. Figure 2 illustrates the boundary conditions; the mesh methods and the dimension choice of the FE model for the substrate DP600 coated with Fe–Zn. This substrate is 1 mm thick, with a 20 μm thick Fe-Zn coating. The dimensions of the digital model were chosen to be 500 times greater than the maximum indentation depth. The mesh is an axisymmetric triangular linear type (CAX3) with three nodes and is refined in the indented zone. The indenter is assumed to be infinitely rigid. In the same context, the different quantities of the elastoplastic behavior of Fe–Zn Coating in loading phase $P_l(h)$
Algorithm 1. Main steps of the parameter extraction using LS.

1. Initialize the experimental data (P, h).
2. PolyLoad is a matrix of loading data.
3. PolyUnload is a matrix of unloading data.
4. k = 2
5. While (k < length (P))
6. if (P(k) – P(k-1) > 0) then PolyLoad receives [P(k), h(k)]
7. if (P(k) – P(k-1) < 0) then PolyUnload receives [P(k), h(k)]
8. k = k + 1
9. End While
10. Evaluate the coefficients for a polynomial (Best Load) of degree 3 that is the best fit in a least-squares sense for the data in PolyLoad.
11. Evaluate the coefficients for a polynomial (BestUnload) of degree 3 that is the best fit in a least-squares sense for the data in PolyUnload.
12. Calculate the real positive solution (h, m) such as BestUnload (h, m) = 0
13. Calculate the values (C) of the BestLoad (in a least-squares sense) where h is the x vector using: BestLoad = C * h^2
14. Calculate the values (α and m) of the BestUnload (in a least-squares sense) where h is the x vector using: BestUnload = α(h – h_0)^m
15. Readjust the value of couple (h_m, P_m) such as C * h_m^3 = α(m(h_m – h_0))^m
16. Calculate (S) using the equation: S = α * m(h_m – h_0)^m+1
17. Calculate (W_t) using the equation: W_t = C * h_m^3/3
18. Deduce (W_p) using the equation: W_p = (h_m/h_0) * W_t
19. Deduce (W_e) using the equation: W_e = W_t – W_p
20. Plot (x, y) data for input P (h) and output P_2 (h) results and evaluate the error.

(C[mN/mm^2], h_m[nm], W_p[mN/mm]) and of the elastic her elastic behavior in unloading phase P_m(h) (α[mN/mm^m], m[–], h_m[nm], S(m/N/mm)) are extracted from the FE P(h) curves.

2.3. P(h) curve extraction optimization methodology

2.3.1. Least-Square (LS) extraction of P(h) curves

To extract the P(h) curve parameters from the experimental results, we consider the measurements (P, h) recorded during the nanoindentation test. This gives us two column vectors named P = {P_1, P_2, …, P_n} and \( h = \{h_1, h_2, \ldots, h_n\} \) in \( \mathbb{R}^2 \) from the indentation of the Fe-Zn coating surfaces. At this step, starting from \( n \) measurements over 2000 pairs \( (h_i, P_i) \) per test, the LS resolutions of the non-linear systems are used to reconstruct the P(h) curve. The \( n \) measurements break down into two separate restrictions: the loading phase \( k \) measurements) and unloading phase \( n-k \) measurements). The measurement of the parameter dispersion governing the evolution of load/unload restrictions is evaluated by the standard deviation chosen as an indication of the precision of the P(h) curve extraction. The use of the LS approach under MATLAB® was established according to a simple methodology for extracting the parameters of the P(h) curves of the Fe-Zn coating. Algorithm 1 regroups a description of the main steps established in the LS extraction of the parameters allowing the experimental reconstruction of the P(h) curves. Note that the selectivity criteria of the extracted P(h) parameters are established by comparing the LS reconstruction with the experimental evolution of the P(h) curves.
The obtained results will be examined hereafter against those of the BSA approach to compare the dispersion and the precision of the two algorithms. In fact, the error threshold in the search for the optimal solution between the experiment and the LS reconstruction of the \( P(h) \) curves is at \( \varepsilon_0 = 0.2\% \), i.e., at the mean precision of the XP\(^{\circ} \) Indenter.

Therefore, we can calculate the extraction error of the parameters governing the two restrictions and thus evaluate the reliability and the precision of the LS approach in extracting the \( P(h) \) curves. The various extractions of \( P(h) \) resulting from the LS approach were evaluated by their reliability and precision compared to referencing quantities established from the experimental results.

### 2.3.2. BSA extraction of the \( P(h) \) curves

The consistent identification of nonlinear equation systems is based on the choice of the objective function to be solved by searching the global optimum and by considering the constraints and the boundary conditions (real or temporal) of the solved problem [21–25]. The objective function to be chosen announces the relation between the system parameters to be identified and the constraints governing its phenomenological evolution. In this regard, we aim to apply the BSA by taking advantage of its speed of convergence, its flexibility, and its robustness in solving various real-value problems [24]. Indeed, the BSA algorithms can calculate optimal solutions during the iterations, progress, and thus, can remedy the problem of local minima [25]. This paper has introduced BSA, a new evolutionary-computing-based global search algorithm. BSA’s algorithmic approach enables it to benefit from previous generation populations by using solutions it has found in the past for a given problem as it searches for solutions with better fitness values [23].

To better exploit evolutionary algorithms in the nanoindentation and \( P(h) \) curve extraction, we use the BSA algorithm developed by Civicioglu [23] to solve the problem of real-value optimization of dedicated models. The particularity of this algorithm lies in the fact of ensuring the storage and memorization of previous populations and managing a single individual for each parameter targeted in the optimization of \( P(h) \) evolutions. However, certain modifications of the original algorithms are established to be compatible with the mathematical model of \( P(h) \) curve identification. To guarantee a certain prediction of the extraction results of the parameters governing the evolution of the \( P(h) \) curves, the chosen objective function (equation (3)) is defined by the following expression:

\[
\text{Fct}_{\text{obj}} = P_{c(h)} - P_{c(h)_{\text{op}}} \leq \varepsilon_0
\]

With \( P_{c(h)} \) is the experimental nanoindentation curve and \( P_{c(h)_{\text{op}}} \) is determined from the BSA extraction approach. Here, the notation \( P_{c(h)} \) is used for the loading phase and \( P_{c(h)} \) for the unloading phase. The synoptic figure 3 summarizes the BSA approach adapted to the extraction and parameterization of the \( P(h) \) curves resulting from the Fe-Zn thin film nanoindented. At the output, an evaluation of the result \((h_{\text{op}}, P_{\text{op}})\) against the input vector \((h, P)\) will be achieved and validated when the error is less than 0.2% to trace the \( P_{\text{op}}(h) \) and \( P(h) \) evolutions.

### 3. Results and discussion

#### 3.1. Experimental results: nanoindentation test

##### 3.1.1. Nanoindentation test of Fe-Zn coating

The SEM observation (figure 4) shows the residual imprint caused by the contact of the indenter. During the nanoindentation test, compressive stress would occur under the indenter, and this could cause piling-up around the residual indentation. In addition, no crack initiation was significantly noticed around the residual indentation. No crack was observed for indentation depths up to 1000 nm performed on the Fe-Zn coatings.

The obtained curves by nanoindentation show two distinct phases: the elastoplastic loading fit by a power law function and the unloading fit by a polynomial function [18, 19]. Moreover, the \( P(h) \) evolutions presented in figure 5 are characterized by a plastic creep during dwell time at the maximum penetration depth 1000 nm of low amplitude followed by an elastic unloading.

The hardness and Young’s modulus evolution analysis (figure 6 and 7) showed that these mechanical properties converge towards their average values from about 400 nm. Indeed, the first instabilities in the penetration depth variation (<50 nm) are caused by the contact vibrations and by the indented surface roughness since no polishing was performed before the test. The hardness curves show a plateau at indentation depths greater than 400 nm with a mean value ranging from 0.5 to 0.92 GPa. Regarding Young’s modulus, we notice that the average varies from 60 to 100 GPa. Thus, the numerical referential combinations are established at two levels of 60–100 GPa for the Young modulus \( E \), 150–300 MPa for the Yield Strength \( (\sigma_Y \approx 3H) \) and 0.10–0.18 for the hardening exponent \( n \).

The CSM mode of the indentation tests allowed us to determine the nanohardness and the modulus of elasticity evolutions as a function of the penetration depth. In this regard, the coupling between the
CSM-indentation, BSA extraction, FE simulation and the dimensionless functions present an ambitious alternative to guarantee the uniqueness of the reverse identification of the coating behavior.

### 3.1.2. Experimental parameters variability of the \( P(h) \) curves

Table 3 illustrates the parameters identified for all the indentations for the three curves extracted respectively from tests \( P(h) \), \( E(h) \) and \( H(h) \). The quantities of variability and dispersions allowed to establish the reference combinations for 16 trials, where the factors are the variables governing the experimental evolution of \( P(h) \) curves. This constitutes a first step to validate the technique of extracting the parameters of the models in loading/unloading phases from the reference data. This approach is developed to build a new approach of reverse identification based on the extraction of \( P(h) \) curves using meta-heuristic BSA algorithms. This can be seen in Figure 3.

![Synoptic diagram of the adapted BSA metaheuristic approach.](image)
Figure 4. Nanoindentation imprint observed by SEM on an indented Fe-Zn coating.

Figure 5. Experimental evolution of $P\text{[mN]}$ load as a function of penetration $h\text{[nm]}$.

Figure 6. Hardness profile $H(h)$ as a function of penetration $h\text{[nm]}$. 
present an ambitious alternative to the classical LS approaches often used in the literature for the extraction and parameterization of \( P(h) \) curves in nanoindentation tests.

In the next paragraph, we focus on the re-construction of the \( P(h) \) curves established by a simulation with an axisymmetric model under the finite element calculation Abaqus® code. The different identifications of the Fe-Zn film behavior by the two approaches LS and BSA are established and discussed. The concept allows the validation of the relevance and the exploitation of meta-heuristic BSA approaches in the identification of the constitutive laws of thin films. Indeed, the standard deviation in the ten selected experimental \( P(h) \) curves is the base considered to define the factors levels in the reverse approach established in this study. Consequently, it is obtained an increase the results accuracy and reduce the dispersion in the physical quantities identification governing the elastoplastic behavior of the Fe-Zn coatings.

3.2. Inverse identification by LS and BSA approaches

In general, no previous study using the nanoindentation test has examined the accuracy and reliability of the extracted quantities governing the response to \( P(h) \) evolution. Indeed, in many cases, the reverse identification approaches are validated by comparing the obtained results with the usual quantities by direct approaches, such as monotonic traction. Moreover, the \( P(h) \) curve extraction is carried out by direct identification using the classical LS concepts, without having a realistic evaluation of the error required in this frequent choice by researchers [7–9, 18, 19, 27–29]. At this stage, we proceed by extracting the required parameters allowing the \( P(h) \) curve reconstruction with two distinct restrictions: elastoplastic loading (\( C \) [mN nm \(^{-2}\)], \( h_{\text{max}} \) [nm]) and elastic unloading (\( m \) [–], \( \alpha \) [mN/nm \(^{-1}\)], \( h_{\text{r}} \) [nm] and \( S \) [mN nm \(^{-1}\)]). Two distinct approaches have been exploited: classical identification with simple non-linear LS algorithms and evolutionary meta-heuristic BSA approach.

3.2.1. \( P(h) \) curve extraction by LS and BSA approaches

Initially, we aim to validate the contribution of the BSA approach compared to that of LS algorithm whose criteria of validation are precision and standard deviation. For this, the variability borders of the different results

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**Table 3. Different levels of the \( P(h) \) curve parameters extracted from the experiment test.**

| Parameters | Variability limits | Standard deviation\(^a\) | Low Level | Top Level |
|------------|-------------------|--------------------------|-----------|-----------|
| Load \( C \) [GPa] | 18–22 | 3.72 | 18 | 22 |
| \( h_{\text{m}} \) [nm] | 1073.4 | 11.37 | 1073.4 | 1073.4 |
| Unload \( \alpha \) [mN/nm \(^{-1}\)] | 0.1–0.18 | 0.004 | 0.05 | 0.30 |
| \( m \) [–] | 1.1–1.15 | 0.06 | 1.05 | 1.30 |
| \( h_{\text{r}} \) [nm] | 980–1020 | 17.35 | 980 | 1020 |
| Hardness \( H \) [GPa] | 0.4–0.96 | 0.07 | 0.4 | 1.00 |
| Rigidity \( E \) [GPa] | 60–100 | 5.82 | 60 | 100 |

\(^a\) The deviation standard from a series of 10 indentations applied on the Fe-Zn coating surface.

Figure 7. Young’s modulus \( E(h) \) profile as a function of penetration \( h \) [nm].
Table 4. Extracted P(h) curve parameters: Least-Square (LS) Approach ($h_m = 1073.4$ nm).

| Test | C [GPa] | hr [nm] | $\alpha$ [mN/nm$^2$] | m [-] | C [GPa] Value | % Error | hr [nm] Value | % Error | $\alpha$ [mN/nm$^2$] Value | % Error | m [-] Value | % Error | S (mN nm$^{-1}$) Value | % Error |
|------|---------|---------|-------------------------|-------|---------------|----------|---------------|----------|----------------------------|----------|---------------|----------|--------------------------|----------|
| 1    | 20      | 980     | 0.1                     | 1.01  | 20.00         | 0.02     | 980.2         | 0.02     | 0.1025                    | 2.50     | 1.0054        | 0.46     | 0.106306                 | 0.65     |
| 2    | 20      | 980     | 0.1                     | 1.3   | 20.00         | 0.00     | 981.6         | 0.16     | 0.1056                    | 5.60     | 1.2755        | 1.88     | 0.427909                 | 7.16     |
| 3    | 20      | 980     | 0.3                     | 1.01  | 20.00         | 0.00     | 980.9         | 0.09     | 0.2868                    | 4.40     | 1.0122        | 0.21     | 0.305692                 | 3.32     |
| 4    | 20      | 980     | 0.3                     | 1.3   | 20.00         | 0.00     | 1030.2        | 0.02     | 0.1027                    | 2.74     | 1.2916        | 0.65     | 0.938800                 | 8.90     |
| 5    | 20      | 1030    | 0.1                     | 1.01  | 20.00         | 0.00     | 1030.7        | 0.07     | 0.1047                    | 4.70     | 1.2787        | 1.64     | 0.431608                 | 6.13     |
| 6    | 20      | 1030    | 0.1                     | 1.3   | 20.00         | 0.00     | 1030.0        | 0.00     | 0.3048                    | 1.59     | 1.0060        | 0.40     | 0.314816                 | 0.59     |
| 7    | 20      | 1030    | 0.3                     | 1.01  | 20.00         | 0.12     | 1030.8        | 0.07     | 0.2819                    | 6.03     | 0.9970        | 1.29     | 0.105429                 | 1.98     |
| 8    | 20      | 1030    | 0.3                     | 1.3   | 20.02         | 0.12     | 980.5         | 0.05     | 0.1077                    | 7.75     | 1.2815        | 1.42     | 0.911662                 | 13.58    |
| 9    | 25      | 980     | 0.1                     | 1.01  | 25.02         | 0.07     | 980.7         | 0.07     | 0.1055                    | 5.50     | 1.2853        | 1.13     | 0.465655                 | 2.37     |
| 10   | 25      | 980     | 0.1                     | 1.3   | 25.00         | 0.00     | 980.0         | 0.00     | 0.3053                    | 1.76     | 1.0057        | 0.42     | 0.315092                 | 0.61     |
| 11   | 25      | 980     | 0.3                     | 1.01  | 25.00         | 0.00     | 980.0         | 0.08     | 0.2869                    | 4.37     | 1.2778        | 1.71     | 0.942794                 | 13.51    |
| 12   | 25      | 980     | 0.3                     | 1.3   | 25.00         | 0.00     | 1030.6        | 0.06     | 0.10966                    | 3.40     | 1.0135        | 0.35     | 0.106968                 | 0.82     |
| 13   | 25      | 1030    | 0.1                     | 1.01  | 25.00         | 0.00     | 1030.7        | 0.07     | 0.1057                    | 5.70     | 1.2815        | 1.42     | 0.467710                 | 4.21     |
| 14   | 25      | 1030    | 0.1                     | 1.3   | 25.05         | 0.21     | 1030.0        | 0.00     | 0.3055                    | 1.83     | 1.0057        | 0.43     | 0.315414                 | 0.61     |
| 15   | 25      | 1030    | 0.3                     | 1.01  | 25.00         | 0.00     | 1030.2        | 0.02     | 0.2859                    | 4.70     | 1.2785        | 1.65     | 0.973102                 | 15.24    |
| 16   | 25      | 1030    | 0.3                     | 1.3   | 25.00         | 0.00     | 1030.2        | 0.02     | 0.2859                    | 4.70     | 1.2785        | 1.65     | 0.973102                 | 15.24    |

Average Identification error for LS approach [%] 0.03 0.06 4.21 0.97 4.90
LS Approaches: Standard deviation of the absolute error 0.06 0.04 1.77 0.60 4.95
in $H(h)$ and $E(h)$ were used to decide on different reference combinations of the measurable $P(h)$ curve quantities. Tables 4 and 5 summarize the results of the $P(h)$ curve extraction using the two approaches LS and BSA. Indeed, the four essential quantities, allowing the reconstruction of the $P(h)$ curves, namely $C[GPa]$, $h_i[mm]$, $\alpha_m[mN/mm]$ and $m[-]$, were extracted using BSA and LS. The different reconstructions were also used to extract the maximum depths $h_m[mm]$ and maximum loads $P_m[mN]$ by the mathematical resolution of the equation $P_i(h) = P_m$. At the output, the mean errors were evaluated by comparison between the $P_{ext}(h)$ curve deduced from the LS or BSA extraction with that of the experimental $P(h)$ evolution. We notice that the average standard deviations for the case of the BSA approach are of the order of 0.02 for $C$, 0.03 for $h_i$, 1.31 for $\alpha$, 0.31 for $m$ and 0.93 for $S$ and this, against 0.03, 0.06, 4.21, 0.97 and 4.90 respectively for the case of the LS approach. This permitted to undertake an appreciable gain in dispersion of around 30% for $C$, 50% for $h_i$, 69% for $\alpha$, 68% for $m$ and 81% for $S$. Concerning the constant curvature of the loading curve $C$, the two dedicated LS and BSA approaches lead to a mean error of zero or insignificant (less than 0.2%). This result demonstrates that the LS approach used in the parameterization of the $P_i(h)$ evolutions is reliable and efficient in contrary to the case of $P_m(h)$ unloading curve evolution.

3.2.2. Validation of the BSA approach

The two restrictions of the loading phase $P_i(h)$ (figure 8(a)) and of the unloading phase $P_u(h)$ (figure 8(b)) deduced from the eight FE simulations showed a typical distribution in the results variability. Moreover, the isovalue associated with the same graphs (figure 8) showed the Von-Mises stress ($\sigma_{VM}$) and the displacement ($U_2$). This permitted to evaluate the stress required by the indenter to progress in the material, and the residual imprint to measure the resulting contact area ($a_{mn}$). The calculation of this contact area is essential to estimate the mechanical properties of the indented surfaces by Dao’s reverse identification approach. We notice here that the resulting pile-up is less than 0.1 $\mu$m (figure 8(b)) which reflects that the influence of DP600 substrate and roughness could be negligible in our case. This agrees with the work discussed by Panich et al [16] for a yield stress ratio equal to 0.5 knowing that the hardness ratio is close to unity. Under these conditions, the authors confirmed that the substrate does not affect the $P(h)$ curve evolution, and which is also confirmed for the case of the Fe-Zn coating/DP600 substrate pair.

The reverse identification results based on the dimensionless functions of Dao et al [7-9] were used. These authors made nanoindentation tests and FE simulations for materials with mechanical characteristics ($E$ from 10 to 210 GPa, $\sigma_{VM}$ from 30 to 3000 MPa) to predict the mechanical behavior from the $P(h)$ evolution. Their approaches are based on dimensionless functions relating the $P(h)$ curve magnitudes to the usual mechanical characteristics of Young’s modulus, yield stress, and work hardening. Despite the convergence towards a response of an acceptable mean value, the dispersion of the prediction results limits the reliability of these reverse predictions. In fact, the reliability of the two approaches (BSA and LS) in reverse identification of the elastoplastic behavior was evaluated for supposed known laws (table 6). In this regard, the results of loading and unloading parameter identification of the two models were used to extract the mechanical characteristics of the Fe-Zn coatings. The results were compared to the reference combinations for the two cases of classical LS approach and meta-heuristic BSA extraction. The identified parameters are those which make it possible to reconstruct the $P(h)$ curve obtained by FE simulation.

For the two reverse identification approaches LS and BSA, the expected results for this study are shown in table 6. At this stage, the BSA approach shows an error of 0.89% (Standard deviation of 0.35) against an error of 3.15% (standard deviation of 1.74) for the LS approach in the case of yield stress reverse identification. This allowed the reverse identification of the Fe-Zn coating yield strength including the reduction of precision errors to 72% and dispersion errors to 82%. For the hardening exponent ($n$), the gains are 60% for precision and 52% for dispersion. Consequently, the validation of the BSA approach is established by considering its impact on the error reduction (in precision and dispersion) in the constitutive law identification $\sigma(\epsilon)$ of the Fe-Zn coating. In the following, we use the two approaches at a third level to identify the constitutive law parameters of Fe-Zn coatings. Before establishing a definitive conclusion on the utility and capacity of the BSA approach, the precision and dispersion quantities in reverse identification are a decision support criterion retained for the approach validation.

3.3. Case study: BSA reverse identification of the Fe-Zn coating behavior

The interest of meta-heuristic BSA algorithms in the reverse identification of the mechanical behavior of thin films was validated in the previous paragraphs. Certainly, considering the known parameters ($E$, $\sigma_{VM}$, $n$) and the FE simulations gave more precision and less dispersion in the optimal BSA solution compared to that of LS approach.
Table 5. Extracted P(h) curve parameters: BSA Approach (hm = 1073.4 nm).

| Test | C [GPa] | hr [nm] | \( \alpha \) [mN nm\(^{-2}\)] | m [-] | C [GPa] | hr [nm] | \( \alpha \) [mN nm\(^{-2}\)] | m [-] | Value | % Error | Value | % Error | Value | % Error |
|------|---------|--------|-------------------------------|-------|---------|--------|-------------------------------|-------|-------|---------|-------|-------|-------|---------|
| 1    | 20      | 980    | 0.1                           | 1.01  | 20.07   | 0.36  | 980.2                         | 0.02  | 0.1007 | 0.67   | 1.0089 | 0.11  | 0.1069 | 0.07   |
| 2    | 20      | 980    | 0.1                           | 1.30  | 20.00   | 0.00  | 980.6                         | 0.06  | 0.0977 | 2.30   | 1.3100 | 0.77  | 0.4599 | 2.35   |
| 3    | 20      | 980    | 0.3                           | 1.01  | 20.00   | 0.00  | 980.0                         | 0.00  | 0.3010 | 0.32   | 1.0093 | 0.07  | 0.3160 | 0.04   |
| 4    | 20      | 980    | 0.3                           | 1.30  | 20.00   | 0.00  | 980.3                         | 0.03  | 0.3022 | 0.73   | 1.3040 | 0.30  | 1.0496 | 1.98   |
| 5    | 20      | 1030   | 0.1                           | 1.01  | 20.00   | 0.00  | 1030.2                        | 0.02  | 0.1007 | 0.74   | 1.0088 | 0.12  | 0.1070 | 0.07   |
| 6    | 20      | 1030   | 0.1                           | 1.30  | 20.00   | 0.00  | 1030.7                        | 0.07  | 0.0967 | 3.30   | 1.3130 | 1.00  | 0.4732 | 2.85   |
| 7    | 20      | 1030   | 0.3                           | 1.01  | 20.00   | 0.00  | 1030.0                        | 0.00  | 0.3011 | 0.37   | 1.0092 | 0.08  | 0.3163 | 0.05   |
| 8    | 20      | 1030   | 0.3                           | 1.30  | 20.04   | 0.20  | 1030.2                        | 0.02  | 0.3049 | 1.63   | 1.2990 | 0.08  | 1.0650 | 1.00   |
| 9    | 25      | 980    | 0.1                           | 1.01  | 25.00   | 0.00  | 980.2                         | 0.03  | 0.1008 | 0.79   | 1.0088 | 0.12  | 0.1074 | 0.10   |
| 10   | 25      | 980    | 0.1                           | 1.30  | 25.00   | 0.00  | 980.7                         | 0.07  | 0.0976 | 2.40   | 1.3096 | 0.74  | 0.4865 | 2.20   |
| 11   | 25      | 980    | 0.3                           | 1.01  | 25.00   | 0.00  | 980.0                         | 0.00  | 0.3013 | 0.45   | 1.0091 | 0.09  | 0.3168 | 0.05   |
| 12   | 25      | 980    | 0.3                           | 1.30  | 25.00   | 0.00  | 980.3                         | 0.03  | 0.3025 | 0.83   | 1.3020 | 0.16  | 1.1019 | 1.42   |
| 13   | 25      | 1030   | 0.1                           | 1.01  | 25.00   | 0.00  | 1030.3                        | 0.03  | 0.1008 | 0.83   | 1.0088 | 0.12  | 0.1077 | 0.12   |
| 14   | 25      | 1030   | 0.1                           | 1.30  | 24.81   | 0.76  | 1030.6                        | 0.06  | 0.0977 | 2.30   | 1.3093 | 0.72  | 0.4974 | 2.27   |
| 15   | 25      | 1030   | 0.3                           | 1.01  | 25.00   | 0.00  | 1030.0                        | 0.00  | 0.3013 | 0.45   | 1.0091 | 0.09  | 0.3175 | 0.06   |
| 16   | 25      | 1030   | 0.3                           | 1.30  | 25.00   | 0.00  | 1030.2                        | 0.02  | 0.3084 | 2.80   | 1.2946 | 0.42  | 1.1159 | 0.28   |

Identification error in the BSA Approach \( E_{id} \) [%]

BSA Approaches: Standard deviation of absolute error

Contribution of the BSA approach: \( E_{id} \) error [%]

Contribution of the BSA approach: Standard Deviation [%]
In this section, we adopt the same approach and apply it in the ten nanoindentations whose mechanical characteristics \((E, \sigma_Y, n)\) and the parameters of the \(P(h)\) curves \((C, P_m, h_m, h_p, \alpha, m, S)\) are initially unknown (figure 9). The extraction results of the quantities governing the \(P(h)\) evolution allow the reconstruction of the experimental results as illustrated in figure 9(a). This allows us to determine the reverse identification whose associated results are presented in tables 7 and 8 when the Dao’s reverse approach \([7, 8]\) is used. Consequently, dimensionless equations are used to determine, on reception, the mechanical characteristics of the Fe-Zn coating deposited on the DP600 steel by the Dao’s reverse approach. Subsequently, to well analyze the identification results, standard deviations and SEM errors were calculated to evaluate the dispersion and reliability of the reverse predictions of the BSA approach. The hardening quantities to be calculated are the yield stress and the hardening exponent.

To identify the Fe-Zn coating mechanical characteristics, the quantitative evolutions of dispersion and precision are detailed in tables 7 and 8. The average values of the rheological quantities identified (figure 9(b)) are: \(E = 77\ \text{GPa}, \ \sigma_Y = 190\ \text{MPa}\) and \(n = 0.118\), which are associated respectively with SEM errors of 5.92 GPa, 7.43 MPa and 0.015. Therefore, the results were developed using the BSA approach whose notable benefits are 84\% for the hardening coefficient \((n)\) and 91\% for the yield strength \((\sigma_Y)\) compared to those of the LS approach.

Figure 8(b) presents the two rational traction curves identified by the inverse approach of the evolutionary algorithms LS and BSA. These curves are obtained by extraction from the \(P(h)\) curves. The BSA algorithms allow an identification of the mechanical characteristics of the Fe-Zn coating with more precision (+10\%) and less dispersion (−85\%) and make it possible to provide manufacturers with a reliable and deterministic approach in the reverse identification of the Fe-Zn coating behavior from the extraction of the \(P(h)\) curves.

The FE model established for the indentation simulation of Fe-Zn layers provided more information on the stress distribution evolution (figure 8(a)), and on displacements (figure 8(b)) when the indenter displacement progresses such as the presence of pile-up \((+8.74 \times 10^{-5} \text{ mm})\) around the residual imprint.
### Table 6. Fe-Zn property identification according to the Dao’s approaches LS & BSA ($h_m = 1073.4$ nm).

| N° | $E$ [GPa] | $\sigma_Y$ [MPa] | $n$ [-] | $C$ [GPa] | hr [hm] | $\alpha$ [mN/nm$^{-1}$] | $m$ [-] | $S$ [mN/nm] | $C$ [GPa] | hr/hm [-] | $\alpha$ [mN/nm$^{-1}$] | $m$ [-] | $S$ [mN nm$^{-1}$] |
|----|---------|---------------|--------|---------|-------|---------------------|------|---------|---------|-------|---------------------|------|---------|
| 1  | 60      | 150           | 0.1    | 17.282  | 0.948 | 0.1329               | 1.2256 | 0.397222 | 17.283  | 0.95  | 0.2093               | 1.1307 | 0.394412 |
| 2  | 60      | 150           | 0.18   | 20.322  | 0.937 | 0.1431               | 1.1887 | 0.371842 | 20.322  | 0.939 | 0.1996               | 1.1236 | 0.372885 |
| 3  | 60      | 300           | 0.1    | 28.582  | 0.914 | 0.1885               | 1.1248 | 0.369942 | 28.601  | 0.913 | 0.1867               | 1.1272 | 0.371225 |
| 4  | 60      | 300           | 0.18   | 31.967  | 0.896 | 0.1148               | 1.2027 | 0.354202 | 31.967  | 0.899 | 0.1743               | 1.1265 | 0.352321 |
| 5  | 100     | 150           | 0.1    | 19.273  | 0.966 | 0.2488               | 1.2255 | 0.674661 | 19.272  | 0.967 | 0.3133               | 1.1722 | 0.671548 |
| 6  | 100     | 150           | 0.18   | 23.818  | 0.958 | 0.2975               | 1.1615 | 0.632445 | 23.818  | 0.958 | 0.3481               | 1.1302 | 0.639521 |
| 7  | 100     | 300           | 0.1    | 32.908  | 0.94  | 0.2118               | 1.2237 | 0.647183 | 32.91   | 0.942 | 0.2994               | 1.1554 | 0.649937 |
| 8  | 100     | 300           | 0.18   | 38.38   | 0.927 | 0.2064               | 1.2115 | 0.618240 | 38.38   | 0.926 | 0.3155               | 1.1277 | 0.612433 |

### Reference’s combinations Laws (Startup: $\sigma(\epsilon)_Y$ known) LS Reverse Identification of $P(h)$ curves

| $E$ [GPa] | $\sigma_Y$ [MPa] | $n$ [-] | Value | % | Error | Value | % | Error | Value | % | Error | Value | % | Error |
|-----------|-----------------|--------|-------|---|-------|-------|---|-------|-------|---|-------|-------|---|-------|
| 1         | 60              | 150    | 0.1   | 59.48 | —     | 153.76 | 2.5 | 0.089 | 10.5  | 59.48 | —     | 149.11 | 0.6 | 0.101 |
| 2         | 60              | 150    | 0.18  | 59.48 | —     | 148.01 | 1.33| 0.177 | 1.89  | 59.48 | —     | 150.52 | 0.35| 0.171 |
| 3         | 60              | 300    | 0.1   | 59.48 | —     | 293.4  | 2.2 | 0.111 | 10.5  | 59.48 | —     | 297.97 | 0.68| 0.104 |
| 4         | 60              | 300    | 0.18  | 59.48 | —     | 310.42 | 3.47| 0.162 | 9.89  | 59.48 | —     | 302.34 | 0.78| 0.174 |
| 5         | 100             | 150    | 0.1   | 103.07| —     | 151.76 | 1.18| 0.095 | 4.90  | 103.07| —     | 148.10 | 1.26| 0.102 |
| 6         | 100             | 150    | 0.18  | 103.07| —     | 139.21 | 7.19| 0.193 | 7.00  | 103.07| —     | 152.00 | 1.33| 0.17  |
| 7         | 100             | 300    | 0.1   | 103.07| —     | 290.91 | 3.03| 0.106 | 5.80  | 103.07| —     | 296.43 | 1.19| 0.099 |
| 8         | 100             | 300    | 0.18  | 103.07| —     | 313.16 | 4.39| 0.157 | 12.94 | 103.07| —     | 297.16 | 0.95| 0.175 |

- Identification error in BSA Approach $E_{id}$ [%]
- BSA Approaches: Standard deviation of absolute error
- Contribution of the BSA approach: $E_{id}$ error [%]
- Contribution of the BSA approach: Standard Deviation (%)
4. Discussion

The main idea discussed in this study is to evaluate the impact of the extraction errors of $P(h)$ curve evolution in the reverse identification analysis of the Fe-Zn coating constitutive laws. The decision criteria to validate such an approach are the dispersion, precision, and SEM errors of the responses. The aim of this approach is to provide quality shaping without decohesion of the Fe-Zn coating which degrades its capability for subsequent processing such as painting. Besides, these precautions allow improving the in-service behavior of the Fe-Zn coatings for better functional integrity of the reinforcement and body components of the automotive structure.

From this perspective, the classical Least-Squares approaches can give satisfaction when identifying power-type models for the elastoplastic loading in nanoindentation $P_s(h)$. However, the extraction of the different quantities governing the $P_s(h)$ restrictions did not give a robust solution to the Fe-Zn behavior prediction, although the associated errors are less than 5%. Indeed, the consequences of these errors in extracting these quantities present a difficulty in the secondary phase of reverse parameter identification of the law constituting the required thin layers whose solution is not necessarily unique. In contrast, the BSA algorithms have given efficient solutions by the convergence towards an optimal combination of parameters for the $P_s(h)$ unloading model. This result is attributed to the possibility of considering the constraints imposed on the borders of $(\alpha)$ and $(m)$ in their respective theoretical intervals of $[0, 1]$ and $[1, 2]$. Consequently, the $E(h)$ and $H(h)$ curves were coupled to BSA algorithms and dimensionless functions used by Dao et al [7–9] in the reverse identification of the Fe-Zn characteristics. This leads to the uniqueness of the solution of the $P(h)$ extraction model and therefore the uniqueness of the law inverse identification. Note here that the intervals of unique solutions of each quantity are established by a stochastic iteration of the BSA algorithm towards the objective solution. In fact, the randomness of the selective solutions starts from an initial value chosen according to the Fe-Zn coating experimental responses. Thus, the $E(h)$ and $H(h)$ curves were used to define the BSA boundary conditions.
Table 7. Elastoplastic properties of Fe-Zn coatings: reverse identification in 10 instrumented nanoindentation tests using the LS approach.

| Test     | C [GPa] | Pm [mN] | hr [mN nm⁻¹] | S [mN nm⁻¹] | α [mN nm⁻²] | m [-] | hm [nm] | E*_exp [GPa] | E [GPa] | n [-] | σ_y [MPa] |
|----------|---------|---------|--------------|-------------|-------------|--------|---------|-------------|---------|-------|-----------|
| Test-01  | 25.21   | 28.09   | 0.937        | 562.699     | 0.1441      | 1.2484 | 1055.6  | 82.40       | 83.470  | 0.117 | 210.40    |
| Test-02  | 25.07   | 27.09   | 0.945        | 636.134     | 0.2311      | 1.2047 | 1039.4  | 91.20       | 93.190  | 0.075 | 228.59    |
| Test-03  | 21.02   | 23.96   | 0.941        | 497.734     | 0.1537      | 1.2334 | 1067.6  | 73.70       | 74.030  | 0.166 | 146.39    |
| Test-04  | 21.35   | 25.30   | 0.943        | 540.032     | 0.1760      | 1.2232 | 1088.5  | 73.30       | 73.600  | 0.057 | 208.20    |
| Test-05  | 23.84   | 28.47   | 0.944        | 467.033     | 0.4492      | 1.0076 | 1092.8  | 69.10       | 69.110  | 0.176 | 175.15    |
| Test-06  | 23.13   | 26.93   | 0.947        | 677.907     | 0.098       | 1.397  | 1079.0  | 92.40       | 94.530  | 0.067 | 211.31    |
| Test-07  | 23.14   | 26.89   | 0.946        | 630.022     | 0.0955      | 1.3846 | 1078.0  | 85.60       | 86.990  | 0.054 | 223.31    |
| Test-08  | 22.96   | 26.42   | 0.933        | 422.879     | 0.2809      | 1.0782 | 1072.7  | 64.10       | 63.790  | 0.181 | 169.61    |
| Test-09  | 21.32   | 25.67   | 0.935        | 457.143     | 0.2169      | 1.1435 | 1097.3  | 61.50       | 61.050  | 0.032 | 232.34    |
| Test-10  | 22.44   | 25.43   | 0.927        | 483.430     | 0.1660      | 1.2030 | 1064.5  | 70.50       | 70.600  | 0.119 | 189.64    |
| Means    | 22.95   | 26.43   | 0.940        | 537.501     | 0.2011      | 1.2123 | 1073.56 | 76.38       | 77.04   | 0.104 | 199.49    |
| standard deviation | 1.48   | 1.37   | 0.01        | 86.74       | 0.1045      | 0.1203 | 17.69   | 10.92       | 11.85   | 0.055 | 28.34    |
Table 8. Elastoplastic properties of Fe-Zn coatings: reverse identification in 10 instrumented nanoindentation tests using the BSA approach.

| Test    | C [GPa] | Pm [mN] | hr hm⁻¹ [-] | S [mN/nm] | σ [mN/nm⁻⁰⁸] | m [-] | hm [nm] | E', exp [GPa] | E [GPa] | n [-] | σy [MPa] |
|---------|---------|---------|-------------|-----------|-------------|-------|---------|-------------|---------|-------|----------|
| Test-01 | 25.19   | 27.81   | 0.942       | 559.816   | 0.1748      | 1.2327 | 1050.6  | 82.40       | 83.470  | 0.118 | 209.78   |
| Test-02 | 25.12   | 27.13   | 0.945       | 607.618   | 0.1498      | 1.2848 | 1039.4  | 91.20       | 93.190  | 0.148 | 182.82   |
| Test-03 | 21.04   | 23.87   | 0.947       | 521.860   | 0.1625      | 1.2566 | 1065.3  | 73.70       | 74.030  | 0.085 | 189.07   |
| Test-04 | 21.35   | 25.31   | 0.943       | 530.201   | 0.1225      | 1.2930 | 1088.7  | 73.30       | 73.600  | 0.083 | 193.92   |
| Test-05 | 23.84   | 28.45   | 0.944       | 470.286   | 0.4425      | 1.0119 | 1092.5  | 69.10       | 69.110  | 0.163 | 182.21   |
| Test-06 | 23.14   | 26.90   | 0.946       | 656.244   | 0.0876      | 1.4113 | 1078.2  | 92.40       | 94.530  | 0.113 | 183.53   |
| Test-07 | 24.34   | 29.25   | 0.949       | 600.123   | 0.2803      | 1.1534 | 1096.2  | 85.60       | 86.990  | 0.148 | 179.55   |
| Test-08 | 22.96   | 26.13   | 0.939       | 445.807   | 0.2427      | 1.1186 | 1066.9  | 64.10       | 63.790  | 0.081 | 224.44   |
| Test-09 | 21.35   | 25.37   | 0.944       | 492.475   | 0.2040      | 1.1754 | 1089.9  | 61.50       | 61.050  | 0.106 | 184.67   |
| Test-10 | 22.45   | 25.54   | 0.922       | 478.919   | 0.1708      | 1.1935 | 1066.7  | 70.50       | 70.600  | 0.137 | 179.44   |
| Means   | 23.07   | 26.57   | 0.942       | 556.335   | 0.2038      | 1.2111 | 1073.44 | 76.38       | 77.040  | 0.118 | 190.94   |
| Standard deviation | 1.53 | 1.64 | 0.01 | 68.40 | 0.1006 | 0.1088 | 18.99 | 10.92 | 11.85 | 0.03 | 14.86 |
| Error MC/BSA % | 0.54 | 0.55 | 0.24 | 0.22 | 1.31 | 0.10 | 0.01 | — | — | 11.67 | 4.48 |
| Gain/ Standard deviations % | 3.60 | 16.69 | 11.78 | 26.81 | 3.92 | 10.59 | 6.84 | — | — | 84.05 | 90.72 |
adopted in the analysis performed under MATLAB. In this regard, the reverse approach established by Dao et al [7–9] is applied for the ten experimental indentations to identify the Fick model parameters (E [GPa], \( \sigma_Y \) [MPa] and \( n \)). On the other hand, figure 10 summarizes all the results obtained for the reverse identification established according to the two \( P(h) \) curves extraction methods and parameterization.

The BSA approach gave less dispersion about the mean value compared to the results obtained by the LS approach for the hardening exponent (figure 10(a)) and the yield stress \( \sigma_Y \) (figure 10(b)). Figure 10(c) represents the variability of the unloading curves identified by the BSA algorithm by comparison with the experimental unloading curves. The various results of the Fe-Zn characteristic identification (figure 11) reveal less error in dispersion and precision. In fact, these results are illustrated in figure 11(a) for the yield strength and in figure 11(b) for the hardening exponent. Hence, the BSA approach led to an average value of 190 MPa for yield stress with a standard deviation of 6.07 MPa, against an average of 199 MPa and a standard deviation of 14.87 MPa for the case of the LS approach. The hardening exponent has a mean value of \( n = 0.104 \) for LS, and \( n = 0.118 \) for the BSA approach. In this regard, the significant gain in terms of mean hardening quantities \( (\sigma_Y \text{ and } n) \) and dispersion precision lies in the power of BSA algorithms to find objective combinations of unloading phase quantities (\( \alpha \) and \( m \)). This helps us to calibrate the certainty in the calculation of the true elastic unloading stiffness (\( S \)) required in the application of the reverse approaches of Dao et al [7–9].

The BSA algorithm may give more accuracy in an approach identifying the Fe-Zn layers behavior according to the inverse algorithms established by X. Long et al [10] as an example. We note that a field of scientific investigation is accessible to better explain the behavior of Fe-Zn coatings and to predict their decohesion mechanisms during forming. As a result, a reliable prediction of their tribological integrity and their service life becomes more accessible and relevant. On the other hand, the continuation of this research will discuss the industrial difficulties to evaluate the service life of base substrates while considering the functional integrity of their associated coatings.
5. Conclusion

In this research, the precision and the dispersion of the inverse identification to extract the constitutive laws from nanoindentation performed on galvanizing Fe-Zn layers were investigated. Two criteria of dispersion (standard deviation) and precision (SEM error) were used to study the reliability of the adopted reverse approach reliability. In fact, this new method is more precise than the other traditional techniques (e.g., regression by the LS method). Therefore, the $P(h)$ curves are extracted with more precision and less dispersion in the inverse identification based on the study of Dao et al [16, 17]. The obtained results are summarized in the following descriptions:

- The BSA approach gives more precision (+66%) and less dispersion (−61%) than the LS method in extracting the quantities governing the evolution of the experimental $P(h)$ curves in the Fe-Zn instrumented nanoindentation test.
- The identification laws of the Fe-Zn coating behavior are very delicate for the accuracy in the unloading stiffness ($S$) calculation. This parameter ($S$) is a function of the accuracy and the dispersion of the two extracted parameters ($\alpha$) and ($m$).
- The use of meta-heuristic BSA approaches improved the precision of the Fe-Zn film constitutive identification laws, i.e., 84.05% for ($n$) and 90.72% for ($\sigma_Y$).

The contribution of this study resides in the reverse identification of the Fe-Zn coating constitutive laws by this first application of BSA algorithms in the $P(h)$ curve extraction. It remains necessary to better exploit the CSM mode in nanoindentation at higher penetration depths coupled with artificial intelligence techniques. Besides, the accuracy and dispersion of the reverse identification of the Fe-Zn coating behavior will be further investigated.
increased by the integration of decision support and artificial intelligence techniques. This will be the subject of a future work keeping the same basis of instrumented indentation $P(h)$, $E(h)$, and $H(h)$ to explore reliable, and deterministic alternatives compared to similar approaches used in (Dao et al [7–9], Iost et al [28], Lee et al [10]) studies.

#### 6. Highlights

- Application of the stochastic BSA approach to improve the precision and reduce the dispersion in the $P(h)$ curve extraction resulting from the Fe-Zn film nanoindentation experiment.
- Resolution of the uniqueness problem in inverse identification by coupling the CSM mode in nanoindentation, the stochastic BSA algorithms and the dimensionless functions for the Fe-Zn layer behavior.
- Establishment of a new methodology for extracting the physical quantities governing the evolution of the $P(h)$ curves in the nanoindentation test to better master the reverse approaches identifying the behavior of industrial coatings.

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#### Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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