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A Method to Extract the Elastoplastic Properties of the Constituent Layers of Multilayer Coatings

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Abstract. This paper presents an approach to characterize the elastoplastic properties of the distinct layers, constituting multilayer coating. The proposed procedure utilizes nanoindentation load-displacement (P-h) curves and a non linear least squares fitting analysis to extract the elastoplastic properties of each layer in the multilayer coating. The accuracy of the optimization results is achieved by choosing initial guess parameters closer to the target values using the modified Jönsson and Hogmark model. The methodology is validated on a CrN/CrAlN multilayer coating systems with varying layer thicknesses from 1 to 0.5 μm, from which the optimal elastoplastic properties: Young's modulus (E), yield stress (σ_Y), and strain hardening exponent (n) of each individual layer were determined. The results show good agreement between the simulated and the experimental (P-h) curves. Furthermore, the results revealed a reduction in the material parameters (E, H and σ_Y) of the constituent layers when the layer thickness decreases. These findings suggest that decreasing the coating layer thickness lead to an increase in the plastic deformation within the coatings, which reduces the stress concentration in this area and improves the adhesion properties of CrN/CrAlN multilayer coatings.

Keywords: Instrumented-indentation testing · Elastoplastic properties · finite element modeling

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1 Introduction

In the last decade, the concepts of multilayer coatings have been proposed as a strategy to improve properties such as hardness, resistance to crack propagation, and toughness of traditional monolayer coatings [1, 2]. In general, the better performance of multilayer coatings against wear damage, scratch and abrasion emanates from incorporation of alternating multilayer interface systems [3].

Due to their remarkable capabilities, multilayer coatings have become an important research objective in the last decade. Among these studies, a particular interest is given to the optimization of the structure and the type of multilayer coatings in order to achieve coatings with the desired mechanical properties. Based on the work of (Kataria et al. 2012) [4], the performance of multilayer coatings is substantially dependent on the elastoplastic properties of the monolayers that constitute them. Furthermore, the distribution of residual stress and deformation at the coating/substrate interface, which control the tribological and adhesion properties of the multilayer coatings, is mainly related to the elastoplastic properties of the monolayers of multilayer [4, 5]. Therefore, the determination of mechanical properties such as hardness, Young's modulus, yiled stress, strain hardening coefficient and fracture toughness of individual monolayer is necessary to achieve the design purposes [6].

Previous works [6, 7], have shown that the assessment of the mechanical properties of multilayer coatings must be performed through nanoindentation under high indentation loads. This approach arises from the fact that the indenter must penetrate through the multiple layers of the coating to separate the contribution of each individual layer from the others ones and also from the substrate. In this context, numerous analytical models have been developed to predict the hardness and young's modulus of monolayer coatings systems from experimental nanoindentation load-displacement curves and composite hardness and composite modulus profiles [7]. However, for the case of multilayer coatings, there has limited development of comparable models that enable the determination of (H) of each individual layers. Notable among these models are the modified Johnsson and hogmark model [6], the modified Puchi-Cabrera model and the modified Korsunsky model [7]. Among these three models, a special attention has been directed award the modified Johnsson and hogmark (JH) model. This model stands out for its minimal number of fitting parameters (just 2), and its high predictive performance compared to the others models [7].

Nevertheless, these analytical models are unable to provide information about the elastoplastic properties of the constituent layers of multilayer coatings. In this case, finite element simulation, especially when integrated with the experimental nanoindentation data, have been successfully used to characterize the true elastoplastic properties of coatings on substrate, such as (E), (σ_y) and (n) [8–12].

The aim of the present work is to evaluate the elastoplastic material parameters of the layers of multilayer coatings. To achieve this goal, an optimization procedure based on the experimental nanoindentation and finite element analysis has been devised for deducing the elastoplastic properties of each individual layer of the multilayer coating (E, σ_y and n). The proposed methodology has been successfully applied to a CrN/CrAlN multilayer coating system with various layers thickness. The results obtained from this work serve as valuable reference for the structural optimization of multilayer coatings.

2 Experimental Details

2.1 CrAlN/CrN Multilayer Coating Deposition

In this study, CrN/CrAlN multilayer coatings with various layer thicknesses were deposited on X50CrMoV8-1 steel samples (20 × 20 × 5 mm^3) using DC reactive magnetron sputtering. During deposition the substrate temperature and the substrate bias voltage were fixed at 300 °C and − 500V, respectively, under 0.5 Pa of working pressure. Nitrogen and Argon gas with flow rate of 33.3 and 68.8 sccm, respectively were used. Then a high-purity Cr and Al target (99.99%) was used to deposit CrN/CrAlN multilayer coatings for a deposition time of 2 h. Figure 1 shows the different multilayer coatings deposited in this study. Two multilayer coatings, denoted as C1 and C2 were developed with a thickness gradient, choosing the same layer thicknesses used in the other coatings (1 and 0.5 μm) and keeping the total thickness fixed at 2 μm.

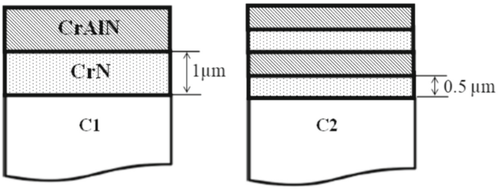


Fig. 1. Scheme of the CrN/CrAlN multilayer systems

2.2 Nanoindentation Tests

The evaluation of the mechanical properties (H) and (E) of CrN/CrAlN multilayer coatings was performed with a nanoindentation system (MTS, USA) equipped with a Berkovich indenter. To explore the impact of the substrate on the elastoplastic properties of the coating layers, large indentation depths, greater than the film thickness were conducted using the CSM mode with a constant strain rate of 0.05 s^{-1}. Each sample underwent nine indents with a maximum load of 750 mN. The estimation of (H) and (E) of the layers and the substrate was performed using the analytical modified (JH) model. The principle of the modified (JH) model will be detailed in the next section.

2.3 Estimation of the Mechanical Properties of Multilayer Coatings by the Modified (JH) Model

The modified (JH) model [7] was used to estimate the (H) and (E) of each monolayer composing the multilayer coatings. In this model, the hardness of the composite structure (H_c) of a multilayer consisting of N layers is evaluated by a mixture law that relates the hardness of i-layer ($H_f^{(i)}$) and the substrate (H_s):

$$H_c = \sum_{i=1}^{N} a_f^{(i)} H_f^{(i)} + a_f^{(s)} H_s \tag{1}$$

where, $a_f^{(i)}$, measure the contribution of the i-layer of the multilayer coating to the composite hardness (H_c). This variable is equal to the ratio of the flow pressure area of the i-layer to the total flow pressure area. For the first layer ($i = 1$), this ratio can be expressed as a function of indentation depth, h, and the thickness of the first layer, $t_f^{(1)}$, by:

$$\begin{cases} a_f^{(1)} = 1 & \text{if } h < C^{(1)}\, t_f^{(1)} \\ a_f^{(1)} = 1 - \left(1 - \dfrac{C^{(1)}\, t_f^{(1)}}{h}\right)^2 & \text{Otherwise} \end{cases} \tag{2}$$

where $C^{(1)}$, represents a constant depending on the indenter geometry and the indentation behavior of the layer under consideration [7]. In this model, the volume fraction (a_f) of the coating material should be between 0 and 1. Consequently, if h is lower than $C^{(1)}$ $t_f^{(1)}$, the substrate does not influence the composite hardness, and $a_f^{(1)}$ is equal to 1. Whereas, for the jth layer of the coating, the volume fraction (a_f) is expressed as follows:

$$\begin{cases} a_f^{(j)} = 1 - \sum\limits_{i=1}^{j-1} a_f^{(i)} & \text{if } h < \sum\limits_{i=1}^{j} C^{(i)}\, t_f^{(i)} \\ a_f^{(j)} = \left\{ 1 - \left\{ 1 - \dfrac{\sum_{i=1}^{j} C^{(i)}\, t_f^{(i)}}{h} \right\}^2 \right\} - \left\{ 1 - \left\{ 1 - \dfrac{\sum_{i=1}^{j-1} C^{(i)}\, t_f^{(i)}}{h} \right\}^2 \right\} & \text{Otherwise} \end{cases} \tag{3}$$

Therefore, once the volume fraction of each layer has been evaluated, the volume fraction corresponding to the substrate material can be obtained:

$$a_f^{(s)} = 1 - \sum_{i=1}^{N} a_f^{(i)} \tag{4}$$

In order to consider the indentation size effect (ISE) in the calculation, the model was then enhanced by incorporating this factor. Hence, the experimental composite hardness data can be described, and the macrohardness can be determined independently of the load for the coating the i-layer ($H_{f0}^{(i)}$) and substrate (H_{S0}), respectively, assuming [7]

$$H_f = H_{f0}^{(i)} + \frac{B_f^{(i)}}{h} \quad \text{and} \quad H_s = H_{s0} + \frac{B_s}{h} \tag{5}$$

where, B_f and B_s represents the parameters which characterize the (ISE).

Furthermore, the yield stress (σ_Y) of the constituent layers and the substrate can be evaluated based on their hardness values using the Tabor relationship [13], H/c, where c is a constant equal to 3 for ideal plastic materials.

Additionally, the same model was used to determine the Young's modulus of the i-layer ($E_f^{(i)}$) and the substrate (E_s), with C considered as a variable [14]. Thus, for a multilayer coating, the composite modulus, (E_c), can be expressed by:

$$E_c = \sum_{i=1}^{N} a_f^{(i)} E_f^{(i)} + a_f^{(s)} E_s \tag{6}$$

Therefore, the modified (JH) model will be used to describe the change in the hardness and Young's modulus of the composite structure with indentation depth. Also, it allows the calculation of (H) and (E) of monolayers composing the multilayer, as well as that of the substrate by means of least square fitting analysis.

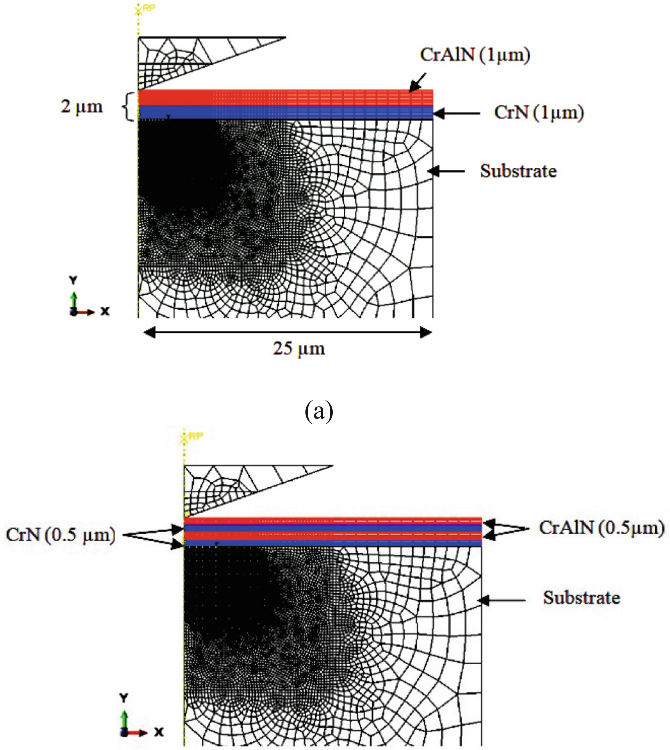
2.4 Finite Element Modeling

In this study, the commercial finite element software Abaqus Standart 2017 was used to simulate the indentation process. The composite structure was composed of 8806 4-node 2D axisymmetric elements (CAX4R). The dimension of the composite system was set to $25 \times 70\ \mu\text{m}^2$. This model used X50CrMoV8-1 as the substrate and featured CrAlN/CrN multilayer coatings with various layers number (2 and 4 layers) and thicknesses (1 and 0.5 μm), as illustrated in Fig. 2. The total thickness of the multilayer coatings remained unchanged at 2 μm. The mesh of the contact area was refined to improve the simulation accuracy ($0.1\ \mu\text{m} \times 0.1\ \mu\text{m}$). The Berkovich indenter was modelled using 4-node 2D axisymmetric deformable conical body (CAX4R) with a face angle of 70.3°, giving the same nominal indental volume as a Berkovich tip. All the interfaces between materials in the composite structure were assumed to be perfectly bonded. The contact between the indenter tip and the top surface layer are assumed to be frictionless [10]. All nodes at the bottom of the model are constraints. Indentation was applied by displacement control with a linear ramp in time and a final tip displacement equal to the experimental depth measurement at maximum load.

2.5 Optimizations Analysis

2.5.1 Optimization Algorithm

In this work, an optimization algorithm is implemented using the MATLAB software. The connection between ABAQUS and MATLAB is provided using Abaqus2Matlab software tool [15], enabling ABAQUS to be run from MATLAB and the results to be post-processed. The proposed optimization algorithm seeks to identify the optimal elastoplastic properties of the constituent layers in a multilayer coating by reducing the gap between the nanoindentation (P-h) curves and the simulated curves using the MATLAB function LSQNONLIN [12]. This function, based on the trust-region algorithm [16] is efficient in solving bound-constrained nonlinear minimization problems, and to ensure the accuracy of the results by using bound constraints for each materials variables. Hence, the prediction error in indentation depth is evaluated iteratively, summed and minimized to obtain the better coincidence between the experimental (P-h) curve and the corresponding simulated ones. To achieve this goal, a cost function F(X), which



(a)

(b)

Fig. 2. FEM geometric model: (a) 2 layers model (C1) (b) 4 layers model (C2).

describes the error criteria, minimized until a minimum convergence value of 10^{-2} is reached. The cost function is expressed as a function of the predicted force $P(X)^{pre}$ and the experimental one $P(X)^{exp}$ by:

$$F(x) = \frac{1}{2}\sum\nolimits_{i=1}^{N}\left[P(x)_i^{pre} - P_i^{\exp}\right]^2 \rightarrow \min \\ LB \leq x \leq UB \tag{7}$$

where X are the elastoplastic properties $X = \left[material\, properties\left(E, \sigma_y, n, ...\right)\right]^T$, which are starting from initial guess parameters $X_0 = \left[material\, properties\left(E_0, \sigma_{y0}, n_0, ...\right)\right]^T$. N is the total number of experimental points, and LB and UB are the lower and upper boundaries constraints of each material parameter X.

2.5.2 Optimization Procedure

In this study, the elastoplastic properties (E, σ_Y and n) of layers, forming the multilayer coating, were parameterized in the ABAQUS input files and adjusted at each iteration of the optimization to generate a new input files. In the simulation, a perfectly-plastic material model was used for the Berkovich tip, with Young's modulus, E = 1024 GPa, a Poisson ratio, $\upsilon = 0.07$, and a yield stress, $\sigma_y = 35.7$ GPa [10]. The deformation of the monolayers and the substrate is modelled using a power law strain hardening model. The (σ-ε) relationship is divided into elastic and plastic parts as follows:

$$\sigma = \begin{cases} E\varepsilon \quad for \quad \varepsilon \le \dfrac{\sigma_y}{E} \\ K\varepsilon^n \quad for \quad \varepsilon > \dfrac{\sigma_y}{E} \end{cases} \tag{8}$$

where E, σ_y, n and K are the Young's modulus, the yield stress, the strain hardening coefficient, and the strength coefficient, respectively.

By decomposing the total strain into elastic and plastic ones, the stress can be expressed as a function of plastic strains as follow:

$$\sigma = K\left(\frac{\sigma_y}{E} + \varepsilon_{pl}\right)^n \tag{9}$$

where K can be expressed by:

$$K = E^n \sigma_y^{1-n} \tag{10}$$

Thus, to define the plastic behaviour of the constituent layers in the ABAQUS input file, the optimization algorithm calculates firstly the coefficient K through Eq. (10) and updates through Eq. (9) the stress data value. The principle of the optimization procedure used in this work is shown in Fig. 3

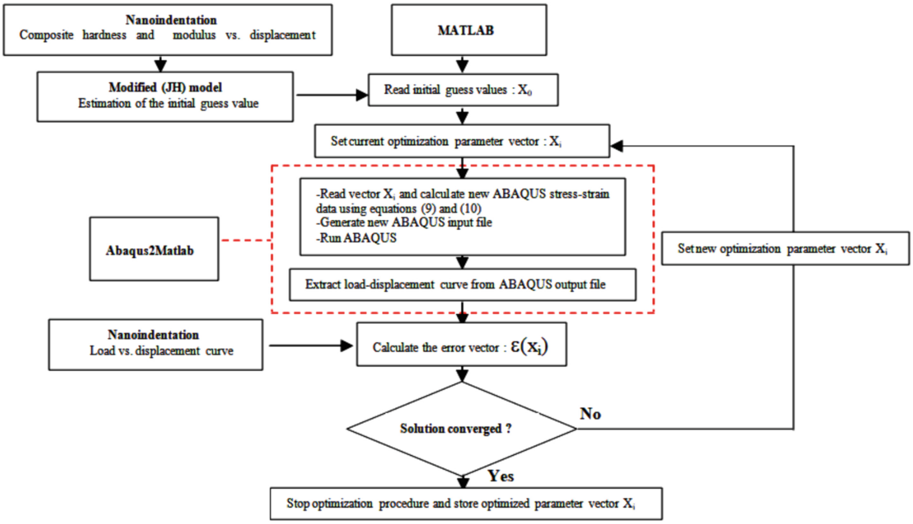


Fig. 3. Principle of the optimization procedure

In this approach, the intial guess parameters $X_0 = \left[elastoplastic\, properties\, \left(E_0, \sigma_{y0}, n_0\right)\right]$ of CrN and CrAlN monolayers were substituted into the finite element model calculation, and the gap between the simulated and the experimental (P-h) curves was calculated through the cost function F(X), and minimized iteratively by modifying the elastoplastic properties $X = \left[elastoplastic\, properties\ \left(E, \sigma_y, n\right)\right]$ of each monolayer. Thus, when the convergence condition reached the specified tolerance (10^{-2}), the optimization process would be completed, and the optimal elastoplastic material parameters of each layer would be determined. To guaranteed the accuracy of the results and the uniqueness of the solution, the initial guess parameters $X_0 = \left[elastoplastic\, properties\ \left(E_0, \sigma_{y_0}, n_0\right)\right]$ of the two monolayers are taken close to the target values [12, 17] by using the estimated mechanical properties provided by the modified (JH) model (E and σ_Y). Only (n) is chosen arbitrary. Thus, two optimization processes will be performed to determine the optimal elastoplastic properties (E, σ_y and n) of CrN and CrAlN monolayers for each multilayer coating systems: C1 and C2. In the simulation, the Poisson ratio υ of CrN and CrAlN is assumed to be equal and constant of magnitude of 0.22 [9]. To reduce the number of unknown parameters in the model, the elastoplastic properties of the substrate are fixed in the simulation, assuming that its (E) and (σ_y) are equal to the estimated values measured by the modified (JH) model and the Tabor assumption, respectively. For the strain hardening coefficient (n) and the Poisson ratio (υ) of the substrate are assumed to be equal to 0.1 and 0.3 [17], respectively.

3 Results and Discussion

3.1 Estimation of the Initial Guess Parameters of the Optimization Procedure Using the Modified (JH) Model

Figure 4 and 5 illustrate the changes in the experimental (Hc) and (Ec) of the two CrN/CrAlN multilayer coating systems: C1 and C2, with the indentation depth. The modified (JH) model is used to describe the experimental composite hardness, assuming that the 2 layers undergo plastic deformation by taking C equal to 0.1746 [7]. Thus, the hardness of the constituent layers of multilayer and the substrate is evaluated by means of least squares fitting analysis of the experimental data (see Fig. 4(a) and Fig. 5 (a)) through the MATLAB function fminsearch. The same approach is used to evaluate their (E) (see Fig. 4(b) and Fig. 5 (b)). Furthermore, the yield stress (σ_y) of each monolayer and substrate is estimated using the estimated value of hardness through the Tabor assumption. Table 1 summarizes the values of the various mechanical properties obtained by the modified (JH) model. Results show that the modified (JH) model provides a good description of the variation in (Hc) and (Ec) of CrN/CrAlN multilayer coatings with indentation depth.

Table 1. Modified (JH) model results

	C1 condition			C2 condition		
	CrAlN	CrN	substrate	CrAlN	CrN	substrate
H (GPa)	9.68	5.92	7.72	7.63	4.86	8.32
E (GPa)	221.41	121.12	218.51	127.2	92.1	233.21
σy (GPa)	3.22	1.98	2.57	2.54	1.65	2.77

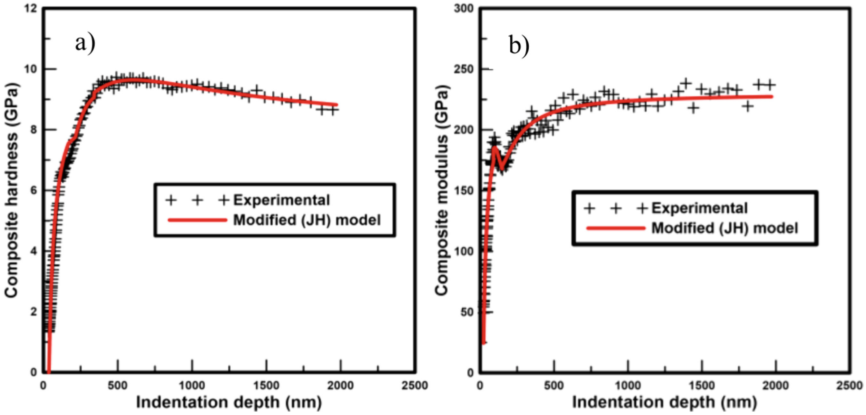


Fig. 4. Variation of (a) (Hc) and, (b) (Ec) with the indentation depth for C1 condition

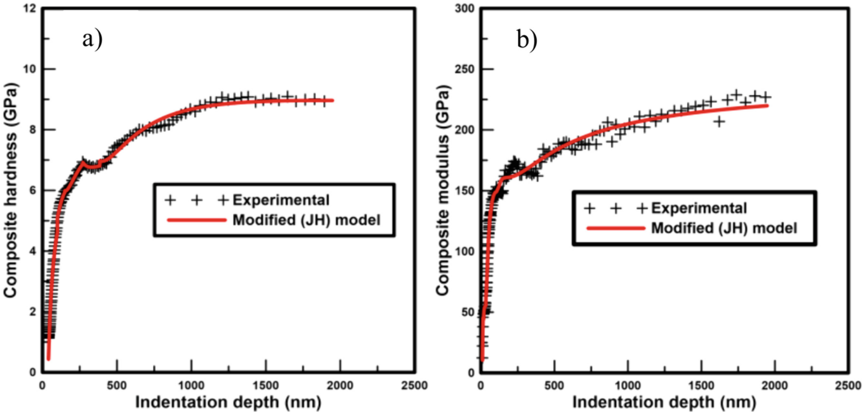


Fig. 5. Variation of (a) (Hc) and, (b) (Ec) with the indentation depth for C2 condition

3.1.1 Optimization Results

In this study, the proposed optimization procedure is validated on CrN/CrAlN multilayer coating systems: C1 and C2, and as a result the optimal elastoplastic properties (E, σ_y

and n) of CrN and CrAlN layers is extracted. The optimization results are summarized in Tables 2 and 3. The initial value parameters of the optimization analysis are defined based on the results of the modified (JH) model (Table 1). Hence, the estimated value of (E) and (σ_Y) of the two layers (CrN and CrAlN) (Table 1) are taken as initial values in the optimization analysis (Tables 2 and 3). For (n), the correspondent initial value was arbitrary taken. Furthermore, the bound constrains of each variable are defined based on previous works [9]. Figure 6 represents the comparison between the experimental (P-h) curves and the corresponding simulated ones, using the optimal solution of elastoplastic material parameters of CrN and CrAlN layers, respectively. The comparison results demonstrate a good agreement between the experimental and the simulated results, indicating the efficiency of the proposed methodology. It's clear that the gap between the optimized values of (E) and (σ_Y) and their initial values (see Tables 2 and 3), was generally low (20%), indicating the accuracy of the proposed approach.s

Table 2. Optimization result for C1 condition

	Parameters	Initial values	Boundaries	Optimized values
CrAlN layer	E	221.41 [GPa]	$50 < E$ [GPa] < 240	192.17 [GPa]
	σy	3.22 [GPa]	$1 < \sigma y$ [GPa] < 14	3.15 [GPa]
	n	0.2	$0 < n < 0.5$	0.33
CrN layer	E	121.12 [GPa]	$50 < E$ [GPa] < 240	128.32 [GPa]
	σy	1.98 [GPa]	$1 < \sigma y$ [GPa] < 14	2.42 [GPa]
	n	0.2	$0 < n < 0.5$	0.29

Table 3. Optimization result for C2 condition

	Parameters	Initial values	Boundaries	Optimized values
CrAlN layer	E	127.2 [GPa]	$50 < E$ [GPa] < 240	131.1 [GPa]
	σy	2.54 [GPa]	$1 < \sigma y$ [GPa] < 14	2.97 [GPa]
	n	0.2	$0 < n < 0.5$	0.23
CrN layer	E	92.1 [GPa]	$50 < E$ [GPa] < 240	98.4 [GPa]
	σy	1.7 [GPa]	$1 < \sigma y$ [GPa] < 14	2.21 [GPa]
	n	0.2	$0 < n < 0.5$	0.21

The results show a decrease in the material properties (H, σ_Y and E) of CrN and CrAlN layers when the layer thickness decreases. These findings suggest that decreasing the coating layer thickness lead to an increase in the plastic deformation within the coatings, which reduces the stress concentration in this area and improves the adhesion properties of CrN/CrAlN multilayer coatings. Indeed, the CrN/CrAlN multilayer coating system with layer thicknesses of 0.5 μm (C2 system) generated the lowest stress and the highest plastic deformation, improving the adhesion properties of the multilayer structure.

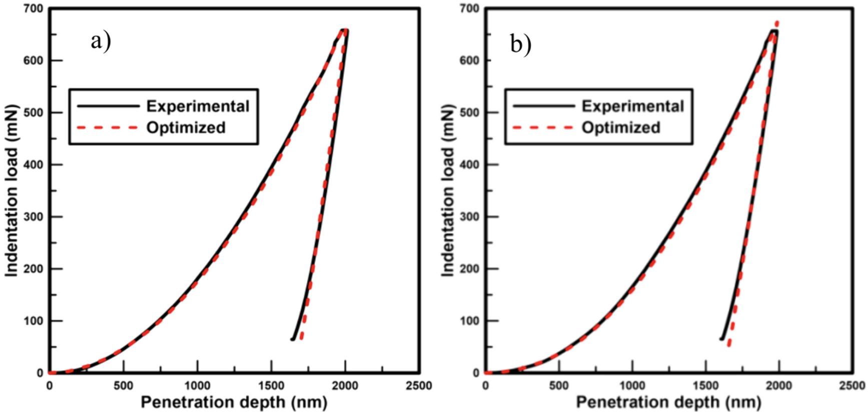


Fig. 6. Comparison of predicted and experimental (P-h) curves of CrN and CrAlN layers: (a) C1 condition; (b) C2 condition

4 Conclusion

In this study, CrN/CrAlN multilayer coatings with various layer thicknesses were prepared using PVD technology. The assessment of the elastoplastic properties of the constituent layers of multilayer coating was performed through nanoindentation using a trust-region algorithm combined with numerical indentation models. The optimization results show that the material properties (H, σ_Y and E) of the two layers decrease when decreasing the layer thickness. The CrN/CrAlN multilayer coating system with monolayer thicknesses of 0.5 μm produced the lowest stress and the highest plastic deformation during the indentation process, leading to the best adhesion properties compared with the other systems.

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