Evaluation of the mechanical properties of plasma-sprayed coating by nanoindentation technology

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Abstract – In this paper, the mechanical properties of plasma-sprayed coatings are evaluated using the technology of nanoindentation. According to the indentation curve (load versus indenter's displacement), which is recorded in an indentation experiment, the mechanical behaviour of the plasma-sprayed coating is investigated via inverse analysis using numerical optimization algorithms. The tip rounding of imperfect indenter is investigated in parameter identification. Subsequently, the hardness on the top surface and on the vertical cross section of the plasma-sprayed coating are investigated. The results show that the hardness on the top surface of the coating depends on the indentation depth. Moreover, the results show that the hardness on the cross section of the coating is significantly affected by the substrate if the indentation impression is too close to the coating-substrate interface.

Key words: Nanoindentation / hardness / plasma-sprayed coating / inverse analysis / numerical method

1 Introduction

The rapid developments of surface engineering technologies and their successful applications in various sectors of industry have led to increasing demands for assessing the mechanical properties of engineering coatings, whose thickness usually ranges from submicron scales to several hundred microns depending on the application requirements and on the processing conditions. In general, plasma-sprayed coatings as a part of thermal spraying are divided into metallic and non-metallic materials and formed through a group of processes which can deposit the coating materials in a molten or semi-molten state onto a prepared substrate [1, 2].

For example, the formation principle of plasma-sprayed coatings adopted in [2] is shown in Figure 1. In a plasma jet, the thermal plasma heat source (direct current arc or radio frequency discharge) with temperatures over 8000 K at atmospheric pressure allows the melting of any material. Powdered materials are injected within the

plasma or plasma jet, where particles are accelerated and molten, or partially molten, see Figure 1a. Before they flatten and solidify onto the substrate, the coating will be built by the layering of splats, see Figure 1b. The thickness of the formed coating normally is in the range of microns or millimetres. At such small scales, the mechanical properties of those coatings, e.g. the toughness, hardness, yield strength, elasticity modulus and the strain-stress law cannot be determined by traditional methods, such as compression and tensile testing, since they do not scale well into the micro- and nanoscales. However, at such scales, nanoindentation technology has already been widely employed to characterize the mechanical properties of thin coatings. For relatively thick coatings (e.g. the thickness $>10\ \mu\text{m}$), conventional indentation technology can provide a simple, efficient and robust means for evaluating the coating's properties [3–8].

In the literature, most of the researchers focused on identification of the hardness on the top surface of coatings [4–6]. However, according to the earlier research carried out by Lin [7] and Leigh [8], the hardness of coatings may exhibit some variability in thickness direction

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Nomenclature

Latin characters	
a_c	Contact area radius in cone (m)
a_s	Contact area radius in sphere (m)
A_{proj}	Projected contact area (m ²)
C_i	Constants
D_0	Diameter of particle
D_f	Diameter of splat
$\mathbf{D}$	Rate of deformation tensor
$\mathbf{D}^e$	Elastic part in rate of deformation
$\mathbf{D}^p$	Inelastic part in rate of deformation
E	Young's modulus of investigated material (Pa)
E_i	Young's modulus of indenter (Pa)
E_r	Reduced modulus (Pa)
f	Yield function (Pa)
$\bar{f}$	Updated yield function (Pa)
F	Objective function (m ²)
h	Indentation depth (m)
$\bar{h}$	Experimental indentation depth (m)
h_c	Contact depth (m)
h_d	Deviation of penetration depth (m)
h_{max}	Maximum indentation depth (m)
G	Shear modulus (Pa)
H	Hardness (Pa)
$\mathbf{H}$	Hooke stress-strain tensor
K	Bulk modulus (Pa)
Lx, Lys, Lzc	Length (m)
m	Viscosity exponent
n	Work-hardening exponent
N	Number of load steps
p	Porosity
P	Load (N)
P_{max}	Maximum load (N)
Q	Material parameter in Voce-type hardening law (Pa)
$\mathbf{s}$	Deviator of the stress tensor
S	Initial unloading stiffness (N.m ⁻¹)
x_j	Material parameters
Greek characters	
β	Material parameter in Voce-type hardening law
δ	Kronecker delta
$\bar{\epsilon}_p$	Plastic strain
$\dot{\bar{\epsilon}}_{vp}$	Rate of effective viscoplastic strain
η	Coefficient of viscosity (Pa/s ^{-1/m})
μ	Friction coefficient
θ	Half apex angle of conical indenter (°)
ρ	Radius of imperfect indenter tip (m)
$\bar{\sigma}$	Effective stress (Pa)
σ_0	Quasi-static flow stress (Pa)
σ_y^0	Initial yield stress (Pa)
σ_v	Current yield stress (Pa)
σ_{visco}	Stress due to viscous effect (Pa)
$\boldsymbol{\sigma}$	Cauchy stress tensor
$\boldsymbol{\sigma}^\nabla$	Objective rate of Cauchy stress tensor
τ	Tolerance
ν	Poisson's ratio of investigated material
ν_i	Poisson's ratio of indenter

Fig. 1. Principle of plasma-sprayed coating formation [1, 2].

due to a heterogeneous microstructure generated during spraying [8] or service conditions producing oxidation or cracking [7]. Therefore, measurement of hardness on the top surface of a coating only is not sufficient enough to explain and describe the mechanical properties, such as the hardness and its relationship with the microstructure of the coating.

In the present paper, the evaluation of a plasma-sprayed coating's hardness and the assessment of its relation with the substrate are carried out. First the material parameters of the plasma-sprayed coating are identified via inverse analysis using numerical optimization algorithms based on the experimental data. Then, according to the identified material parameters, the hardness on the top surface and on the vertical cross section of the plasma-sprayed coating are investigated.

2 Theoretical considerations

2.1 Theory of hardness measurement

Usually, for hardness measurement, an indentation instrument is employed. It can continuously register the load p versus the indentation depth h , during loading, holding (not necessary) and unloading processes, see Figure 2a. Then, the hardness and Young's modulus can be inferred from the $p-h$ curve by some corresponding estimation methods. In current applications, the most widely used method is proposed by Oliver and Pharr [9], which has been adopted as a standard reference method in ISO-14577 [10],

$$H = \frac{P}{A_{\text{proj}}} \quad (1)$$

$$E_r = \frac{1}{2} \sqrt{\frac{\pi}{A_{\text{proj}}}} \frac{dP}{dh} \quad (2)$$

$$h_c = h_{\text{max}} - \frac{2}{\pi}(\pi - 2) \frac{P_{\text{max}}}{S} \quad (3)$$

$$A_{\text{proj}} = 24.5 h_c^2 + \sum_{i=1}^8 C_i h_c^{1/2^{i-1}} \quad (4)$$

where, A_{proj} is the projected contact area, see Figure 2b, which can be calculated by Equation (4). The constants, C_i in Equation (4) describe the deviation caused by an imperfect indenter tip. In practice, C_i are calibrated by some known materials in experiments, e.g. fused silicon, aluminium [9, 10]. P_{max} is the maximum load and h_{max} is the maximum indentation depth. h_c is the contact depth, see the details in Figure 2b. In the following numerical simulations, h_c is obtained from the modelled deformed surface and C_i are set as zero if the indenter is designed with a perfect sharp tip. $S = dP/dh$ is the initial unloading stiffness, which is calculated at the maximum indentation depth, see Figure 2a. E_r is the so-called reduced modulus, which includes the Young's modulus and Poisson's ratio of the indenter (E_i, ν_i) and of the investigated material (E, ν). The relationship of them is described by

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \quad (5)$$

For a diamond indenter, $E_i = 1140$ GPa and $\nu_i = 0.07$. Thus, in order to calculate E by Equation (5), ν should be known. Normally, ν is set as 0.3 or 0.25 for unknown materials. In this paper, $\nu = 0.3$ is assumed for the investigated plasma-sprayed coating.

2.2 Material model

Through the nanoindentation experimental measurement, the hardness and reduced Young's modulus of the sample are obtained. Besides this, the further mechanical

Fig. 2. Schematics of load versus indentation depth curve and indentation profiles with sinking-in and piling-up.

behaviour of the plasma-sprayed coating, e.g. the elasto-plastic and viscous behaviour is also interested. In the present paper, the investigated plasma-sprayed coating is made of Y_2O_3 -stabilized ZrO_2 . Through the experimental data, see Figure 5, the creep behaviour during the holding process can be seen clearly, which shows that this kind of coating not only has elasto-plastic behaviour, but also exhibits viscous behaviour.

Therefore, according to the mechanical behaviour of the plasma-sprayed coating, a constitutive formulation will be presented for obtaining more mechanical properties the plasma-sprayed coating. The establishment of the formulation in this section considers the work of Ponthot [11], who introduced a unified stress update algorithm for elasto-plastic constitutive equations and then extended it to elasto-viscoplasticity in a finite deformation framework using a corotational formulation, within an updated Lagrangian scheme.

According to [11], the rate of deformation, $\mathbf{D}$ can be additively decomposed into an elastic, $\mathbf{D}^e$ (reversible) and an inelastic, $\mathbf{D}^p$ (irreversible) part, i.e.

$$\mathbf{D} = \mathbf{D}^e + \mathbf{D}^p \quad (6)$$

The relationship between the rate of strain and the rate of stress is postulated as

$$\sigma_{ij}^\nabla = H_{ijkl}(\mathbf{D}_{kl} - \mathbf{D}_{kl}^p) \quad \text{or} \quad \sigma^\nabla = \mathbf{H} : (\mathbf{D} - \mathbf{D}^p) \quad (7)$$

where, σ^∇ is an objective rate of the Cauchy stress tensor σ . $\mathbf{H}$ is the Hooke stress-strain tensor (elastic stiffness tensor) which is given by

$$H_{ijkl} = K\delta_{ij}\delta_{kl} + 2G\left(\delta_{ik}\delta_{jl} - \frac{1}{3}\delta_{ij}\delta_{kl}\right) \quad (8)$$

where, δ is the Kronecker delta symbol, K is the bulk modulus and G is the shear modulus of the material.

Plastic deformation is triggered when the stress in the material reaches a given limit. In the material model, the

yield function is used to detect an increase of the plastic deformations. It defines a surface which envelops all physically possible stress states in rate-independent plasticity. Stress states inside this contour cause only elastic deformations, while stress states on this yield surface give rise to elastic-plastic deformations. By definition, rate-independent plastic stress states outside the yield contour f are not admissible.

Moreover, the von Mises yield function with J_2 flow theory for isotropic materials will be chosen as the yield criterion in the following numerical calculations, more details can be seen in [11, 12]. This yield criterion is frequently assumed for metals and alloys [12]. Furthermore, it offers the numerical advantage that the gradients of the von Mises yield surface, which are used for the numerical solution procedure, are always uniquely defined. Mathematically, the yield function is expressed as,

$$f(\sigma, \sigma_\nu) = \bar{\sigma} - \sigma_\nu = 0 \quad (9)$$

where, $\bar{\sigma}$ is the effective stress, i.e. $\bar{\sigma} = \sqrt{\frac{3}{2}s:s}$; s is the deviator of the stress tensor; σ_ν is the current yield stress.

While $f < 0$, there is elastic material behaviour. For $f = 0$, rate-independent plastic material behaviour takes place. Herein, plastic hardening with an associative flow rule is considered, including non-linear isotropic hardening. The evolution of yield stress, σ_ν can be closely approximated by the Voce-type hardening law,

$$\sigma_\nu = \sigma_y^0 + Q[1 - \exp(-\beta\epsilon_p)] \quad (10)$$

where, σ_y^0 is the initial yield stress. Q and β are non-linear isotropic hardening parameters. In addition, if the materials are considered with viscosity, e.g. if the viscosity is described as the Cowper-Symonds law,

$$\sigma_{\text{visco}} = \sigma_0 \left(\frac{\dot{\epsilon}_{vp}}{\eta} \right)^{\frac{1}{m}} \quad (11)$$

Fig. 3. Graphic depiction of the forward and inverse problems in indentation for elasto-plastic materials [20].

where, σ_0 is the quasi-static flow stress. $\dot{\bar{\epsilon}}_{vp}^*$ is the rate of effective viscoplastic strain. η is a constant and m is the viscosity exponent. A new constraint is defined in the viscoplastic range according to [11, 13],

$$\bar{f} = \bar{\sigma} - \sigma_\nu - \sigma_{\text{visco}} = 0 \quad (12)$$

In the elastic regime, both f and $\bar{f}$ are equivalent because in this case, $\dot{\bar{\epsilon}}_{vp}^* = 0$ and $\bar{\sigma} \leq \sigma_\nu$. So that one has $\bar{f} \leq 0$.

3 Methods for parameter identification

3.1 Forward and inverse analyses

The problems solved by indentation testing are generally divided into two kinds. One is the so-called “forward problem” [14], another is the “inverse problem” [15]. In the forward algorithm, a unique indentation response (e.g. $P-h$ curve) from a given set of material properties (e.g. E, σ_y^0 and ν for elasto-plastic materials) [16, 17] can be calculated, whereas the inverse algorithm enables the extraction of material properties from a given set of indentation data [15, 18–20]. The relationship between these two algorithms can be seen in Figure 3.

In the literature, many researchers focused on using the forward analysis to calculate the hardness [9, 19], and using the inverse indentation method to evaluate the relation of strain-stress [21] and analyze the elasto-plastic and elasto-viscoplastic behaviour [22–25] etc. In the following parts, first, an algorithm is presented to identify the material parameters of the investigated plasma-sprayed coating through inverse analysis. Subsequently, the hardness on the top surface and on the cross section of the

plasma-sprayed coating are investigated utilizing forward analysis based on the identified material parameters.

3.2 Optimization algorithm for parameter identification

In order to identify the material parameters from the $p-h$ curve registered during the loading and unloading processes in an experiment, the inverse analysis mentioned in the foregoing part and based on numerical optimization has to be performed. The key idea of this procedure is to model the indentation test with a numerical method, i.e. the finite-element method, using a starting set of material parameters and quantifying the difference between the modelled indentation curve and the experimental curve through an objective function. Subsequently, the material parameters used in the finite element model are updated using some algorithms from numerical optimization in order to minimize the objective function. When they lead to a good agreement between the experimental and numerical results, the identified material parameters can be used as equivalent values for the investigated material.

Herein, the objective function to be minimized by numerical optimization algorithm is made up of the difference between the experimental and modelled displacement-into-surface, $\bar{h}$ and h , respectively, which are the function of the independent load, P .

$$F = \frac{1}{2N} \sum_{i=1}^N [h(p_i) - \bar{h}(P_i)]^2 \quad (13)$$

Fig. 4. The process of optimization.

Therefore, the sensitivity analysis is defined as the derivative of the objective function with respect to these material parameters.

$$\frac{\partial F}{\partial x_j} = \frac{1}{N} \sum_{i=1}^N [h(P_i) - \bar{h}(P_i)] \frac{\partial h(P_i)}{\partial x_j} \quad (14)$$

where, x_j represents the material parameters. After defining the objective function and sensitivities, the objective function can be minimized using some gradient-based numerical optimization algorithms. In the present research, the cascade optimization methodology which is a combination of Levenberg-Marquardt and conjugate gradient algorithms is adopted. This optimization methodology is proposed by Ponthot, its efficiency and robustness are compared with other optimization algorithms seen in [26]. When F drops below a predefined tolerance, the optimization process will be stopped and optimized material parameters will be written down. The whole procedure is shown in Figure 4.

4 Results and discussion

4.1 Experimental results

In the real indentation experiment, a sample with $\sim 100 \mu\text{m}$ thickness of plasma-sprayed coating ($\text{ZrO}_2\text{-Y}_2\text{O}_3$) formed on a stainless steel substrate was measured. The proportion of ZrO_2 and Y_2O_3 is 93:7 (mol) and the porosity of the coating is in the range of 5–10%. In this experiment, the nano-triboindenter instrument [27] equipped with a Berkovich indenter was used. The measurement procedure involves continuous monitoring of the load and of the indenter's displacement during the loading, holding and unloading processes; see left figure in Figure 5, where the experimental data are represented by squares. The indentation processes are executed by force control and the maximum load equals $4908 \mu\text{N}$. The loading history can be seen in the right figure in Figure 5. From 0 to 5 s, the loading process takes place. Then the loading is held at maximum load for 2 s. After that, the unloading process is performed from 7 to 12 s.

Fig. 5. Optimized $p-h$ curves obtained by perfect conical indenter compared to the experimental data.

Correspondingly, the measured hardness, H equals 16.5 GPa, and reduced Young's modulus, E_r equals 192 GPa. If the Poisson's ratio of the plasma-sprayed coating is assumed as 0.3, its Young's modulus, $E = 209.9$ GPa, which is calculated according to Equation (5).

4.2 Identification of the material parameters of the plasma-sprayed coating

According to the experimental data, the material parameters of the plasma-sprayed coating can be evaluated by inverse analysis using numerical optimization. In the following parts, all the simulation studies are performed in the finite-element code METAFOR, which performs powerfully in computations of nonlinear materials and large deformations [28].

Because the coating's thickness of the measured sample is $\sim 100 \mu\text{m}$, the maximum indentation depth is lower than 120 nm, see Figure 5. The indentation depth is below $1/800$ of the coating's thickness. Therefore, it can be said that the experimental $P-h$ curve is not affected by the substrate as the researchers demonstrated that for hard coating and soft substrate material, normally, the measured results are no longer affected by the substrate when the indentation depth is under $1/30$ of the thickness of coating [29]. Thus, herein, in order to simplify the problem and save the computer time, 2D finite-element model is adopted and it is modelled only with the coating material, see Figure 6. The 2D axisymmetric finite-element is defined with $7 \mu\text{m}$ radius and $7 \mu\text{m}$ height. It is established using 1062 four-node quadrilateral elements, where a finer mesh near the contact region and a gradually coarser mesh further from the contact region are designed to ensure numerical accuracy. Moreover, the horizontal and vertical displacements are fixed at central and bottom lines as boundary conditions. In the experiment, the used indenter is a three-sided Berkovich indenter. Therefore, as proposed by Albrecht [21] for 2D computations, the Berkovich indenter can be modelled by a

conical indenter with the half apex angle of $\theta = 70.3^\circ$. Furthermore, the Coulomb's friction model is assumed in the contact region with the friction coefficient, $\mu = 1.5$. The loading history, exactly corresponds to the one performed in the experiment, which can be seen in the right figure in Figure 5.

The optimized $p-h$ curve compared to the experimental data is shown in Figure 5a. It is clearly seen that the optimized $p-h$ curve has a good agreement with the experimental one. The final identified coating's material parameters are listed in the first rows of Table 1. The simulated hardness equals 15.2 GPa. The difference is 7.9% compared to the experimental hardness, $H = 16.5$ GPa. Moreover, the identified Young's modulus is compared to the experimental one, $E = 209.9$ GPa, the difference is 2.9%.

However, we can see in Figure 5 that, when the indenter starts penetrating into the specimen, the optimized loading curve is lower than the experimental curve. This is supposed to be due to the tip rounding of the indenter used in the experiment because every indenter never has a perfect sharp tip as designed in practical application [31–33]. For this reason, a conical indenter with a rounded tip is modelled like the one shown in Figure 7. The radius of the indenter rounded tip, ρ is defined as a variable parameter in the optimization procedure. According to the geometry of this imperfect conical indenter, the contact radius, a_c can be written as

$$a_c = (h_c + h_d) \tan \theta \quad (15)$$

where, h_d is the deviation of penetration depth for the imperfect indenter. It is expressed by

$$h_d = \rho(\sin^{-1} \theta - 1) \quad (16)$$

Moreover, the contact radius a_s which is produced by the rounded tip can be calculated as

$$a_s = \rho \cos \theta \quad (17)$$

Fig. 6. 2D axisymmetric finite-element model for parameter identification.

Table 1. The identified material parameters for the investigated plasma-sprayed coating.

ρ (nm)	E (GPa)	σ_y^0 (GPa)	Q (GPa)	β (GPa)	η (GPa/s ^{1/m})	m	H (GPa)
0	203.8	8.99	15.89	5.41	0.046	0.54	15.2
219	167.6	7.70	0.03	8.34	0.056	2.06	11.5

The optimized load-displacement curve compared to the experimental data is shown in Figure 8. It is clear that the optimized load-displacement curve has a good agreement with the experimental curve. The corresponding identified material parameters for this coating and the optimized radius of the rounded tip are listed in Table 1. As can be seen in the second row of this table, the optimized radius of the rounded tip equals 219 nm. It is in the reasonable range according to the investigation [10]. However, it is noted that at the maximum penetration depth $h_{\max} = 118$ nm, the contact depth $h_c = 114$ nm is directly measured from the finite-element model. Thus the ratio of $a_s/a_c \approx 0.21$ can be calculated according to Equations (15) and (17). This means that the conical indenter used in the experiment is worn out very much and about 21% part of the contact area is a spherical contact area.

In order to calculate the projected contact area produced by the imperfect indenter, the constants C_i in Equation (4) have to be calibrated. Here, only C_i ($i = 1, 2, 3, 4$) are calibrated because the latter terms in Equation (4) affect A_{proj} less. The identified material parameters listed in the second rows of Table 1 are used to calibrate C_i . As a consequence, A_{proj} for this imperfect indenter is written as

$$A_{\text{proj}} = 24.5h_c^2 + 677.5h_c - 385.1h_c^{1/2} + 157.7h_c^{1/4} + 3891.5h_c^{1/8} \quad (18)$$

The corresponding calculated hardness and Young's modulus can be seen in the second rows of Table 1. The differences of the hardness and Young's modulus are over than 30% and 18%, respectively, in comparison with the

Fig. 7. Schematic of the indentation profiles for an imperfect conical indenter with the sinking-in and the piling-up [30].

experimental ones. Concerning to the large differences between simulated data and experimental data, they may be led by the calculation of A_{proj} . In the experiment A_{proj} is calculated using Equation (4). However, the constants C_i in Equation (4) may be calibrated when the indenter was new. After the indenter was worn, the constants C_i no longer were exact for this worn indenter. That is due to the fact that in the experiment, the calculated A_{proj} equals 3.0×10^5 nm². However, according to Equation (18), for the imperfect indenter with the tip radius $\rho = 219$ nm, at the same penetration depth, A_{proj} equals 4.03×10^5 nm². Thus the calculated hardness for identified material parameters is smaller than the hardness obtained in the experiment.

Fig. 8. Optimized $p-h$ curves obtained by imperfect conical indenter compared to the experimental data.

Furthermore, when plasma-sprayed coating is being generated, porosity exists in the coating. According to the investigations [34,35], the Young's modulus of a coating is dependent on this porosity. The Young's modulus will decrease significantly with an increase of the porosity. The relationship of them can be fitted according to experimental results for a $\text{ZrO}_2\text{-Y}_2\text{O}_3$ coating [34],

$$E = E_0 \exp(a_e p) \quad (19)$$

with $E_0 = 221 \pm 4$ GPa and $a_e = 2.7 \pm 0.5$, where p is the porosity. Thus, for a coating with 10% porosity, its Young's modulus would decrease to 169 GPa. Simultaneously, it is reminded that the Young's modulus equals 167.6 GPa obtained by the imperfect conical indenter in the previous part. They have a good agreement. This denotes that the experimental Young's modulus may have a large error.

Furthermore, it is clear that the optimized load-displacement curve which takes into account the tip rounding is close to the experimental one. Therefore, it is believed that the identified material parameters determined by the imperfect conical indenters are more reliable because the corresponding load-displacement curve is almost identical compared to the experimental curve. As a consequence, the material properties of the plasma-sprayed coating can be presented by the material parameters which are evaluated by the imperfect conical indenter. Thus in the following hardness investigations, the mechanical behaviour of the coating is described by the identified material parameters in the second row of Table 1.

5 Hardness testing

5.1 Hardness on top surface of coating

The hardness on the top surface of coating is focused on in many of the published papers [6–8]. It represents the

Fig. 9. 2D axisymmetric finite-element model for coating material.

work condition of coatings in practical applications. However, it is reported that hardness depends on the indentation depth. In order to investigate this problem, another 2D axisymmetric finite-element model with a perfect conical indenter is modelled using 958 four-noded quadrilateral elements. There are 737 elements for the coating and 221 elements for the substrate. A finer mesh near the contact region and a gradually coarser mesh further from the contact region are designed to ensure numerical accuracy, see Figure 9. The model is designed with the radius, $Lx = 7 \mu\text{m}$. The thickness of the coating is presumed as $Lyc = 1 \mu\text{m}$ and the thickness of the substrate is presumed as $Lys = 6 \mu\text{m}$. Herein, the substrate is stainless steel, its plastic behaviour is approximated by the power law description according to Mata [19],

$$\sigma_\nu = \sigma_y^0 \left(\frac{E}{\sigma_y^0 \bar{\epsilon}_p} \right)^n \quad (20)$$

The work-hardening exponent, $n = 0.19$, the Young's modulus, $E = 200$ GPa, the initial yield stress, $\sigma_y^0 = 675$ MPa and the Poisson's ratio is set as 0.3.

Because a perfect sharp conical indenter is used in this part, the projected contact area will be calculated by Equation (4) and the constants C_i are set to zero. The hardness is identified at the maximum load, $P_{\max}$ varies from 100 to 13 195 μN , respectively. The hardness versus the ratio of the indentation depth and the coating's thickness is shown in Figure 10. It can be seen, at the range of ultimate low loads, i.e. the indentation depth is lower than about 7.5% of the coating's thickness, the hardness is nearly constant and agrees well with the calculated hardness, which is already obtained by the pure coating model shown in Figure 6. However, the hardness decreases significantly with an increase of $h_{\max}$ as expected, because when $h_{\max}$ increases more, the identified hardness of the

Fig. 10. Hardness versus the ratio of the indentation depth versus the coating's thickness (blue dash line represents the hardness of 11.5 GPa. It is obtained by the pure coating model shown in Fig. 6).

coating is influenced more by the substrate. Therefore, as usual, the indentation depth is required that it should not exceed 10% of the thickness of coating [36] for eliminating the substrate effects. Whereas, on the other hand, an indentation depth as large as possible should be used in order to eliminate indentation size effect (ISE) and specimen roughness effect which are usually more significant at lower indentation depth. Thus, the definition of the critical ratio of coating thickness to indentation depth is a challenge in identification of thin coatings. Anyway, the ISE and specimen roughness effect are beyond the aims of this paper.

5.2 Hardness on vertical cross section

In [6–8], researchers measured the hardness on the top surface of a coating, as stated in the previous section. The hardness is normally reported as an average value of a certain number of readings from random indentations on the top surface of the coating. However, such hardness is not sufficient to understand the mechanical properties. It is necessary to evaluate the hardness within coating and assess its relation with the substrate. Herein, a 3D model is designed and shown in Figure 11 to describe the schematic of different indentation impressions and micro-structural characteristics of the finite-element models, which are used in the following investigations.

Herein, a perfect conical indenter with the half apex angle of $\theta = 70.3^\circ$ is used. In order to obtain the same projected contact areas, in the following computations, the load is executed through control of the displacement of the indenter. The maximum indentation depth is set to $h_{\max} = 118$ nm, which is nearly same as the depth of the indenter reached in the experiment. As a consequence,

Fig. 11. Schematic of different indentation impressions and microstructural characteristics of the used model.

Fig. 12. 3D finite-element model for hardness tests. The indentation position is on the distance of 50 μm from the coating-substrate interface.

the radius of projected contact area at the maximum indentation depth is about 329 nm evaluated according to Equation (4). Thus, for decreasing the number of elements and saving computer time, the size of the used model is designed with 200 μm length and 4 μm height. The thickness of the coating is 100 μm and the thickness of the substrate is assumed as 70 μm , see Figure 11.

On the cross section of the coating, seven hardness values are calculated, which are positioned at a distance of 0.52, 1.25, 2.5, 25, 50, 75, 97.5 and 98.96 μm from the coating-substrate interface, respectively. Besides this, on the cross section of the substrate, the hardness positioned at 0.52, 5, 10 and 40 μm from the coating-substrate interface are calculated. Therefore, a total of twelve 3D finite-element models is designed for the foregoing hardness investigations. The half symmetric 3D finite-element

Fig. 13. Hardness distributions on the cross section of the sample (blue dash line represents the hardness of 11.5 GPa. It is obtained by the pure coating model shown in Fig. 6).

model for the hardness testing on the distances of 50 μm from the coating-substrate interface within coating is shown in Figure 12. This model is established by 11 870 eight-noded 3D solid elements. There are 11 510 elements in the coating area and 360 elements for the substrate. A finer mesh is designed near the contact region to ensure the calculation accuracy. At maximum load, more than 800 contact nodes are active in the contact region. The bottom surface of the model located on the X - Y plane is fixed as boundary condition and the surface on Y - Z plane is defined as symmetric surface.

The hardness distributions on the cross section of the sample are shown in Figure 13. It can be seen, within the coating, where the indentation positions are far from the coating-substrate interface and the top surface, the hardness is less affected by boundary effects. The maximum hardness is 11.5 GPa, which appears in the middle of the coating. It is equivalent to the calculated hardness, $H = 11.5$ GPa which is obtained by the pure coating model shown in Figure 6.

However, it must be noted that the close to the interface, coating's hardness is affected significantly by the substrate. It can be seen that the hardness of the coating decreases when the indentation impression is close to the coating-substrate interface, because the substrate is much softer than the coating. Particularly, the hardness at a distance of 0.52 μm from the coating-substrate interface within the coating decreases to 6.5 GPa as the substrate is less stiff than the coating. Besides this, when the indentation position is too close to the top surface of the coating, the hardness decreases again because the top surface is not constrained.

6 Conclusions

In the present work, the mechanical properties of a plasma-sprayed coating are evaluated using inverse analysis based on nanoindentation testing. The tip rounding

of an imperfect indenter is investigated in the parameter identification. First, the material parameters of the plasma-sprayed coating are determined by a conical indenter with a perfect sharp tip. The optimized $p-h$ curve has a good agreement with the experimental data. Second, the tip rounding of imperfect conical indenter is considered because the optimized $p-h$ curve is lower than the experimental data at the beginning of loading curve. The result shows that the optimized $p-h$ curve, which includes the effect of tip rounding, is close to the experimental data. Subsequently, based on the identified material parameters, the hardness on the top surface of the plasma-sprayed coating as a function of the indentation depths is studied. The results show that the measured hardness will decrease significantly with an increase of the indentation depth. Furthermore, the relation of the hardness on the vertical cross section of the coating and the substrate is assessed. The results show that the hardness on the cross section of the coating will decrease largely under the effect of the soft substrate, if the indentation impression is close to the coating-substrate interface.

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